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Runoff Water Quality and Crop Growth as Affected by Wastewater-derived Struvite

A thesis submitted in partial fulfillment  
of the requirements for the degree of  
Master of Science in Crop, Soil, and Environmental Sciences

by

Machaela Morrison  
University of Arkansas  
Bachelor of Science in Environmental, Soil, and Water Sciences, 2021

August 2023  
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This thesis is approved for recommendation to the Graduate Council.

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## Abstract

Mined phosphate rock, used to produce phosphorus (P) fertilizers, is a finite resource. Struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) that has been synthetically produced from a stock solution of known P and nitrogen (N) concentrations has been shown to be an effective, alternative fertilizer-P source for various crops. However, little is known about the runoff-water-quality implications from and the crop response to soil application of struvite created from an actual municipal wastewater source. This study consisted of two objectives: i) to evaluate the effects of soil [i.e., Creldon (Oxyaquic Fragiudalf), Dapue (Fluventic Hapludoll), Roxana (Typic Udifluent), and Calloway (Aquic Fraglossudalf) series], fertilizer-P source [i.e., synthetically produced electrochemically precipitated struvite (ECSTsyn), real-wastewater-derived ECST (ECSTreal), chemically precipitated struvite (CPST), and monoammonium phosphate (MAP)], and water source (i.e., rain water, groundwater, and struvite-removed real wastewater) over time on runoff-water-quality parameters from laboratory-conducted, rainfall-runoff simulations, and ii) to evaluate the effects of soil (i.e., Creldon silt-loam and Calloway silt-loam series), fertilizer-P source [i.e., ECSTsyn, ECSTreal, CPST, MAP, and an unamended control (UC)], and irrigation water source (i.e., tap water and struvite-removed real wastewater) on corn (*Zea mays* L.) and soybean [*Glycine max* (L.) Merr.] growth and N, P, and magnesium (Mg) concentrations and uptakes in a 60-day, greenhouse potted-plant study. For Objective 1, mesh tea bags containing each soil-fertilizer treatment combination were rained on with each water source (Trial 1), incubated for 6 months, and rained on again (Trial 2) to evaluate runoff-water quality changes over time. In general, in both trials, runoff-water-quality properties from the struvite fertilizers (i.e., CPST, ECSTreal, and ECSTsyn) were similar to those from MAP. In Trial 1, runoff total P (TP) concentration differences (i.e., soil-fertilizer-water-type response minus the

UC response minus the blank response) from ECSTsyn or ECSTreal were one to five times larger than MAP and CPST for all water-soil-fertilizer-P source treatment combinations, except for the Creldon soil-groundwater and Roxana soil-wastewater combinations. In both Trial 1 and 2, runoff TP decreased over time in all water-soil and soil-fertilizer-P source treatment combinations, except for in the Roxana soil-CPST treatment combination, where TP increased over time by 46%. For Objective 2, crop growth and N, P, and Mg concentrations and uptakes for the struvite treatments (i.e., CPST, ECSTsyn, and ECSTreal) were generally similar to MAP or at least 1.2 to 2.5 times greater than MAP. The ECSTsyn material commonly had up to five times greater N, P, and Mg concentration and uptake in corn and soybean than any other fertilizer-P source. Struvite-removed wastewater tended to result in N, P, and Mg concentrations, uptakes, and dry matter that were at least 1.3 times lower than tap water. Results showed that, despite having larger concentrations of many plant nutrients than tap water, struvite-removed wastewater does not appear to be a viable source of nutrients for corn or soybean growth. The similar water-quality and corn and soybean responses from the struvite fertilizers compared to MAP suggest that struvite has similar runoff-water-quality and agronomic implications as at least one widely used, commercially available, multi-nutrient fertilizer-P source.

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## **Introduction**

Phosphorus (P) is a limiting essential plant nutrient in many agricultural production systems, and the usage of P fertilizers in agriculture has allowed for the increase in food production along with a growing world population (Ashley et al., 2011). Most P fertilizers are derived from phosphate rock, which is a non-renewable resource. At the current rate of increasing fertilizer-P demand, the world's phosphate rock reserves will be depleted within 150 years (Cordell & White, 2013).

Environmental impacts of P in runoff from fertilized, agricultural systems are also a major concern. Phosphorus is not readily soluble and usually accumulates in the soil in a particulate form, making P susceptible to erosion and easily lost to water bodies in large quantities. Potter et al. (2006) estimated that over 9.6 million metric tons of P are translocated from agricultural systems each year, of which 63% is sediment-bound and 20% is soluble. When P is moved from soils into waterways, excess nutrients can cause harmful algal blooms that severely degrade water quality and harm aquatic life (Carpenter, 2008).

Another substantial source of P is P contained in human and animal waste. Much of the nutrients consumed by humans and animals are excreted and not recycled, instead ending up as leachate and runoff, ultimately further contributing to excess nutrient loads in the environment. A potential solution to both the depletion of phosphate rock deposits and the large amounts of P contained in human and animal waste is to harvest P from waste to be reused as a fertilizer-P source, such as struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) (Cordell & White, 2013). Struvite is generally a slow-release, fertilizer-P source derived from both solid and liquid forms of human and animal waste, as well as other sources.

A wide variety of research is currently being conducted to evaluate struvite's potential source materials, uses, benefits, applications, and production processes. The behavior of

synthetic, electrochemically precipitated struvite (ECST) in various soils without plants (Anderson et al., 2020a,b; Anderson et al., 2021) and rice (Omidire et al., 2022a), corn (Omidire et al., 2022b), and soybean (Omidire, 2021) response to ECST have been recently evaluated for potential use in various agronomic cropping systems. However, ECST derived from municipal wastewater, as a logical next step beyond the creation of ECST from a solution of known nutrient concentrations, has been minimally studied to date. Therefore, the goal of this thesis is to evaluate the environmental and agronomic implications of ECST derived from municipal wastewater through a rainfall-runoff simulation study and a greenhouse, potted-plant study.

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**Chapter 1**  
**Literature Review**

## Phosphorus Sources

As the world's population has grown exponentially over the last few centuries, so has the demand for agriculture to feed the growing human population. With increased agricultural production has also come a need for increased fertilizer inputs in those agricultural systems, specifically for phosphorus (P). Widespread use of P fertilizers in agricultural systems has been documented since the 1940s when the exponential population growth in the United States (US) created a demand for increased food production (Ashley et al., 2011). Since P is a limiting plant-essential nutrient in many soils, the addition of P fertilizers into agricultural systems allowed for a large increase in crop production and yield. The incorporation of synthetic fertilizers into crop systems is estimated to have increased worldwide yield by over 50% (Smil, 2000).

Currently, most fertilizer-P sources are derived from phosphate rock (PR). Phosphate rock is mined from deposits worldwide, but the majority is controlled by Morocco, China, and the US (Cordell et al., 2009). There is an estimated  $5 \times 10^{12}$  kg P present in mineable PR in North America (Stevenson & Cole, 1999). Phosphate rock does not contain pure phosphate. Apatite is the most common phosphate mineral in PR, and the majority of PR is mined from sedimentary deposits. After processing, the range of actual phosphate derived from PR is 5 to 40% (Steen, 1998). In the US, more than 95% of the phosphate derived from PR goes to agricultural use, including fertilizer production and animal feed supplements. Fifty percent of the phosphate goes directly to commercially available fertilizer-P production, specifically diammonium phosphate (DAP) and monoammonium phosphate (MAP; USGS, 2021). Fertilizer derived directly from PR is known as superphosphate, containing 10% P (Stevenson & Cole, 1999).

With the current rate of human population growth, the demand for fertilizers will increase at a proportional rate. Crop yield demands are expected to increase annually by approximately 2.5% (Steen, 1998). In 2020, the demand for fertilizer-P increased by 7% (IFA, 2021). However, demand for all fertilizer is expected to increase at an annual rate of only 0.9% (IFA, 2021). Phosphate rock reserves and demand for fertilizer-P fluctuate year to year. Peak P, when demand outpaces supply, is set to occur by 2040 (Cordell & White, 2013). Depending on demand, economic viability, and P concentrations within the current reserves, PR reserves are likely to be depleted in the next 30 to 150 years (Cordell & White, 2013).

## **Phosphorus Cycle**

Phosphorus is an irreplaceable nutrient in agricultural systems. Phosphorus is essential for nucleic acid structure in deoxyribonucleic acid (DNA) and ribonucleic acid (RNA). Phosphorus also occurs in enzymes, which play an important role in the transfer and storage of energy in plants (Johnston, 2000; Smil, 2000). Phosphorus cycles through the environment in two forms, inorganic and organic. The inorganic P cycle happens on a geologic time scale, which starts with rock weathering. Phosphorus is released from weathered sediments and runs off soil into rivers and lakes, where the P eventually reaches the ocean and precipitates out as calcium phosphate and sinks to the sea floor (Follimi, 1996). At the global scale, P is transported from terrestrial ecosystems to freshwater bodies and ultimately to the ocean at a rate of  $17 \times 10^9 \text{ kg yr}^{-1}$  (Stevenson & Cole, 1999). Some particulate P, an estimated  $4.3 \times 10^9 \text{ kg yr}^{-1}$ , is also carried into the atmosphere by wind, but the P cycle does not have a gaseous component like the nitrogen (N), carbon (C), or sulfur (S) cycles (Stevenson & Cole, 1999). After hundreds of



millions of years, the precipitated material forms sedimentary rock and is uplifted to form new land (Liu et al., 2008), completing the inorganic P cycle.

The greatest quantity of P is adsorbed onto marine sediments ( $\sim 840,000 \times 10^{12}$  kg P),  $\sim 19 \times 10^{12}$  kg P are contained in minable PR, and P stored in the soil ranges from  $\sim 96$  to  $160 \times 10^{12}$  kg (Stevenson & Cole, 1999). In both terrestrial and marine systems,  $\sim 80 \times 10^{12}$  kg P are dissolved (Stevenson & Cole, 1999). Whether P at the Earth's surface is contained in the soil or mineable PR, P eventually makes its way to the sea where the P is deposited as ocean sediment and effectively removed from the P cycle. Phosphorus translocation from the soil is mainly due to soil erosion or runoff, as P does not readily leach in large quantities through the soil profile due to P's low solubility and general immobility in soil (Brye et al., 2002; Stevenson & Cole, 1999). Phosphorus leaching potential is even lower in silt-loam soils that have an increasing clay content with depth (Haggard et al., 2003).

The organic P cycle is the process of P moving from soil to plants to animals and then back to the soil. The organic P cycle also consists of a water-based cycle that moves P among aquatic organisms and rivers, lakes, and oceans. The land-based organic P cycle takes approximately a year, while the water-based organic P cycle takes only weeks to complete (Liu et al., 2008). As part of the water-based cycle, P precipitates and falls out of suspension as to the ocean floor, much like in the inorganic P cycle. The precipitation results in an annual loss of available P on land (Liu et al., 2008). Due to low solubility and importance in many life functions, the majority of P in the environment is unavailable for plant and fertilizer use (Smil, 2000).

Phosphorus in the soil is mainly present in its insoluble orthophosphate form as complexes with calcium (Ca), iron (Fe), aluminum (Al), and silicates, depending on the soil pH.

At a low pH, P complexes with Fe, Al at a neutral pH, and Ca at a more alkaline pH. The greatest fixation of P occurs in acidic soils. Phosphate ( $\text{PO}_4^{3-}$ ) is taken up by plants after dissociation of phosphoric acid ( $\text{H}_3\text{PO}_4$ ). The form of the phosphate ion varies with pH, where  $\text{H}_3\text{PO}_4$  is present at a low pH (pH 4-5) and  $\text{PO}_4^{3-}$  is present at an alkaline pH (pH 7-8). The most common plant-available phosphate ions are  $\text{H}_2\text{PO}_4^-$  and  $\text{HPO}_4^{2-}$ , available around pH 6 (Stevenson & Cole, 1999). On average, US soils typically contain 500 to 800 mg P  $\text{kg}^{-1}$ , although the P concentration can be smaller or larger based on region. Topsoils in the southern US usually have a smaller P concentration ( $\sim 200$  mg P  $\text{kg}^{-1}$ ), while topsoils in the northwest region of the US can contain upwards of 1000 mg P  $\text{kg}^{-1}$  (Stevenson & Cole, 1999). However, only a small portion of the soil-P concentration is plant available. In Arkansas, a low soil-test P is considered to be less than 25 mg  $\text{kg}^{-1}$  (Espinoza et al., 2013)

Humans accelerate and alter the P cycle in various ways. The first mechanism is by exacerbated runoff and erosion caused by human activities, including agricultural practices and the construction of roads and buildings. The second human activity that influences the P cycle is fertilization by animal or livestock wastes in agricultural systems (Smil, 2000; Liu et al., 2008). Human waste that is left untreated also contributes to an accelerated P cycle, especially considering that human waste is comprised of 10% P on average. Finally, the addition of inorganic P to agricultural systems in the form of fertilizers also causes changes in the P cycle (Smil, 2000; Liu et al., 2008). Liu et al. (2008) also reported that the harvest and removal of crop residues further eliminates a source of P in the soil. All of the human-induced alterations to the P cycle are directly related to the increase in population and demand for agriculture.

## **Phosphorus and Environmental Impacts**

Human-induced changes in the P cycle have allowed for the opportunity for increased P losses. Since P is not easily soluble, P tends to accumulate in the soil in a particulate form. Consequently, sediment-bound P is easily eroded, which is a main mechanism by which P is transported from the soil. As of the early 2000s, 60% of global soil erosion was caused by human activities (Liu et al., 2008). Potter et al. (2006) estimated that 327,000 metric tons (MT) of P are translocated from agricultural fields each year, which is 16% of the total fertilizer-P amount applied each year. Potter et al. (2006) also estimated that approximately 63% of the total P translocation from agricultural systems is through sediment runoff. Another 20% was soluble P lost through water runoff, and 15% of total P lost was lost due to wind erosion (Potter et al., 2006). Overall, 9.6 million MT (MMT) of P are estimated to be translocated from agricultural systems each year (Potter et al., 2006).

Both P and nitrogen (N) runoff from cropland, and the majority of the N and P eroded from agricultural systems, flows into surface waterways, such as rivers and lakes, and ultimately ends up in the ocean. Nutrient excess in the surface waterways contributes to water quality impairment, particularly by eutrophication (Carpenter, 2008; Withers & Haygarth, 2007; Correll, 1998). Eutrophication is the growth of dense plant life in water due to excessive nutrient concentrations (i.e., P and N) and is considered one of the most significant global water pollution problems (Gilbert et al., 2005). In freshwater systems, P is the limiting nutrient that is responsible for primary production and eutrophication in large concentrations, and the same is true for N in the ocean. Historically, there has been a focus on N as the cause of eutrophication, but in more recent decades, the importance of P, and the combination of both P and N, as the cause of eutrophication has been recognized (Correll, 1998).

Excess P in water can cause harmful algal blooms that, when decaying, consumes and reduces the dissolved oxygen (DO) in the water system. The decrease in DO leads to the death of aquatic species, the algae can become toxic to both animals and humans, and the overall quality of water becomes foul (Carpenter, 2008). One large-scale example of eutrophication is the dead zone in the northern Gulf of Mexico. This area is a huge hypoxic zone that kills many marine animals each year and negatively impacts the fishing industry. Both N and P run off from agricultural lands, into the Mississippi River, and ultimately end up in the Gulf of Mexico (Dodds, 2006). The P concentration in the Mississippi River increased 50% from 1993 to 2003 (Rittmann et al., 2011). Stopping harmful algal blooms by limiting nutrient runoff is especially important in freshwater systems, since humans rely immensely on freshwater as a drinking water source and for agriculture and recreation. Reducing non-point-source nutrient pollution where it originates would substantially decrease nutrient runoff into the ocean as well.

There are two major ways to regulate nutrient runoff in order to decrease eutrophication. The first way to control runoff-P concentrations and loads is to control non-point P sources. Controlling P sources means reducing P inputs to the soil in agricultural systems (Bennett et al., 2001). Reducing erosion is also an important way to control pollution from P sources. Some methods to reduce erosion include cover cropping, conservation tillage, and strip cropping. Along waterways, the addition of filter strips, riparian zones, and buffer areas can help control sediment and P inputs into rivers and lakes. Although these methods are effective in controlling sediment P, they are not as effective in controlling dissolved P (Mullins, 2009).

To control dissolved P, fertilizer must be applied to the soil more responsibly. One way to be more responsible with fertilizer-P applications is to conduct routine soil tests and develop nutrient management plans, which help producers more responsibly add or maintain P for crops

(Mullins, 2009). Incorporating P into the soil also helps P bind with the soil to become less mobile in the environment and applying fertilizer-P to actively growing crops facilitates plant-P uptake to be less likely succumb to runoff (Mullins, 2009).

The second way to decrease P runoff and control eutrophication is to monitor soil-test P levels and increase P sinks. Monitoring soil-test P is related to nutrient management planning. Producers perform regular (i.e., annual) soil tests to determine whether soil P availability is sufficient for crop growth each year. If the soil P is sufficient for crop growth, no fertilizer needs to be added and there is a decreased opportunity for excess P to run off into nearby waterways. If the soil P availability is insufficient for optimal crop growth and productivity, then a fertilizer-P rate needs to be applied to the crop (Mullins, 2009). Adding a calibrated fertilizer-P rate to meet annual crop needs reduces excess P in the soil that can runoff into waterways in dissolved or particulate-bound forms while ensuring adequate crop production and farming profitability.

Increasing P sinks also can help reduce P runoff. Sinks are soils that have a large capacity to securely bind and store P in the soil so that P does not enter rivers and lakes. Examples of sinks include wetlands, detention basins, riparian buffers, and agricultural fields under conservation management practices (Bennett et al., 2001). The overall goal with both controlling P sources and monitoring soil-test P levels is to regulate P flows. Human activities have thoroughly disrupted the P cycle balance, so the hope is that by managing P inputs and outputs, the P cycle will return to its natural balance (Liu et al., 2008).

### **Phosphorus in Human and Animal Waste**

As a result of the need to produce enough food to sustain an increasing human population, there is a concomitant substantial increase in the production of human and animal

wastes, which further disrupts the natural P cycle and balance among major P pools. Agricultural runoff and animal waste make up the majority of P discharge, but approximately 15% is also due to human waste (Rittmann et al., 2011). Ninety-eight percent of the N and P in the human diet is lost through waste (Smil, 2000), thus human waste has a large P and N content. Every year, 300 MMT of human waste is produced globally, but less than a third of that is reused. Currently, human waste accounts for 22% of the global P economy by weight (Mihelcic et al., 2011).

In most urban areas of the world, human waste is collected by sewer networks. The sewers carry the sewage to wastewater treatment plants where the sewage can be treated to varying levels of desired water quality (Englande et al., 2015). Part of the treatment process is to remove the majority of the N and P from the sewage to create a sludge and keep excess nutrients out of the resulting processed, or treated, wastewater (Moree et al., 2013). Treated wastewater is normally reused, especially for agricultural irrigation, and the removed N and P stays part of the sewage sludge. The remaining sludge gets dehydrated and is either sent to a landfill, used as biosolid fertilizer, or gets incinerated (Englande et al., 2015). Before incineration, sewage sludge contains around 1.4% P, while incinerated sewage sludge contains approximately 3.5% P (Cordell & White 2013). Wastewater treatment plants in the US produce over 100 MMTs of sewage sludge each year (Linville et al., 2015), which means that incinerated sewage sludge contains around 3.5 MMT of P.

Animal waste makes up the second largest flow of P lost to the environment at 40% (Rittmann et al., 2011). The majority of animal waste in the US comes from confined animal-feeding operations (CAFOs), which have waste management systems in place designed to collect, store, and treat the waste. Animal waste is not only manure, but the waste also consists of dead animals, wasted feed, sometimes bedding materials, and wash water (Zhang & Schroder,

2014). The US alone produces over 300 MMTs of animal manure each year (Linville et al., 2015). Of all the P taken in, animals usually excrete around 70% of the consumed P as waste (Greaves et al., 2010). Although much of the animal waste from CAFOs is collected, some of the waste is still lost. Because 65% of P in animal wastes is in an organic form, the P is unable to be fixed in the soil, thus the soil-P load has the potential to be mobile in the environment and end up in surface waterways (Greaves et al., 2010). Moree et al. (2013) estimated that as much as 30 to 50% of nutrients in animal waste may be lost by leaching and runoff. Reuse of P from animal wastes is also a major issue. Only 50% of P that animals consume is returned as a fertilizer to where food is produced (Childers et al., 2011). Overall, there are large P inefficiencies among fertilizer, food production, human and animal consumption, and waste treatment and reuse that have perpetuated the significant P imbalance that exists in the environment today.

### **Alternative Phosphorus Sources**

Many agricultural scientists emphasize that recycling P is imperative to continue producing food after PR reserves are depleted and in order to close the inefficiencies of the P cycle (Cordell et al., 2009; Steen, 1998; Cordell & White, 2013; Liu et al., 2008; Johnston, 2000). Since the largest P loss is through human and animal waste, P recovery from waste is the focus of many P-recycling efforts. There is a large divide between agriculture, where P is a necessary fertilizer-nutrient input, and sanitation, where P is a pollutant to be removed (Cordell et al., 2009). However, there are a few ways that waste materials can be better used and/or reused as fertilizers.

One way is to apply human urine directly as a fertilizer, but this method has not been extensively considered in modern times due to issues with collection. However, urine is almost

entirely sterile when not combined with the other wastes that are in sewage sludge. Because of the relative sterility of urine, urine also does not contain the heavy metals that sewage sludge sometimes contains (Cordell & White, 2013). It has been estimated that human urine alone could provide half of the P needed to grow cereal crops each year (Childers et al., 2011).

Animal manures have historically been widely utilized as an organic crop fertilizer. Manures are an excellent source of available nutrients. Nitrogen availability in manures ranges from 30 to 50% and P and K availability is around 90% (Zhang & Schroder, 2014). Soil organic matter increases considerably with manure application (Zhang & Schroder, 2014; Gross & Glaser, 2021), thereby increasing soil aggregation, increasing both buffering capacity and cation exchange capacity, and leading to a general improvement in physical soil properties (Zhang & Schroder, 2014; Gross & Glaser, 2021). However, application of animal manures to the soil can also lead to a buildup of soil-available P pools that can easily become a pollutant in waterways (Zhang et al., 2017).

Some animal manures also contain large heavy metal concentrations due to feed additives that are consumed. The heavy metals, including arsenic (As), selenium (Se), chromium (Cr), copper (Cu), and zinc (Zn), can become concentrated in the soil and lead to phytotoxicity and plant death (Zhang & Schroder, 2014). However, the most limiting factor to using widespread manure application as fertilizer is the bulkiness and cost of transportation of the waste to agricultural fields (Liu et al., 2008).

Sewage treatment, removal of P, and use of that removed P as a fertilizer is currently the most feasible method to recycle P from waste (Cordell & White, 2013). Removal of N and P are already part of the wastewater treatment process that creates sewage sludge. The resulting sewage sludge, also referred to as biosolids, is sometimes already reused in its raw form as a



fertilizer. However, since biosolids are not specifically produced for agricultural use, heavy metals may be a major concern depending on the wastewater sources (Cordell & White, 2013). Wastewater treatment is concerned with removing pollutants only, not specifically separating useful nutrients for crop use (Liu et al., 2008). In some areas, infrastructure is specifically planned to avoid mixing human waste with industrial wastes that contain large concentrations of heavy metals (Cordell et al., 2009). Presently, removal of nutrients, specifically N and P, from wastewater effluents for agricultural fertilizer use is a primary research focus.

## **Struvite**

Technology that removes P from wastewater effluent and sewage sludge has been in development since the 1950s, where processes include biological and chemical removal and crystallization, in order to recycle P (Morse et al., 1998). The term ‘struvite’ was first used in the 17<sup>th</sup> century for a type of urinary stone made of  $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$  (Griffith, 1978). Historically, struvite has been more well-known for being a nuisance for wastewater treatment plants. In 1939, struvite was identified as the material precipitating out of solution in wastewater treatment plant pipes and causing clogs. One of the most infamous examples of struvite clogging was in 1963, when pipes at the Hyperion wastewater treatment plant in Los Angeles, California were reduced in diameter from 30 to 15 cm (Doyle & Parsons, 2002). Removal of struvite deposits from pipes, or altogether replacement of clogged pipes, can be extremely expensive, costing hundreds of thousands of dollars annually for a mid-sized wastewater treatment plant (Baur et al., 2002). Unwanted struvite formation has been recorded in both animal and human wastewater treatment plants (Doyle & Parsons, 2002).

Struvite use as a fertilizer-nutrient source was first proposed in 1858 (Johnston & Richards, 2003), although extensive research was not conducted on the topic of struvite as a fertilizer until the mid-1900s. Since the mid-1900s, struvite removal from a wide variety of wastewaters have been identified, including manure, industrial wastes, landfill leachate, and sewage sludge (Rahman et al., 2014). In the past decade, struvite formation from waste sources has gained interest as an option for nutrient removal, especially for N and P recovery and as an alternative for the traditional, non-renewable P source, phosphate rock.

### **Struvite Properties**

Struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) is a white, crystalline substance, containing a 1:1:1 equimolar ratio of magnesium ( $\text{Mg}^{2+}$ ), ammonium ( $\text{NH}_4^+$ ), and phosphate ( $\text{PO}_4^{3-}$ ), that is somewhat soluble in neutral and alkaline conditions, but more readily soluble in acidic conditions (Liu et al., 2012). The solubility of struvite in water is generally low, around 1 to 5%, but research shows that the low solubility of struvite does not decrease its effectiveness as a fertilizer-P source for plants (Talboys et al., 2016). Struvite has been characterized as a slow-release fertilizer due to its low solubility, although more recent research shows that struvite in powder form has a similar dissolution rate in soil as MAP (Degryse et al., 2017). However, struvite's slow-release properties may benefit crops, as the P will become available to crops over time, in a controlled-release manner, where P release and plant uptake will remain steady across the growing season (Bonvin et al., 2015). Although struvite has been shown to be an effective, potential fertilizer source, the nutrient content of struvite varies depending on what waste source was used and how the struvite was actually created.

On average, struvite has 5.7% N, 12.6% P, and 9.9% Mg (Nongqwegna et al., 2017). However, the nutrient concentrations can vary based on the waste that the struvite was precipitated from and what method was used to precipitate the struvite. The appearance of the struvite also varies among sources and methods. Johnston & Richards (2003) compiled a list of various struvite materials and their compositions. Naturally precipitating struvite in wastewater treatment plant pipes had a large crystal formation and contained 12.4% P (Johnston & Richards, 2003). Struvite recovered from industrial dye waste was a brown powder and contained only 8.1% P (Johnston & Richards, 2003). Alternatively, synthetically produced struvite in pellet form had a P concentration of 13.4% (Johnston & Richards, 2003).

### **Struvite Formation**

Struvite formation relies on the solution pH and the solution concentrations of Mg, ammonium, and phosphate. Struvite formation and purity can also be affected by the method used to precipitate the struvite or the presence of  $\text{Ca}^{2+}$  ions or heavy metals in solution. Struvite generally precipitates out of solution about pH 7.5 and increases in precipitation rate up to pH 10.5 (Doyle & Parsons, 2002). The optimum pH for struvite purity > 90% is from 7.5 to 9 (Hao et al., 2008). Hao et al. (2008) also discovered that precipitating struvite out of solution with a lower pH can result in differential phosphate form,  $\text{HPO}_4^{2-}$  instead of  $\text{PO}_4^{3-}$  formation. It was also reported that at approximately pH 8.5, the presence of  $\text{Ca}^{2+}$  ions in the struvite crystals increased, leading to a more impure substance or calcium phosphate  $[\text{Ca}_3(\text{PO}_4)_2]$  instead of struvite at the greater pH (Hao et al., 2008). Wastewater normally has a low Mg concentration, thus Mg typically needs to be added for struvite precipitation to occur. In many cases, the Mg is

added in the form of magnesium chloride ( $\text{MgCl}_2$ ), magnesium oxide ( $\text{MgO}$ ), or magnesium hydroxide [ $\text{Mg}(\text{OH})_2$ ], all of which also increase the solution pH (Moussa et al., 2006).

Another source of potential struvite contamination is heavy metals. Heavy metals are a common source of pollution in wastewater, thus heavy metals are also present in sewage sludge (Liu et al., 2021). Over 70% of the Mn and Cu present in wastewater ends up in the treated sewage sludge. Anywhere from 47 to 63% of the original cadmium (Cd), chromium (Cr), lead (Pb), and iron (Fe) in wastewater is also present in sewage sludge (Liu et al., 2021). Since a main source of struvite formation is wastewater and subsequently sewage sludge, heavy metals have the potential for negative effects. At low concentrations, heavy metals were only adsorbed into the struvite during crystallization (Li et al., 2020). However, at larger concentrations, the metals led to the formation of non-struvite compounds that affected the purity of the struvite formed (Li et al., 2020). To prevent heavy metal adsorption or formation of alternate, non-struvite compounds, heavy metals should be removed prior to intentional struvite precipitation (Li et al., 2020).

Struvite formation occurs in two steps: nucleation followed by growth. Nucleation itself has a primary and secondary step. In primary nucleation, the first struvite crystal forms. More struvite crystals form in and around the initial crystal in secondary nucleation. Nucleation is driven by a supersaturation ratio of Mg:P and the pH of the solution (LeCorre et al., 2009; Siciliano, 2020). After nucleation, each crystal becomes larger in the growth step of struvite formation. Struvite growth is dictated by the solution pH, temperature, supersaturation, and the presence of potential contaminants (Mehta & Batstone, 2013). These various factors must be considered when designing a crystallizer and investigating struvite formation methods.

## **Chemical and Electrochemical Precipitation**

The addition of compounds such as  $\text{MgCl}_2$  or  $\text{MgO}$  allow for struvite crystallization once the solution becomes supersaturated with  $\text{Mg}^{2+}$ ,  $\text{NH}_4^+$ , and  $\text{PO}_4^{3-}$  (Siciliano et al., 2020). The process of adding chemicals to an aqueous solution to precipitate struvite out of solution is known as chemical precipitation. Historically, chemical precipitation of struvite was the main method used for struvite formation. Today, there is a commercially available, chemically precipitated struvite fertilizer known as Crystal Green (CPST), which is produced by Ostara Nutrient Technologies, Inc. According to the Crystal Green website (Ostara, 2021), Crystal Green is a slow-release fertilizer in pellet form with a fertilizer grade of 5-28-0 and 10% Mg. Crystal Green also has low heavy metal and salt concentrations (Ostara, 2021).

A more recently developed technique for precipitating struvite from wastewaters is electrochemical precipitation. Electrochemical precipitation of struvite is achieved by electrochemically releasing Mg via sacrificing a Mg anode plate (Kékedy-Nagy et al., 2021). Electrochemical precipitation avoids the chemical dosing that chemical struvite precipitation requires and instead requires only an energy input for Mg dissolution. Kékedy-Nagy et al. (2020) used electrochemical precipitation to more efficiently recover P from synthetic wastewater compared to chemical precipitation. Overall, electrochemical precipitation of struvite has the potential to be more energy efficient and more effective than other P recovery methods.

## **Struvite, Soil, Crop, and Water Interactions**

Since struvite formation relies on the pH of the solution, it is logical that struvite solubility also relies on the pH of the soil. Results of Massey et al. (2009) and Ackerman et al. (2013) show that struvite solubility in soil is dependent on soil pH. Acidic soils enhance struvite

solubility, while alkaline soils lower struvite solubility (Massey et al., 2009; Ackerman et al., 2013). As soil pH affects struvite solubility, the struvite itself can also change the soil pH, as well as the soil-P concentration. Various studies have been conducted to evaluate the extractable nutrient concentrations of struvite compared to other commercially available fertilizers, such as triple superphosphate (TSP), MAP, DAP and rock phosphate (RP; Anderson et al., 2021a; Anderson et al., 2021b; Anderson et al., 2021c). In a flooded-soil incubation experiment performed by Anderson et al. (2021c), the pH of an acidic silt-loam soil increased significantly under struvite application. Anderson et al. (2021c) also reported that the soil pH increased with time incubated. The change in water-soluble-P concentrations in struvite-treated soils was reported to be double the change in water-soluble-P concentrations under any other fertilizer treatment (i.e., TSP and MAP; Anderson et al., 2021c). The change in water-soluble-P concentrations decreased with time (Anderson et al., 2021c). The same trend was reported with Mehlich-3-extractable P concentrations (Anderson et al., 2021c). Nongqwuegna et al. (2017) performed a similar soil-struvite incubation study with soils wetted to 50% of their water holding capacity instead of being under flooded conditions. Like Anderson et al. (2021c), Nongqwuegna et al. (2017) reported an increase in extractable-P concentration over time in both fertilizer treatments (i.e., struvite and single superphosphate; SSP).

Similar to being incubated with various moisture contents, rainfall onto and runoff from fertilized soils can also affect soil-P concentrations, both by water interaction and nutrient runoff. Shigaki et al. (2007) performed a small-scale tray rainfall-runoff study to evaluate various rainfall intensities (i.e., 25, 50, and 75 mm h<sup>-1</sup>), fertilizer-P sources (TSP, SSP, RP, and swine manure), and the effect on P concentration in runoff water from the soil. In all fertilizer-P treatments, the P concentration in the runoff water exponentially increased with an increase in

rainfall intensity. However, if rainfall occurred days or weeks after the fertilizer application, the runoff-P concentration exponentially decreased (Shigaki et al., 2007). For example, the runoff-P concentration in the swine manure treatment at the largest rainfall intensity (75 mm hr<sup>-1</sup>) decreased from 21.9 mg P L<sup>-1</sup> after one day passed between fertilization and rainfall to only 2.9 mg P L<sup>-1</sup> after 56 days passed between fertilization and rainfall (Shigaki et al., 2007). A similar study conducted by Smith et al. (2007) reported similar results where runoff-P concentration decreased exponentially over time. Consequently, the longer the time that passed between fertilizer application and the rainfall event, the lower the runoff-P concentration, likely due to P adsorption onto soil particles (Smith et al., 2007; Shigaki et al., 2007).

Plant response to struvite also depends on soil pH. In many different studies (Hilt et al., 2016; Ackerman et al., 2013; Talboys et al., 2016; Johnston & Richards, 2003), no difference was reported in crop response between plants fertilized with commercially available fertilizers and plants fertilized with struvite. Ackerman et al. (2013) grew canola (*Brassica napus* L) in a sandy-loam soil with pH 7.7 comparing commercially available fertilizers, such as MAP, to struvite derived from liquid swine manure. No crop response difference was recorded among fertilizer treatments due to struvite's low solubility under alkaline conditions (Ackerman et al., 2013). Talboys et al. (2016) had a similar result when growing spring wheat (*Triticum aestivum*) and buckwheat (*Fagopyrum esculentum*) fertilized with a combination of CPST (i.e., Crystal Green) and DAP under alkaline soil conditions. Johnston & Richards (2003) reported no difference in dry matter yield among ryegrass (*Lolium perenne*) treated with commercially available P fertilizer, synthetically produced struvite, or struvite recovered from municipal or animal waste, where the soils used in this study had neutral pH. Hilt et al. (2016) compared corn (*Zea mays*) response to MAP and struvite recovered from dairy manure in both an acidic and an

alkaline soil, where corn response in the alkaline soil did not differ between MAP and struvite. However, in the acidic soil, the corn treated with struvite had a greater P uptake and P tissue concentration compared to plants treated with MAP (Hilt et al., 2016), further illustrating struvite's solubility dependency on soil pH. Similarly, Robles-Aguilar et al. (2020) grew corn in acidic soil to compare crop response between TSP and struvite. Corn treated with struvite had a larger average biomass than corn treated with TSP (Robles-Aguilar et al., 2020). Omidire & Brye (2022) compared soybean (*Glycine max*) and wheat fertilized with CPST to soybean and wheat fertilized with TSP and recorded no significant difference in response between the two treatments. Similarly, Omidire et al. (2022) grew rice treated with both synthetic electrochemically precipitated struvite (ECSTsyn) and CPST compared to other commercially available fertilizer-P sources (i.e., TSP, MAP, DAP, and RP) and again recorded no difference in response among struvite and the commercially available fertilizers. Hertzberger et al. (2020) conducted a meta-analysis and review of struvite as a potential fertilizer and reported that struvite-fertilized crops generally resulted in larger biomass, tissue-P concentration, and P uptake than plants fertilized with ammonium phosphates or superphosphates, especially in soils with pH < 6. In many studies, crop response to struvite increased as soil pH decreased, and struvite was recorded to be just as effective as commercially available fertilizers in soils with a neutral or alkaline pH (Hertzberger et al., 2020).

## **Justification**

Currently, agriculture depends heavily on RP as the main source for P fertilizers. However, RP is a non-renewable resource, and the world's minable RP deposits will likely be depleted within 150 years. Therefore, it is necessary to establish sustainable, alternative



fertilizer-P sources. Struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ), a fertilizer-P material precipitated from a synthetic solution or wastewater, has emerged as a potential renewable fertilizer-P source. Struvite can either be precipitated chemically or electrochemically, where chemically precipitated struvite (CPST) has already been shown to be a viable commercially available struvite fertilizer. Using electrochemical precipitation is a more recently developed method to create struvite, where much less research has been conducted on electrochemically precipitated struvite (ECST) compared to CPST. Furthermore, more studies have been conducted on synthetically produced ECST than struvite derived from other sources materials (i.e., animal or municipal wastewater). Consequently, further research is needed to evaluate municipal-wastewater-derived ECST and its effects on soil and water interactions and crop response compared to synthetic ECST, CPST, and other commercially available fertilizer-P sources.

### **Objectives and Testable Hypotheses**

The overall goal of this study is to evaluate the environmental and agronomic effects of wastewater-derived ECST relative to other struvite and commercially available fertilizer-P sources. This goal will be addressed through two study objectives. The first objective is to evaluate the effects of soil (i.e., Crelton, Dapue, Roxana, and Calloway series), fertilizer-P source (i.e., synthetically produced struvite, real wastewater derived struvite, CPST, and MAP), and water source (i.e., rain water, struvite-removed wastewater, and groundwater) over time on runoff water quality parameters from laboratory-conducted, rainfall-runoff simulations. It is hypothesized that runoff water quality parameters will differ among soils, fertilizer-P sources, and water sources over time. More specifically, it is hypothesized that the largest runoff-P concentration from the ECST treatments will occur in the soil with the lowest pH soil treated

with water with the lowest pH due to struvite's larger solubility in low-pH solutions. It is also hypothesized that all struvite treatments will, in general, have the lowest runoff-P concentrations among the fertilizer-P treatments due to struvite's reported slow-release nature. The second objective is to evaluate the effects of fertilizer-P source (i.e., synthetically produced struvite, real wastewater derived struvite, CPST, and MAP) and irrigation water source (i.e., tap water and struvite-removed wastewater) on corn and soybean growth in various soils over 60 days in a greenhouse potted-plant study. It is hypothesized that struvite-amended crops will have similar or greater growth than the crops treated with MAP. It is also hypothesized that plants watered with struvite-removed wastewater will have similar growth to plants watered with tap water.

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## **Chapter 2**

### **Runoff-water Properties from Various Soils as Affected by Fertilizer-phosphorus Sources and Water Types**

## Abstract

Mined phosphate rock, used to produce phosphorus (P) fertilizers, is a finite resource. Struvite can be produced from municipal wastewater and has been shown to be an effective, renewable, alternative fertilizer-P source for various crops, but little is known about the runoff-water-quality implications from soil application of struvite. The objective of this study was to evaluate the effects of soil [i.e., Creldon (Oxyaquic Fragiudalf), Dapue (Fluventic Hapludoll), Roxana (Typic Udifluent), and Calloway (Aquic Fraglossudalf) series], fertilizer-P source [i.e., synthetically produced electrochemically precipitated struvite (ECSTsyn), real-wastewater-derived ECST (ECSTreal), chemically precipitated struvite (CPST), and monoammonium phosphate (MAP)], and water source (i.e., rainwater, groundwater, and struvite-removed real wastewater) over time on runoff-water-quality parameters from laboratory-conducted, rainfall-runoff simulations. Mesh tea bags containing each soil-fertilizer treatment combination were rained on with each water source (Trial 1), incubated for 6 months, and rained on again (Trial 2) to evaluate runoff-water quality. In general, in both trials, all measured runoff-water-quality properties from the struvite fertilizers (i.e., CPST, ECSTreal, and ECSTsyn) were similar to those from MAP. In Trial 1, runoff total P (TP) concentration differences (i.e., soil-fertilizer-water-type response minus unamended control response minus blank response) from ECSTsyn or ECSTreal were one to five times larger than MAP and CPST for all water-soil-fertilizer-P source treatment combinations, except for the Creldon soil-groundwater and Roxana soil-wastewater combinations. In both Trial 1 and 2, runoff TP decreased over time in all water-soil and soil-fertilizer-P source treatment combinations, except for in the Roxana soil-CPST treatment combination, where TP increased over time by 46%. The similar water-quality responses of the struvite fertilizers among the various soils and water types compared to MAP

suggest that struvite has similar runoff-water-quality implications as at least one widely used, commercially available fertilizer-P sources.

## Introduction

As the world's population has grown exponentially over the last few centuries, so has the demand for agriculture to feed the growing human population. With increased agricultural production has also come a need for increased fertilizer inputs, specifically for phosphorus (P). Widespread use of P fertilizers in agricultural systems has been documented since the 1940s, when the exponential population growth in the United States (US) created a demand for increased food production (Ashley et al., 2011). Since P is a limiting plant-essential nutrient in many soils, the addition of P fertilizers into agricultural systems allowed for a large increase in crop production and yield. The incorporation of synthetic fertilizers into crop systems is estimated to have increased worldwide yield by over 50% (Smil, 2000).

Currently, most fertilizer-P sources are derived from phosphate rock (PR). Phosphate rock is mined from deposits worldwide, but the majority is controlled by Morocco, China, and the US (Cordell et al., 2009). There is an estimated  $5 \times 10^{12}$  kg P present in mineable phosphate rock in North America (Stevenson & Cole, 1999). However, PR does not contain pure phosphate. Apatite is the most common phosphate mineral in PR, and the majority of PR is mined from sedimentary deposits. After processing, the range of actual phosphate derived from PR is 5 to 40% (Steen, 1998). In the US, more than 95% of the phosphate derived from PR goes to agricultural use, including fertilizer production and animal feed supplements. Fifty percent of the phosphate goes directly to commercially available fertilizer-P production, specifically diammonium phosphate (DAP) and monoammonium phosphate (MAP; USGS, 2021). Fertilizer derived directly from PR is known as superphosphate, which contains ~ 10% P (Stevenson & Cole, 1999).

With the current rate of human population growth, the demand for fertilizers will increase at a proportional rate to keep up with increased food production needs. Crop yield demands are expected to increase annually by approximately 2.5% (Steen, 1998). In 2020, the demand for fertilizer-P increased by 7% (IFA, 2021). However, demand for all fertilizer is expected to increase at an annual rate of only 0.9% (IFA, 2021). Phosphate rock reserves and demand for fertilizer-P fluctuate year to year. Peak P, when demand outpaces supply, is set to occur by 2040 (Cordell & White, 2013). Depending on demand, economic viability, and P concentrations within the current reserves, PR reserves are likely to be depleted in the next 30 to 150 years (Cordell & White, 2013). Therefore, it is necessary to establish sustainable, alternative fertilizer-P sources for the future of agricultural production.

Struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) is a crystalline mineral, containing a 1:1:1 equimolar ratio of magnesium ( $\text{Mg}^{2+}$ ), ammonium ( $\text{NH}_4^+$ ), and phosphate ( $\text{PO}_4^{3-}$ ), that is somewhat soluble in neutral and alkaline conditions, but more readily soluble in acidic conditions (Liu et al., 2012). The solubility of struvite in water is generally low, around 1 to 5%, but research shows that the low solubility of struvite does not decrease its effectiveness as a fertilizer-P source for plants (Talboys et al., 2016). Struvite has been characterized as a slow-release fertilizer due to its low water solubility, although more recent research shows that struvite in powder form has a similar dissolution rate in soil as MAP (Degryse et al., 2017). However, struvite's slow-release properties may benefit crops, as the P becomes available to crops as the rhizosphere develops and slightly acidifies over time, in a controlled-release manner (Bonvin et al., 2015). Although struvite has been shown to be an effective, potential fertilizer source (Anderson et al., 2020b; Omidire & Brye, 2022; Ylagan et al., 2020), struvite's composition is somewhat variable depending on method of creation and source material.

The addition of compounds such as  $\text{MgCl}_2$  or  $\text{MgO}$  allow for struvite crystallization once the solution becomes supersaturated with  $\text{Mg}^{2+}$ ,  $\text{NH}_4^+$ , and  $\text{PO}_4^{3-}$  (Siciliano et al., 2020). The process of adding chemicals to an aqueous solution to precipitate struvite out of solution is known as chemical precipitation. Historically, chemical precipitation of struvite was the main method used for struvite formation. Today, there is a commercially available, chemically precipitated struvite fertilizer known as Crystal Green (CPST), which is produced by Ostara Nutrient Technologies, Inc. (Vancouver, British Columbia). According to Ostara (2021), Crystal Green is a slow-release fertilizer in pellet form with a fertilizer grade of 5-28-0 and 10% Mg. Crystal Green also has low heavy metal and salt concentrations (Ostara, 2021).

A more recently developed technique for precipitating struvite from wastewaters is electrochemical precipitation. Electrochemical precipitation of struvite is achieved by electrochemically releasing Mg via sacrificing a Mg anode plate (Kékedy-Nagy et al., 2021). Electrochemical precipitation avoids the chemical dosing that chemical struvite precipitation requires and instead requires only an energy input for Mg dissolution. Kékedy-Nagy et al. (2020) used electrochemical precipitation to more efficiently recover P from synthetic wastewater compared to chemical precipitation. Overall, electrochemical precipitation of struvite has the potential to be more energy efficient and more effective than other P recovery methods.

In either method, struvite formation relies on the solution pH and the solution concentrations of Mg,  $\text{NH}_4^+$ , and phosphate. Struvite formation and purity can also be affected by the method used to precipitate the struvite and/or the presence of calcium ( $\text{Ca}^{2+}$ ) ions or heavy metals in solution. Struvite generally precipitates out of solution at about pH 7.5 and increases in precipitation rate until pH 10.5 (Doyle & Parsons, 2002). The optimum pH for struvite purity > 90% is from 7.5 to 9 (Hao et al., 2008). Hao et al. (2008) also discovered that precipitating



struvite out of solution with a lower pH can result in differential phosphate forms, such as  $\text{HPO}_4^{2-}$  instead of  $\text{PO}_4^{3-}$  formation. It was also reported that, at approximately pH 8.5, the presence of  $\text{Ca}^{2+}$  ions in the struvite crystals increased, leading to a more impure substance or calcium phosphate instead of struvite at the greater pH (Hao et al., 2008). Wastewater normally has a low Mg concentration; thus, Mg typically needs to be added as a Mg salt for chemical struvite precipitation to occur. In many cases, the Mg is added in the form of magnesium chloride ( $\text{MgCl}_2$ ), magnesium oxide ( $\text{MgO}$ ), or magnesium hydroxide [ $\text{Mg}(\text{OH})_2$ ], all of which also increase the solution pH (Moussa et al., 2006).

Since struvite formation relies on the solution pH, struvite solubility will depend on the soil pH once land-applied as a fertilizer-P source (Massey et al., 2009; Ackerman et al., 2013). Acidic soils enhance struvite solubility, while alkaline soils lower struvite solubility (Massey et al., 2009; Ackerman et al., 2013). As soil pH affects struvite solubility, the struvite itself can also change the soil pH, as well as the soil-P concentration.

Various studies have been conducted to evaluate the extractable nutrient concentrations of struvite compared to other commercially available fertilizers, such as triple superphosphate (TSP), MAP, DAP and rock phosphate (RP; Anderson et al., 2021a,b,c). In a flooded-soil incubation experiment performed by Anderson et al. (2021c), the pH of an acidic silt-loam soil increased significantly under struvite application. Anderson et al. (2021c) also reported that the soil pH increased with time incubated. The change in water-soluble-P concentrations in struvite-treated soils were reported to be double the change in water-soluble-P concentrations under any other fertilizer treatment (i.e., TSP and MAP; Anderson et al., 2021c). The change in water-soluble-P concentrations decreased with time (Anderson et al., 2021c). The same trend was reported with Mehlich-3-extractable P concentrations (Anderson et al., 2021c). Nongqwuegna et

al. (2017) performed a similar soil-struvite incubation study with soils wetted to 50% of their moisture holding capacity instead of being under flooded conditions. Like Anderson et al. (2021c), Nongqwegna et al. (2017) reported an increase in extractable-P concentration over time in both fertilizer treatments [i.e., struvite and single superphosphate (SSP)].

Similar to being incubated with various moisture contents, rainfall onto and runoff from fertilized soils can also affect soil-P concentrations, both by water interaction and nutrient runoff. Shigaki et al. (2007) performed a small-scale tray rainfall-runoff study to evaluate various rainfall intensities (i.e., 25, 50, and 75 mm h<sup>-1</sup>), fertilizer-P sources (TSP, SSP, RP, and swine manure), and their effects on runoff-P concentration from the soil. In all fertilizer-P treatments, the runoff-P concentration exponentially increased with an increase in rainfall intensity (Shigaki et al., 2007). However, if rainfall occurred days or weeks after the fertilizer application, the runoff-P concentration exponentially decreased (Shigaki et al., 2007). For example, the runoff-P concentration in the swine manure treatment at the largest rainfall intensity (75 mm hr<sup>-1</sup>) decreased from 21.9 mg L<sup>-1</sup> after one day passed between fertilization and rainfall to only 2.9 mg L<sup>-1</sup> after 56 days passed between fertilization and rainfall (Shigaki et al., 2007). A similar study conducted by Smith et al. (2007) reported similar results, where runoff-P concentration decreased exponentially over time. Consequently, the longer the time that passed between fertilizer application and the rainfall event, the lower the runoff-P concentration (Smith et al., 2007; Shigaki et al., 2007).

Currently, agriculture depends heavily on phosphate rock as the main source for P fertilizers. However, phosphate rock is a non-renewable resource, and the world's minable phosphate rock deposits will likely be depleted within 150 years. Therefore, the objective of this study was to evaluate the effects of soil [i.e., Crelton (Oxyaquic Fragiudalf), Dapue (Fluventic

Hapludoll), Roxana (Typic Udifluvent), and Calloway (Aquic Fraglossudalf) series], fertilizer-P source [i.e., synthetically produced ECST (ECSTsyn), real-wastewater-derived ECST (real), CPST, and MAP)], and water source (i.e., rain water, groundwater, and struvite-removed wastewater) over time on runoff-water quality parameters from laboratory-conducted, rainfall-runoff simulations. It was hypothesized that runoff-water quality parameters will differ among soils, fertilizer-P sources, and water sources over time. More specifically, it was hypothesized that the largest runoff-P concentration from the ECST treatments will occur in the soil with the lowest-pH soil treated with water with the lowest pH due to struvite's larger solubility in low-pH solutions. It was also hypothesized that all struvite treatments will, in general, have the lowest runoff-P concentrations among the fertilizer-P treatments due to struvite's reported slow-release nature (Talboys et al., 2016). However, it was hypothesized that struvite (i.e., ECSTsyn, ECSTreal, and CPST) runoff-water quality parameters will be similar to MAP.

## **Materials and Methods**

### **Soil Collection**

Soil was collected from agriculturally relevant areas of southwestern Missouri and western and eastern Arkansas to use in a laboratory rainfall-runoff simulation study. Bulk soil was collected from a 0- to 15-cm depth at all locations. A Roxana fine sandy loam (coarse-silty, mixed, superactive, nonacid, thermic Typic Udifluvent; NCSS, 2001) was collected from a row-crop-cultivated field at the University of Arkansas, Division of Agriculture's Vegetable Research Station near Kibler, AR (35°22'50" N; 94°14'01" W; Anderson et al., 2020a). A Calloway silt loam (fine-silty, mixed, active, thermic Aquic Fraglossudalf; NCSS, 2021) was collected from within an approximate 3-m<sup>2</sup> area from the edge of a row-crop-cultivated field at the University of

Arkansas, Division of Agriculture's Pine Tree Research Station near Colt, AR (35°07'23" N; 90°55'46" W). The Roxana soil was collected in December 2017 and the Calloway soil was collected in early spring 2021. Two soils were collected from managed pasturelands at the University of Missouri's Southwest Research Center near Mount Vernon, MO in June 2021. One soil was a Creldon silt loam (fine, mixed, active, mesic Oxyaquic Fragiudalf; 37°04'45" N; 93°52'17" W; NCSS, 2006) and the second soil was a Dapue silt loam (fine-silty, mixed, active, mesic Fluventic Hapludoll; 37°05'07" N; 93°52'17" W; NCSS, 1997). All four soils represent various typical agricultural areas in the mid-Southern US.

### **Soil Processing and Analyses**

After collection, all moist soil was gently, manually pushed through a 6-mm sieve to remove large plant or/and rock debris and to unify the aggregate-size distribution to facilitate use in the rainfall-runoff simulation tests. Sieved soil was then air-dried in a greenhouse for approximately one week at approximately 37.8°C and stored in 20-L plastic buckets. Three soil sub-samples were gathered from each of the four air-dried bulk soils. Soil sub-samples were oven-dried at 70°C for 48 hours to determine gravimetric water content, manually crushed using a mortar and pestle, and sieved through a 2-mm sieve before performing chemical analyses.

Soil pH and electrical conductivity (EC) were measured potentiometrically in a 2:1 water volume:soil mass slurry. Soil organic matter (SOM) concentration was determined gravimetrically through weight-loss-on ignition after 2 hours of combustion at 360°C in a muffle furnace (Zhang & Wang, 2014). Total N (TN) and C (TC) were determined by high-temperature combustion with a VarioMAX CN analyzer (Elementar Americas Inc., Mt. Laurel, NJ). Particle-

size analyses were conducted using a modified 12-hr hydrometer method (Gee & Or, 2002) to determine sand, silt, and clay fractions and confirm soil textural classes.

Extractable soil nutrient concentrations were also determined for each of the four soils. A water extraction was performed with a 1:10 soil mass:water volume ratio, where the soil suspensions were agitated for 1 hour, filtered through a 0.45- $\mu$ m filter, and analyzed by inductively coupled, argon-plasma spectrometry (ICAPS; Spectro Arcos ICP, Spectro Analytical Instruments, Inc., Kleve, Germany; Zhang et al., 2014) to determine water-soluble (WS) elemental (i.e., P, K, Ca, Mg, S, Na, Fe, Mn, Zn, and Cu) concentrations. A Mehlich-3 (M3) extraction (Tucker, 1992) was conducted with a 1:10 (mass:volume) soil:extractant solution ratio to determine weak-acid-extractable nutrient (i.e., P, K, Ca, Mg, S, Na, Fe, Mn, Zn, and Cu) concentrations. A strong-acid digestion was conducted using the Environmental Protection Agency (EPA) method 3050B (USEPA, 1996) and analyzed by ICAPS to determine total-recoverable elemental (i.e., P, K, Ca, Mg, S, Na, Fe, Mn, Zn, and Cu) concentrations. All measured initial soil physical and chemical properties are summarized in Table 1.

## **Fertilizer Treatments**

The fertilizer-P treatments used in the rainfall-runoff simulation were CPST (i.e., Crystal Green; Ostara, 2021), MAP, ECSTreal, ECSTsyn, and an unamended control (UC). The CPST material is pelletized and has nutrient concentrations of 11.7% P, 6% N, and 8.3% Mg (Anderson et al., 2020a). Monoammonium phosphate is a pelletized, commonly used, commercially available blended P and N fertilizer that has a reported fertilizer grade of 11-52-0 and contains 20.9% P, 11% N, and 1.5% Mg (Anderson et al., 2021c). Both ECSTsyn and ECSTreal were electrochemically precipitated struvite materials, but ECSTsyn was precipitated from a synthetic

wastewater source, produced to have a similar average P and N composition as typical municipal wastewater (Kékedy-Nagy et al., 2020). The nutrient composition of ECSTsyn was 18.5% P, 3.3% N, and 13.3% Mg (Anderson et al., 2020b). Using similar methods as for the creation of the ECSTsyn material, the ECSTreal material was created from an actual municipal wastewater source collected from the West Side Wastewater Treatment Facility in Fayetteville, AR and contained 15.4% P, 3.3% N, and 13.6% Mg.

Chemical analyses had been previously performed on the ECSTsyn, CPST, and MAP fertilizer-P sources by Anderson et al. (2020b), and similar procedures were used to chemically characterize the ECSTreal fertilizer-P source. Since both the ECSTsyn and ECSTreal materials were electrochemically precipitated, the material produced was a crystalline powder. Since CPST and MAP were originally in pellet form, both were finely ground to match the powder consistency of the ECSTsyn and ECSTreal materials for more valid fertilizer comparisons among chemical characteristics and behavior once soil-applied. Briefly, fertilizer pH and EC were determined potentiometrically in a 1:2 fertilizer mass:water volume ratio. Total N and TC concentrations were measured by high-temperature combustion (Elementar VarioMax CN Analyzer). Water-soluble elemental concentrations (i.e., P, K, Ca, Mg, Na, S, Fe, Mn, Zn, B, and Cu) were determined using a 1:10 fertilizer mass:water volume ratio, where the mixture was agitated for 1 hour, filtered through a 0.45- $\mu$ m filter, and analyzed by ICAPS. Plant-available nutrient concentrations were determined by weak-acid extraction, in which elemental concentrations (i.e., P, K, Ca, Mg, Na, S, Fe, Mn, Zn, B, and Cu) were evaluated after M3 extraction using a 1:10 fertilizer mass:extractant volume ratio (Tucker, 1992) and analyzed by ICAPS (Zhang et al., 2014). A strong-acid digestion (EPA, 1996) was conducted, with analysis

by ICAPS, to evaluate the total-recoverable (TR) elemental concentrations (i.e., P, K, Ca, Mg, Na, S, Fe, Mn, Zn, B, and Cu).

### **Water Collection and Processing**

Three water sources were used for the rainfall-runoff simulations. Rainfall was collected in July 2021 from a single rainfall event. The rainwater used in the rainfall-runoff simulation was to mimic natural rainfall events on the soil. Groundwater was obtained from an existing well in July 2021 from the Savoy Research Center west of Fayetteville, AR (36°07'52"N; 94°18'54"W) to represent an irrigation-water source. The third water source was struvite-removed wastewater (SRW) produced in July 2021 as result of the precipitation of the ECSTreal material described above. The purpose of using the SRW was to evaluate its effectiveness as a potential irrigation-water source after struvite removal. All water sources were refrigerated at 4°C until used. Chemical characterization of the three water sources occurred from the analyses of blanks as part of the rainfall-runoff simulations described below.

### **Rainfall-runoff Simulation Experiment**

To simulate rainfall onto fertilizer-soil mixtures and generate runoff, a wooden rainfall-runoff simulator was constructed measuring 91.1-cm tall by 87.0-cm wide at the front by 73.7-cm deep (Figure 1). A frame containing four sets of seven 31.5-mL min<sup>-1</sup> drip emitters (MLD-AAD, Mister Landscaper Inc., Dundee, FL), separated by approximately 15.9 cm, rested on top of the simulator. For each of the four rows of emitters, three drip emitters were installed in a row 10 cm apart starting at 10.8 cm from the water-source end of the series of emitters and four additional drip emitters were installed 5 cm apart 10.5 cm from the third emitter (Figure 2). All

emitters were connected with 4.8-mm-inside-diameter plastic tubing. Tapered, plastic trays (i.e., short sections of rain gutters for houses), 62-cm long by 12-cm wide at the top by 7-cm wide at the base, were manually placed free-standing at a slope of 22.1% below each row of drip emitters to collect runoff (Figure 1).

On a day of a set of rainfall-runoff simulations, water stored in a 20-L plastic carboy was removed from the refrigerator approximately 1 hr prior to use. The carboy was set on top of a ladder 176.5 cm above and immediately adjacent to the rainfall simulator. The carboy's spigot was connected to 6.4-mm-inside-diameter plastic tubing to gravity-flow to and through the drip emitters. The water delivery rate was approximately  $3.5 \text{ cm hr}^{-1}$  to represent a substantial, high-intensity storm event.

Tea bags (TamBee, B07TCDT76Q), 15-cm wide by 20-cm long and made from a synthetic fiber, were filled with 175 g of air-dried soil that had previously been sieved through the 6-mm mesh screen. An agriculturally relevant fertilizer-P rate ( $56 \text{ kg P}_2\text{O}_5 \text{ ha}^{-1}$ ) was determined based on the fertilizer-P recommendation for soybeans (*Glycine max* L. [Merr.]) grown on a loamy Arkansas soil (Slaton et al., 2013). The appropriate mass of each fertilizer material (i.e., CPST, ECSTsyn, ECSTreal, and MAP) was added to the soil-filled tea bags to deliver the fertilizer-P rate of  $56 \text{ kg P}_2\text{O}_5 \text{ ha}^{-1}$  based on the air-dry mass of soil added to the tea bag, but the nitrogen rate was not balanced among fertilizer treatments since the objective of the study did not include evaluating any plant response. The tea bags were then gently, manually massaged to mix the fertilizer in with the soil. Since air-dried soil was initially used, once fertilizers were mixed with the soil, each tea bag was pre-wet using a squirt bottle with 20 mL of the appropriate water source (i.e., rainwater, groundwater, or SRW) one week before conducting the rainfall simulation to increase the soil moisture content and to minimize potential soil and



fertilizer loss through the tea bags upon handling. Three replications of each soil-fertilizer combination were prepared for a total of 60 tea bags for each of three water sources, totaling 180 tea bags.

Rainfall simulations were performed on 21, 22, and 23 July 2021 (Trial 1). Each day, a single water source was used for rainfall simulations. The sequence of tea bags containing each soil-fertilizer combination used for rainfall simulations were randomized within each water source. Prior to any rainfall simulations, the plastic tubing was primed with the water source to be used for raining on treatment-filled tea bags. Prior to raining on each set of four treatment-filled tea bags, rainfall was simulated onto the four empty trays and then rainfall was simulated into the four trays with an empty, dry tea bag in the tray to serve as two sets of blanks.

Treatment-filled tea bags were placed four at a time directly under the four drip emitters that were closest together and near the down-slope end of the tray to simulate raindrops impacting the soil and water moving underneath and through the soil from up-slope. Each set of four rainfall simulations were conducted for 6 minutes. Pre-trial tests were conducted to determine that 6 minutes of rainfall would yield an estimated 220 mL of runoff water for conducting all planned chemical analyses and to store extra in case any analyses needed to be re-performed in the future.

Runoff water from each of the four trays was collected in 2400-mL rectangular containers at the end of each tray and poured into 250-mL plastic cups for immediate processing for water quality attributes. The process was repeated until each sample was rained on and all runoff water samples were collected.

After raining on each set of four tea bags, tea bags were laid out on plastic bags to air-dry at room temperature ( $\sim 22^{\circ}\text{C}$ ) for one week. Each day the tea bags were gently, manually turned over to facilitate uniform drying on each side of the tea bag. Once air-dried, the three replications

of each treatment-filled tea bag were placed into its own separate plastic bag for incubation for approximately six months when a second rainfall-runoff simulation was performed. The incubated samples for the rainwater and groundwater water sources were wetted using a beaker with 5 to 10 mL of each respective water source once a month.

Following procedures described above, the second rainfall-runoff simulation experiment was conducted on 15 and 16 December 2021 (Trial 2). Only the rainwater and groundwater sources were used in the second rainfall-runoff simulation experiment due to insufficient quantity of the SRW source.

### **Water Sample Processing and Analyses**

Runoff-sample pH and EC measurement, filtering, and acidification were conducted immediately after sample collection. For pH and EC measurements, approximately 25 mL of each sample were poured into a 50-mL glass beaker. A combination pH/EC probe (Orion Star™ A215 pH/Conductivity Benchtop Multiparameter Meter, Thermo Scientific, Beverly, MA) was placed in the runoff sample for 30 seconds, after which pH and EC readings were recorded. The pH/EC probe was rinsed with distilled water between each individual runoff sample. The pH and EC for all runoff samples were measured within seven minutes of sample collection.

Following pH/EC measurements, approximately 100 mL of each runoff water sample were poured into a small plastic vessel (UC475, Ultimate Clean Environmental Express, Charleston, SC) and filtered through a 0.45- $\mu$ m filter (SC0409, FilterMate, Environmental Express, Charleston, SC) attached to the bottom of a plunger. Approximately 20 mL of filtered runoff sample were poured into four, 20-mL, plastic scintillation vials. Two of the four vials were acidified with two drops of concentrated hydrochloric acid for preservation and analysis of

total soluble elemental concentrations (i.e., TP, Ca, Mg, and Fe), while the other two vials were left unacidified for nitrate + nitrite ( $\text{NO}_3 + \text{NO}_2$ ),  $\text{NH}_4^+$ , and soluble-reactive P (SRP; primarily orthophosphates) concentration analyses. Nitrate + nitrite was measured using the salicylate method (EPA 351.2; USEPA, 1993a),  $\text{NH}_4^+$  was measured using the cadmium-reduction method (EPA 353.2; USEPA, 1993b), and SRP was measured using the acid-molybdate method (EPA 365.1; USEPA, 1993c). When a resulting measured concentration was below the instrument's detection limit, the measured value was replaced with one-half of the instrument's concentration detection limit.

To prepare the resulting runoff-water data for statistical analyses, separately for each water source, the average of all eight measured blank values for a water quality parameter was subtracted from the raw water quality parameter measurement to create a blank-corrected value. Since each of the four soils used had somewhat differing initial soil properties, to eliminate potential data bias from dissimilar initial properties, the average was also calculated for the UC replications for each water-source-soil-fertilizer-treatment combination and was also subtracted from the blank-corrected value. Correcting the raw water quality parameter measurements for blanks and subtracting the average UC value resulted in a final water quality parameter data set that varied from negative to positive values for all measured water quality parameters, which is hereafter referred to as the change in water quality data to better address actual the soil-fertilizer interactions without being skewed by somewhat dissimilar initial soil properties.

## **Statistical Analyses**

Based on a completely random design, a one-factor analysis of variance (ANOVA) was conducted in SAS (version 9.4, SAS Institute, Inc., Cary, NC) using PROC GLIMMIX to

evaluate the differences in initial soil properties among the four soils used in the rainfall-runoff simulations. A gamma distribution was used for statistical analyses of initial soils and water data.

It was impractical to randomize the water sources for the rainfall-runoff simulations, but the soil-fertilizer treatment combinations were randomized within a water source. Therefore, a randomized block design was assumed for the purposes of statistical analyses of runoff-water-quality data. Consequently, a three-factor ANOVA was performed using PROC GLIMMIX in SAS to evaluate the effects of water source, soil, fertilizer-P source, and their interactions on the change in runoff water pH, EC, total soluble P, K, Ca, Mg, S, Na, Fe, Mn, Zn, and Cu, and  $\text{NO}_3 + \text{NO}_2$ ,  $\text{NH}_4$ , and SRP separately for the first rainfall-runoff trial. Since the change in water quality parameters varied between negative and positive values, a normal distribution was used for statistical analyses of water-quality data.

To evaluate treatment effects for Trial 2 and changes over time (i.e., results from the first compared to the second rainfall-runoff trial) on the soil-fertilizer treatment combinations for the rainwater and groundwater sources only, a four-factor ANOVA was performed using PROC GLIMMIX in SAS evaluate the effects of water source, soil, fertilizer-P source, time (i.e., trial), and their interactions on the change in runoff water pH, EC, total soluble P, K, Ca, Mg, S, Na, Fe, Mn, Zn, and Cu, and  $\text{NO}_3 + \text{NO}_2$ ,  $\text{NH}_4$ , and SRP. Since the change in water-quality parameters varied between negative and positive values, a normal distribution was also used for analyses of water-quality data for both trials. For all data analyses, significance was judged at  $P < 0.05$ . When appropriate, means were separated by the least significant difference at the 0.05 level.

## **Results and Discussion**

### **Initial soil property differences**

All initial soil properties evaluated differed ( $P < 0.01$ ) among soils (Table 1). Percent clay was largest in the Calloway soil, smallest in the Dapue soil, and was intermediate for the Roxana and Creldon soils, which did not differ (Table 1). Similar to percent clay, soil pH was largest and most alkaline in the Calloway soil, smallest and most acidic in the Dapue soil, and was intermediate for the Roxana and Creldon soils (Table 1). Total C, TN, SOM, and  $\text{NO}_3\text{-N}$  were numerically largest in the Dapue soil and smallest in the Roxana soil (Table 1).

For WS nutrients, P, Ca, and Fe concentrations differed ( $P < 0.01$ ) among all soils (Table 1). Water-soluble P, Mg, and Fe were numerically largest in the Roxana soil and numerically smallest in the Calloway soil (Table 1). However, unlike P and Fe, Mg did not differ between the Calloway and Creldon soils. Water-soluble Ca was largest in the Dapue soil and smallest in the Roxana soil (Table 1).

For M3-extractable nutrients, P, Mg, Ca, and Fe concentrations differed ( $P < 0.01$ ) among all soils (Table 1). Mehlich-3 Ca and Fe were largest in the Calloway soil, but M3 Ca was smallest in the Roxana soil, while M3 Fe was smallest in the Creldon soil (Table 1). Mehlich-3 P was largest in the Roxana soil and smallest in the Calloway soil, while M3 Mg was largest in the Creldon soil and smallest in the Roxana soil (Table 1).

For total-recoverable nutrients, P, Mg, and Fe concentrations differed ( $P < 0.01$ ) among all soils (Table 1). Total-recoverable Fe and Ca were largest in the Calloway soil, but TR Fe was smallest in only Roxana, whereas TR Ca was smallest in both Roxana and Creldon, which did not differ (Table 1). Total-recoverable P was largest in the Dapue soil and smallest in the Creldon soil, while TR Mg was largest in the Roxana soil and smallest in the Dapue soil (Table 1).

Soil nutrient concentrations vary depending on which analysis technique is used. Water-soluble concentrations tend to be lowest, while TR concentrations are largest, where, in the current study, are around 100 to 200 times larger than WS concentrations, and M3 concentrations reflect plant-available nutrient concentrations. However, WS concentrations are most environmentally relevant for studying the impacts of fertilizer-P effects on runoff-water quality.

### **Initial water property differences**

All initial water properties evaluated, except Fe, differed ( $P < 0.01$ ) among water types (Table 2). Water pH, EC, TP, K, Ca, Mg, S, Na, Fe, Mn, Zn, Cu, B,  $\text{NO}_3 + \text{NO}_2$ ,  $\text{NH}_4$ , and SRP concentrations differed among all water types (Table 2). Of the water properties evaluated, most were largest in struvite-removed wastewater, except for Ca and  $\text{NO}_3 + \text{NO}_2$ , which were largest in groundwater (Table 2). Water pH, Ca, Mg, and  $\text{NO}_3 + \text{NO}_2$  were numerically smallest in rainwater, while TP,  $\text{NH}_4$ , and SRP were numerically smallest in groundwater (Table 2).

Since responses from both the unamended controls and blanks were subtracted from individual treatment replicates, pH was the main initial water property that affected resulting water quality among soil-fertilizer-water-type combinations. Initial water pH was most alkaline for the struvite-removed wastewater and least alkaline for rainwater. The alkalinity differences among initial water types affected elemental and compound solubilities, particularly for the rainfall simulation conducted after 6 months of incubation.

### **Initial treatment effects on runoff water quality (Trial 1)**

With the exception of runoff EC, every other measured water quality parameter (i.e., pH, TP, Ca, Mg, Fe,  $\text{NO}_3 + \text{NO}_2$ ,  $\text{NH}_4$ , and SRP) change was affected ( $P < 0.05$ ) by a combination of

two or more treatments (i.e., water type, soil, and/or fertilizer-P source; Table 3). Runoff water quality changes are being presented where responses from blanks without soil and the UC were subtracted so that the change results isolate the actual effect of the fertilizer addition rather than being complicated by the inherent differences among initial soil (Table 1) and water (Table 2) properties.

Averaged across fertilizer-P sources, runoff-water pH,  $\text{NH}_4$ , Ca, Mg, and Fe concentration changes differed ( $P < 0.05$ ) among water type-soil treatment combinations (Figure 3; figure 4). Water pH influences the fraction of soluble nutrients compared to what might remain sorbed to soil solids. Of the three water types, runoff-water pH changes in response to rainwater differed from zero in all soils except for Creldon, where runoff pH decreased in the Dapue and Calloway soils, but increased in the Roxana soil (Figure 3). In addition, runoff-pH changes decreased from zero in the Dapue soil-wastewater treatment combination (Figure 3). Runoff-pH changes from all other water type-soil treatment combinations did not differ from zero or from each other (Figure 3). The decrease in runoff pH among the Dapue- and Calloway-rainwater and Dapue-wastewater combinations, which did not differ among themselves, was lower than the runoff pH increase from rainwater in the Roxana soil (Figure 3).

The exact reasons for the differential change in runoff pH are likely complicated by the complex interaction among numerous initial soil and water properties. However, it is likely that the runoff pH response in the Dapue soil was dominated by the lowest initial soil pH and largest initial soil TC and SOM concentrations that impacted an acidifying effect to the runoff water. The runoff pH increase from rainwater in the Roxana soil was likely related to low buffer capacity from the coarsest soil texture so that the alkaline rainwater dominated the response. For the Calloway soil, the decreased runoff pH from rainwater was likely due to the lower initial

rainwater pH compared to the initial soil pH, whereas the initial pH for the groundwater and wastewater were both greater than the initial soil pH.

Excluding the groundwater-Calloway soil and rainwater-Roxana soil combinations, runoff-water  $\text{NH}_4$  concentration changes for all other water type-soil combinations differed ( $P < 0.05$ ) from zero (Figure 3). The greatest runoff-water  $\text{NH}_4$  concentration decrease occurred from the wastewater treatment within each soil (Figure 3), in which all runoff-water  $\text{NH}_4$  concentration changes for the wastewater-soil combinations also differed from each other (Figure 3).

All significant  $\text{NH}_4$  soil-water responses were likely due to the initial soil (Table 1) and water  $\text{NH}_4$  concentrations (Table 2). However, the wastewater-soil treatment combinations had the greatest decrease in  $\text{NH}_4$ . The large initial  $\text{NH}_4$  concentration in the wastewater was likely retained in the soil during rainfall and was responsible for the runoff  $\text{NH}_4$  decrease in all soil-wastewater treatment combinations.

In addition, averaged across water types and soils, runoff-water  $\text{NH}_4$  concentration changes differed ( $P = 0.02$ ) among fertilizer treatments (Table 3). Runoff-water  $\text{NH}_4$  concentration changes for all four fertilizer treatments (i.e., CPST, MAP, ECSTreal, and ECSTsyn) differed from zero (i.e., -1.35, -1.14, -1.28, and -1.25  $\text{mg L}^{-1}$ , respectively). The MAP treatment had the numerically smallest runoff-water  $\text{NH}_4$  concentration change, which differed from ECSTreal and CPST, while CPST had the numerically largest runoff-water  $\text{NH}_4$  concentration change (Table 3). Runoff-water  $\text{NH}_4$  concentration differences from ECSTsyn did not differ from any other fertilizer treatment (Table 3). Added  $\text{NH}_4$  from the fertilizers was uniformly retained by the soils, likely due to cation exchange sites from the clays, TC, and/or SOM.



In contrast to  $\text{NH}_4$ , runoff-water Ca concentration changes differed from zero in three of four groundwater-soil combinations (i.e., Crelton, Dapue, and Roxana), but did not differ from zero for any of the rainwater- or wastewater-soil combinations (Figure 3). However, runoff-water Ca concentration changes did not differ from each other in the groundwater-Calloway soil combination or among any other rainwater-soil combination (Figure 3). Also, in contrast to  $\text{NH}_4$ , runoff-water Ca concentrations decreased the most in the groundwater-soil combinations, which was a 5 to 30 times greater decrease compared to all other water type-soil combinations (Figure 3).

Groundwater had a large initial pH (7.9) in addition to having the largest initial Ca concentration (Table 2). The combination of large initial pH and large initial Ca concentration likely facilitated the precipitation of a portion of the Ca as Ca-P compounds to retain Ca and result in lower runoff Ca for the Crelton, Dapue, and Roxana soils. However, the Calloway soil was unaffected by all three water types, possibly due to its large initial pH leading to no additional precipitation of Ca.

Similar to  $\text{NH}_4$ , runoff-water Mg concentration changes differed from zero in all wastewater-soil combinations and in the groundwater-Roxana soil combination (Figure 4). Runoff-water Mg concentration changes in all rainwater- and groundwater-soil combinations did not differ from each other. Runoff-water Mg concentration changes in the wastewater treatment across the four soils decreased 5 to 25 times more than any other water type-soil combination (Figure 4). Wastewater had the largest initial pH of the three water types (9.8; Table 2), which likely caused a fraction of the Mg to precipitate as Mg-P and/or Ca-Mg-P compounds in all four soils, resulting in the runoff-Mg decrease from zero. There was also a significant runoff-Mg increase in the Roxana-groundwater combination, but the increase was small.

Runoff-water Fe concentration changes differed from zero in three of four rainwater-soil combinations, excluding the Dapue soil, and was greater than zero for the groundwater-Calloway soil combination (Figure 4). Runoff-water Fe concentration from all water type-soil combinations, except for those that differed from zero and the wastewater-Dapue soil combination, did not differ from each other (Figure 4). Of the water type-soil combination whose runoff-water Fe concentration change differed from zero, the rainwater-Calloway and -Roxana soil combinations did not differ from each other and both changes decreased from zero (Figure 4). Similarly, runoff-water Fe concentration changed for the rainwater-Creldon and groundwater-Dapue soil combinations did not differ from each other, but both changes were greater than zero (Figure 4). The decrease in runoff Fe in the Calloway- and Roxana-rainwater treatment combinations was likely due to the relatively large initial soil and water pHs, as Fe solubility in the soil decreases as pH increases. For similar reasons, in combination with the low initial Fe in the Calloway and Creldon soils (Table 1), there was an increase in runoff Fe in the Creldon-rainwater and Calloway-groundwater combinations.

Averaged across soils, runoff-water Fe concentration changes also differed ( $P = 0.04$ ) among water type-fertilizer-P source combinations (Figure 5). Runoff-water Fe concentration changes differed from zero in three of four rainwater-fertilizer-P source combinations, excluding CPST (Figure 5). Runoff-water Fe concentration changes were less than zero in all wastewater-fertilizer-P source combinations but were greater than zero in all groundwater-fertilizer-P source combinations (Figure 5). In general, the rainwater treatment caused the greatest runoff-water Fe concentration change from zero among all water treatments (Figure 5). In all rainwater-fertilizer-P source combinations, except for ECSTreal, the runoff-water Fe concentration change was less than zero (Figure 5). Similarly, the runoff-water Fe concentration change for all struvite

fertilizer-P sources (i.e., CPST, ECSTreal, and ECSTsyn) was similar to MAP in all treatment combinations, except for the rainwater-ECSTreal combination (Figure 5). All treatments did not differ from each other, except for the rainwater-CPST, -MAP, and -ECSTsyn and wastewater-ECSTsyn combinations, which did not differ from each other (Figure 5).

The increase in runoff Fe from only ECSTreal in the rainwater treatment was likely due to the original conditions under which the ECSTreal was formed. Unlike ECSTsyn, ECSTreal was precipitated from a real municipal wastewater source, while CPST and MAP were also created from a source with known composition. There could have been unknown or unexpected elemental concentrations within the wastewater from which ECSTreal was originally created. Because Fe is more available at more acidic pHs, the non-significant runoff Fe change that occurred in the groundwater and wastewater treatments was likely due to the groundwater's and wastewater's initial pHs > 7.8 (Table 2).

Runoff-water TP,  $\text{NO}_3 + \text{NO}_2$ , and SRP concentration changes differed ( $P < 0.05$ ) among water type-soil-fertilizer-P source treatment combinations (Table 4). There were few consistent trends among water type-soil-fertilizer-P source treatment combinations, and most were complex. Most runoff-water TP concentration changes differed from zero among treatment combinations, particularly within the struvite-removed wastewater treatment (Table 4). All runoff-water TP concentration changes from the struvite-removed wastewater were negative in three of four soil-fertilizer-P source combinations, excluding the Roxana soil (Table 4). The numerically largest runoff-water TP concentration changes were in the rainwater-Roxana soil combination, excluding the CPST fertilizer-P source. The numerically smallest runoff-water TP concentration changes occurred in the wastewater-Calloway soil combinations (Table 4). Few runoff-water TP concentration changes differed from each other among fertilizer-P sources

within water type-soil combinations (Table 4). In most treatment combinations, runoff-water TP concentration changes from the struvite-P sources (i.e., CPST, ECSTsyn, and ECSTreal) were similar to MAP (Table 4). However, within the rainwater-Creldon and groundwater-Dapue soil combinations, runoff-water TP concentration changes from ECSTsyn differed from all other fertilizer treatments. Within the rainwater-Roxana soil combination, runoff-water TP concentration changes did not differ among MAP, ECSTreal, or ECSTsyn (Table 4).

The negative runoff-TP concentration changes in the wastewater-soil-fertilizer treatment combinations could have been caused by the large initial wastewater Ca and Mg concentrations binding with P due to the wastewater's alkaline pH (Table 2). However, in the wastewater-Roxana soil combination, the TP concentration change was positive. The positive TP concentration change, in contrast to the other three soils' negative TP concentration change, was likely due to the large initial TP, but small initial Ca (Table 1), causing no precipitation of Ca-P compounds. In every water-soil-fertilizer combination but one (i.e., wastewater-Roxana), either ECSTsyn or ECSTreal consistently had the largest runoff-TP concentration (Table 4). The large runoff-TP concentration of ECSTreal and ECSTsyn was unexpected since the initial P concentration of the ECST fertilizers was the intermediate among the four fertilizer treatments, but the initial Mg concentration for ECSTsyn and ECSTreal was the largest (i.e., 13.3% and 13.6%). The large runoff-TP concentration in the ECSTsyn and ECSTreal treatments, in contrast to CPST and MAP, may have been due to the timing of when each fertilizer material solubilized and subsequently ran off during the rainfall event. Being readily soluble, MAP may have dissolved within the first few days of pre-wetting and had sufficient time for Ca-Mg-P compounds to form, lowering the runoff-TP concentration. In contrast, the slow-release nature of

ECST, as reported by Anderson et al. (2021c), could have led to the simulated rainfall promoting ECST solubilization, increasing the runoff-TP concentration.

Runoff-water SRP concentration changes were similar to runoff-water TP concentration changes in which treatments differed from zero and which treatments differed from each other (Table 4). Similar to runoff-water TP, runoff-water SRP concentration changes differed from zero in almost all water type-soil-fertilizer-P source combinations, except for the groundwater-Calloway soil combination (Table 4). Also similar to runoff-water TP, the numerically largest runoff-water SRP concentration changes occurred for the rainwater-Roxana soil treatment combination, excluding CPST (Table 4). The numerically smallest runoff-water SRP concentration changes occurred for the wastewater-Calloway soil combinations (Table 4). Few runoff-water SRP concentration changes differed from each other among fertilizer-P sources within water type-soil combinations (Table 4). In general, except for the rainwater-Calloway and Creldon soil and wastewater-Dapue soil combinations, runoff-water SRP concentration changes were numerically smaller than runoff-water TP concentration changes. In most treatment combinations, runoff-water SRP concentration changes from the struvite-P sources were similar to MAP (Table 4).

Runoff-SRP concentration responses were likely the result of reasons similar to that of runoff-TP concentrations. The negative runoff-SRP concentration changes in the wastewater-soil-fertilizer combinations could have been caused by the large initial wastewater Ca and Mg concentrations binding with P due to the wastewater's alkaline pH. However, in the wastewater-Roxana soil combination, the SRP concentration change was generally positive. The positive TP concentration change, in contrast to the other three soils' negative SRP concentration change, was likely due to the large initial TP, but small initial Ca causing no precipitation of Ca-P

compounds. Runoff-SRP concentration changes were smaller than for runoff TP in almost all instances, which was consistent with TP measuring all P forms and SRP only measuring plant-available P forms.

In contrast to runoff-water TP and SRP, most runoff-water  $\text{NO}_3 + \text{NO}_2$  concentration changes did not differ from zero (Table 4). Runoff-water  $\text{NO}_3 + \text{NO}_2$  concentration changes differed from zero in three of four rainwater-Calloway soil-fertilizer-P source combinations, excluding ECSTreal (Table 4). Runoff-water  $\text{NO}_3 + \text{NO}_2$  concentration changes for most treatment combinations also did not differ from each other, except the numerically largest and smallest runoff-water  $\text{NO}_3 + \text{NO}_2$  concentration changes (Table 4). The largest runoff-water  $\text{NO}_3 + \text{NO}_2$  concentration changes occurred for the wastewater-Calloway soil-CPST and the rainwater-Calloway soil-CPST and -MAP combinations (Table 4). The smallest runoff-water  $\text{NO}_3 + \text{NO}_2$  concentration change occurred for the groundwater-Dapue soil-ECSTreal combination (Table 4). In most treatment combinations, runoff-water  $\text{NO}_3 + \text{NO}_2$  concentration changes for the struvite-P sources were similar to MAP (Table 4). Complex interactions between soil, water type, and fertilizer properties made it challenging to explain runoff  $\text{NO}_3 + \text{NO}_2$  concentration changes, as there were no consistent patterns in the results. In contrast to all other measured water-quality parameters, runoff-EC changes were unaffected by water type, soil, or fertilizer-P source (Table 3) and averaged  $4.32 \text{ dS m}^{-1}$  across all treatments.

### **Treatment effects on runoff water quality over time (Trial 2)**

After a 6-month period of incubation between rainfall simulations, with monthly wetting and in contrast to Trial 1, every measured water quality parameter, with the exception of runoff-Mg concentration changes, differed ( $P < 0.05$ ) over time (Table 5). For Trial 2, the struvite-

removed wastewater was not used, thus the water types only consisted of rainwater and groundwater. The remainder of the results in this section will focus on changes over time and differences among treatment combinations from Trial 2 results only.

Changes in runoff-pH, which partially dictates the fractionation between WS and sorbed nutrient concentrations, and runoff-TP, -Ca, and -SRP concentration changes differed ( $P < 0.02$ ; Table 5) among water-soil treatment combinations over time (Table 5). Averaged across fertilizer-P sources, runoff-pH changes decreased over time in all water type-soil combinations, where the decrease was larger for rainwater- compared to groundwater-soil combinations (Table 6). In response to rainwater and groundwater, after 6 months of incubation (Trial 2), all runoff-pH changes differed from zero (Table 6). Runoff-pH changes in groundwater-soil combinations did not differ from each other in Trial 2 (Table 6). In contrast, runoff-pH changes in all rainwater-soil combinations differed from each other in Trial 2, except for the Roxana soil that did not differ from the Dapue or Creldon soils (Table 6). The decrease in runoff pH over time was likely due to the nitrifying processes that occurred during incubation, producing  $H^+$  ions and thus lowering runoff pH. The general decrease in runoff pH was similar to the results of Anderson et al. (2021c), who reported a decrease in soil pH over time in a silty clay loam soil with large initial SOM and clay concentration.

Similar to runoff-pH, runoff-TP concentration changes also decreased over time in all water type-soil combinations, with the larger decreases over time generally occurring in the rainwater-soil combinations (Table 6). In six of the eight water type-soil combinations in Trial 2, with the exception of the groundwater- and rainwater-Roxana soil combinations, runoff-TP concentration changes were small and did not differ from zero and did not differ among themselves (Table 6). However, runoff-TP concentration changes the groundwater- and

rainwater-Roxana soil combinations were largest than for the other six water-type combinations and differed from zero, but did not differ from one another. Soluble TP may have reacted with Ca and/or Mg to form Ca-Mg-P compounds, partially explaining the decrease in runoff TP over time from Trial 1 to Trial 2. However, due to the decrease in runoff pH, it is more likely that runoff TP simply adsorbed onto soil particles over the 6-month incubation period. Shigaki et al. (2007) and Smith et al. (2007) reported a similar decrease in water-soluble P over time in a loam and silt loam soil, respectively.

In contrast to runoff-pH and -TP, runoff-water Ca concentrations at least numerically increased over time in all groundwater-soil and two of four rainwater-soil combinations, excluding the rainwater-Creldon, which did not change over time, and the rainwater-Calloway soil combination, which significantly decreased over time (Table 6). For Trial 2 only, the largest runoff-water Ca concentration increase occurred in the rainwater-Dapue soil, while the largest decrease occurred in the rainwater-Calloway soil combination (Table 6). The increase in runoff Ca over time was likely due to the decrease in runoff pH and Ca being released from cation exchange sites. The runoff pH of the Dapue soil-rainwater combination was already low, likely resulting in no change of the cation exchange site components and explaining the decrease in runoff Ca in the rainwater-Dapue treatment.

Similar to runoff-water TP, runoff-water SRP concentration changes also decreased significantly over time in all water type-soil combinations, with the larger decreases generally occurring in response to rainwater compared to groundwater (Table 6). Similar to runoff-water TP, in Trial 2, runoff-water SRP concentration changes were generally small, where most did not differ from a change of zero and many did not differ from one another (Table 6). For Trial 2 only, the largest runoff-water SRP concentration increases occurred in the groundwater-Roxana



and rainwater-Creldon soil, combinations, which differed from zero, but did not differ from one another (Table 6). The explanation for changes in runoff SRP is similar to that of runoff TP, where it was likely that runoff SRP adsorbed onto soil particles over time. Runoff-SRP was lower than runoff-TP concentration changes in almost all instances, which was consistent with TP measuring all P forms and SRP only measuring plant-available P forms.

Averaged across water types, runoff-water TP and SRP concentration changes also differed ( $P < 0.01$ ; Table 5) among soil-fertilizer-P source combinations over time (Table 7). Treatment combination effects on runoff-water TP and SRP concentration changes over time were the same (Table 7). Runoff-water TP and SRP concentration changes were generally small for Trial 2, where, with the exception of the same two treatment combinations, most changes did not differ from zero, and decreased over time in all soil-fertilizer-P source treatment combinations (Table 7). The largest runoff-water TP and SRP concentration changes from Trial 2 occurred in the Roxana soil-CPST and -ECSTsyn combinations, which were both larger than zero, but did not differ from one another, and both were larger than the changes measured in all other soil-fertilizer-P source combinations (Table 7). Runoff-water TP and SRP concentration changes for Trial 2 did not differ between the three struvite-P sources in the Creldon, Dapue, or Calloway soils and behaved similar to MAP (Table 7). However, runoff-water TP and SRP concentration changes for Trial 2 from the Roxana soil-CPST and -ECSTsyn were both greater than changes from Roxana soil-ECSTreal and -MAP combinations, which did not differ (Table 7).

The explanation for changes in runoff-SRP and -TP concentration changes were similar, where it was likely that runoff TP and SRP adsorbed to soil particles over time. In most instances, runoff-TP and -SRP concentration changes were largest in the ECSTsyn or ECSTreal

combinations in Trial 1. The large runoff-TP and -SRP concentration changes were unexpected since the initial P concentration of the ECST fertilizers was intermediate among the four fertilizer treatments, but the initial Mg concentration for ECSTsyn and ECSTreal was the largest (i.e., 13.3% and 13.6%). As previously, the large runoff-TP concentration change in the ECSTsyn and ECSTreal treatments, in contrast to CPST and MAP, may have been due to the timing of when each fertilizer material solubilized and subsequently ran off during the rainfall event. Being readily soluble, MAP may have dissolved within the first few days of pre-wetting and had sufficient time to form Ca-Mg-P compounds, lowering the runoff-TP concentration. The increase in runoff-TP concentration for ECST may have been due to the slow-release nature of ECST (Anderson et al., 2021c) and the promotion of ECST dissolution by rainfall. After the 6-month incubation period, there was likely sufficient time for Ca-Mg-P compounds to form in all fertilizer treatment combinations. Anderson et al. (2021c) reported that CPST, not ECST, had the greatest increase in water-soluble P over nine months. However, Anderson et al. (2021c) also reported that, in general, there was a decrease in water-soluble P after the initial wetting event, which was similar to the results of this study.

In contrast to the other soluble nutrients, averaged across water type and fertilizer-P source, runoff-water Fe and NH<sub>4</sub> concentration changes differed ( $P < 0.01$ ; Table 5) among soils over time (Figure 6). For three of the four soils (i.e., Crelton, Dapue, and Roxana), runoff-water Fe concentration changes did not differ over time, changes for Trial 2 did not differ from zero, and changes were all similar to one another (Figure 6). However, for the Calloway soil, runoff-water Fe concentration changes decreased  $> 0.5 \text{ mg L}^{-1}$  over the six months between trials, in which the decrease differed from zero and differed from all other soil-time combinations (Figure

6). The decrease in runoff Fe over time in the Calloway soil was likely due to the large initial pH of the Calloway soil (Table 1), where Fe availability decreased at the large pH.

In contrast to Fe, averaged across water type and fertilizer-P sources, runoff-water  $\text{NH}_4$  concentration changes decreased over time for the Creldon, Dapue, and Roxana soils, where the change for Trial 2 only was greater than zero for the Dapue soil, but did not differ from the change in the Creldon or Roxana soils (Figure 6). However, the runoff-water  $\text{NH}_4$  concentration change did not differ over time for the Calloway soil, though the change for Trial 2 only differed from zero, did not differ from the change in the Roxana soil, but the change was smaller than in the Creldon and Dapue soils (Figure 6). The decrease in runoff- $\text{NH}_4$  concentrations over time was likely due to nitrification occurring during the incubation process. There was no change over time in runoff- $\text{NH}_4$  concentration in the Calloway soil, but the Calloway soil had the smallest initial  $\text{NH}_4$  concentration and largest initial pH (Table 1), which may have contributed to an increased nitrification rate and the runoff- $\text{NH}_4$  concentration decrease from zero.

In contrast to all other measured water-quality parameters, runoff-water EC and  $\text{NO}_3 + \text{NO}_2$  concentration changes were complex and differed ( $P < 0.03$ ; Table 5) among water type-soil-fertilizer-P source combinations over time (Table 8). Runoff-water EC changes for Trial 2 did not differ from zero, except for the groundwater-Calloway-MAP and -ECSTsyn, rainwater-Dapue-MAP, - ECSTreal, and -ECSTsyn combinations, where all changes were greater than zero (i.e., an increase in runoff-water EC; Table 8). Similarly, most runoff-water EC changes did not change over time, except for the groundwater-Calloway-MAP, rainwater-Dapue-MAP, and - ECSTsyn combinations that increased over time, where the Trial 2 change did not differ among themselves, and the rainwater-Calloway-CPST and -MAP combinations that decreased over time, where the Trial 2 changes did not differ between them (Table 8). Runoff-water EC changes

for most other water type-soil-fertilizer-P source combinations did not differ among themselves (Table 8). Most of the changes and interactions in EC among treatment combinations were likely due to the initial EC among the soils (Table 1) and water types (Table 3).

Similar to EC, runoff-water  $\text{NO}_3 + \text{NO}_2$  concentration changes for Trial 2 did not differ from zero, except for the groundwater-Calloway-MAP, -ECSTreal, and -ECSTsyn, and rainwater-Dapue-MAP, ECSTreal, and -ECSTsyn combinations, where all Trial 2 changes were greater than zero (i.e., an increase in runoff-water  $\text{NO}_3 + \text{NO}_2$  concentration; Table 8). Similarly, most runoff-water  $\text{NO}_3 + \text{NO}_2$  concentration changes did not change over time, except for the groundwater-Calloway-MAP and rainwater-Dapue-MAP and -ECSTsyn combinations that increased over time, where the Trial 2 change did not differ among themselves, and the rainwater-Calloway-MAP and -ECSTsyn combinations that decreased over time, where the Trial 2 changes did not differ between them (Table 8). Runoff-water  $\text{NO}_3 + \text{NO}_2$  concentration changes for most other water type-soil-fertilizer-P source combinations did not differ among themselves (Table 8). The change in runoff  $\text{NO}_3 + \text{NO}_2$  concentrations over time was likely due to mineralization of organic matter, nitrification of  $\text{NH}_3$ , and/or the denitrification of added fertilizer materials, especially in the Calloway soil, which had the greatest pH and greatest runoff-water  $\text{NO}_3 + \text{NO}_2$  concentration changes among the four soil treatments.

## **Implications**

On a global scale, capturing and reusing P from wastewater instead of converting mined phosphate rock into fertilizer could potentially lead to an increase in global food security due to lowered reliance on a finite resource. Struvite also has the potential to maintain or increase crop yields, potentially further increasing food production and security. On a regional scale, struvite

could lower nutrient loads to already sensitive environments and resources. Recovery of struvite from wastewater also leads to a reduction of nutrients in wastewater effluent and sewage sludge, and struvite nutrient runoff concentrations are no different from runoff concentrations of commercially available fertilizers, such as MAP. However, as there is not yet a pelletized form of ECST, this study showed that timing of ECST application may influence runoff water quality. Furthermore, there were no differences between runoff nutrient concentrations from MAP and ECST, thus the application of and potential runoff from ECST would not exacerbate current environmental, nutrient-input issues. Because of reduced nutrient loads from both recovery of nutrients in ECST and similar nutrient concentrations from MAP compared to ECST, having an alternative fertilizer-P source to traditional, commercially available fertilizers with struvite may lead to a decrease in eutrophication and increase in overall water quality in many areas.

Currently, it is too expensive to produce ECST on a commercial scale. Struvite is more costly than traditional fertilizers to produce, partially due to the experimental state of the fertilizer and that commercial-scale cost information is still unavailable (Brye et al., 2022). However, struvite generally contains more N than many traditional fertilizer-P-only sources, such as TSP, which can reduce the cost of extra needed urea inputs (Omidire et al., 2022). Furthermore, struvite also has the potential to outperform traditional fertilizer-P sources in terms of crop yield (Omidire et al., 2022). Continued research is needed to compare the cost of struvite, particularly ECST, production to the production of traditional, commercially available fertilizer-P sources, but, as more research is conducted, there will be opportunity to increase the efficiency and decrease the cost of struvite production.

## **Conclusions**

This laboratory study evaluated the effects of soil, fertilizer-P source, and water source over a six-month period on runoff-water quality parameters from laboratory-conducted, rainfall-runoff simulations. Numerous runoff-water-quality parameters responded similarly across multiple soils. Most significantly, with respect to P, the struvite fertilizers (i.e., CPST, ECSTreal, and ECSTsyn) responded similarly to MAP in most treatment combinations. When struvite did not respond similarly to MAP, the differences in interactions were likely due to the slow-release nature of struvite and/or the unpredictable elemental composition of the wastewater from which ECSTreal was derived.

To date, the environmental impacts of struvite, as a relatively new, potential alternative fertilizer-P source generated from recycled nutrient in simulated and real wastewater stream, have been under-studied. The similar water-quality responses of the struvite fertilizers among the various soils and water types compared to MAP suggest that struvite has similar runoff-water-quality implications as at least one widely used, commercially available fertilizer-P source. However, more long-term soil and field studies are needed to fully understand the environmental and agronomic implications of using struvite as an alternative fertilizer-P source to commonly used, commercially available fertilizer-P sources.

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**Table 1.** Summary of soil physical and chemical property differences among the Calloway, Roxana, Creldon, and Dapue soil series used in the rainfall-runoff simulations.

| Soil Property   | <i>P</i> | Calloway            | Roxana  | Dapue   | Creldon |
|---|----------|---------------------|---------|---------|---------|
| Sand (g g <sup>-1</sup> )                                   | < 0.01   | 0.09 d <sup>†</sup> | 0.44 a  | 0.20 c  | 0.24 b  |
| Silt (g g <sup>-1</sup> )                                   | < 0.01   | 0.79 a              | 0.47 d  | 0.74 b  | 0.67 c  |
| Clay (g g <sup>-1</sup> )                                   | < 0.01   | 0.12 a              | 0.10 b  | 0.07 c  | 0.09 b  |
| pH  | < 0.01   | 7.46 a              | 6.17 b  | 5.77 d  | 6.03 c  |
| Electrical conductivity (dS m <sup>-1</sup> )               | < 0.01   | 0.17 b              | 0.11 d  | 0.19 a  | 0.13 c  |
| Total C (%)   | < 0.01   | 1.14 c              | 0.28 d  | 2.57 a  | 1.65 b  |
| Total N (%)   | < 0.01   | 0.11 c              | 0.03 d  | 0.2 a   | 0.1 b   |
| C:N ratio   | < 0.01   | 10.0 c              | 10.5 b  | 11.8 a  | 11.5 a  |
| Soil organic matter (%)                                     | < 0.01   | 2.6 c               | 0.7 d   | 4.2 a   | 3.4 b   |
| NO <sub>3</sub> -N (mg kg <sup>-1</sup> )                   | < 0.01   | 10.6 c              | 9.0 d   | 35.8 a  | 15.3 b  |
| NH <sub>4</sub> -N (mg kg <sup>-1</sup> )                   | < 0.01   | 5.3 c               | 6.4 b   | 17.9 a  | 19.5 a  |
| Water-soluble concentrations (mg kg <sup>-1</sup> )         |          |                     |         |         |         |
| P   | < 0.01   | 1.8 d               | 11.9 a  | 5.7 b   | 3.5 c   |
| K   | < 0.01   | 6.2 c               | 44.8 a  | 19.7 b  | 20.3 b  |
| Ca  | < 0.01   | 58.8 b              | 34.0 d  | 83.0 a  | 43.7 c  |
| Mg  | < 0.01   | 8.9 c               | 21.7 a  | 16.7 b  | 16.3 b  |
| S   | < 0.01   | 12.0 a              | 4.9 c   | 12.2 a  | 9.5 b   |
| Na  | < 0.01   | 19.5 a              | 4.1 d   | 4.8 c   | 5.9 b   |
| Fe  | < 0.01   | 1.5 d               | 49.7 a  | 1.9 c   | 12.6 b  |
| Mn  | < 0.01   | 0.12 d              | 0.57 c  | 2.2 a   | 1.7 b   |
| Zn  | < 0.01   | 0.52 b              | 0.33 c  | 0.90 a  | 0.47 bc |
| Cu  | < 0.01   | 0.03 c              | 0.08 a  | 0.06 b  | 0.04 c  |
| Mehlich-3-extractable concentrations (mg kg <sup>-1</sup> ) |          |                     |         |         |         |
| P   | < 0.01   | 11.4 d              | 93.3 a  | 41.7 b  | 17.0 c  |
| K   | < 0.01   | 46.1 d              | 145.3 a | 102.0 c | 113.0 d |
| Ca  | < 0.01   | 2006 a              | 933 d   | 1601 b  | 1115 c  |
| Mg  | < 0.01   | 276.3 b             | 194.3 d | 216.0 c | 328.0 a |
| S   | < 0.01   | 12.0 b              | 5.7 c   | 17.0 a  | 13.0 b  |
| Na  | < 0.01   | 29.8 a              | 10.0 b  | 7.7 c   | 10.4 b  |
| Fe  | < 0.01   | 303.8 a             | 200.7 b | 139.3 c | 112.0 d |
| Mn  | < 0.01   | 244.3 a             | 32.9 c  | 98.7 b  | 101.3 b |
| Zn  | < 0.01   | 2.6 c               | 2.4 c   | 30.9 a  | 4.2 b   |
| Cu  | < 0.01   | 1.6 b               | 1.1 c   | 2.6 a   | 1.2 c   |
| Total-recoverable concentrations (mg kg <sup>-1</sup> )     |          |                     |         |         |         |
| P   | < 0.01   | 342.9 c             | 370.7 b | 426.3 a | 248.0 d |
| K   | < 0.01   | 597 d               | 1730 a  | 1436 b  | 1160 c  |
| Ca  | < 0.01   | 2424 a              | 1440 c  | 2251 b  | 1494 c  |
| Mg  | < 0.01   | 1318 c              | 2433 a  | 1202 d  | 1372 b  |
| S   | < 0.01   | 137.2 c             | 42.3 d  | 245.7 a | 154.0 b |
| Na  | < 0.01   | 61.6 a              | 52.7 b  | 26.7 c  | 25.7 c  |
| Fe  | < 0.01   | 16705 a             | 8340 c  | 11059 b | 18023 a |
| Mn  | < 0.01   | 1337 a              | 177 d   | 687 c   | 1163 b  |
| Zn  | < 0.01   | 32.9 b              | 22.3 c  | 97.0 a  | 31.3 b  |
| Cu  | < 0.01   | 6.3 b               | 4.2 d   | 9.0 a   | 4.7 c   |

<sup>†</sup>Means in a row with different letters are different at *P* < 0.05

**Table 2.** Summary of water chemical property differences among the rainwater, groundwater, and struvite-removed wastewater sources used in the rainfall-runoff simulations.

| <b>Water Property</b>                                    | <b><i>P</i></b> | <b>Rainwater</b> | <b>Groundwater</b> | <b>Wastewater</b> |
|--|-----------------|------------------|--------------------|-------------------|
| pH   | < 0.01          | 7.22 c           | 7.89 b             | 9.77 a            |
| Electrical conductivity (dS m <sup>-1</sup> )            | < 0.01          | 14.8 c           | 461 b              | 578 a             |
| P (mg kg <sup>-1</sup> )                                 | < 0.01          | 0.14 b           | 0.01 c             | 1.25 a            |
| K (mg kg <sup>-1</sup> )                                 | < 0.01          | 0.41 c           | 1.17 b             | 20.2 a            |
| Ca (mg kg <sup>-1</sup> )                                | < 0.01          | 0.37 c           | 77.9 a             | 27.2 b            |
| Mg (mg kg <sup>-1</sup> )                                | < 0.01          | < 0.01 c         | 1.05 b             | 35.6 a            |
| S (mg kg <sup>-1</sup> )                                 | < 0.01          | 0.16 c           | 0.92 b             | 12.1 a            |
| Na (mg kg <sup>-1</sup> )                                | < 0.01          | 0.41 c           | 13.2 b             | 35.8 a            |
| Fe (mg kg <sup>-1</sup> )                                | 0.38            | 0.18 a           | 0.15 a             | 0.19 a            |
| Mn (mg kg <sup>-1</sup> )                                | < 0.01          | < 0.01 b         | < 0.01 b           | < 0.01 a          |
| Zn (mg kg <sup>-1</sup> )                                | < 0.01          | 0.06 b           | 0.11 a             | 0.05 c            |
| Cu (mg kg <sup>-1</sup> )                                | < 0.01          | < 0.01 b         | < 0.01 b           | < 0.01 a          |
| NO <sub>3</sub> + NO <sub>2</sub> (mg kg <sup>-1</sup> ) | < 0.01          | < 0.01 c         | 5.79 a             | 3.05 b            |
| NH <sub>4</sub> (mg kg <sup>-1</sup> )                   | < 0.01          | 0.27 b           | 0.03 c             | 7.83 a            |
| SRP (mg kg <sup>-1</sup> )                               | < 0.01          | 0.05 b           | < 0.01 c           | 1.17 a            |

<sup>†</sup>Means in a row with different letters are different at  $P < 0.05$ .

**Table 3.** Analysis of variance summary of the effects of water type (W), soil (S), fertilizer (F), and their interactions on the change ( $\Delta$ ) in runoff-water quality properties for Trial 1 of the rainfall-runoff simulation experiment.

| Source of Variation | $\Delta\text{pH}$ | $\Delta\text{EC}^\dagger$ | $\Delta[\text{TP}]^\dagger$ | $\Delta[\text{Ca}]^\dagger$ | $\Delta[\text{Mg}]^\dagger$ | $\Delta[\text{Fe}]^\dagger$ | $\Delta[\text{NO}_3 + \text{NO}_2]^\dagger$ | $\Delta[\text{NH}_4]^\dagger$ | $\Delta[\text{SRP}]^\dagger$ |
|---------------------|-------------------|---------------------------|-----------------------------|-----------------------------|-----------------------------|-----------------------------|---|-------------------------------|------------------------------|
| Water type          | 0.01              | 0.09                      | < 0.01                      | < 0.01                      | < 0.01                      | < 0.01                      | 0.16  | < 0.01                        | < 0.01                       |
| Soil                | < 0.01            | 0.07                      | < 0.01                      | < 0.01                      | < 0.01                      | 0.14                        | < 0.01                                      | < 0.01                        | < 0.01                       |
| Fertilizer          | 0.99              | 0.74                      | < 0.01                      | 0.36                        | 0.55                        | 0.31                        | 0.40  | 0.02                          | < 0.01                       |
| W x S               | < 0.01            | 0.19                      | < 0.01                      | < 0.01                      | < 0.01                      | < 0.01                      | < 0.01                                      | < 0.01                        | < 0.01                       |
| W x F               | 0.14              | 0.35                      | 0.05                        | 0.41                        | 0.47                        | 0.04                        | 0.53  | 0.22                          | 0.01                         |
| S x F               | 0.33              | 0.23                      | < 0.01                      | 0.15                        | 0.58                        | 0.35                        | 0.49  | 0.27                          | < 0.01                       |
| W x S x F           | 0.11              | 0.12                      | < 0.01                      | 0.10                        | 0.88                        | 0.28                        | 0.04  | 0.29                          | 0.02                         |

$^\dagger$  Electrical conductivity, EC; total phosphorus, TP; calcium, Ca; magnesium, Mg; iron, Fe; nitrate + nitrite,  $\text{NO}_3 + \text{NO}_2$ ; ammonium,  $\text{NH}_4$ ; soluble-reactive phosphorus, SRP

**Table 4.** Summary of mean changes ( $\Delta$ ) in water-quality parameters among water type-soil-fertilizer-phosphorus-(P)-source treatment combinations from rainfall-runoff simulation Trial 1.

| Water Type                  | Soil     | Fertilizer-P source <sup>‡</sup> | $\Delta$ [TP] <sup>†,‡</sup><br>(mg L <sup>-1</sup> ) | $\Delta$ [NO <sub>3</sub> + NO <sub>2</sub> ] <sup>†,‡</sup><br>(mg L <sup>-1</sup> ) | $\Delta$ [SRP] <sup>†,‡</sup><br>(mg L <sup>-1</sup> ) |
|-----------------------------|----------|----------------------------------|---|---|--|
| Groundwater                 | Crelton  | CPST                             | 0.16*e-k  | 0.91 c-h  | 0.15*e-k   |
|                             |          | MAP                              | 0.06 jk   | -0.52 e-i   | 0.06 jkl   |
|                             |          | ECSTreal                         | 0.09 h-k  | -0.60 e-i   | 0.08 ijk   |
|                             |          | ECSTsyn                          | 0.23*d-j  | 2.48*b-e  | 0.14*e-k   |
|                             | Dapue    | CPST                             | 0.11 g-k  | 0.83 c-h  | 0.09*ijk   |
|                             |          | MAP                              | 0.12*g-k  | 0.51 c-i  | 0.10*h-k   |
|                             |          | ECSTreal                         | 0.15*e-k  | -2.52*i   | 0.14*e-k   |
|                             |          | ECSTsyn                          | 0.30*cde  | -0.59 e-i   | 0.26*cde   |
|                             | Calloway | CPST                             | 0.06 k  | -0.08 d-i   | 0.03 kl  |
|                             |          | MAP                              | 0.12*g-k  | -0.98 f-i   | 0.07 i-l   |
|                             |          | ECSTreal                         | 0.12 g-k  | 1.08 c-h  | 0.08 ijk   |
|                             |          | ECSTsyn                          | 0.12*g-k  | -0.31 d-i   | 0.08 ijk   |
|                             | Roxana   | CPST                             | 0.14*e-k  | 0.73 c-i  | 0.12*g-k   |
|                             |          | MAP                              | 0.16*e-k  | 1.91 b-g  | 0.14*e-k   |
|                             |          | ECSTreal                         | 0.30*c-f  | 0.64 c-i  | 0.25*c-f   |
|                             |          | ECSTsyn                          | 0.23*d-i  | -0.36 d-i   | 0.19*d-j   |
| Rainwater                   | Crelton  | CPST                             | 0.07 ijk  | -0.52 e-i   | 0.06 jkl   |
|                             |          | MAP                              | 0.15*e-k  | 0.63 c-i  | 0.14*e-k   |
|                             |          | ECSTreal                         | 0.14*e-k  | 0.98 c-h  | 0.18*e-k   |
|                             |          | ECSTsyn                          | 0.37*bcd  | -0.80 e-i   | 0.36*abc   |
|                             | Dapue    | CPST                             | 0.24*d-i  | 0.98 c-h  | 0.20*d-i   |
|                             |          | MAP                              | 0.11 g-k  | -1.04 f-i   | 0.12*g-k   |
|                             |          | ECSTreal                         | 0.26*d-g  | 2.08 c-f  | 0.25*c-g   |
|                             |          | ECSTsyn                          | 0.20*d-k  | -0.60 e-i   | 0.23*d-h   |
|                             | Calloway | CPST                             | 0.12*g-k  | 3.53*abc  | 0.11*h-k   |
|                             |          | MAP                              | 0.12 g-k  | 5.08*ab   | 0.13*f-k   |
|                             |          | ECSTreal                         | 0.04 k  | 1.44 c-h  | 0.13*e-k   |
|                             |          | ECSTsyn                          | 0.13*f-k  | 2.88*bcd  | 0.14*e-k   |
|                             | Roxana   | CPST                             | 0.09 h-k  | -1.16 f-i   | 0.04 kl  |
|                             |          | MAP                              | 0.48*ab   | -0.75 e-i   | 0.40*ab  |
|                             |          | ECSTreal                         | 0.59*a  | 1.03 c-h  | 0.47*a   |
|                             |          | ECSTsyn                          | 0.45*abc  | -0.19 d-i   | 0.43*a   |
| Struvite-removed wastewater | Crelton  | CPST                             | -0.79*n   | 0.01 d-i  | -0.87*no   |
|                             |          | MAP                              | -0.64*lmn   | -0.08 d-i   | -0.74*mn   |
|                             |          | ECSTreal                         | -0.68*mn  | -1.39 ghi   | -0.77*mn   |
|                             |          | ECSTsyn                          | -0.50*l   | -1.03 f-i   | -0.71*m  |
|                             | Dapue    | CPST                             | -0.72*mn  | 0.17 d-i  | -0.77*mn   |
|                             |          | MAP                              | -0.58*lm  | 0.75 c-i  | -0.72*m  |
|                             |          | ECSTreal                         | -0.66*lmn   | 0.32 c-i  | -0.73*m  |
|                             |          | ECSTsyn                          | -0.56*lm  | 0.59 c-i  | -0.73*m  |
|                             | Calloway | CPST                             | -1.22*o   | 6.43*a  | -1.19*q  |
|                             |          | MAP                              | -1.19*o   | 0.43 c-i  | -1.07*pq   |



|        |          |          |           |          |
|--------|----------|----------|-----------|----------|
| Roxana | ECSTreal | -1.15*o  | 0.46 c-i  | -1.10*pq |
|        | ECSTsyn  | -1.11*o  | -0.34 d-i | -0.98*po |
|        | CPST     | 0.16*e-k | -1.84 hi  | -0.06 l  |
|        | MAP      | 0.52*ab  | -0.62 e-i | 0.28*bcd |
|        | ECSTreal | 0.11 g-k | -0.95 f-i | 0.11*h-k |
|        | ECSTsyn  | 0.25*d-h | -1.17 f-i | 0.19*d-j |

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† Means in a column across water types, soils, and fertilizer-P sources with different letters are different at  $P < 0.05$

\* An asterisk (\*) indicates the mean change differs from zero at  $P < 0.05$

‡ Chemically precipitated struvite, CPST; monoammonium phosphate, MAP; real-wastewater-derived electrochemically precipitated struvite, ECSTreal; synthetic electrochemically precipitated struvite, ECSTsyn; total phosphorus, TP; nitrate + nitrite,  $\text{NO}_3 + \text{NO}_2$ ; soluble-reactive phosphorus, SRP

**Table 5.** Analysis of variance summary of the effects of water type (W), soil (S) and fertilizer (F) over time (T) and their interactions on the change ( $\Delta$ ) in runoff-water quality properties for Trial 2 of the rainfall-runoff simulation experiment.

| Source of Variation | $\Delta[\text{pH}]^\dagger$ | $\Delta[\text{EC}]^\dagger$ | $\Delta[\text{TP}]^\dagger$ | $\Delta[\text{Ca}]^\dagger$ | $\Delta[\text{Mg}]^\dagger$ | $\Delta[\text{Fe}]^\dagger$ | $\Delta[\text{NO}_3 + \text{NO}_2]^\dagger$ | $\Delta[\text{NH}_4]^\dagger$ | $\Delta[\text{SRP}]^\dagger$ |
|---------------------|-----------------------------|-----------------------------|-----------------------------|-----------------------------|-----------------------------|-----------------------------|---|-------------------------------|------------------------------|
| Water type          | < 0.01                      | 0.66                        | 0.02                        | < 0.01                      | 0.05                        | 0.42                        | 0.80  | 0.16                          | 0.01                         |
| Soil                | 0.10                        | 0.12                        | < 0.01                      | < 0.01                      | 0.79                        | < 0.01                      | 0.06  | < 0.01                        | < 0.01                       |
| W*S                 | < 0.01                      | < 0.01                      | 0.16                        | < 0.01                      | 0.02                        | 0.15                        | 0.01  | < 0.01                        | 0.91                         |
| Fertilizer          | 0.03                        | 0.48                        | 0.01                        | 0.73                        | 0.87                        | 0.15                        | 0.92  | 0.04                          | < 0.01                       |
| W*F                 | 0.03                        | 0.29                        | 0.81                        | 0.33                        | 0.59                        | 0.07                        | 0.43  | 0.22                          | 0.11                         |
| S*F                 | < 0.01                      | 0.35                        | 0.35                        | 0.13                        | 0.97                        | 0.04                        | 0.95  | 0.92                          | 0.08                         |
| W*S*F               | 0.12                        | 0.79                        | 0.38                        | 0.71                        | 0.73                        | 0.20                        | 0.40  | 0.62                          | 0.01                         |
| Time                | < 0.01                      | 0.46                        | < 0.01                      | < 0.01                      | 0.85                        | 0.12                        | 0.06  | < 0.01                        | < 0.01                       |
| W*T                 | < 0.01                      | 0.06                        | 0.01                        | < 0.01                      | 0.81                        | 0.98                        | 0.08  | 0.17                          | < 0.01                       |
| S*T                 | < 0.01                      | 0.19                        | 0.02                        | < 0.01                      | 0.39                        | < 0.01                      | 0.13  | < 0.01                        | < 0.01                       |
| W*S*T               | < 0.01                      | 0.01                        | 0.02                        | < 0.01                      | 0.17                        | 0.32                        | < 0.01                                      | 0.82                          | < 0.01                       |
| F*T                 | 0.37                        | 0.71                        | < 0.01                      | 0.33                        | 0.83                        | 0.40                        | 0.71  | 0.12                          | < 0.01                       |
| W*F*T               | 0.99                        | 0.96                        | 0.51                        | 0.66                        | 0.91                        | 0.26                        | 0.83  | 0.07                          | 0.15                         |
| S*F*T               | 0.28                        | 0.67                        | < 0.01                      | 0.60                        | 0.76                        | 0.07                        | 0.60  | 0.88                          | < 0.01                       |
| W*S*F*T             | 0.89                        | 0.03                        | 0.09                        | 0.07                        | 0.55                        | 0.85                        | 0.02  | 0.27                          | 0.16                         |

<sup>†</sup> Electrical conductivity, EC; total phosphorus, TP; calcium, Ca; magnesium, Mg; iron, Fe; nitrate + nitrite,  $\text{NO}_3 + \text{NO}_2$ ; ammonium,  $\text{NH}_4$ ; soluble-reactive phosphorus, SRP

**Table 6.** Summary of mean changes ( $\Delta$ ) in water-quality parameters among water type-soil combinations over the 6-month period from rainfall-runoff simulation Trial 1 to Trial 2.

| Water Type  | Soil Type | Trial | $\Delta \text{pH}^{\dagger, \ddagger}$ | $\Delta[\text{TP}]^{\dagger, \ddagger}$<br>(mg L <sup>-1</sup> ) | $\Delta[\text{Ca}]^{\dagger, \ddagger}$<br>(mg L <sup>-1</sup> ) | $\Delta[\text{SRP}]^{\dagger, \ddagger}$<br>(mg L <sup>-1</sup> ) |
|-------------|-----------|-------|--|--|--|---|
| Groundwater | Creldon   | 1     | -0.01 b                                | 0.14*bcd   | -35.5*g  | 0.11*de   |
|             |           | 2     | -1.95*e                                | < 0.01 e   | 1.18 bc  | < 0.01 gh   |
|             | Dapue     | 1     | 0.03 ab                                | 0.17*bc  | -27.8*f  | 0.15*bcd  |
|             |           | 2     | -1.89*e                                | < 0.01 e   | 0.08 bcd   | -0.01 gh  |
|             | Calloway  | 1     | -0.01 b                                | 0.10*cd  | 0.64 bcd   | 0.07*ef   |
|             |           | 2     | -1.90*e                                | < 0.01 e   | 1.45*abc   | -0.03 h   |
|             | Roxana    | 1     | -0.02 b                                | 0.21*b   | -8.34*e  | 0.18*bc   |
|             |           | 2     | -1.96*e                                | 0.10*cd  | 0.33 bcd   | 0.07*ef   |
|             | Creldon   | 1     | 0.01 b                                 | 0.18*b   | 0.40 bcd   | 0.18*bc   |
|             |           | 2     | -2.58*g                                | 0.01 e   | 0.39 bcd   | 0.04*fg   |
| Rainwater   | Dapue     | 1     | -0.19*c                                | 0.20*b   | 1.21 bc  | 0.20*b  |
|             |           | 2     | -2.73*h                                | < 0.01 e   | 3.45*a   | 0.02 fgh  |
|             | Calloway  | 1     | -0.34*d                                | 0.10*cd  | 1.68*ab  | 0.13*cd   |
|             |           | 2     | -2.33*f                                | < 0.01 e   | -0.98 d  | 0.01 gh   |
|             | Roxana    | 1     | 0.15*a                                 | 0.40*a   | -0.58 cd   | 0.34*a  |
|             |           | 2     | -2.66*gh                               | 0.07*ed  | -0.16 bcd  | 0.02 fgh  |

<sup>†</sup> Means in a column across water types and soils over time with different letters are different at  $P < 0.05$

\* An asterisk (\*) indicates the mean value differs from zero at  $P < 0.05$

<sup>‡</sup> Total phosphorus, TP; calcium, Ca; soluble-reactive phosphorus, SRP

**Table 7.** Summary of mean changes ( $\Delta$ ) in water-quality parameters among soil-fertilizer-phosphorus-(P)-source treatment combinations over the 6-month period from rainfall-runoff simulation Trial 1 to Trial 2.

| Soil     | Fertilizer | Trial | $\Delta[\text{TP}]^{\dagger, \ddagger}$<br>(mg L <sup>-1</sup> ) | $\Delta[\text{SRP}]^{\dagger, \ddagger}$<br>(mg L <sup>-1</sup> ) |
|----------|------------|-------|--|---|
| Creldon  | CPST       | 1     | 0.12*efg   | 0.10*efg  |
|          |            | 2     | < 0.01kl   | 0.02 j-m  |
|          | MAP        | 1     | 0.11*e-k   | 0.10*e-h  |
|          |            | 2     | < 0.01kl   | 0.04 g-m  |
|          | ECSTreal   | 1     | 0.12*e-h   | 0.13*def  |
|          |            | 2     | 0.01jkl  | 0.01 j-m  |
|          | ECSTsyn    | 1     | 0.30*bc  | 0.25*bd   |
|          |            | 2     | 0.01h-l  | 0.02 j-m  |
| Dapue    | CPST       | 1     | 0.17*def   | 0.15*de   |
|          |            | 2     | < 0.01kl   | 0.01 j-m  |
|          | MAP        | 1     | 0.12*e-h   | 0.11*efg  |
|          |            | 2     | < 0.01kl   | 0.02 i-m  |
|          | ECSTreal   | 1     | 0.21*cde   | 0.19*cd   |
|          |            | 2     | < 0.01kl   | 0.02 j-m  |
|          | ECSTsyn    | 1     | 0.25*bcd   | 0.24*bc   |
|          |            | 2     | 0.01jkl  | < 0.01 klm  |
| Calloway | CPST       | 1     | 0.09*f-l   | 0.07*e-k  |
|          |            | 2     | < 0.01kl   | < 0.01 j-m  |
|          | MAP        | 1     | 0.12*efg   | 0.10*e-h  |
|          |            | 2     | < 0.01lk   | -0.01l m  |
|          | ECSTreal   | 1     | 0.08*f-l   | 0.11*efg  |
|          |            | 2     | 0.01i-l  | -0.01l m  |
|          | ECSTsyn    | 1     | 0.13*efg   | 0.11*efg  |
|          |            | 2     | < 0.01 l   | -0.02 m   |
| Roxana   | CPST       | 1     | 0.11*e-j   | 0.08*e-j  |
|          |            | 2     | 0.16*def   | 0.07*e-l  |
|          | MAP        | 1     | 0.32*b   | 0.27*bc   |
|          |            | 2     | 0.03g-l  | 0.01 j-m  |
|          | ECSTreal   | 1     | 0.44*a   | 0.36*a  |
|          |            | 2     | 0.04g-l  | 0.02 h-m  |
|          | ECSTsyn    | 1     | 0.34*ab  | 0.31*ab   |
|          |            | 2     | 0.10*f-l   | 0.06*f-l  |

<sup>†</sup> Means in a column across soils and fertilizer-P sources over time with different letters are different at  $P < 0.05$

\* An asterisk (\*) indicates the mean value differs from zero at  $P < 0.05$

<sup>‡</sup> Chemically precipitated struvite, CPST; monoammonium phosphate, MAP; real-wastewater-derived electrochemically precipitated struvite, ECSTreal; synthetic electrochemically precipitated struvite, ECSTsyn; total phosphorus, TP; soluble-reactive phosphorus, SRP

**Table 8.** Summary of mean changes ( $\Delta$ ) in water-quality parameters among water type-soil-fertilizer-phosphorus-(P)-source treatment combinations over the 6-month period from rainfall-runoff simulation Trial 1 to Trial 2.

| Water Type  | Soil Type | Fertilizer-P source | Trial | $\Delta[\text{EC}]^{\dagger, \ddagger}$<br>( $\text{dS m}^{-1}$ ) | $\Delta[\text{NO}_3 + \text{NO}_2]^{\dagger, \ddagger}$<br>( $\text{mg L}^{-1}$ ) |
|-------------|-----------|---------------------|-------|---|---|
| Groundwater | Crelton   | CPST                | 1     | 6.66 d-k  | 0.91 d-l  |
|             |           |                     | 2     | 12.3 b-j  | 1.41 d-k  |
|             |           | MAP                 | 1     | -5.00 g-k   | -0.52 g-l   |
|             |           |                     | 2     | 18.7 a-g  | 1.73 c-j  |
|             |           | ECSTreal            | 1     | 7.66 d-k  | -0.60 g-l   |
|             |           |                     | 2     | 18.7 a-g  | 1.57 c-j  |
|             |           | ECSTsyn             | 1     | 4.00 e-k  | 2.48 a-i  |
|             |           |                     | 2     | 10.7 c-k  | 2.12 a-j  |
|             | Dapue     | CPST                | 1     | -13.0 ijk   | 0.83 d-l  |
|             |           |                     | 2     | -5.67 g-k   | 0.45 e-l  |
|             |           | MAP                 | 1     | -7.67 g-k   | 0.51 d-l  |
|             |           |                     | 2     | -15.7 jk  | -2.07 kl  |
|             |           | ECSTreal            | 1     | -10.0 h-k   | -2.52 l   |
|             |           |                     | 2     | 0.33 e-k  | 0.38 e-l  |
|             |           | ECSTsyn             | 1     | -5.67 g-k   | -0.59 g-l   |
|             |           |                     | 2     | 10.7 c-k  | 1.73 c-j  |
|             | Calloway  | CPST                | 1     | 3.00 e-k  | -0.08 e-l   |
|             |           |                     | 2     | 18.7 a-g  | 0.64 d-l  |
|             |           | MAP                 | 1     | 8.67 d-k  | -0.98 i-l   |
|             |           |                     | 2     | 38.7*abc  | 5.71*a  |
|             |           | ECSTreal            | 1     | 11.0 c-k  | 1.08 d-l  |
|             |           |                     | 2     | 13.7 a-i  | 2.83*a-h  |
|             |           | ECSTsyn             | 1     | 12.0 c-j  | -0.31 g-l   |
|             |           |                     | 2     | 24.7*a-f  | 3.35*a-f  |
|             | Roxana    | CPST                | 1     | 10.0 d-k  | 0.73 d-l  |
|             |           |                     | 2     | 0.83 e-k  | 0.70 d-l  |
|             |           | MAP                 | 1     | 9.00 d-k  | 1.91 b-l  |
|             |           |                     | 2     | 4.17 e-k  | 0.38 e-l  |
|             |           | ECSTreal            | 1     | 6.33 d-k  | 0.64 d-l  |
|             |           |                     | 2     | -4.83 g-k   | -0.12 f-l   |
|             |           | ECSTsyn             | 1     | 6.66 d-k  | -0.36 g-l   |
|             |           |                     | 2     | 3.17 e-k  | 1.37 d-k  |
| Rainwater   | Crelton   | CPST                | 1     | 4.60 e-k  | -0.52 g-l   |
|             |           |                     | 2     | 12.4 b-j  | 1.34 d-k  |
|             |           | MAP                 | 1     | 16.2 a-h  | 0.63 d-l  |
|             |           |                     | 2     | 7.23 d-k  | 0.40 e-l  |
|             |           | ECSTreal            | 1     | 13.6 a-i  | 0.98 d-l  |
|             |           |                     | 2     | 0.93 e-k  | 0.05 e-l  |
|             |           | ECSTsyn             | 1     | -6.07 g-k   | -0.80 h-l   |
|             |           |                     | 2     | 3.60 e-k  | 0.47 d-l  |
|             | Dapue     | CPST                | 1     | 16.0 a-h  | 0.98 d-l  |

|          |          |   |           |           |
|----------|----------|---|-----------|-----------|
| Calloway | MAP      | 2 | -1.70 f-k | 0.09 e-l  |
|          |          | 1 | -1.40 f-k | -1.04 i-l |
|          | ECSTreal | 2 | 40.6*ab   | 5.43*ab   |
|          |          | 1 | 34.3*a-d  | 2.08 a-j  |
|          | ECSTsyn  | 2 | 28.6*a-e  | 4.10*a-d  |
|          |          | 1 | -0.27 f-k | -0.60 g-l |
|          | CPST     | 2 | 28.4*a-e  | 4.09*a-d  |
|          |          | 1 | 19.7*a-g  | 3.52*a-e  |
|          | MAP      | 2 | -5.03 g-k | 0.12 e-l  |
|          |          | 1 | 41.7*a    | 5.08*abc  |
|          | ECSTreal | 2 | -2.53 f-k | -0.24 f-l |
|          |          | 1 | -4.44 g-k | 1.44 d-k  |
| Roxana   | ECSTsyn  | 2 | 0.03 f-k  | 0.01 e-l  |
|          |          | 1 | 6.96 d-k  | 2.88*a-g  |
|          | CPST     | 2 | -16.4 k   | -1.21 jkl |
|          |          | 1 | -13.8 ijk | -1.16 jkl |
|          | MAP      | 2 | 6.70 d-k  | 0.94 d-l  |
|          |          | 1 | -3.47 f-k | -0.75 g-l |
|          | ECSTreal | 2 | -2.47 f-k | -0.37 g-l |
|          |          | 1 | 15.8 a-h  | 1.03 d-l  |
|          | ECSTsyn  | 2 | -1.97 f-k | -0.29 g-l |
|          |          | 1 | 1.66 e-k  | -0.19 f-l |
|          |          | 2 | -3.07 f-k | -0.56 g-l |
|          |          |   |           |           |

† Means in a column across soils, water types, and fertilizer-P sources over time with different letters are different at  $P < 0.05$

\* An asterisk (\*) indicates the mean value differs from zero at  $P < 0.05$

‡ Chemically precipitated struvite, CPST; monoammonium phosphate, MAP; real-wastewater-derived electrochemically precipitated struvite, ECSTreal; synthetic electrochemically precipitated struvite, ECSTsyn; electrical conductivity, EC; nitrate + nitrite,  $\text{NO}_3 + \text{NO}_2$

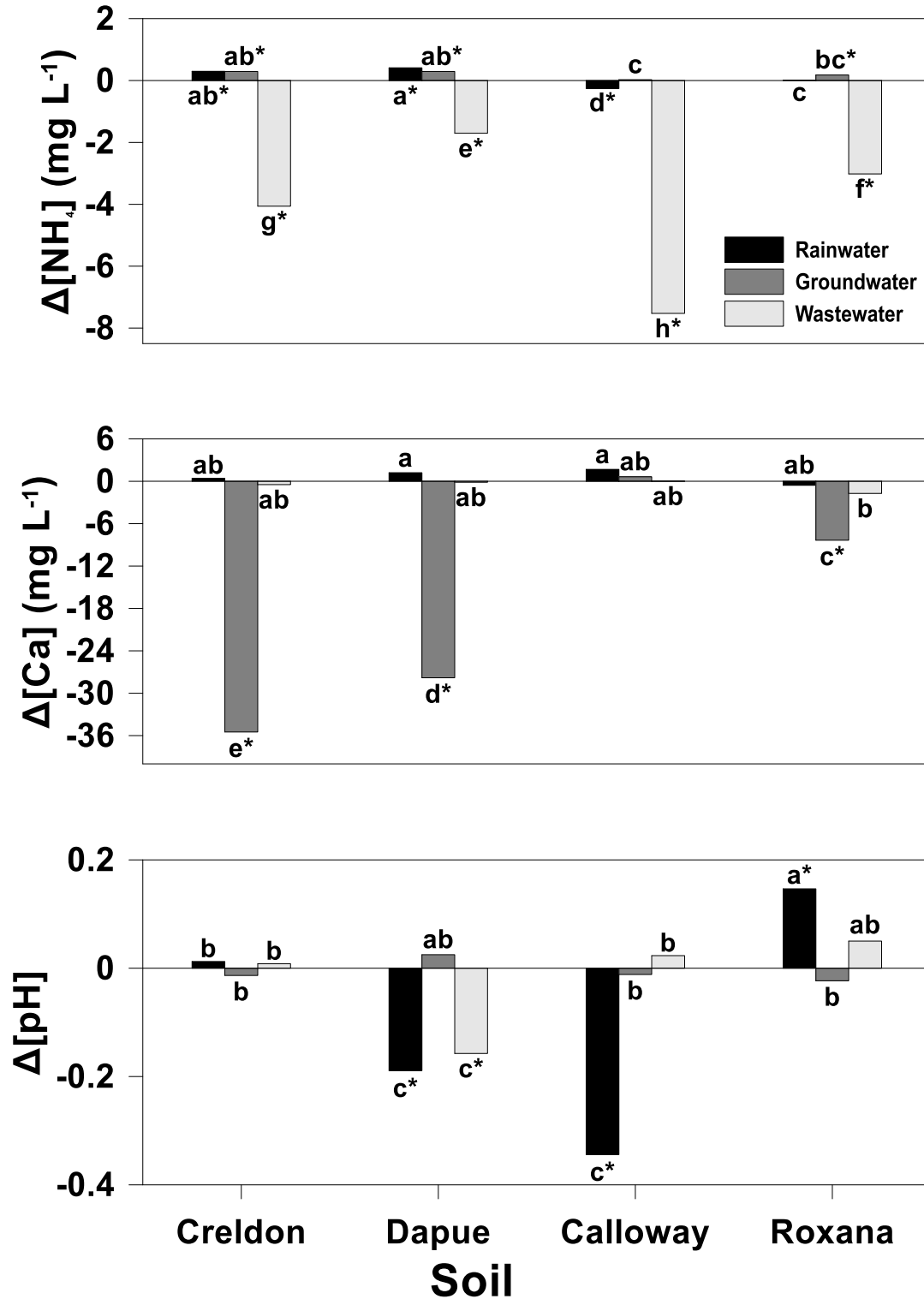


**Figure 1.** Image of the rainfall-runoff simulator setup. The tapered plastic trays were 62-cm long, 12-cm across at the top, and 7-cm across at the base. The slope of the trays was set to 22.1%.

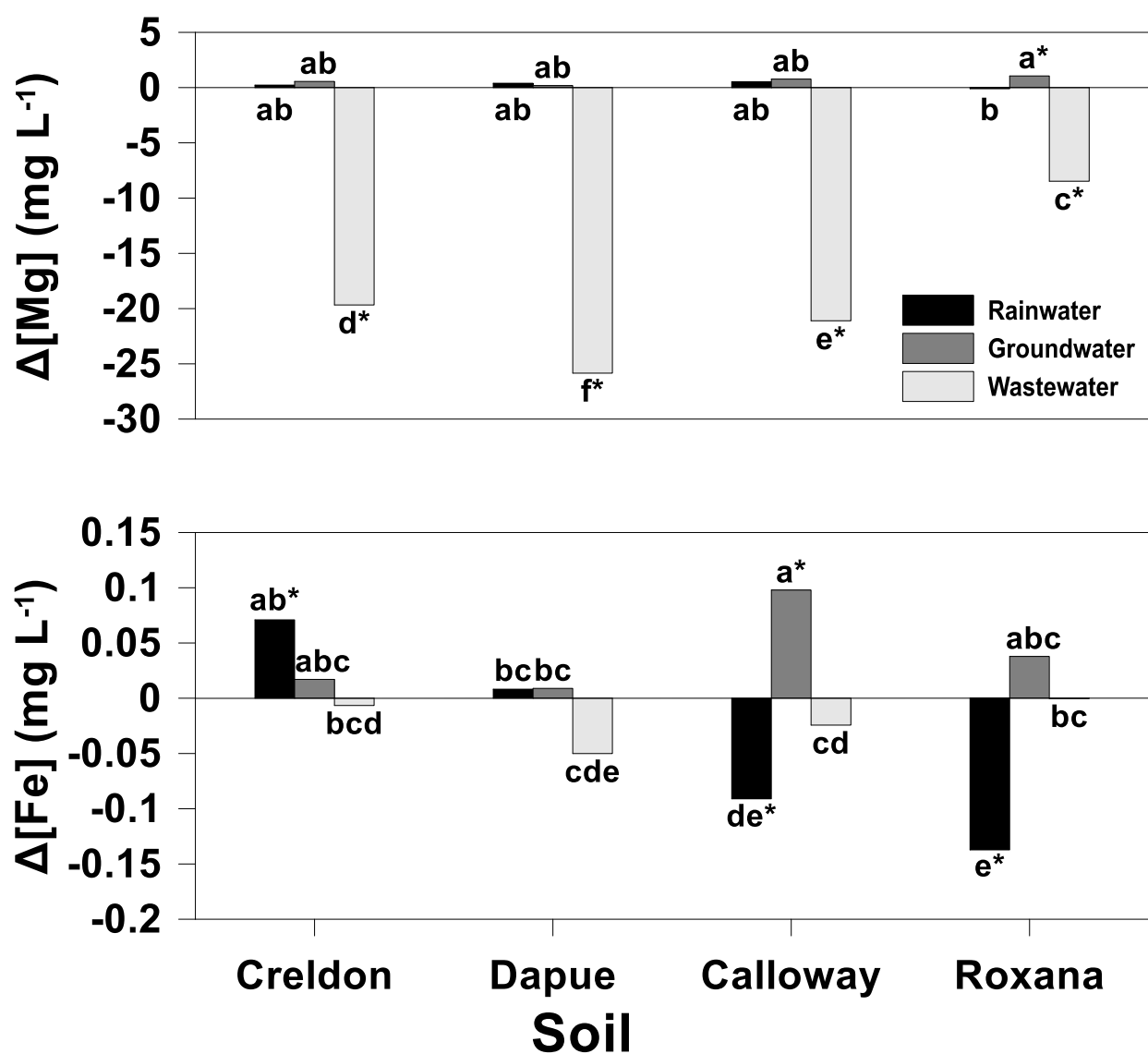


**Figure 2.** Close-up image of the drip emitters and tubing in the rainfall simulator. The four down-slope drip emitters are 5 cm apart, while the other three up-slope drip emitters are 10 cm apart. Tea bags were placed directly underneath the four closer-oriented, down-slope drip emitters.

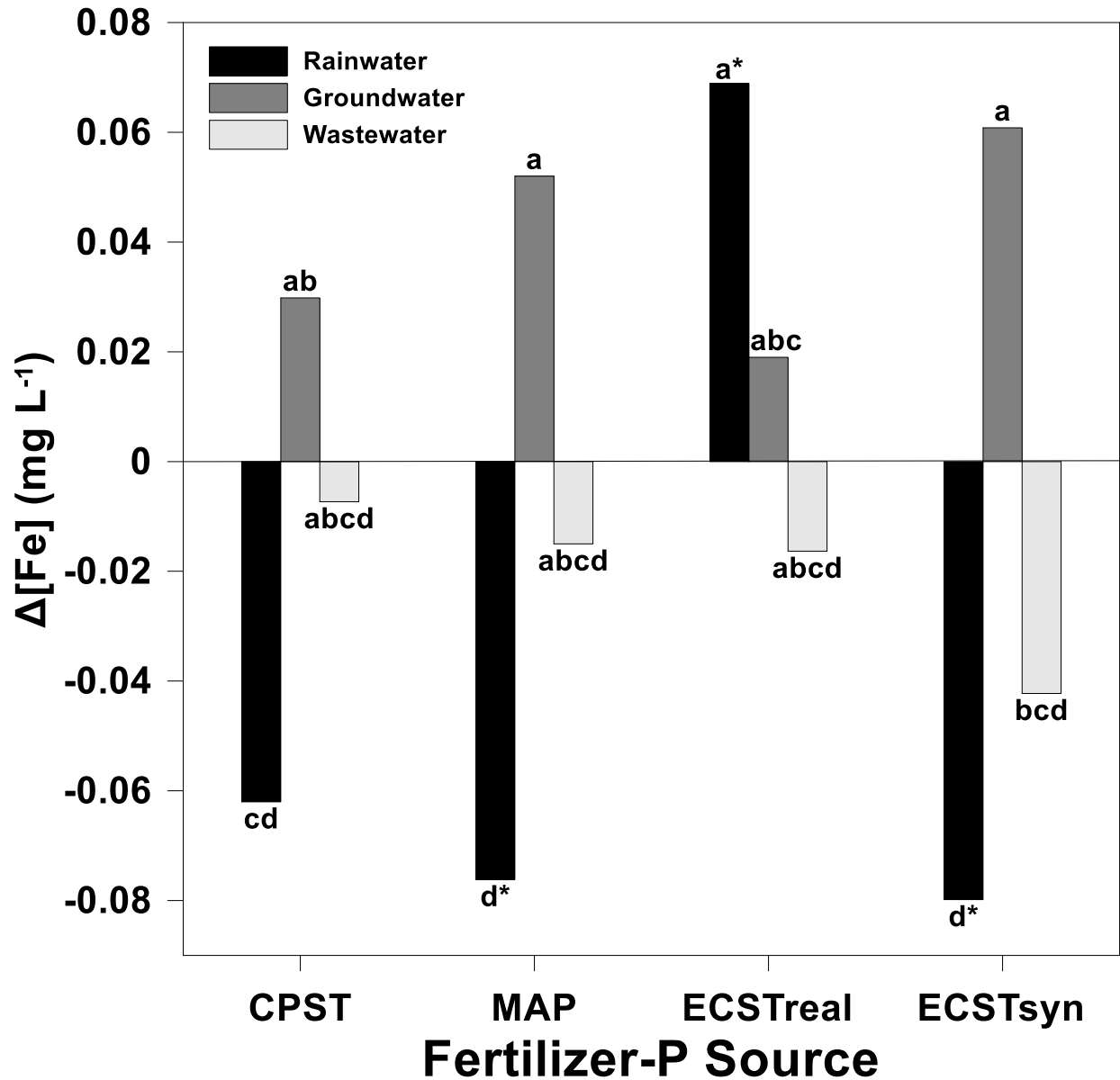




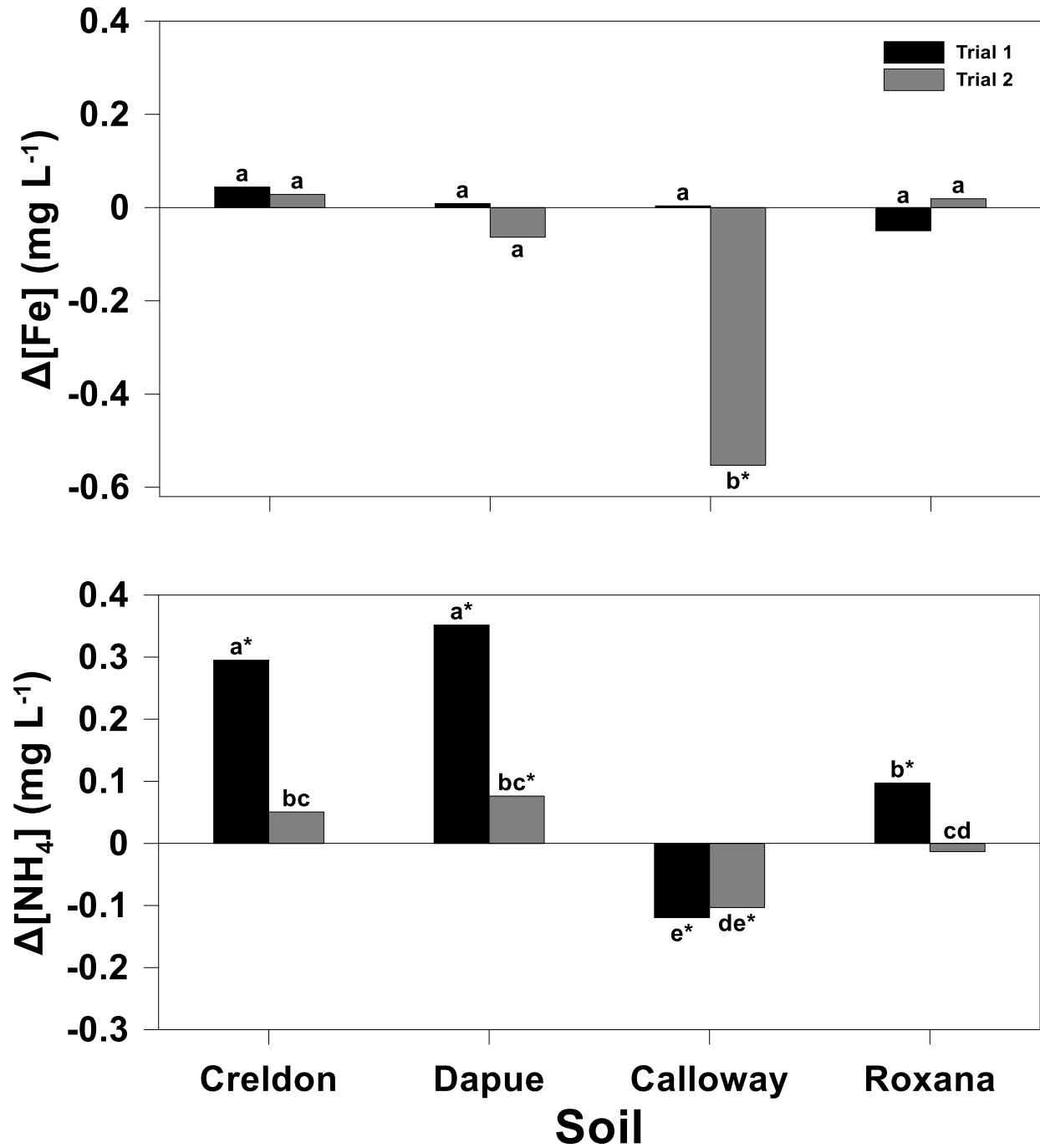
**Figure 3.** Summary of changes ( $\Delta$ ) in runoff-water ammonium ( $\text{NH}_4$ ), calcium ( $\text{Ca}$ ), and pH concentrations among water type (i.e., groundwater, rainwater, and struvite-removed wastewater)-soil series (i.e., Crelton, Dapue, Calloway, and Roxana) combinations for Trial 1. Bars with different lower-case letters across all treatment combinations are different at  $P < 0.05$ . Asterisks (\*) indicate the change is significantly different from zero at  $P < 0.05$ .



**Figure 4.** Summary of changes ( $\Delta$ ) in runoff-water magnesium (Mg) and iron (Fe) concentrations among water type (i.e., groundwater, rainwater, and struvite-removed wastewater)-soil series (i.e., Crelton, Dapue, Calloway, and Roxana) combinations for Trial 1. Bars with different lower-case letters across all treatment combinations are different at  $P < 0.05$ . Asterisks (\*) indicate the change is significantly different from zero at  $P < 0.05$ .



**Figure 5.** Summary of changes ( $\Delta$ ) in runoff-water iron (Fe) concentrations among water type (i.e., groundwater, rainwater, and struvite-removed wastewater)-fertilizer-phosphorus (P) source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), and synthetic electrochemically precipitated struvite (ECSTsyn)] combinations for Trial 1. Bars with different lower-case letters across all treatment combinations are different at  $P < 0.05$ . Asterisks (\*) indicate the change is significantly different from zero at  $P < 0.05$ .



**Figure 6.** Summary of changes ( $\Delta$ ) in runoff-water iron (Fe) and ammonium ( $\text{NH}_4$ ) concentrations among soil series (i.e., Credon, Dapue, Calloway, and Roxana) over the 6-month period from rainfall-runoff simulation Trial 1 to Trial 2. Bars with different lower-case letters across all treatment combinations are different at  $P < 0.05$ . Asterisks (\*) indicate the change is significantly different from zero at  $P < 0.05$ .

### **Chapter 3**

#### **Corn and Soybean Growth in Various Soils as Affected by Fertilizer-phosphorus Source and Irrigation Water Type**

## Abstract

Phosphorus (P) fertilizers are presently, primarily produced using mined phosphate rock, a non-renewable resource. Struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) that has been synthetically produced from a stock solution of known P and nitrogen (N) concentrations has been shown to be an effective, alternative fertilizer-P source for various crops, but little is known about struvite created from an actual municipal wastewater source. The objective of this study was to evaluate the effects of soil [i.e., Creldon silt loam (Oxyaquic Fragiudalf) and Calloway silt loam (Aquic Fraglossudalf) series], fertilizer-P source [i.e., synthetically produced electrochemically precipitated struvite (ECSTsyn), real-wastewater-derived ECST (ECSTreal), chemically precipitated struvite (CPST), monoammonium phosphate (MAP), and an unamended control (UC)], and irrigation water source (i.e., tap water and struvite-removed wastewater) on corn (*Zea mays* L.) and soybean [*Glycine max* (L.) Merr.] growth and N, P, and magnesium (Mg) concentration and uptake in a 60-day, greenhouse potted-plant study. Crop growth and N, P, and Mg concentrations and uptakes for the struvite treatments (i.e., CPST, ECSTsyn, and ECSTreal) were generally similar to MAP or at least 1.2 to 2.5 times greater than MAP. The ECSTsyn material commonly had up to five times greater N, P, and Mg concentration and uptake in corn and soybean than any other fertilizer-P source. Struvite-removed wastewater tended to result in N, P, and Mg concentrations, uptakes, and dry matter that were at least 1.3 times lower than tap water. The similar corn and soybean responses from the struvite fertilizers among the various soils and irrigation water types compared to MAP suggest that struvite has similar agronomic implications as at least one widely used, commercially available, multi-nutrient fertilizer-P source.

## Introduction

With the current rate of human population growth, the demand for food fertilizers will increase at a proportional rate. Crop yield demands are expected to increase annually by approximately 2.5% (Steen, 1998; Hemathilake & Gunathilake, 2022). Consequently, a larger input of phosphorus (P), a plant-essential macronutrient, will be needed in order to grow more crops to feed the growing human population. In 2020, the demand for P fertilizer increased by 7% from 2020 to 2021 (IFA, 2021). However, demand for all fertilizers is expected to increase at an annual rate of only 0.9% (IFA, 2021), which means the raw materials to make fertilizers will increase as well.

The majority of P fertilizers are derived from mined phosphate rock (PR). Phosphate rock reserves and demand for fertilizer-P fluctuate year to year. Peak P, when demand outpaces supply, is predicted to occur by 2040 (Cordell & White, 2013; Hemathilake & Gunathilake, 2022). Depending on demand, economic viability, and P concentrations within the current reserves, PR reserves are likely to be depleted in the next 30 to 150 years (Cordell & White, 2013; Hemathilake & Gunathilake, 2022).

As a result of the need to produce enough food to sustain an increasing human population, there is a concomitant substantial increase in the production of human and animal wastes, which further disrupts the natural P cycle and balance among major P pools. Agricultural runoff and animal waste make up the majority of P discharge to surface water bodies, but approximately 15% is also due to human waste (Rittmann et al., 2011). Ninety-eight percent of the N and P in the human diet is lost through waste (Smil, 2000), thus human waste has a large P and N concentration. Every year, 300 million megatons of human waste is produced globally, but

less than a third of that is reused. Human waste accounts for approximately 22% of the global P economy by weight (Mihelcic et al., 2011).

Struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) is a white, crystalline substance, containing a 1:1:1 equimolar ratio of magnesium ( $\text{Mg}^{2+}$ ), ammonium ( $\text{NH}_4^+$ ), and phosphate ( $\text{PO}_4^{3-}$ ), that is somewhat soluble in neutral and alkaline conditions, but more readily soluble in acidic conditions (Liu et al., 2012). The solubility of struvite in water is generally low, around 1 to 5%, but research shows that the low solubility of struvite does not decrease its effectiveness as a fertilizer-P source for plants (Talboys et al., 2016). Struvite has been characterized as a slow-release fertilizer due to its low solubility, although more recent research shows that struvite in powder form has a similar dissolution rate in soil as monoammonium phosphate (MAP; Degryse et al., 2017). However, struvite's slow-release properties may benefit crops, as the P will become available to crops over time, in a controlled-release manner (Bonvin et al., 2015). Although struvite has been shown to be an effective, potential fertilizer source, the nutrient content of struvite varies depending on what source material was used and how the struvite was actually created.

Struvite crystallization can occur in two ways. For one method, compounds such as magnesium chloride ( $\text{MgCl}_2$ ) or magnesium oxide ( $\text{MgO}$ ) allow for struvite crystallization once the solution becomes supersaturated with  $\text{Mg}^{2+}$ ,  $\text{NH}_4^+$ , and  $\text{PO}_4^{3-}$  (Siciliano et al., 2020). The process of adding chemicals to an aqueous solution to precipitate struvite out of solution is known as chemical precipitation. Historically, chemical precipitation of struvite was the main method used for struvite formation. Today, there is a commercially available, chemically precipitated struvite (CPST) fertilizer known as Crystal Green, which is produced by Ostara Nutrient Technologies, Inc. (Vancouver, British Columbia). According to Ostara (2021), Crystal



Green is a slow-release fertilizer in pellet form with a fertilizer grade of 5-28-0 and 10% Mg. Crystal Green also has low heavy metal and salt concentrations (Ostara, 2021).

For a second method of struvite crystallization, electrochemical precipitation of struvite is achieved by electrochemically releasing Mg via sacrificing a Mg anode plate (Kékedy-Nagy et al., 2021). The creation of electrochemically precipitated struvite (ECST) avoids the chemical dosing that chemical struvite precipitation requires and instead requires only an energy input for Mg dissolution. Kékedy-Nagy et al. (2020) used electrochemical precipitation to more efficiently recover P from synthetic wastewater compared to chemical precipitation. Overall, electrochemical precipitation of struvite has the potential to be more energy efficient and more effective than other P recovery methods (Kékedy-Nagy et al., 2020).

Plant response to struvite often depends on soil pH. In many different studies (Hilt et al., 2016; Ackerman et al., 2013; Talboys et al., 2016; Johnston & Richards, 2003), no difference was reported in crop response between plants fertilized with commercially available fertilizers and plants fertilized with struvite. Ackerman et al. (2013) grew canola (*Brassica napus L*) in a sandy-loam soil with pH 7.7 comparing commercially available fertilizers, such as MAP, to struvite derived from liquid swine manure. No crop response difference was recorded among fertilizer treatments due to struvite's low solubility under alkaline conditions (Ackerman et al., 2013). Talboys et al. (2016) had a similar result when growing spring wheat (*Triticum aestivum*) and buckwheat (*Fagopyrum esculentum*) fertilized with a combination of CPST (i.e., Crystal Green) and diammonium phosphate (DAP) under alkaline soil conditions. Johnston & Richards (2003) reported no difference in dry matter yield among ryegrass (*Lolium perenne*) treated with commercially available P fertilizer, synthetically produced struvite, or struvite recovered from municipal or animal waste, where the soils used had neutral pHs. Hilt et al. (2016) compared

corn (*Zea mays*) response to MAP and struvite recovered from dairy manure in both an acidic and an alkaline soil, where corn response in the alkaline soil did not differ between MAP and struvite. However, in the acidic soil, the corn treated with struvite had a greater P uptake and P tissue concentration compared to plants treated with MAP (Hilt et al., 2016), further illustrating struvite's solubility dependency on soil pH. Similarly, Robles-Aguilar et al. (2020) grew corn in an acidic soil to compare crop response between triple superphosphate (TSP) and struvite. Corn treated with struvite had a larger average biomass than corn treated with TSP (Robles-Aguilar et al., 2020). Omidire & Brye (2022) compared soybean (*Glycine max*) and wheat fertilized with CPST to soybean and wheat fertilized with TSP and recorded no difference in response between the two treatments. Similarly, Omidire et al. (2022a) grew rice (*Oryza sativa*) treated with both synthetic electrochemically precipitated struvite (ECSTsyn) and CPST compared to other commercially available fertilizer-P sources [i.e., TSP, MAP, DAP, and rock phosphate (RP)] and again recorded no difference in response among struvite and the commercially available fertilizers. Hertzberger et al. (2020) conducted a meta-analysis and review of struvite as a potential fertilizer and reported that struvite-fertilized crops generally resulted in larger biomass, tissue-P concentration, and P uptake than plants fertilized with ammonium phosphates or superphosphates, especially in soils with  $\text{pH} < 6$ . In many studies, crop response to struvite increased as soil pH decreased, and struvite was recorded to be just as effective as commercially available fertilizers in soils with a neutral or alkaline pH (Hertzberger et al., 2020).

The exponential population growth and the rapidly declining PR reserves will push the development and application of new and/or alternative fertilizer-P sources. Therefore, the objective of this study was to evaluate the effects of soil [i.e., Crelton (Oxyaquic Fragiudalf) and Calloway (Aquic Fraglossudalf)], irrigation water source (i.e., tap water and struvite-removed

wastewater), and fertilizer-P source [i.e., synthetically produced ECST (ECSTsyn), real-wastewater-derived ECST (ECSTreal), CPST, MAP, and an unamended control (UC)] on corn and soybean growth and tissue nutrient concentrations and uptakes in a 60-day greenhouse potted-plant experiment. It was hypothesized that the corn and soybean dry matter and tissue nutrient concentrations and uptakes in the struvite treatments (i.e., CPST, ECSTsyn, and ECSTreal) will be similar to or greater than that for MAP. It was also hypothesized that corn and soybean properties will be unaffected by irrigation water source. Furthermore, it was hypothesized that, for ECSTreal and ECSTsyn, greater crop growth and nutrient properties will occur in the lower-pH soil (i.e., Creldon).

## **Materials and Methods**

A corn and soybean potted-plant study was conducted in the greenhouse on the University of Arkansas campus in Fayetteville, AR. Similar procedures to Ylagan et al. (2020) were used, who recently, successfully evaluated corn and soybean response to various fertilizer-P-sources, including ECSTsyn and CPST, in a potted-plant study in the greenhouse.

### Soil Collection, Processing, and Analyses

Soil was collected from agriculturally relevant areas of southwestern Missouri and eastern and western Arkansas for use in the greenhouse potted-plant studies. Bulk soil was collected from a 0- to 15-cm depth at all locations. A low-soil-test-P Calloway silt loam (fine-silty, mixed, active, thermic Aquic Fraglossudalf; NCSS, 2021) was collected in early spring 2021 from within an approximate 3-m<sup>2</sup> area from the edge of a row-crop-cultivated field at the University of Arkansas, Division of Agriculture's Pine Tree Research Station near Colt, AR

(35°07'23" N; 90°55'46" W). A Creldon silt loam (fine, mixed, active, mesic Oxyaquic Fragiudalf; NCSS, 2006) was collected from managed pasturelands at the University of Missouri's Southwest Research Center near Mount Vernon, MO (37°04'45" N; 93°53'13" W) in June 2021. Both soils represent typical agricultural areas in the mid-southern US with a low soil-test P concentration in the upper 15 cm that would typically receive a fertilizer-P rate recommendation to maximize crop yield.

The two low-soil-test-P soils [i.e., < 18 mg Mehlich-3 (M3)-extractable P kg<sup>-1</sup>] had already been collected, air-dried, and sieved to < 6 mm before use in this study. Initial soil subsamples were oven-dried, crushed, and sieved to < 2 mm and extracted with M3 extraction solution and water in a 1:10 soil mass:extraction volume ratio and analyzed for extractable soil nutrients (i.e., P, K, Ca, Mg, Na, S, Fe, Mn, Zn, B, and Cu) by extraction in a 10:1 extractant-volume/soil mass ratio. Digested and extracted solutions were analyzed by inductively coupled, argon-plasma, optical emissions spectrometry (ICAP-OES; Soltanpour, 1996). Soil pH and electrical conductivity (EC) were determined with an electrode on a 1:2 (mass/volume) soil-to-water paste. Nitrate and NH<sub>4</sub> concentrations were measured using a SKALAR autoanalyzer (SAN+ System Segmented Flow Analyzer, Skalar Analytical B.V., Breda, The Netherlands; SERA-IEG-6, 2014; Krom, 1980). The soil organic matter (SOM) concentration was determined gravimetrically through weight-loss-on-ignition after 2 hours of combustion at 360°C in a muffle furnace (Zhang & Wang, 2014). Total soil N and C were determined by high-temperature combustion with an Elementar VarioMAX CN analyzer (Elementar Americas Inc.). Soil particle-size analyses were conducted using a modified 12-hour hydrometer method (Gee & Or, 2002). Table 1 summarizes all measured soil physical and chemical properties.

## Fertilizer Treatments

Corn and soybean were treated with MAP, CPST, ECSTsyn, ECSTreal, and a UC as the fertilizer-P sources. Similar procedures were used to chemically analyze the ECSTreal fertilizer material as those used in Anderson et al. (2020a) to chemically analyze ECSTsyn, CPST, and MAP. Due to ECSTsyn and ECSTreal being electrochemically precipitated, both ECST fertilizers were in powder form, while CPST and MAP were in pellet form. For the purposes of chemical comparison, CPST and MAP were finely ground to match the powder consistency of ECSTsyn and ECSTreal. A 1:2 fertilizer mass:water volume ratio was used to potentiometrically determine pH and EC. Using a 1:10 fertilizer mass:water volume ratio that was agitated for 1 hour, filtered through a 0.45- $\mu$ m filter, and then analyzed by ICAP-OES for water-soluble elemental concentrations (i.e., P, K, Ca, Mg, Na, S, Fe, Mn, Zn, B, and Cu). A 1:10 fertilizer mass:extractant volume ratio (Tucker, 1992) was used for M3 extraction, after which elemental concentrations (i.e., P, K, Ca, Mg, Na, S, Fe, Mn, Zn, B, and Cu) were measured by ICAP-OES (Zhang et al., 2014). Total-recoverable (TR) elemental concentrations (i.e., P, K, Ca, Mg, Na, S, Fe, Mn, Zn, B, and Cu) were determined after strong-acid digestion (USEPA, 1996) and analysis by ICAP-OES. High-temperature combustion (Elementar VarioMax CN Analyzer) was used to measure TN and TC concentrations.

Monoammonium phosphate (fertilizer grade: 11-52-0) is a pelletized, commonly used, commercially available P and N fertilizer that contains actual nutrient concentrations of 20.9% P, 11% N, and 1.5% Mg (Anderson et al., 2021). The CPST material is pelletized and has nutrient concentrations of 11.7% P, 6% N, and 8.3% Mg (Anderson et al., 2020b). A synthetic wastewater source, produced to have a similar average P and N concentration as typical municipal wastewater (Kékedy-Nagy et al., 2020), was used to precipitate ECSTsyn (18.5% P,

3.3% N, and 13.3% Mg; Anderson et al., 2020a). An actual municipal wastewater source collected from the West Side Wastewater Treatment Facility in Fayetteville, AR was used to electrochemically precipitate the ECSTreal material (15.4% P, 3.3% N, and 13.6% Mg). Both ECSTsyn and ECSTreal were precipitated electrochemically by sacrificing a Mg anode plate (Kékedy-Nagy et al., 2020).

### Greenhouse Experiment Preparation and Management

A consistent mass of air-dried, sieved soil (1500 g) for the two soils was added to large plastic bags, to which differing masses of ECSTreal, ECSTsyn, CPST, and MAP were added to result in a consistent fertilizer-P application rate. The fertilizer application rates for corn and soybean were based on the resulting mean M3-soil-test-P concentration of the initial soil for corn, with an appropriate yield goal of 12.5 MT ha<sup>-1</sup>, and soybean grown in a silt-loam soil in Arkansas (Slaton et al., 2013; Espinoza & Ross, 2008). The resulting fertilizer rates were 100.9 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup>, 235.4 kg N ha<sup>-1</sup>, and 168.1 kg K<sub>2</sub>O ha<sup>-1</sup> for corn grown in the Calloway soil; 100.9 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup>, 235.4 kg N ha<sup>-1</sup>, and 100.9 kg K<sub>2</sub>O ha<sup>-1</sup> for corn grown in the Creldon soil; 89.7 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup>, 0 kg N ha<sup>-1</sup>, and 179.3 kg K<sub>2</sub>O ha<sup>-1</sup> for soybeans grown in the Calloway soil; and 67.3 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup>, 0 kg N ha<sup>-1</sup>, and 67.3 kg K<sub>2</sub>O ha<sup>-1</sup> for soybeans grown in the Creldon soil. Six replications of each soil-fertilizer treatment combination for each crop were prepared, including six replications per crop of a UC treatment that received no fertilizer-P addition, but received N and K as recommended. Based on the differential N concentrations of the various fertilizer materials, uncoated urea (46% N) was used to balance the N additions at the time the soil-fertilizer mixtures were prepared. Similarly, muriate of potash (60% K<sub>2</sub>O) was added to all soil-fertilizer combinations. The soil and fertilizer materials were manually mixed in the bags to

simulate incorporation by tillage. The soil-fertilizer mixtures were added to small plastic pots (14.6 cm in diameter and 17.8 cm tall) with glass fiber filter paper placed at the bottom of each pot to contain the soil-fertilizer material in the pots. Pots were arranged separately by crop in a randomized complete block design (RCB) with three blocks (i.e., replications), where each crop's pots were on separate, but adjacent greenhouse benches.

Three seeds of each crop were initially seeded in a triangular arrangement in each pot on 21 May 2022 to a depth of 1 cm. After germination, emergence, and approximately one week of growth, the pots were thinned to only one plant per pot. Both corn and soybean plants were grown for 60 days from the date of planting, at which time the greenhouse study was terminated, the pots were disassembled, and soil and plant samples were collected.

Three of the six replicates of soil in the pots were watered exclusively with regular tap water from the greenhouse facility. The other three replicates were watered once a week with struvite-removed wastewater and tap water approximately three times per week due to the limited volume of struvite-removed wastewater available to use. The struvite-removed wastewater was prepared during Summer 2021 by electrochemically precipitating and removing struvite from a local wastewater source from a municipal wastewater treatment plant in Fayetteville, AR. The struvite-removed wastewater and tap water were chemically characterized approximately mid-way through the 60-day plant growth period. Pots were watered approximately four times per week, where each time the soil was watered to the estimated field moisture capacity, 35.2% (v/v) for the Calloway soil and 24.7% (v/v) for the Creldon soil. Similar to recent studies (Brye et al., 2006; Durre et al., 2019; Ylagan et al., 2020), multiple regression relationships (Saxton et al., 1986), as part of the Soil Water Characteristics subroutine of the Soil-Plant-Atmosphere-Water (SPA-W) model (version 6.02.75; USDA, 2017), were used

to estimate field moisture capacity for each soil using measured sand, clay, and SOM concentrations.

#### Water Sample Processing and Analyses

Two water sources were used for the rainfall-runoff simulations. Tap water was obtained from a spigot in the greenhouse in which the potted-plant study was conducted. The second water source was struvite-removed wastewater produced in July 2021 as a result of the precipitation of the ECSTreal material described above. The purpose of using the wastewater was to evaluate its effectiveness as a potential irrigation-water source after struvite removal. The struvite-removed wastewater was refrigerated at 4°C until used. Tap water and struvite-removed wastewater samples were analyzed for total soluble elemental concentrations (i.e., P, K, Ca, Mg, S, Na, Fe, Mn, Zn, and Cu) using ICP-OES (Zhang et al., 2014).

#### Plant Sampling, Processing, and Analyses

After the 60-day growth period, plant heights were measured. Plants were then cut at the soil surface to separate the above- and belowground plant biomass. Roots were manually removed from the soil by washing and sieving. Above- and belowground plant samples were dried separately at 70°C for approximately one week to determine dry matter. Sub-samples of above- and belowground dry matter were ground and sieved through a 2-mm mesh screen for P, N, and Mg concentration analyses. Plant tissue samples were chemically analyzed to determine total tissue N by high-temperature combustion (Elementar VarioMAX CN analyzer, Elementar Americas Inc.) and total tissue P and Mg concentrations by acid digestion (USEPA, 1996) followed by analysis with ICP-OES (Soltanpour et al., 1996). Nutrient uptakes were calculated



using measured nutrient concentrations and dry matter for each replicate. Total plant dry matter and total nutrient uptake were calculated by adding the above- and belowground dry matters and nutrient uptakes.

### Statistical Analyses

Separately by crop and based on a RCB design, the effects of soil, fertilizer-P source, water type, and their interaction on plant height, above- and belowground dry matter, tissue nutrient concentrations and uptakes, and total plant dry matters and nutrient uptakes were analyzed by analysis of variance (ANOVA) using SAS (version 9.4, SAS Institute, Inc., Cary, NC). A one-factor ANOVA was used to determine differences in initial soil properties and mid-experiment water properties between the two soils and the two water types used, respectively. A gamma distribution was used for all plant, soil, and water property analyses. Significance was judged at  $P < 0.05$ . When appropriate, means were separated by the least significant difference at the 0.05 level.

## **Results and Discussion**

### **Initial soil properties**

All initial soil properties evaluated differed ( $P < 0.01$ ) between soils (Table 1). Percent silt and clay and soil pH and EC were larger in the Calloway than in the Creldon soil, while percent sand, TN, TC, C:N ratio, SOM,  $\text{NO}_3\text{-N}$ , and  $\text{NH}_4\text{-N}$  were larger in the Creldon than in the Calloway soil (Table 1).

All WS nutrients differed between soils ( $P < 0.01$ ; Table 1). Water-soluble soil P, K, Mg, Fe, and Mn concentrations were larger in the Creldon than the Calloway soil (Table 1). Water-

soluble soil Ca, S, and Na concentrations were larger in the Calloway than in the Creldon soil. Soil Cu and Zn did not differ between soils.

For M3-extractable concentrations, all soil nutrient concentrations differed ( $P < 0.01$ ) between soils (Table 1). Mehlich-3 extractable soil P, K, Mg, and Zn concentrations were larger in the Creldon than in the Calloway soil, while all other M3 nutrient concentrations were larger in the Calloway than in the Creldon soil, except S, which did not differ between soils (Table 1).

All total-recoverable soil nutrients differed ( $P < 0.01$ ) between soils (Table 1). Total-recoverable soil K, Mg, and S concentrations were largest in the Creldon than in the Calloway soil, while TR soil P, Ca, Na, and Cu were largest in the Calloway soil (Table 1). Soil Fe, Mn, and Zn did not differ between soils.

Soil nutrient concentrations varied depending on which analysis technique was used. Water-soluble concentrations tended to be lowest, while TR concentrations were largest, where, in the current study, TR concentrations were 100 to 200 times larger than WS concentrations. Mehlich-3 concentrations reflect plant-available nutrients, which are the most relevant for studying the effects of fertilizer-P sources on corn and soybean growth.

### **Initial water properties**

All initial water properties evaluated, except for Ca, differed ( $P < 0.01$ ) between tap water and wastewater (Table 2). All water properties evaluated were larger in the struvite-removed wastewater than in the tap water (Table 2). Macro- and micro-nutrients were both larger in the struvite-removed wastewater than tap water, which may result in various unexpected plant responses for interactions between water types, soils, and fertilizer-P sources. However, results

clearly indicate that struvite-removed wastewater, as a irrigation water source for plants, has greater concentrations of numerous essential plant nutrients that could benefit plant growth.

## **Corn response**

### Belowground

Every measured belowground corn parameter (i.e., dry matter and N, P, and Mg concentration and uptake) was affected ( $P < 0.05$ ) by one or more treatments (i.e., irrigation water source, soil, and/or fertilizer-P source; Table 3). Belowground dry matter and N concentration differed ( $P < 0.05$ ) among fertilizer-P sources but were unaffected by soil and water type (Table 3). Averaged across soils and water types, belowground corn dry matter was numerically largest for MAP, which did not differ from the other three P-fertilized treatments, and was numerically smallest from the UC, which did not differ from ECSTsyn (Table 4). Belowground dry matter from ECSTreal, MAP, and CPST was at least 1.2 times greater than from the UC (Table 4). Without receiving any fertilizer-P addition, it stands to reason that the UC treatment would have the lowest belowground dry matter. Belowground dry matter did not differ among the three struvite treatments (Table 4). In contrast to the results of the current study, Ylagan et al. (2020) reported that ECSTsyn had the numerically largest belowground corn dry matter. However, similar to Ylagan et al. (2020), the UC had the numerically smallest belowground corn dry matter due to the UC being unfertilized.

Similar to dry matter, averaged across soils and water types, belowground N concentration was numerically largest for CPST, which did not differ from ECSTreal and the UC, and was numerically smallest from MAP, which did not differ from ECSTsyn and ECSTreal (Table 4). Belowground N concentration from CPST was 1.3 times greater than from MAP

(Table 4). Similar to dry matter, belowground N concentration did not differ among the three struvite treatments (Table 4). Similar to results of the current study, Ylagan et al. (2020) reported that belowground corn N concentration differed among fertilizer-P sources, where N concentration was largest for CPST. However, Omidire et al. (2022b) reported no effect of fertilizer-P source on belowground corn N concentration.

In contrast to dry matter and N concentration, belowground P and Mg concentrations differed ( $P < 0.05$ ) among fertilizer-P sources between soils (Table 3). Averaged across water types, belowground P concentration was 1.5 times larger from CPST in the Creldon soil than from all other soil-fertilizer-P-source combinations and was smallest from the UC in the Creldon soil among all soil-fertilizer-P-source combinations (Figure 1). Belowground P concentration was greater in the Creldon than in the Calloway soil only for CPST, but was greater in the Calloway than in the Creldon soil for MAP, ECSTreal, and the UC, while there was no difference between soils for ECSTsyn (Figure 1). Belowground P concentrations from all three struvite materials differed from one another in both soils, where  $CPST > ECSTsyn > ECSTreal$  (Figure 1).

Similar to P, averaged across water types, belowground Mg concentration was 1.3 times larger from CPST in the Creldon soil than from all other soil-fertilizer-P-source combinations and was numerically smallest from MAP in the Creldon soil, which did not differ from MAP in the Calloway soil (Figure 1). Similar to P, belowground Mg concentration was greater in the Creldon than in the Calloway soil only for CPST, but was greater in the Calloway than in the Creldon soil for only ECSTreal, while there was no difference between soils for MAP, ECSTsyn, or the UC (Figure 1). In the Creldon soil, belowground Mg concentration was larger from CPST than from the ECSTsyn and ECSTreal, which did not differ, while, in the Calloway soil,

belowground Mg concentration in all three struvite materials were similar to one another (Figure 1).

In contrast to dry matter and P and Mg, belowground P concentrations also differed ( $P < 0.05$ ) among fertilizer-P sources between water types (Table 3). Averaged across soils, similar to Ylagan et al. (2020), belowground P concentration was 1.3 times larger from CPST with tap water than from all other water-fertilizer-P-source combinations and was numerically smallest from the UC with tap water, which did not differ from the UC with wastewater (Figure 1). Belowground P concentration was greater with tap water than wastewater for CPST and ECSTreal, while there was no difference between water types for MAP, ECSTsyn, or the UC (Figure 1). With tap water and wastewater, similar to Ylagan et al. (2020), belowground P concentration was larger from CPST than from ECSTsyn, which was larger than from ECSTreal (Figure 1). In contrast to the current results, Omidire et al. (2022b) did not report a significant fertilizer-P source effect on belowground corn P concentration.

In contrast to N concentration, belowground N uptake differed ( $P < 0.05$ ) among fertilizer-P sources between soils (Table 3). Averaged across water types, belowground N uptake was numerically largest from CPST in the Creldon soil, which did not differ from ECSTreal in the Creldon soil, and was numerically smallest from the UC in the Creldon soil, which did not differ from the UC-Calloway, CPST-Calloway, MAP-Calloway, ECSTreal-Calloway, and ECSTsyn-Calloway and -Creldon combinations (Figure 2). Belowground N uptake for the CPST-Creldon combination was at least 1.2 times greater than for all other soil-fertilizer-P-source combinations, except for ECSTreal in the Creldon soil (Figure 2). Belowground N uptake was greater for the Creldon than the Calloway soil for CPST, MAP, and ECSTreal, while there was no difference between soils for ECSTsyn or the UC (Figure 2). In the Creldon soil,

belowground N uptake was at least 1.3 times greater for CPST and ECSTreal, which did not differ, than for ECSTsyn, while there was no difference in belowground N uptake among struvite sources in the Calloway soil (Figure 2).

In contrast to the other belowground corn properties, belowground P and Mg uptakes differed ( $P < 0.05$ ) among soil-water-fertilizer-P-source combinations (Table 3). Belowground P uptake differences among treatment combinations were complex, but belowground P uptake was numerically largest from the Creldon-tap water-CPST combination, which did not differ from the Creldon-wastewater-CPST combination and was numerically smallest from the Creldon-tap water-UC combination, which did not differ from the Creldon-wastewater-UC combination (Table 5). Belowground P uptake differed among the struvite materials in the following order: CPST > ECSTsyn > ECSTreal for the Creldon-tap water combination, CPST = ECSTreal = ECSTsyn for the Calloway-tap water combination, CPST > ECSTreal = ECSTsyn for the Creldon-wastewater combination, and CPST = ECSTsyn > ECSTreal for the Calloway-wastewater combination (Table 5).

Similar to P, belowground Mg uptake differences among treatment combinations were complex, but belowground Mg uptake was numerically largest from the Creldon-tap water-CPST combination, which did not differ from the Calloway-tap water-ECSTreal and Creldon-wastewater-CPST combinations, and was numerically smallest from the Creldon-tap water-UC combination, which did not differ from eight other soil-water-fertilizer-P-source combinations (Table 5). Belowground Mg uptake differed among the struvite materials in the following order: CPST > ECSTsyn = ECSTreal for the Creldon-tap water combination, ECSTreal > CPST = ECSTsyn for the Calloway-tap water combination, CPST = ECSTreal = ECSTsyn for the

Creldon-wastewater combination, and  $CPST = ECST_{syn} > ECST_{real}$  for the Calloway-wastewater combination (Table 5).

For belowground corn, CPST generally did not differ from the other struvite treatments or resulted in the greatest response for nutrient concentration and uptake. Specifically for N and P concentration and N uptake, CPST was largest, which could partially have been due to struvite's slow-release behavior or the pelletized form of CPST resulting in slower dissolution, keeping CPST-derived nutrients plant-available for longer than the other fertilizer-P sources. The Creldon soil, in general, resulted in a greater plant response than Calloway, which could be explained by the greater initial N and P concentrations of the Creldon soil. In contrast, tap water, which had a much lower initial nutrient concentration than wastewater (Table 2), resulted in the greater plant response between the two water types. The large initial micro- and macro-nutrient concentrations in the wastewater could have caused unexpected interactions among the water, soils, and fertilizer-P sources, causing the greater plant response to tap water.

### Aboveground

Similar to belowground dry matter, aboveground corn dry matter differed ( $P < 0.05$ ) among water-soil-fertilizer-P source treatment combinations (Table 3). Aboveground corn dry matter interactions among treatment combinations were complex, but aboveground corn dry matter was numerically largest from the Calloway-tap water-MAP combination, which did not differ from any other water-soil-MAP, water-soil- $ECST_{syn}$ , or the Calloway-wastewater- $ECST_{syn}$  treatment combinations (Table 5). Aboveground corn dry matter was numerically smallest from the Creldon-tap water-UC treatment combination (Table 5). Aboveground corn dry matter differed among struvite treatments in the following order:  $ECST_{syn} = ECST_{real} > CPST$

for the Creldon-tap water combination,  $ECST_{syn} > ECST_{real} = CPST$  for the Calloway-tap water combination,  $ECST_{syn} > ECST_{real} = CPST$  for the Creldon-wastewater combination, and  $ECST_{syn} = ECST_{real} > CPST$  for the Calloway-wastewater combination (Table 5). Results of the current study differed from Omidire et al. (2022b), in which aboveground corn dry matter did not differ among treatment combinations. However, results of the current study were similar to Ylagan et al. (2020), in which the traditional, commercially available, fertilizer-P source (i.e., TSP) produced the largest aboveground corn dry matter.

Averaged across water types and fertilizer treatments, aboveground corn Mg concentration differed ( $P < 0.05$ ) between soils (Table 3). The Calloway soil had a larger Mg concentration (0.58%) than the Creldon soil (0.55%).

Aboveground corn N and P concentrations differed ( $P < 0.05$ ) between soils across fertilizer-P sources. Averaged across water types, aboveground N concentration was 1.3 times larger from the UC in the Creldon soil, which did not differ from CPST in the Creldon soil (Figure 3). Aboveground N concentration was greater with Creldon for all fertilizer-P treatments, except for  $ECST_{syn}$ , where Creldon and Calloway did not differ (Figure 3). Aboveground corn N concentration was numerically smallest from MAP in the Calloway soil, which did not differ from either soil- $ECST_{syn}$  or the UC-Calloway combination (Figure 3). For aboveground N concentration from the Creldon soil,  $CPST > ECST_{real} > ECST_{syn}$ , while  $CPST = ECST_{real} > ECST_{syn}$  for Calloway (Figure 3). It is unclear why the corn N concentration was greater in the unfertilized UC treatment than the other fertilizer-P source treatments, but it is possible corn translocated more N to the aboveground tissue due to the P deficiency in the UC. Similar to the results of the current study, Ylagan et al. (2020) reported differences in N concentration among fertilizer-P treatments where CPST had the largest aboveground corn N concentration. However,



in contrast to both the results of Ylagan et al. (2020) and the current results, Omidire et al. (2022b) reported that aboveground corn N concentration was unaffected by fertilizer-P source.

In contrast to aboveground N concentration and to the results of Omidire et al. (2022b), averaged across water types, aboveground corn P concentration was at least 1.3 times larger from ECSTsyn in the Creldon soil and differed from all other soil-fertilizer treatment combinations (Figure 3). The numerically smallest P concentration was from UC in the Creldon soil, which also differed from all other soil-fertilizer treatment combinations (Figure 3). Aboveground P concentration was greater from the Creldon than the Calloway soil for ECSTreal and ECSTsyn, was greater with Calloway for MAP and the UC, and did not differ for CPST (Figure 3). With both the Creldon and Calloway soils, aboveground P concentration was larger from ECSTsyn than from ECSTreal, which was larger than CPST (Figure 3). The results of the current study were similar to those of Ylagan et al. (2020), where ECSTsyn had the largest aboveground corn P concentration among fertilizer-P sources evaluated.

Unlike the results of Omidire et al. (2022b), aboveground corn Mg concentration differed ( $P < 0.05$ ) between water types across fertilizer treatments (Table 3). Averaged across soils, aboveground Mg concentration was 1.1 times larger from ECSTsyn with tap water than all other water-fertilizer-P source combinations and was numerically smallest from MAP with tap water, which did not differ from MAP with wastewater or the UC with tap water (Figure 3). Aboveground Mg concentration was larger with tap water than wastewater for ECSTsyn and did not differ between water types for all other fertilizer treatments (Figure 3). With tap water and wastewater, aboveground Mg concentration was larger from ECSTsyn than from ECSTreal, which did not differ from CPST (Figure 3). Ylagan et al. (2020) also reported that aboveground

Mg concentration from ECSTsyn was numerically largest and did not differ among struvite treatments.

In contrast to the results of Omidire et al. (2022b), aboveground N and P uptake differed ( $P < 0.05$ ) between soils across fertilizer treatments (Table 3). Averaged across water types, aboveground N uptake was numerically largest from ECSTsyn in the Creldon soil, which did not differ from ECSTsyn in the Calloway soil, ECSTreal in either soil, Creldon-CPST or -MAP (Figure 4). The numerically smallest aboveground N uptake was from the UC in the Creldon soil, which differed from all other treatment combinations (Figure 4). Aboveground N uptake was greater from the Creldon than the Calloway soil for CPST, greater with Calloway than Creldon for the UC, but similar between soils in MAP, ECSTreal, and ECSTsyn (Figure 4). With both Creldon and Calloway soils, aboveground N uptake was the same in ECSTreal as ECSTsyn and with the Creldon soil for CPST, which all were larger than with the Calloway soil for CPST (Figure 4).

Averaged across water types and unlike the results of Omidire et al. (2022b), aboveground P uptake was numerically at least 1.2 times larger from ECSTsyn in the Creldon soil than all other treatment combinations (Figure 4). Aboveground P uptake was numerically smallest from the UC in the Creldon soil, which also differed from all other treatment combinations (Figure 4). Aboveground P uptake was larger with the Calloway than Creldon soil for CPST, MAP, and the UC, but larger in the Creldon than Calloway soil for ECSTreal and ECSTsyn (Figure 4). With both Creldon and Calloway soils, aboveground P uptake from ECSTsyn was larger than ECSTreal, which was larger than CPST (Figure 4).

Unlike the results of Omidire et al. (2022b), aboveground corn Mg uptake differed ( $P < 0.05$ ) among water-soil-fertilizer-P source treatment combinations (Table 3). Aboveground Mg

uptake treatment interactions were complex, but aboveground Mg uptake was numerically largest from the Calloway-tap water-ECSTsyn combination, which did not differ from any other soil-water-ECSTsyn or from the Calloway-wastewater-ECSTreal treatment combinations (Table 5). Aboveground Mg uptake was numerically smallest from the Creldon-tap water-UC combination, which differed from all other soil-water-fertilizer-P source treatment combinations (Table 5). Aboveground Mg uptake differed among the struvite materials in the following order: ECSTsyn > ECSTreal > CPST for the Creldon-tap water combination, ECSTsyn > CPST = ECSTreal for the Calloway-tap water combination, ECSTsyn > ECSTreal > CPST for the Creldon-wastewater combination, and ECSTsyn = ECSTreal > CPST for the Calloway-wastewater combination (Table 5).

In general, the UC had the smallest aboveground corn response among all fertilizer treatments, which was expected since the UC was unfertilized. In most cases, ECSTsyn had the largest plant response, especially for P and Mg concentration and uptake, due to its crystalline form and large initial P and Mg. The crystalline form of ECSTsyn had a larger surface area than the other pelletized fertilizer-P sources, which could have resulted in easier uptake for the plant. Corn response in the Creldon soil was greater than in the Calloway soil in most instances, especially for ECSTsyn and ECSTreal, likely due to struvite's greater solubility under more acidic conditions (pH 7.5 for Calloway vs. pH 6.0 for Creldon; Table 1). Similar to belowground corn and the reasons explained above, aboveground corn also had a greater response to tap water than wastewater.

#### Total

Total corn dry matter differed ( $P < 0.05$ ) between water types (Table 3). Averaged across fertilizer-P sources and soils, total dry matter was larger (41.5 g) from wastewater than from tap water (39.3 g).

Unlike the results of Omidire et al. (2022b), total dry matter also differed ( $P < 0.05$ ) among fertilizer-P sources between soils (Table 3). Averaged across water types, total dry matter was largest from MAP in the Calloway soil and did not differ from MAP in the Creldon soil, ECSTreal, or ECSTsyn (Figure 5). The numerically smallest total dry matter was from the UC in the Creldon soil, which differed from all other soil-fertilizer-P source treatment combinations (Figure 5). Total corn dry matter was greater with the Calloway than the Creldon soil for the UC, but did not differ between soils for all other soil-fertilizer-P source combinations (Figure 5). With both the Creldon and Calloway soil, total corn dry matter from ECSTreal and ECSTsyn did not differ, but both were greater than CPST (Figure 5). In contrast to result of the current study, Ylagan et al. (2020) reported that ECSTsyn, not MAP, had the largest total corn dry matter. However, similar to the result of the current study, Ylagan et al. (2020) reported that ECSTsyn had a larger total corn dry matter than CPST.

Total N and P uptake differed ( $P < 0.05$ ) among fertilizer treatments between soils (Table 3). Averaged across water types, total N uptake was numerically largest from ECSTreal in the Creldon soil, which did not differ from the Creldon-CPST, -MAP, or either ECSTsyn-soil combinations (Figure 6). The numerically smallest total N uptake was from the UC in the Creldon soil, which differed from all other soil-fertilizer-P source combinations (Figure 6). Total N uptake was greater with the Creldon than the Calloway soil for CPST and ECSTreal, greater with Calloway than Creldon for the UC, and did not differ between soils for MAP and ECSTsyn (Figure 6). With both Calloway and Creldon soils, total N uptake did not differ for ECSTreal,

ECSTsyn, or Creldon-CPST, while Calloway-CPST was lower than all other soil-struvite treatment combinations (Figure 6).

In contrast to total N uptake, total P uptake in almost all treatment combinations differed from each other (Figure 6). Averaged across water types, total P uptake was 1.3 times larger from ECSTsyn in the Creldon soil than in all other combinations, while the numerically smallest total P uptake was from the UC in the Creldon soil, which differed from all other combinations (Figure 6). Total P uptake was greater with the Creldon than the Calloway soil for CPST, ECSTreal, and ECSTsyn, while total P uptake from the Calloway was greater than the Creldon soil for MAP and the UC (Figure 6). With both Creldon and Calloway soils, total P uptake ECSTsyn was larger than CPST and ECSTreal, which did not differ (Figure 6). Omidire et al. (2022b) reported that N and P uptake was unaffected by fertilizer-P source, which was unlike the results of the current study.

Unlike the results of Omidire et al. (2022b), total Mg uptake differed ( $P < 0.05$ ) among water-soil-fertilizer-P source treatment combinations (Table 3). Total Mg uptake interactions among treatment combinations were complex, but total Mg uptake was numerically greatest from Calloway-tap water-ECSTsyn, which did not differ from any other soil-water-ECSTsyn combination (Table 5). The numerically smallest total Mg uptake was from the Creldon-tap water-MAP, which did not differ from Creldon-wastewater-MAP, Creldon-tap water-CPST, Calloway-tap water-CPST, -ECSTreal, -UC, or Calloway-wastewater-UC combinations (Table 5). Total Mg uptake differed among the struvite materials in the following order: ECSTsyn > ECSTreal = CPST for the Creldon-tap water combination, ECSTsyn > ECSTreal = CPST for the Calloway-tap water combination, ECSTsyn > ECSTreal > CPST for the Creldon-wastewater

combination, and ECSTsyn > ECSTreal > CPST for the Calloway-wastewater combination (Table 5).

The largest total corn dry matter was for MAP, which was reasonable as MAP had the largest solubility of all fertilizer-P sources. The nutrients in MAP would have been released faster than the other fertilizer-P sources and caused greater foliage growth more quickly. For similar reasons to above- and belowground corn, the greatest total corn response was, in general, to ECSTsyn or ECSTreal, to the Creldon soil, especially for the struvite treatments, and to tap water compared to the other fertilizer-P sources, the Calloway soil, and wastewater, respectively. Since the total corn dry matter and uptakes were calculated from the above- and belowground responses, total corn plant responses mirrored the aboveground corn response results.

## **Soybean response**

### Belowground

Similar to corn, every measured belowground soybean parameter (i.e., dry matter and N, P, and Mg concentration and uptake) was affected ( $P < 0.05$ ) by a combination of one or more treatments (i.e., water type, soil, and/or fertilizer-P source; Table 6). Belowground soybean dry matter differed ( $P < 0.05$ ) between soils among fertilizer-P sources (Table 6). Averaged across water types, belowground dry matter was numerically largest for the UC in the Calloway soil, which did not differ from Calloway-CPST, -MAP, and both soil-ECSTreal and -ECSTsyn treatment combinations (Figure 7). Belowground dry matter was smallest for the UC in the Creldon soil, which differed from all other soil-fertilizer-P source treatment combinations (Figure 7). Belowground dry matter was greater in the Calloway than in the Creldon soil for CPST and the UC, and belowground soybean dry matter did not differ between soils for MAP,

ECSTreal, or ECSTsyn (Figure 7). With both the Calloway and Creldon soils, belowground soybean dry matter from the struvites did not differ from each other, except for CPST in the Creldon soil, which was smaller than the other soil-struvite treatment combinations (Figure 7). It is unclear why the soybean belowground dry matter was numerically greater in the unfertilized UC treatment than the other fertilizer-P source treatments. In contrast to the current results, Ylagan et al. (2020) reported that ECSTsyn had the largest corn belowground dry matter and MAP had the smallest belowground dry matter.

In contrast to Omidire et al. (2023) and Ylagan et al. (2020), belowground soybean Mg concentration differed ( $P < 0.05$ ) among fertilizer-P sources (Table 6). Averaged across soils and water types, belowground Mg concentration was numerically largest for ECSTsyn, which did not differ from CPST or ECSTreal, and was smallest for the UC, which did not differ from MAP (Table 4).

Belowground soybean P concentration differed ( $P < 0.05$ ) between soils across fertilizer treatments (Table 6). Averaged across water types, belowground P concentration was 1.8 times larger for ECSTsyn in Creldon than all other soil-fertilizer-P source treatment combinations (Figure 8). Belowground P concentration was smallest for the UC in the Creldon soil, which differed from all other soil-fertilizer-P source treatment combinations (Figure 8). Belowground P concentration was greater in Creldon than in Calloway for CPST, ECSTreal, and ECSTsyn, greater for Calloway than Creldon for the UC, and did not differ between soils for MAP (Figure 8). With both Calloway and Creldon soils, ECSTsyn was larger than CPST and ECSTreal, which did not differ (Figure 8). In contrast to the results of the current study, Omidire et al. (2023) reported no significant treatment effect on belowground P concentration. However, Ylagan et al. (2020) reported that belowground P concentration was largest for MAP, which differed from

results of the current study where ECSTsyn had the largest belowground P concentration. Ylagan et al. (2020) also reported no difference in belowground soybean P concentration between ECSTsyn or CPST, which was similar to results of the current study.

In contrast to the results of Omidire et al. (2022b), belowground N concentration differed ( $P < 0.05$ ) among water-soil-fertilizer-P source treatment combinations (Table 6). Belowground N concentrations among treatment interactions were complex, but belowground N concentration was numerically largest in the Creldon-tap water-ECSTsyn and Calloway-tap water-ECSTsyn treatment combinations, which did not differ from each other or from any other soil-water-ECSTreal or -ECSTsyn combination, Creldon-tap water-MAP, Calloway-tap water-CPST or -MAP, or Calloway-wastewater-CPST or -MAP (Table 7). Belowground N concentration was numerically smallest in the Creldon-wastewater-UC treatment combination, which did not differ from any other soil-water-UC combination, Creldon-tap water-CPST, Creldon-wastewater-CPST, or Creldon-wastewater-MAP (Table 7). Belowground N concentration differed among the struvite materials in the following order: ECSTsyn = ECSTreal > CPST for the Creldon-tap water combination, ECSTsyn = ECSTreal = CPST for the Calloway-tap water combination, ECSTsyn = ECSTreal > CPST for the Creldon-wastewater combination, and ECSTsyn = ECSTreal = CPST for the Calloway-wastewater combination (Table 7). In contrast to result of the current study, Ylagan et al. (2020) reported that belowground soybean N concentration was largest in CPST rather than ECSTsyn, while belowground N concentration for ECSTsyn was smallest.

Nitrogen and Mg uptake differed ( $P < 0.05$ ) between soils among fertilizer-P treatments (Table 6). Averaged across water types, belowground N uptake was numerically largest for the UC in the Calloway soil, which did not differ from any other Calloway-fertilizer-P source



combination or for ECSTsyn in the Creldon soil (Figure 9). Belowground N uptake was numerically smallest for the UC in the Creldon soil, which differed from all other soil-fertilizer-P source treatment combinations (Figure 9). Belowground N uptake was larger in the Calloway than the Creldon soil for the UC and CPST, and was similar between the Creldon and Calloway soils for MAP, ECSTreal, and ECSTsyn (Figure 9). Belowground N uptake for CPST, ECSTreal, and ECSTsyn were similar between the Calloway and Creldon soils, except for CPST, where CPST in the Creldon soil was smaller than ECSTreal and ECSTsyn (Figure 9).

Averaged across water types, belowground Mg uptake was numerically largest for CPST in the Calloway soil, which did not differ from any other Calloway-fertilizer-P source treatment combination (Figure 9). Belowground Mg uptake was numerically smallest for the UC in the Creldon soil, which differed from all other soil-fertilizer-P source treatment combinations (Figure 9). Belowground Mg uptake was larger in the Calloway than the Creldon soil for all treatment combinations (Figure 9). Belowground Mg uptake did not differ between struvite treatments (Figure 9).

In contrast to N and Mg, belowground P uptake differed ( $P < 0.05$ ) among water-soil-fertilizer-P source treatment combinations (Table 6). Belowground P uptake interactions between treatment combinations were complex, but the numerically largest P uptake was from the Calloway-tap water-CPST combination, which did not differ from 10 other soil-water-fertilizer-P source treatment combinations (Table 7). Belowground P uptake was numerically smallest in the Creldon-wastewater-UC combination, which did not differ from the Creldon-tap water-UC or -CPST combinations (Table 7). Belowground P uptake differed among the struvite materials in the following order: ECSTsyn = ECSTreal > CPST for the Creldon-tap water combination, ECSTsyn = ECSTreal = CPST for the Calloway-tap water combination, ECSTsyn = ECSTreal =

CPST for the Creldon-wastewater combination, and  $ECST_{syn} = ECST_{real} = CPST$  for the Calloway-wastewater combination (Table 7). In contrast to the results of the current study, Omidire et al. (2023) reported that belowground soybean N, Mg, and P uptakes did not differ among fertilizer-P sources.

Similar to the corn response,  $ECST_{syn}$  had, in general, the largest soybean response among all fertilizer-P sources and tap water had a greater soybean response than wastewater. The largest response to  $ECST_{syn}$  was likely due to the crystalline form of  $ECST_{syn}$  having a greater surface area than the other pelletized fertilizers and making greater contact with the soil for dissolution and uptake by soybean roots, promoting growth. In most cases, soybean responses in the Creldon soil were greater than in the Calloway soil for the struvite treatments, which was expected due to the increased solubility of struvite under acidic conditions. The greater response to tap water than wastewater was likely due to the larger initial nutrient concentrations in the wastewater (Table 2). The large initial micro- and macro-nutrient concentrations, large pH, and potential unknown contaminants in the real wastewater could have caused unexpected, antagonistic interactions among the water, soils, and fertilizer-P sources, causing the greater plant response to tap water. In general, soybean response to the UC was smallest, which was expected due to the UC being unfertilized.

### Aboveground

Every measured aboveground soybean parameter (i.e., dry matter and N, P, and Mg concentration and uptake) was affected ( $P < 0.05$ ) by a combination of one or more treatments (i.e., water type, soil, and/or fertilizer-P source; Table 6). Aboveground dry matter differed ( $P < 0.05$ ) between soils across fertilizer-P sources (Table 6). Averaged across water types,

aboveground dry matter was numerically largest for ECSTsyn in the Calloway soil, which did not differ from ECSTsyn in the Creldon soil or ECSTreal or MAP in the Calloway soil (Figure 10). Aboveground dry matter was numerically smallest for the UC in the Creldon soil, which differed from all other soil-fertilizer-P source treatment combinations (Figure 10). Aboveground dry matter was larger in the Calloway soil than the Creldon soil for CPST, MAP, and the UC, and did not differ between soils for ECSTreal and ECSTsyn (Figure 10). With both the Calloway and Creldon soils, aboveground dry matter from ECSTreal and ECSTsyn did not differ, while CPST was smaller than ECSTsyn or ECSTreal (Figure 10). In contrast to the results of the current study, Omidire et al. (2023) and Ylagan et al. (2020) both reported that aboveground soybean dry matter was unaffected by fertilizer-P source.

Aboveground dry matter also differed ( $P < 0.05$ ) between water types across fertilizer-P sources (Table 6). Averaged across soils, aboveground dry matter was largest for ECSTsyn with tap water, which did not differ from MAP or ECSTreal with tap water (Figure 10). Aboveground dry matter was numerically smallest for the UC with tap water, which did not differ for the UC with wastewater (Figure 10). Aboveground dry matter was larger with tap water than wastewater for MAP and ECSTsyn, but did not differ between water types for the other fertilizer-P source treatments (Figure 10). With tap water and wastewater, aboveground dry matter from ECSTreal and ECSTsyn did not differ from each other, but both were larger than CPST (Figure 10).

Aboveground soybean N concentration differed ( $P < 0.05$ ) between water types across fertilizer-P treatments (Table 6). Averaged across soils, aboveground N concentration was numerically largest for ECSTsyn with tap water, which did not differ from ECSTsyn or ECSTreal with wastewater (Figure 11). Aboveground N concentration was numerically smallest for CPST with tap water, which did not differ from CPST or MAP with wastewater, ECSTreal

with tap water, or either water-UC combination (Figure 11). Aboveground N concentration was larger with tap water than wastewater for MAP but did not differ between water types for all other water-fertilizer-P source treatment combinations (Figure 11). With tap water and wastewater, aboveground N concentrations for ECSTsyn and ECSTreal were similar to each other, while CPST in the Calloway soil was similar to ECSTsyn and ECSTreal, and CPST in the Creldon soil was smaller than ECSTsyn or ECSTreal (Figure 11).

Aboveground soybean P concentration differed ( $P < 0.05$ ) between water types within soils (Table 6). Averaged across fertilizer-P sources, aboveground P concentration in the Creldon-tap water combination was numerically largest and did not differ from the Creldon-wastewater combination, while the Calloway-tap water combination was numerically smallest and differed from all other soil-water treatment combinations (Figure 11).

Aboveground soybean N, P, and Mg concentrations also differed ( $P < 0.05$ ) between soils among fertilizer-P sources (Table 6). In contrast to both Omidire et al. (2023) and Ylagan et al. (2020), averaged across water types, aboveground N concentration was numerically largest for ECSTsyn in the Calloway soil, which did not differ from ECSTsyn in the Creldon soil or MAP in the Calloway soil (Figure 12). Aboveground N concentration was numerically smallest for CPST in the Creldon soil, which did not differ from MAP in the Creldon soil (Figure 12). Aboveground N concentration was larger for the Calloway than Creldon soil for CPST and MAP, while the soils did not differ for ECSTsyn, ECSTreal, and the UC. With both the Calloway and Creldon soils, aboveground N concentration for ECSTsyn was larger than ECSTreal, which did not differ from the CPST-Calloway combination, and the CPST-Creldon combination was smallest among the struvite materials (Figure 12).

Averaged across water types, aboveground P concentration was 1.3 times larger for ECSTsyn in the Creldon soil than any other treatment combination, while aboveground P concentration was numerically smallest for the UC in the Creldon soil, which differed from all other soil-fertilizer-P source treatment combinations (Figure 12). Aboveground P concentration was larger in the Creldon soil than the Calloway soil for CPST, ECSTreal, and ECSTsyn, smaller in the Creldon soil than the Calloway soil in the UC and did not differ between soils for MAP (Figure 12). With both the Creldon and Calloway soils, aboveground P concentration for ECSTsyn was larger than ECSTreal, which was larger than CPST (Figure 12). Similar to the results of the current study, Omidire et al. (2023) and Ylagan et al. (2020) both reported that aboveground soybean P concentration differed among fertilizer-P sources. Omidire et al. (2023) reported that ECSTsyn had the greatest aboveground soybean P concentration, which did not differ from CPST, while Ylagan et al. (2020) reported that MAP had the greatest aboveground soybean P concentration, which did not differ from ECSTsyn or CPST.

Averaged across water types, aboveground Mg concentration was 1.1 times larger for ECSTsyn in the Creldon soil than any other soil-fertilizer-P source combination, while the numerically smallest Mg concentration was for the UC in the Calloway soil, which did not differ from CPST or MAP in the Calloway soil (Figure 12). Aboveground Mg concentration was larger in the Creldon soil than the Calloway soil for all soil-fertilizer-P source treatment combinations (Figure 12). With both the Calloway and Creldon soils, aboveground Mg concentration for ECSTsyn was larger than ECSTreal and CPST, which did not differ from each other in the Creldon soil, but ECSTreal was larger than CPST for the Calloway soil (Figure 12). Similar to the results of the current study, Ylagan et al. (2020) also reported that ECSTsyn had the numerically largest aboveground Mg concentration among all fertilizer-P sources. However, both

the results of the current study and Ylagan et al. (2020) differed from results of Omidire et al. (2023), in which aboveground Mg concentration was unaffected by fertilizer-P source.

Aboveground soybean N, P, and Mg uptake differed ( $P < 0.05$ ) between soils among fertilizer-P sources (Table 6). Averaged across water types, aboveground N uptake was numerically largest for ECSTsyn in the Calloway soil, which did not differ from MAP in the Calloway soil (Figure 13). The numerically smallest aboveground N uptake was for the UC in the Creldon soil, which did not differ from CPST in the Creldon soil (Figure 13). Aboveground N uptake was larger in the Calloway than Creldon soil for CPST, MAP, ECSTsyn, and the UC, but did not differ between soils for ECSTreal (Figure 13). With both the Calloway and Creldon soils, aboveground N uptake from ECSTsyn was larger than ECSTreal, which did not differ from the Calloway-CPST combination, while the Creldon-CPST combination was smallest among the struvite treatment combinations (Figure 13).

Averaged across water types, aboveground P uptake was two times larger for ECSTsyn in both soils than in any other soil-fertilizer-P source combination, while ECSTsyn in Creldon was numerically largest and differed from all other treatment combinations (Figure 13). The numerically smallest aboveground P uptake was for the UC in the Creldon soil, which differed from all other soil-fertilizer-P source combinations (Figure 13). Aboveground P uptake was larger in the Creldon than Calloway soil for ECSTreal and ECSTsyn, smaller in the Creldon than the Calloway soil for the UC, and did not differ between soils for CPST and MAP. For both the Creldon and Calloway soils, aboveground P uptake from ECSTsyn was larger than ECSTreal, which was larger than CPST (Figure 13).

Averaged across water types, aboveground Mg uptake was numerically largest for ECSTsyn in the Creldon soil, which did not differ from ECSTsyn in the Calloway soil (Figure

13). Aboveground Mg uptake was numerically smallest for the UC in the Creldon soil, which differed from all other soil-fertilizer-P source combinations (Figure 13). Aboveground Mg uptake was larger for the Creldon than the Calloway soil for MAP and ECSTreal, while Mg uptake was larger for the Calloway than the Creldon soil for the UC, and Mg uptake did not differ between soils for CPST and ECSTsyn (Figure 13). With both the Creldon and Calloway soils, aboveground Mg uptake from ECSTsyn was larger than ECSTreal, which was larger than CPST (Figure 13).

Aboveground soybean N, P, and Mg uptakes also differed ( $P < 0.05$ ) between water types among fertilizer-P sources (Table 6). Averaged across soils, aboveground N uptake was 1.3 times larger for ECSTsyn with tap water than any other water-fertilizer-P source combination, and N uptake was smallest for the UC with tap water, which did not differ from the UC-wastewater, either CPST-water combination, or MAP-wastewater combinations (Figure 14). Aboveground N uptake was larger with tap water than wastewater for MAP and ECSTsyn, but tap water and wastewater did not differ for all other fertilizer-P sources (Figure 14). With tap water and wastewater, aboveground N uptake from ECSTsyn was larger than ECSTreal, which was larger than CPST (Figure 14).

Averaged across water types, aboveground P uptake was numerically largest for ECSTsyn with tap water, which did not differ from ECSTsyn with wastewater (Figure 14). The smallest aboveground P uptake was for the UC with tap water, which differed from all other water-fertilizer-P source combinations (Figure 14). Aboveground P uptake was larger with tap water than wastewater with CPST and MAP, larger with wastewater than tap water for the UC, and did not differ between water types for ECSTsyn and ECSTreal (Figure 14). With tap water and wastewater, aboveground P uptake from ECSTsyn was larger than ECSTreal, which was

larger than CPST (Figure 14). Similar to the results of the current study, Omidire et al. (2023) reported that aboveground N and P uptake were largest for ECSTsyn.

In contrast to the results of Omidire et al. (2023), aboveground Mg uptake differed among water type-fertilizer-P source combinations. Averaged across soils, aboveground Mg uptake was numerically largest for ECSTsyn with tap water, which did not differ from ECSTsyn with wastewater (Figure 14). Aboveground Mg uptake was numerically smallest for the UC with tap water, which did not differ from the UC with wastewater (Figure 14). Aboveground Mg uptake was larger with tap water than wastewater for MAP, but did not differ between water types for all other fertilizer-P sources (Figure 14). With tap water and wastewater, aboveground Mg uptake from ECSTsyn was larger than ECSTreal, which was larger than CPST (Figure 14).

Aboveground soybean responses to fertilizer-P sources, water types, and soils were due to similar reasons as corn and belowground soybean responses. Generally, the largest soybean response was to ECSTsyn due to its crystalline nature as described for belowground soybean. The soybean properties had a greater response to the struvite treatments in the Creldon (pH 6.0) than the Calloway (pH 7.5) soil due to the greater solubility of struvite under more acidic conditions. The soybean properties also had a greater response to tap water than wastewater in all fertilizer treatments for the same reasons explained for belowground soybean, except the UC, which had a greater response with wastewater due to its nutrient addition. The UC had the smallest soybean response in most cases due to being unfertilized.

### Total

Every calculated total soybean parameter (i.e., total dry matter and total N, P, and Mg uptake) was affected ( $P < 0.05$ ) by a combination of two or more treatments (i.e., water type,



soil, and/or fertilizer-P source; Table 6). Total soybean dry matter differed ( $P < 0.05$ ) between soils among fertilizer-P sources (Table 6). Averaged across water types, total dry matter was numerically largest for ECSTsyn in the Calloway soil, which did not differ from ECSTsyn in the Creldon soil or any other Calloway-fertilizer-P source combination (Figure 7). Total dry matter was smallest for the UC in the Creldon soil, which differed from all other soil-fertilizer-P source treatment combinations (Figure 7). Total dry matter was larger in the Calloway than in the Creldon soil for CPST, MAP, and the UC, and did not differ between soils for ECSTsyn or ECSTreal (Figure 7). For both soils, total soybean dry matter for ECSTsyn and ECSTreal did not differ from each other or from CPST in the Calloway soil, which were all larger than CPST in the Creldon soil (Figure 7). In contrast to results of the current study, Omidire et al. (2023) and Ylagan et al. (2020) reported that total soybean dry matter was unaffected by fertilizer-P source.

Total soybean dry matter also differed ( $P < 0.05$ ) between water types among fertilizer-P sources (Table 6). Averaged across soils, total soybean dry matter was numerically largest for ECSTsyn with tap water, which did not differ from MAP with tap water (Figure 7). The numerically smallest total dry matter was for the UC with tap water, which did not differ from the UC with wastewater (Figure 7). Total soybean dry matter was larger with tap water than wastewater for MAP and ECSTsyn, but did not differ between water types for CPST, ECSTreal, or the UC (Figure 7). With tap water, total soybean dry matter from ECSTsyn was larger than ECSTreal, which was larger than CPST. For wastewater, total soybean dry matter did not differ among struvite materials (Figure 7).

Total soybean N and P uptake differed ( $P < 0.05$ ) between soils among fertilizer-P sources (Table 6). Averaged across water types, total N uptake was 1.3 times larger for ECSTsyn in the Calloway soil than in any other soil-fertilizer-P source combination and numerically

smallest for the UC in the Creldon soil, which differed from all other soil-fertilizer-P source combinations (Figure 15). Total N uptake was larger for the Calloway than the Creldon soil in CPST, MAP, ECSTsyn, and the UC and did not differ between soils for ECSTreal (Figure 15). With the Calloway and Creldon soils, total N uptake from ECSTsyn was larger than ECSTreal, which was larger than CPST (Figure 15).

Averaged across water types and similar to the results of Omidire et al. (2023), total P uptake was 1.2 times larger for ECSTsyn in the Creldon soil than in any other soil-fertilizer-P source combinations, while total P uptake was numerically smallest for the UC in the Creldon soil, which differed from all other soil-fertilizer-P source combinations (Figure 15). Total P uptake was larger in the Creldon than in the Calloway soil for ECSTreal and ECSTsyn, larger in the Calloway than in the Creldon soil for the UC, and did not differ between soils for CPST and MAP (Figure 15). With both the Calloway and Creldon soils, total P uptake from ECSTsyn was larger than ECSTreal, which was larger than CPST (Figure 15).

Total soybean N uptake differed ( $P < 0.05$ ) between water types among fertilizer-P sources (Table 6). Averaged across soils, total N uptake was 1.4 times larger for ECSTsyn with tap water than any other water-fertilizer-P source combination (Figure 15). The numerically smallest total N uptake was for the UC with tap water, which did not differ from the UC with wastewater, MAP with wastewater, or CPST with tap water (Figure 15). Total N uptake was larger with tap water than wastewater for MAP and ECSTsyn and did not differ between water types for CPST, ECSTreal, and the UC (Figure 15). With tap water, total N uptake from ECSTsyn was larger than ECSTreal, which was larger than CPST (Figure 15). For wastewater, total N uptake from ECSTsyn and ECSTreal did not differ, but were larger than CPST (Figure

15). Omidire et al. (2023) also reported a significant fertilizer-P source effect, in which ECSTsyn had the largest total soybean N uptake and did not differ from CPST.

Total soybean Mg uptake differed ( $P < 0.05$ ) among water-soil-fertilizer-P source treatment combinations (Table 6). Total Mg uptake interactions varied among treatment combinations were complex, but total Mg uptake was numerically largest for the Creldon-tap water-ECSTsyn combination, which did not differ from any other soil-water-ECSTsyn treatment combination (Table 7). The numerically smallest total Mg uptake was in the Creldon-wastewater-UC combination, which did not differ from the Creldon-tap water-UC combination (Table 7). Total Mg uptake differed among the struvite materials in the following order: ECSTsyn > ECSTreal > CPST for the Creldon-tap water combination, ECSTsyn > ECSTreal = CPST for the Calloway-tap water combination, ECSTsyn > ECSTreal > CPST for the Creldon-wastewater combination, and ECSTsyn > ECSTreal > CPST for the Calloway-wastewater combination (Table 7). In contrast to results of the current study, Omidire et al. (2023) reported that total soybean Mg uptake was unaffected by fertilizer-P source.

For similar reasons to below- and aboveground corn and soybean response, the greatest total soybean response was, in general, to ECSTsyn or ECSTreal due to their crystalline nature. The greater total soybean response was also to the Creldon soil rather than the Calloway soil due to soil pH differences, especially for the struvite treatments, and to tap water, as explained for below- and aboveground soybean responses. Since total soybean dry matter and uptakes were calculated from the above- and belowground responses, total soybean responses mirrored aboveground results.

## **Implications**

As demonstrated in the current study and others (Omidire et al., 2022a,b, 2023; Ylagan et al., 2020), struvite can perform similarly to traditional fertilizer-P sources in terms of crop growth and yield. Struvite may also reduce the cost of extra needed urea-N inputs due to struvite containing more N than many traditional P-only fertilizer sources, namely TSP (Omidire et al., 2022a). Due to ECST still being in an experimental state, ECST is more costly than traditional fertilizers to produce. According to Omidire et al. (2023), in 2019, the cost of producing ECSTsyn was greater than the cost to produce TSP. However, depending on the source used to create struvite, ECSTsyn may contain more N and P than TSP, resulting in ECSTsyn-fertilized crops producing a greater yield than TSP-fertilized crops, and therefore ECSTsyn had the greatest value in 2019 (Omidire et al., 2023). However, as more research is conducted on both the potential crop growth efficiency and large-scale production costs, there will be opportunities to lower the cost of ECST production. The opportunity to lower ECST production costs, coupled with the growing need for sustainable fertilizer-P sources that do not rely on mined rock phosphate to produce, makes ECST a potentially environmentally and economically viable alternative fertilizer-P source.

Despite having numerous greater initial nutrient concentrations (Table 2), corn and soybean responses to struvite-removed wastewater as part of the irrigation water were often lower than for tap water. Based on results of this study, and only using struvite-removed wastewater as an irrigation water source approximately one out of every fourth irrigation, it appears that the potential to use struvite-removed wastewater as an irrigation-water and a nutrient source is not warranted. Further research will need to be conducted to ascertain the cause of reduced corn and soybean responses using struvite-removed wastewater as an irrigation-water and nutrient source.

Results of this study showed that, as a viable, alternative fertilizer-P source, struvite, particularly ECSTreal, recorded many similar corn and soybean responses as other traditional fertilizer-P sources, namely MAP and TSP. Results of the current study also show that, although ECSTsyn resulted in the numerically greater crop response, the response to ECSTreal was still at least similar to or greater than the response to CPST and MAP. More research with ECSTreal in field studies and with crop yield will provide a better understanding of the viability of ECSTreal as a replacement for commercially available fertilizer-P sources. Struvite can also lower excess nutrient loads into the environment by recovering P and N from human and animal waste streams. Reduced nutrient input to the environment could also decrease eutrophication in waterways. Because corn and soybean were grown for only 60 days, the nutrient tissue concentration and uptake may have been different had a full-season greenhouse or field study been conducted, where crops were allowed to fully mature. Therefore, more economic and practical field research is needed before struvite can be considered for widespread use as a replacement for traditional fertilizer-P sources, such as TSP, MAP, and DAP. However, having a potential, alternative, renewable fertilizer-P source such as struvite would be beneficial for both agricultural production and the environment.

## **Conclusions**

This greenhouse potted-plant study evaluated the combined effects of two silt-loam soils, two water types, and five fertilizer-P sources on corn and soybean properties over a 60-day period. Along with below- and aboveground dry matter, many below- and aboveground N, P, and Mg concentrations and uptakes responded similarly across the various soil-water-fertilizer-P source combinations. The hypothesis that the struvite materials (i.e., CPST, ECSTreal, and

ECSTsyn) would have the greater corn and soybean response in the lower-pH soil (i.e., the Creldon soil) was partially supported, as only a sub-set of measured plant properties were larger in the Creldon than in the Calloway soil. Struvite-removed wastewater was initially studied to investigate its potential as a nutrient-supplying, irrigation-water source, but after watering corn and soybean with the wastewater only a few times a week throughout the 60-day period, wastewater often negatively affected corn and soybean properties, where tap water resulted in larger corn and soybean N, P, and Mg concentrations and uptakes, despite the wastewater having numerous greater initial nutrient concentrations than tap water. Therefore, the hypothesis that plant properties would remain unaffected by water source was not supported. Most significantly, the hypothesis that corn and soybean properties would be greater in the struvite-P sources than in MAP was also only partially supported. In almost every fertilizer-P source treatment interaction, struvite-P sources behaved similarly to MAP, the traditional, commercially available fertilizer-P source. More specifically, ECSTsyn consistently had greater corn and soybean N, P, and Mg concentrations and uptakes than any other fertilizer-P source, likely due to ECST's large initial P and Mg concentration and crystalline application form providing greater surface area for reactions than pellets.

Since struvite is a relatively new, potential alternative fertilizer-P source, struvite's full potential as a replacement for traditional, commercially available fertilizer-P sources has been under-studied, especially crop nutrient concentration and uptake response in the belowground plant matter. In this study, the similar corn and soybean responses to struvite compared to MAP suggest that struvite has similar crop response to at least one commercially available, fertilizer-P source. However, more long-term soil and field studies are needed to fully understand the

agronomic and environmental implications of using struvite as an alternative fertilizer-P source to commonly used, commercially available fertilizer-P sources.

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**Table 1.** Summary of soil physical and chemical property differences between the Calloway and Creldon soil series used in the greenhouse potted-plant study.

| Soil Property   | <i>P</i> | Calloway            | Creldon |
|---|----------|---------------------|---------|
| Sand (g g <sup>-1</sup> )                                   | < 0.01   | 0.09 b <sup>†</sup> | 0.24 a  |
| Silt (g g <sup>-1</sup> )                                   | < 0.01   | 0.79 a              | 0.67 b  |
| Clay (g g <sup>-1</sup> )                                   | < 0.01   | 0.12 a              | 0.09 b  |
| pH  | < 0.01   | 7.46 a              | 6.03 b  |
| Electrical conductivity (dS m <sup>-1</sup> )               | < 0.01   | 0.17 a              | 0.13 b  |
| Total C (%)   | < 0.01   | 1.14 b              | 1.65 a  |
| Total N (%)   | < 0.01   | 0.11 b              | 0.1 a   |
| C:N ratio   | < 0.01   | 10.0 b              | 11.5 a  |
| Soil organic matter (%)                                     | < 0.01   | 2.6 b               | 3.4 a   |
| NO <sub>3</sub> -N (mg kg <sup>-1</sup> )                   | < 0.01   | 10.6 b              | 15.3 a  |
| NH <sub>4</sub> -N (mg kg <sup>-1</sup> )                   | < 0.01   | 5.3 b               | 19.5 a  |
| Water-soluble concentrations (mg kg <sup>-1</sup> )         |          |                     |         |
| P   | < 0.01   | 1.8 b               | 3.5 a   |
| K   | < 0.01   | 6.2 b               | 20.3 a  |
| Ca  | < 0.01   | 58.8 a              | 43.7 b  |
| Mg  | < 0.01   | 8.9 b               | 16.3 a  |
| S   | < 0.01   | 12.0 a              | 9.5 b   |
| Na  | < 0.01   | 19.5 a              | 5.9 b   |
| Fe  | < 0.01   | 1.5 b               | 12.6 a  |
| Mn  | < 0.01   | 0.12 b              | 1.7 a   |
| Zn  | 0.52     | 0.52 a              | 0.47 a  |
| Cu  | 0.09     | 0.03 a              | 0.04 a  |
| Mehlich-3-extractable concentrations (mg kg <sup>-1</sup> ) |          |                     |         |
| P   | < 0.01   | 11.4 b              | 17.0 a  |
| K   | < 0.01   | 46.1 b              | 113.0 a |
| Ca  | < 0.01   | 2006 a              | 1115 b  |
| Mg  | < 0.01   | 276.3 b             | 328.0 a |
| S   | 0.15     | 12.0 a              | 13.0 a  |
| Na  | < 0.01   | 29.8 a              | 10.4 b  |
| Fe  | < 0.01   | 303.8 a             | 112.0 b |
| Mn  | < 0.01   | 244.3 a             | 101.3 b |
| Zn  | < 0.01   | 2.6 b               | 4.2 a   |
| Cu  | < 0.01   | 1.6 a               | 1.2 b   |
| Total-recoverable concentrations (mg kg <sup>-1</sup> )     |          |                     |         |
| P   | < 0.01   | 342.9 a             | 248.0 b |
| K   | < 0.01   | 597 b               | 1160 a  |
| Ca  | < 0.01   | 2424 a              | 1494 b  |
| Mg  | < 0.01   | 1318 b              | 1372 a  |
| S   | < 0.01   | 137.2 b             | 154.0 a |
| Na  | < 0.01   | 61.6 a              | 25.7 b  |
| Fe  | 0.12     | 16705 a             | 18023 a |
| Mn  | 0.05     | 1337 a              | 1163 a  |
| Zn  | 0.36     | 32.9 a              | 31.3 a  |
| Cu  | < 0.01   | 6.3 a               | 4.7 b   |

<sup>†</sup>Means in a row with different letters are different at *P* < 0.05

**Table 2.** Summary of the chemical property differences between the tap water and struvite-removed wastewater sources used in the greenhouse potted-plant study.

| <b>Water Property</b>     | <b><i>P</i></b> | <b>Tap water</b>      | <b>Wastewater</b> |
|---------------------------|-----------------|-----------------------|-------------------|
| P (mg kg <sup>-1</sup> )  | < 0.01          | < 0.01 b <sup>†</sup> | 10.6 a            |
| K (mg kg <sup>-1</sup> )  | < 0.01          | 1.25 b                | 23.3 a            |
| Ca (mg kg <sup>-1</sup> ) | 0.51            | 24.4 a                | 28.2 a            |
| Mg (mg kg <sup>-1</sup> ) | < 0.01          | 1.86 b                | 45.6 a            |
| S (mg kg <sup>-1</sup> )  | < 0.01          | 7.52 b                | 13.2 a            |
| Na (mg kg <sup>-1</sup> ) | < 0.01          | 5.85 b                | 39.6 a            |
| Fe (mg kg <sup>-1</sup> ) | < 0.01          | 0.04 b                | 0.28 a            |
| Mn (mg kg <sup>-1</sup> ) | < 0.01          | < 0.01 b              | 0.16 a            |
| Zn (mg kg <sup>-1</sup> ) | < 0.01          | < 0.01 b              | 0.02 a            |
| Cu (mg kg <sup>-1</sup> ) | < 0.01          | < 0.01 b              | 0.03 a            |

<sup>†</sup>Means in a row with different letters are different at  $P < 0.05$

**Table 3.** Analysis of variance summary of the effects of water type (W), soil (S), fertilizer-phosphorus source (F), and their interactions on corn properties for the greenhouse potted-plant study.

| Source of variation | BG <sup>†</sup><br>dry<br>matter | AG <sup>†</sup><br>dry<br>matter | Total<br>dry<br>matter | BG<br>concentration |                |                 | AG concentration |       |       | BG uptake |       |       | AG uptake |       |       | Total uptake |       |       |
|---------------------|----------------------------------|----------------------------------|------------------------|---------------------|----------------|-----------------|------------------|-------|-------|-----------|-------|-------|-----------|-------|-------|--------------|-------|-------|
|                     |                                  |                                  |                        | N <sup>†</sup>      | P <sup>†</sup> | Mg <sup>†</sup> | N                | P     | Mg    | N         | P     | Mg    | N         | P     | Mg    | N            | P     | Mg    |
| Water (W)           | 0.21                             | 0.05                             | 0.04                   | 0.39                | 0.18           | 0.17            | 0.06             | 0.37  | 0.83  | 0.28      | 0.95  | 0.92  | 0.98      | 0.33  | 0.03  | 0.71         | 0.71  | 0.05  |
| Soil (S)            | 0.68                             | <0.01                            | <0.01                  | 0.16                | 0.47           | 0.90            | <0.01            | 0.44  | <0.01 | 0.01      | 0.95  | 0.65  | 0.16      | <0.01 | <0.01 | 0.04         | 0.01  | <0.01 |
| Fertilizer (F)      | <0.01                            | <0.01                            | <0.01                  | 0.01                | <0.01          | <0.01           | <0.01            | <0.01 | <0.01 | <0.01     | <0.01 | <0.01 | <0.01     | <0.01 | <0.01 | <0.01        | <0.01 | <0.01 |
| W x S               | 0.07                             | 0.61                             | 0.44                   | 0.24                | 0.09           | 0.43            | 0.76             | 0.38  | 0.71  | 0.38      | 0.74  | 0.35  | 0.23      | 0.38  | 0.85  | 0.43         | 0.79  | 0.98  |
| W x F               | 0.96                             | 0.95                             | 0.96                   | 0.57                | 0.04           | 0.74            | 0.57             | 0.46  | 0.02  | 0.06      | 0.54  | 0.93  | 0.25      | 0.07  | 0.03  | 0.21         | 0.06  | 0.06  |
| S x F               | 0.26                             | <0.01                            | <0.01                  | 0.52                | <0.01          | <0.01           | 0.01             | <0.01 | 0.85  | 0.01      | <0.01 | 0.02  | 0.04      | <0.01 | <0.01 | 0.01         | <0.01 | <0.01 |
| W x S x F           | 0.44                             | 0.04                             | 0.19                   | 0.22                | 0.09           | 0.06            | 0.10             | 0.68  | 0.85  | 0.19      | 0.01  | 0.03  | 0.20      | 0.17  | 0.03  | 0.31         | 0.16  | 0.04  |

<sup>†</sup>Belowground, BG; aboveground, AG; nitrogen, N; phosphorus, P; magnesium, Mg

**Table 4.** Corn and soybean properties among fertilizer-phosphorus (P) sources for the 60-day greenhouse potted-plant study.

| Fertilizer-P<br>Source | Corn                                   |                                       | Soybean                                |
|------------------------|--|---------------------------------------|--|
|                        | DM <sub>BG</sub> <sup>†,‡</sup><br>(g) | N <sub>BG</sub> <sup>†,‡</sup><br>(%) | Mg <sub>BG</sub> <sup>†,‡</sup><br>(%) |
| CPST                   | 12.9 a                                 | 0.99 a                                | 0.55 a                                 |
| MAP                    | 13.9 a                                 | 0.78 c                                | 0.45 b                                 |
| ECSTreal               | 13.3 a                                 | 0.86 abc                              | 0.53 a                                 |
| ECSTsyn                | 12.2 ab                                | 0.85 bc                               | 0.57 a                                 |
| UC                     | 10.5 b                                 | 0.98 ab                               | 0.41 b                                 |

<sup>†</sup> Means in a column with different letters are different at  $P < 0.05$

<sup>‡</sup>Chemically precipitated struvite, CPST; monoammonium phosphate, MAP; real-wastewater-derived electrochemically precipitated struvite, ECSTreal; synthetic electrochemically precipitated struvite, ECSTsyn; dry matter, DM; nitrogen, N; magnesium, Mg; belowground, BG



**Table 5.** Corn properties among water-soil-fertilizer-phosphorus-(P)-source treatment combinations for the 60-day greenhouse potted-plant study.

| Water Type | Soil Series | Fertilizer-P Source | P <sub>BG</sub> uptake <sup>†,‡</sup><br>(mg cm <sup>-2</sup> ) | Mg <sub>BG</sub> uptake <sup>†,‡</sup><br>(mg cm <sup>-2</sup> ) | DM <sub>AG</sub> <sup>†,‡</sup><br>(g) | Mg <sub>AG</sub> uptake <sup>†,‡</sup><br>(mg cm <sup>-2</sup> ) | Mg <sub>tot</sub> uptake <sup>†,‡</sup><br>(mg cm <sup>-2</sup> ) |
|------------|-------------|---------------------|---|--|--|--|---|
| Tap water  | Creldon     | CPST                | 0.28 a  | 0.32 a   | 116.0 i                                | 0.88 fg  | 1.20 def  |
|            |             | MAP                 | 0.06 gh   | 0.13 hi  | 131.0 a-f                              | 0.98 efg   | 1.11 f  |
|            |             | ECSTreal            | 0.08 fg   | 0.16 d-h   | 124.3 b-g                              | 1.11 de  | 1.27 de   |
|            |             | ECSTsyn             | 0.15 bc   | 0.19 c-f   | 127.0 ab                               | 1.49 ab  | 1.68 ab   |
|            |             | UC                  | 0.03 i  | 0.12 i   | 91.9 k                                 | 0.53 i   | 0.65 h  |
|            | Calloway    | CPST                | 0.13 cd   | 0.18 c-h   | 113.7 f-i                              | 1.06 de  | 1.24 def  |
|            |             | MAP                 | 0.09 ef   | 0.15 e-i   | 133.6 a-d                              | 1.15 d   | 1.30 de   |
|            |             | ECSTreal            | 0.12 cde  | 0.24 abc   | 112.6 e-i                              | 1.03 def   | 1.26 def  |
|            |             | ECSTsyn             | 0.10 def  | 0.18 c-h   | 127.6 abc                              | 1.59 a   | 1.77 a  |
|            |             | UC                  | 0.06 h  | 0.15 e-i   | 121.3 e-i                              | 1.02 d-g   | 1.17 def  |
| Wastewater | Creldon     | CPST                | 0.21 ab   | 0.27 ab  | 119.6 hi                               | 0.88 g   | 1.14 ef   |
|            |             | MAP                 | 0.08 fg   | 0.15 f-i   | 124.0 a-e                              | 1.07 de  | 1.22 def  |
|            |             | ECSTreal            | 0.09 def  | 0.21 b-e   | 110.3 d-h                              | 1.12 de  | 1.33 d  |
|            |             | ECSTsyn             | 0.12 cde  | 0.18 c-g   | 129.5 ab                               | 1.34 bc  | 1.53 b  |
|            |             | UC                  | 0.04 i  | 0.13 hi  | 95.5 j                                 | 0.68 h   | 0.81 g  |
|            | Calloway    | CPST                | 0.15 bc   | 0.22 bcd   | 120.2 c-h                              | 1.11 de  | 1.33 cd   |
|            |             | MAP                 | 0.08 fg   | 0.13 ghi   | 132.9 ab                               | 1.18 cd  | 1.31 de   |
|            |             | ECSTreal            | 0.08 fg   | 0.16 d-i   | 132.1 abc                              | 1.36 abc   | 1.52 bc   |
|            |             | ECSTsyn             | 0.12 cde  | 0.22 bcd   | 128.3 a                                | 1.48 ab  | 1.70 ab   |
|            |             | UC                  | 0.06 h  | 0.14 ghi   | 107.3 ghi                              | 1.03 def   | 1.16 def  |

<sup>†</sup> Means in a column with different letters are different at  $P < 0.05$

<sup>‡</sup>Chemically precipitated struvite, CPST; monoammonium phosphate, MAP; real-wastewater-derived electrochemically precipitated struvite, ECSTreal; synthetic electrochemically precipitated struvite, ECSTsyn; belowground ground, BG; above ground, AG; phosphorus, P; magnesium, Mg; dry matter, DM

**Table 6.** Analysis of variance summary of the effects of water type (W), soil (S), fertilizer-phosphorus source (F), and their interactions on soybean properties for the greenhouse potted-plant study.

| Source of Variation | BG <sup>†</sup><br>dry<br>matter | AG <sup>†</sup><br>dry<br>matter | Total<br>dry<br>matter | BG<br>concentration |                |                 | AG<br>concentration |       |       | BG uptake |       |       | AG uptake |       |       | Total uptake |       |       |
|---------------------|----------------------------------|----------------------------------|------------------------|---------------------|----------------|-----------------|---------------------|-------|-------|-----------|-------|-------|-----------|-------|-------|--------------|-------|-------|
|                     |                                  |                                  |                        | N <sup>†</sup>      | P <sup>†</sup> | Mg <sup>†</sup> | N                   | P     | Mg    | N         | P     | Mg    | N         | P     | Mg    | N            | P     | Mg    |
| Water (W)           | 0.06                             | 0.01                             | <0.01                  | 0.74                | 0.05           | 0.21            | 0.67                | 0.29  | 0.76  | 0.06      | 0.13  | 0.65  | 0.07      | 0.20  | 0.02  | 0.04         | 0.90  | 0.09  |
| Soil (S)            | <0.01                            | <0.01                            | <0.01                  | <0.01               | 0.03           | 0.06            | <0.01               | <0.01 | <0.01 | <0.01     | <0.01 | <0.01 | <0.01     | 0.74  | 0.71  | <0.01        | 0.13  | 0.05  |
| Fertilizer (F)      | 0.10                             | <0.01                            | <0.01                  | <0.01               | <0.01          | <0.01           | <0.01               | <0.01 | <0.01 | 0.10      | <0.01 | 0.05  | <0.01     | <0.01 | <0.01 | <0.01        | <0.01 | <0.01 |
| W*S                 | 0.86                             | 0.16                             | 0.26                   | 0.46                | 0.07           | 0.17            | 0.48                | 0.01  | 0.14  | 0.86      | 0.36  | 0.17  | 0.97      | 0.38  | 0.75  | 0.86         | 0.14  | 0.73  |
| W*F                 | 0.13                             | 0.01                             | <0.01                  | 0.14                | 0.50           | 0.85            | 0.04                | 0.14  | 0.11  | 0.13      | 0.06  | 0.06  | <0.01     | 0.01  | 0.01  | <0.01        | 0.12  | 0.02  |
| S*F                 | <0.01                            | <0.01                            | <0.01                  | <0.01               | <0.01          | 0.20            | 0.03                | <0.01 | <0.01 | <0.01     | <0.01 | 0.02  | <0.01     | <0.01 | <0.01 | <0.01        | <0.01 | <0.01 |
| W*S*F               | 0.19                             | 0.39                             | 0.14                   | 0.03                | 0.88           | 0.43            | 0.65                | 0.60  | 0.16  | 0.19      | <0.01 | 0.14  | 0.45      | 0.20  | 0.08  | 0.05         | 0.52  | 0.02  |

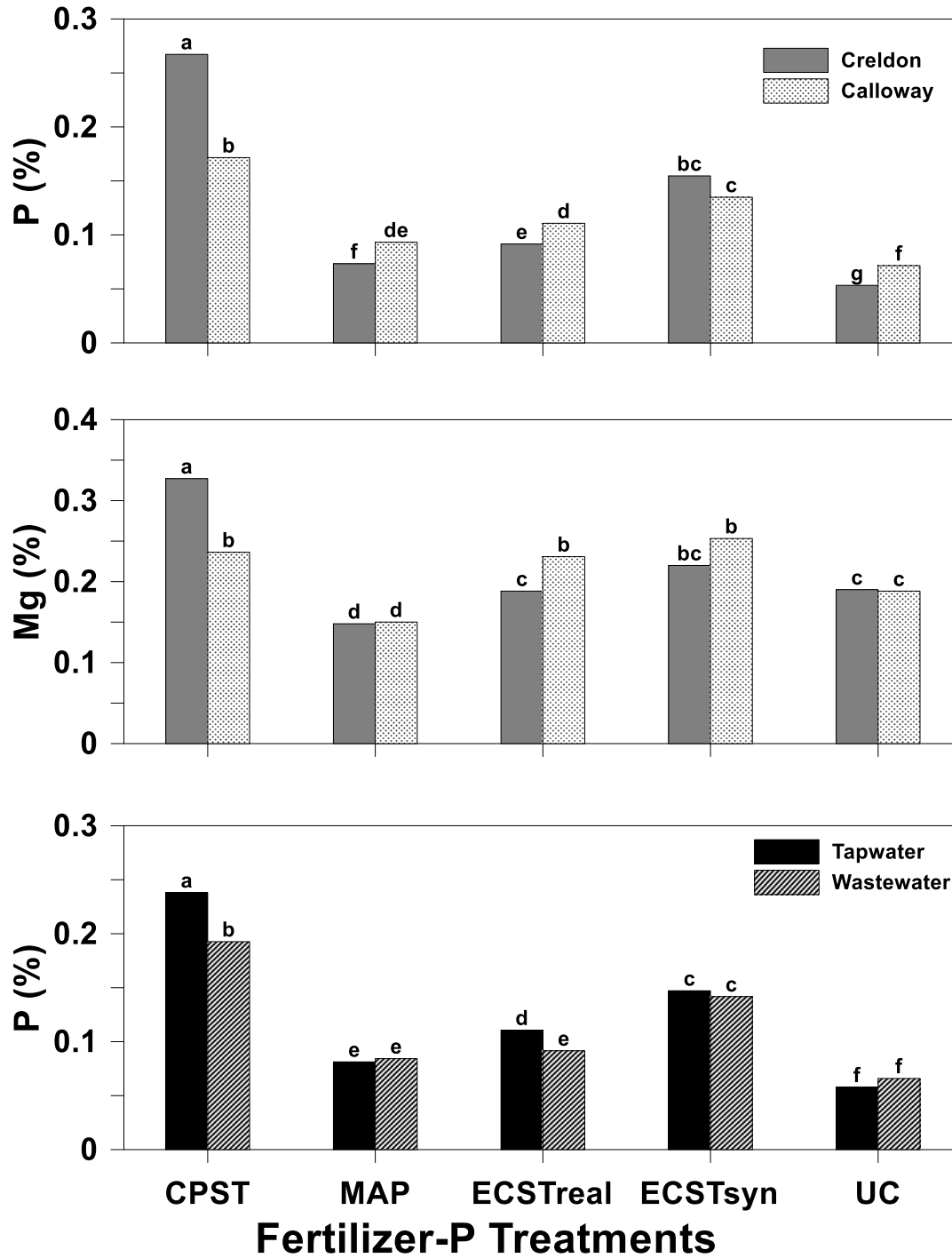
<sup>†</sup>Belowground, BG; aboveground, AG; nitrogen, N; phosphorus, P; magnesium, Mg

**Table 7.** Soybean properties among water-soil-fertilizer-phosphorus-(P)-source treatment combinations for the 60-day greenhouse potted-plant study.

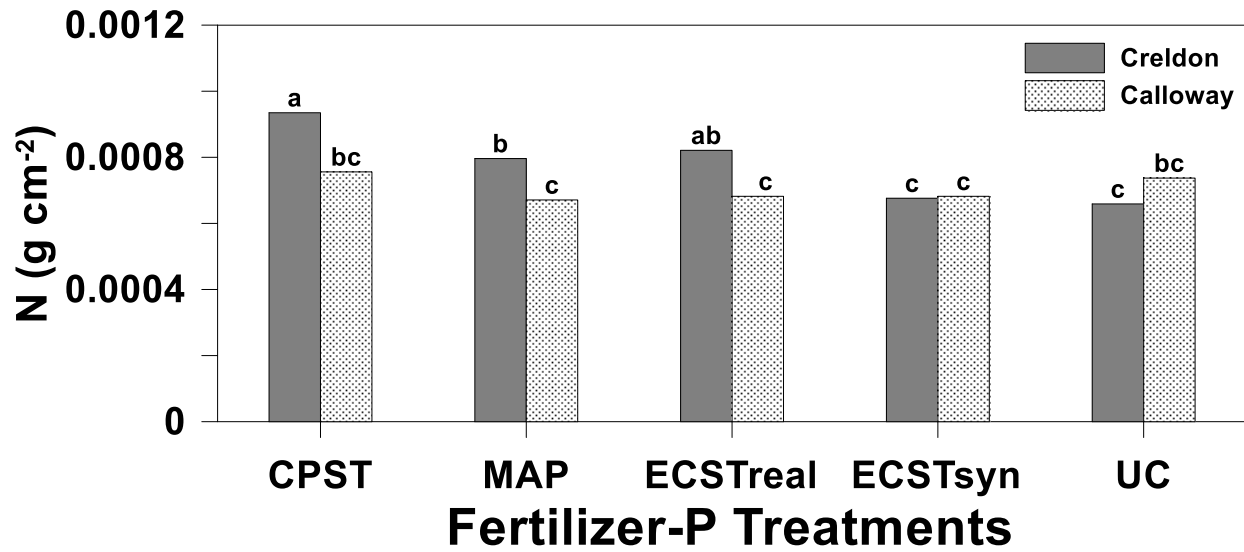
| Water Type | Soil Series | Fertilizer-P Source | N <sub>BG</sub> <sup>†,‡</sup> (%) | P <sub>BG</sub> uptake <sup>†,‡</sup> (mg cm <sup>-2</sup> ) | Mg <sub>tot</sub> uptake <sup>†,‡</sup> (mg cm <sup>-2</sup> ) |
|------------|-------------|---------------------|------------------------------------|--|--|
| Tap water  | Crelton     | CPST                | 1.07 e                             | 6.45 e   | 0.57 fgh   |
|            |             | MAP                 | 1.94 abc                           | 11.14 abc  | 0.72 bc  |
|            |             | ECSTreal            | 2.14 a                             | 10.43 abc  | 0.74 b   |
|            |             | ECSTsyn             | 2.40 a                             | 11.25 abc  | 0.95 a   |
|            |             | UC                  | 1.36 d                             | 5.97 e   | 0.39 i   |
|            | Calloway    | CPST                | 1.98 ab                            | 13.00 a  | 0.67 b-e   |
|            |             | MAP                 | 1.94 abc                           | 10.84 abc  | 0.65 b-f   |
|            |             | ECSTreal            | 2.00 ab                            | 10.55 abc  | 0.68 bcd   |
|            |             | ECSTsyn             | 2.40 a                             | 12.14 ab   | 0.90 a   |
|            |             | UC                  | 1.49 d                             | 9.60 bc  | 0.51 h   |
| Wastewater | Crelton     | CPST                | 1.53 cd                            | 8.96 cd  | 0.64 c-f   |
|            |             | MAP                 | 1.41 d                             | 6.94 de  | 0.59 e-h   |
|            |             | ECSTreal            | 2.28 a                             | 9.38 bc  | 0.71 bc  |
|            |             | ECSTsyn             | 2.07 ab                            | 9.51 bc  | 0.91 a   |
|            |             | UC                  | 1.31 de                            | 5.49 e   | 0.37 i   |
|            | Calloway    | CPST                | 1.95 abc                           | 10.15 abc  | 0.56 fgh   |
|            |             | MAP                 | 2.20 a                             | 9.94 bc  | 0.55 gh  |
|            |             | ECSTreal            | 2.13 a                             | 11.26 abc  | 0.71 bcd   |
|            |             | ECSTsyn             | 2.25 a                             | 11.21abc   | 0.87 a   |
|            |             | UC                  | 1.65 bcd                           | 12.01ab  | 0.61 deg   |

<sup>†</sup> Means in a column with different letters are different at  $P < 0.05$

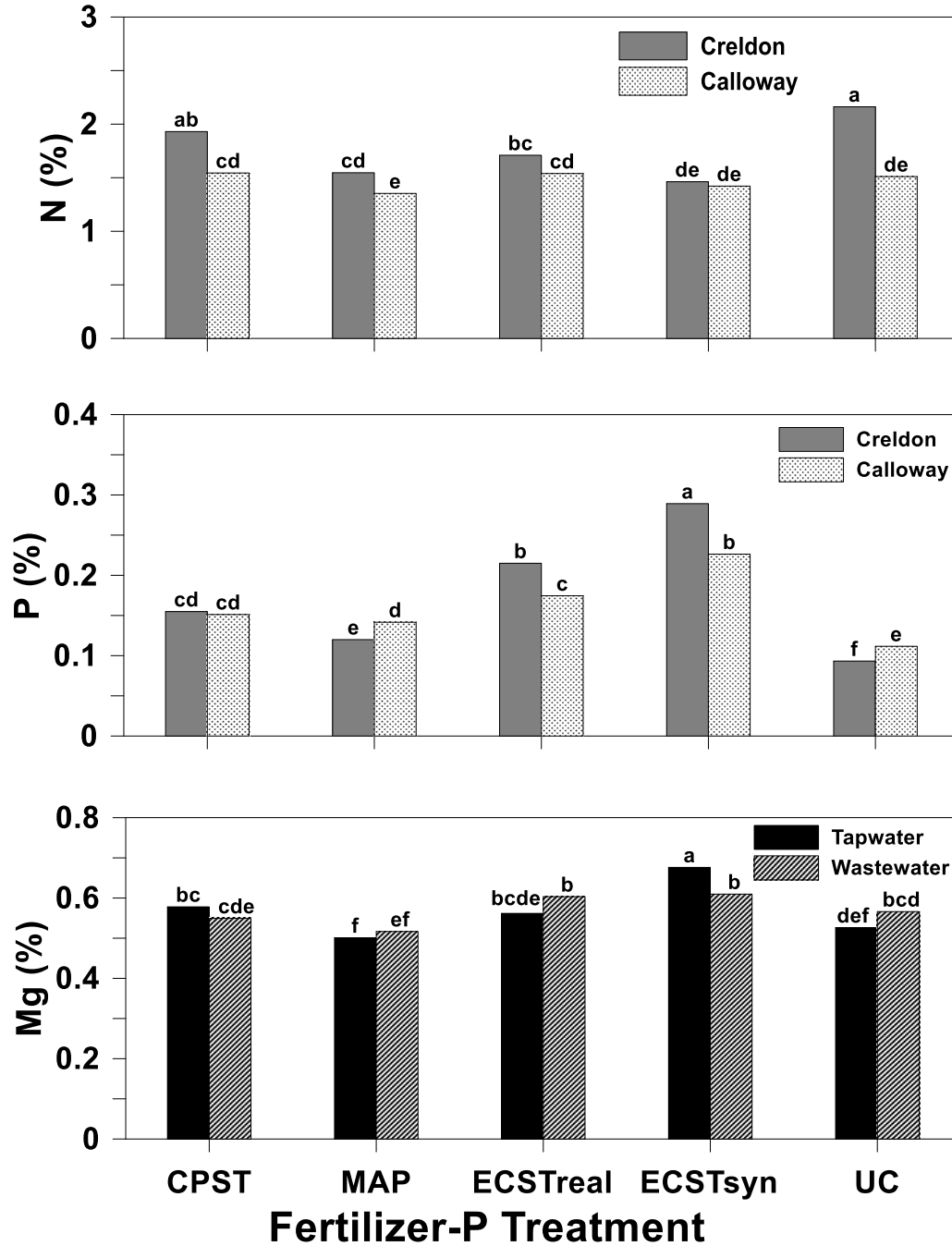
<sup>‡</sup>Chemically precipitated struvite, CPST; monoammonium phosphate, MAP; real-wastewater-derived electrochemically precipitated struvite, ECSTreal; synthetic electrochemically precipitated struvite, ECSTsyn; belowground ground, BG; nitrogen, N; phosphorus, P; magnesium, Mg



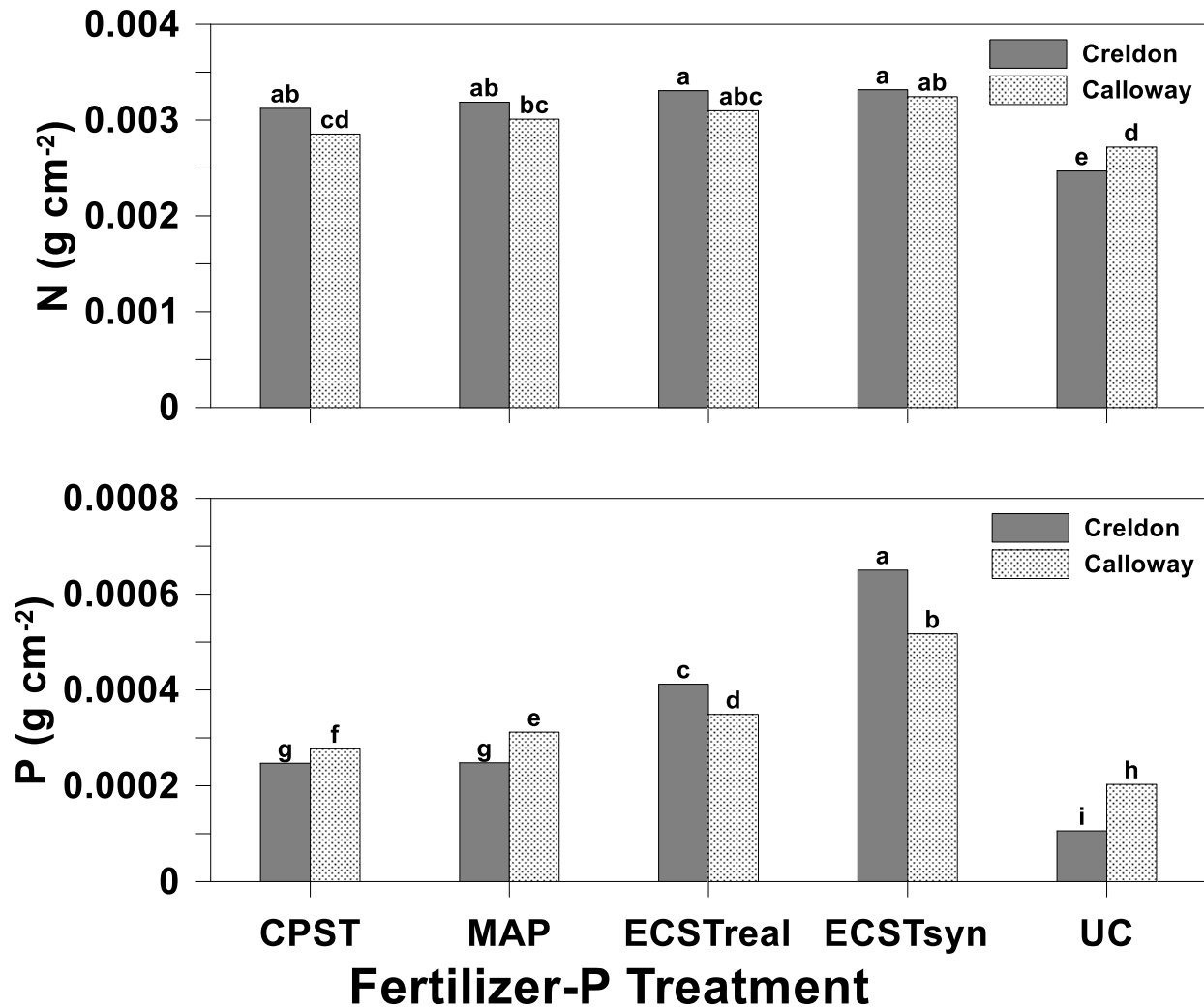
**Figure 1.** Belowground corn phosphorus (P) and magnesium (Mg) concentrations among soil-fertilizer-P-source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations and belowground corn P concentration between water type-fertilizer-P-source combinations for the 60-day greenhouse potted-plant study. Within a panel, bars with different lower-case letters are different at  $P < 0.05$ .



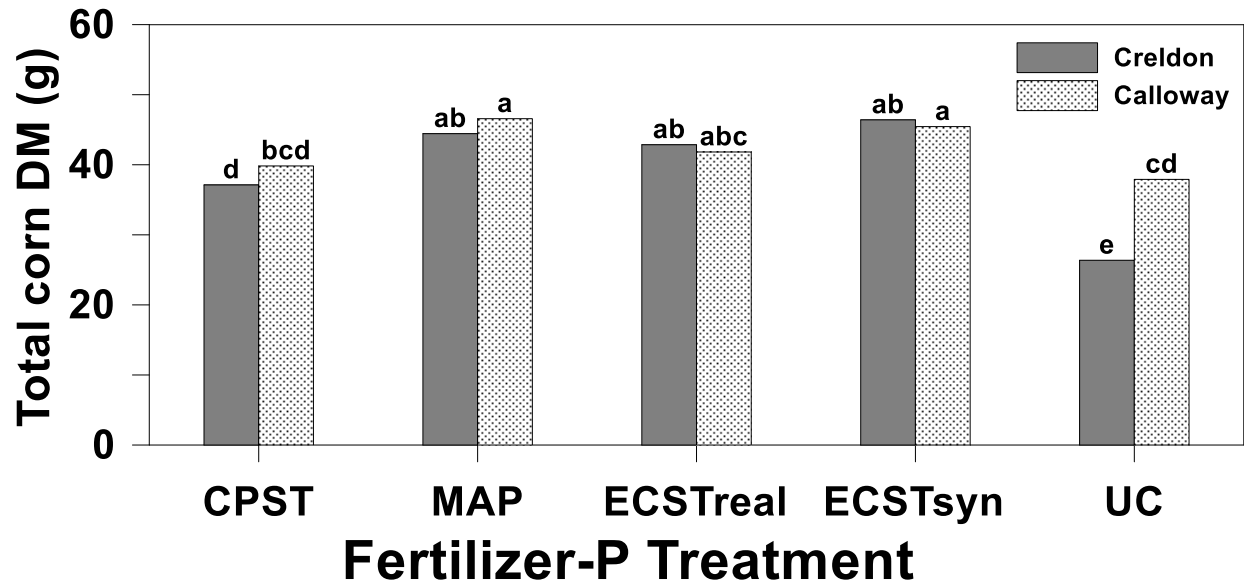
**Figure 2.** Belowground corn nitrogen (N) uptake among soil-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations for the 60-day greenhouse potted-plant study. Bars with different lower-case letters are different at  $P < 0.05$ .



**Figure 3.** Aboveground corn nitrogen (N) and phosphorus (P) concentrations among soil-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations and aboveground corn magnesium (Mg) concentration among water type-fertilizer-P source treatment combinations for the 60-day greenhouse potted-plant study. Within a panel, bars with different lower-case letters are different at  $P < 0.05$ .

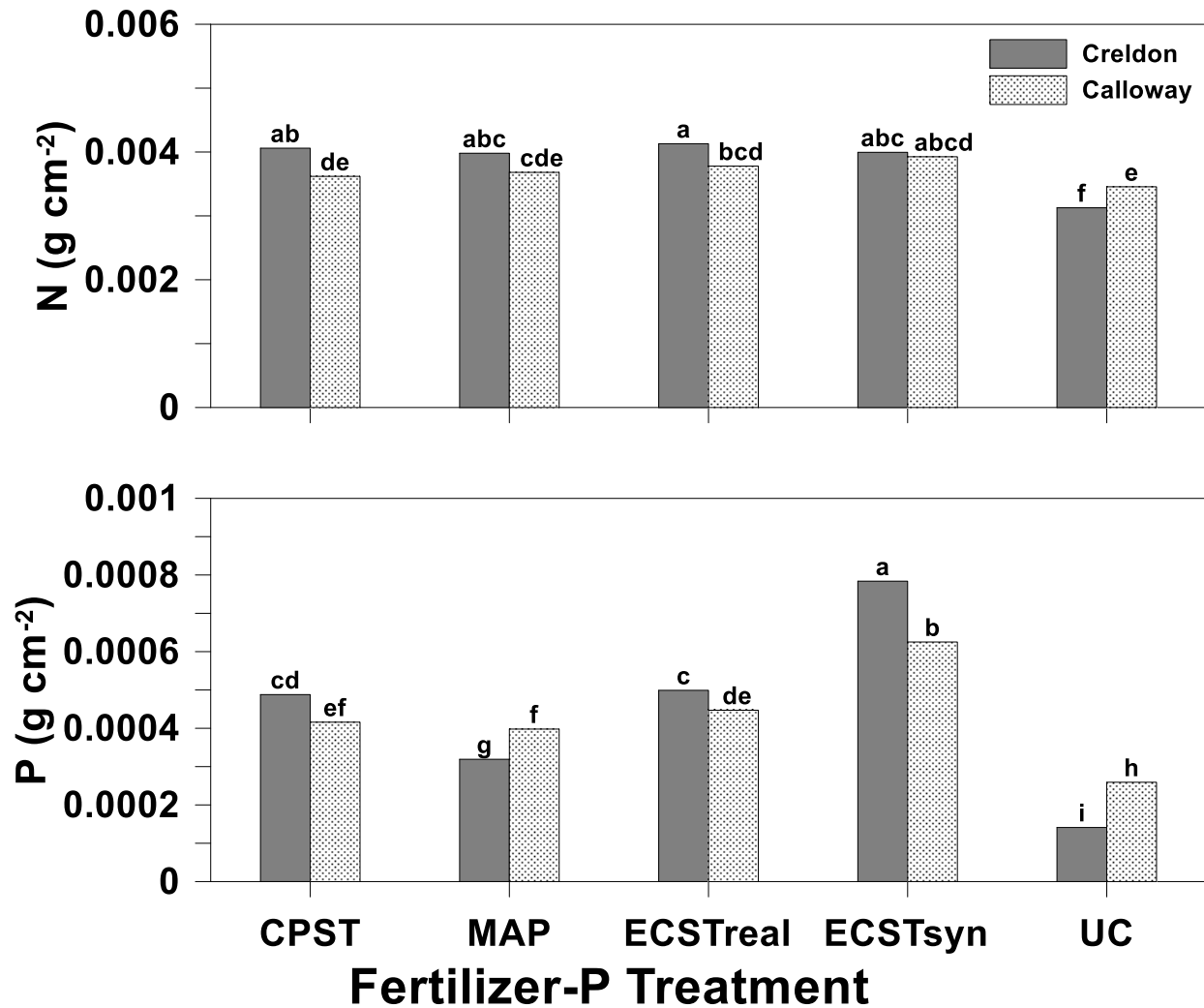


**Figure 4.** Aboveground corn nitrogen (N) and phosphorus (P) uptakes among soil-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations for the 60-day greenhouse potted-plant study. Within a panel, bars with different lower-case letters are different at  $P < 0.05$ .

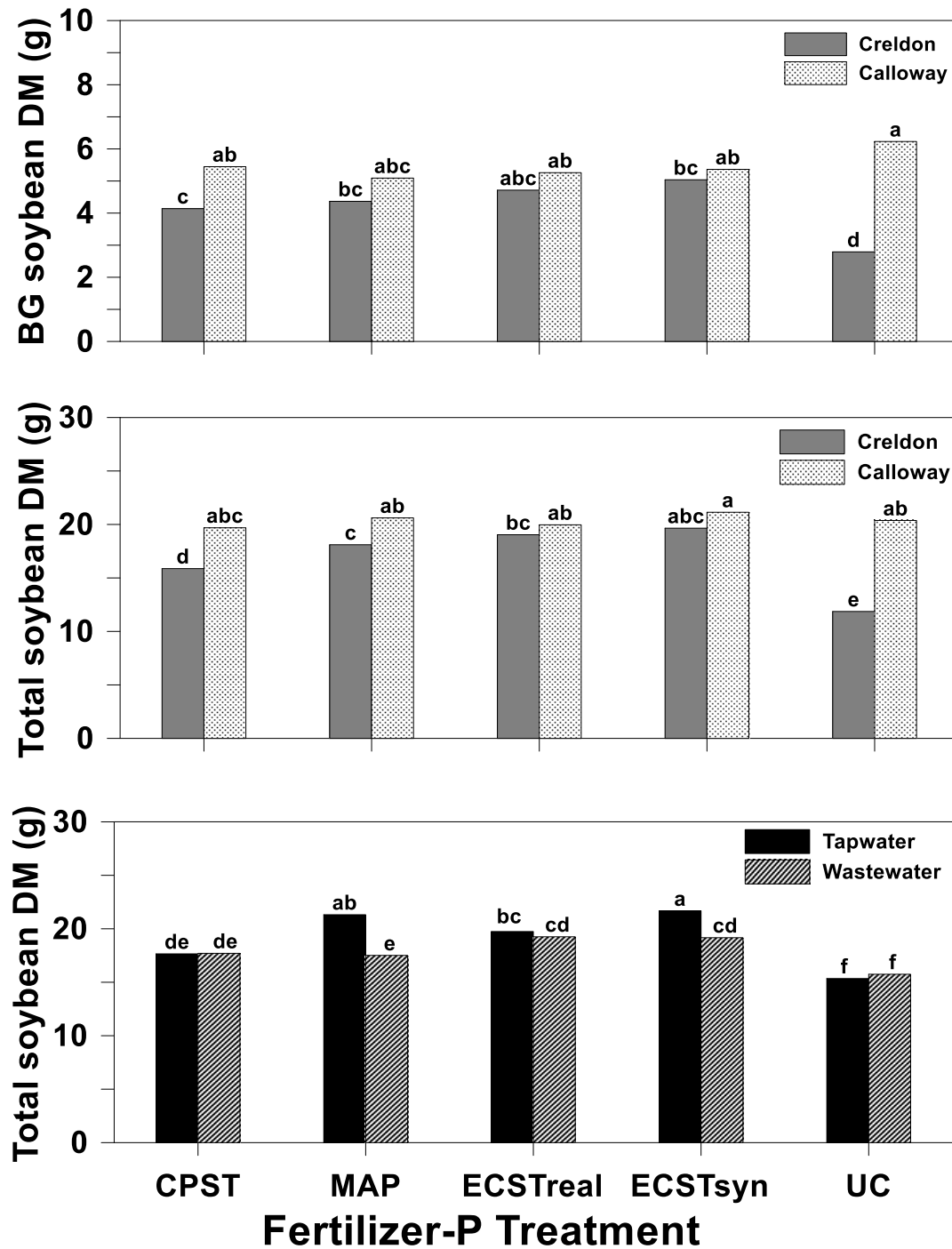


**Figure 5.** Total corn dry matter (DM) among soil-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations for the 60-day greenhouse potted-plant study. Bars with different lower-case letters are different at  $P < 0.05$ .

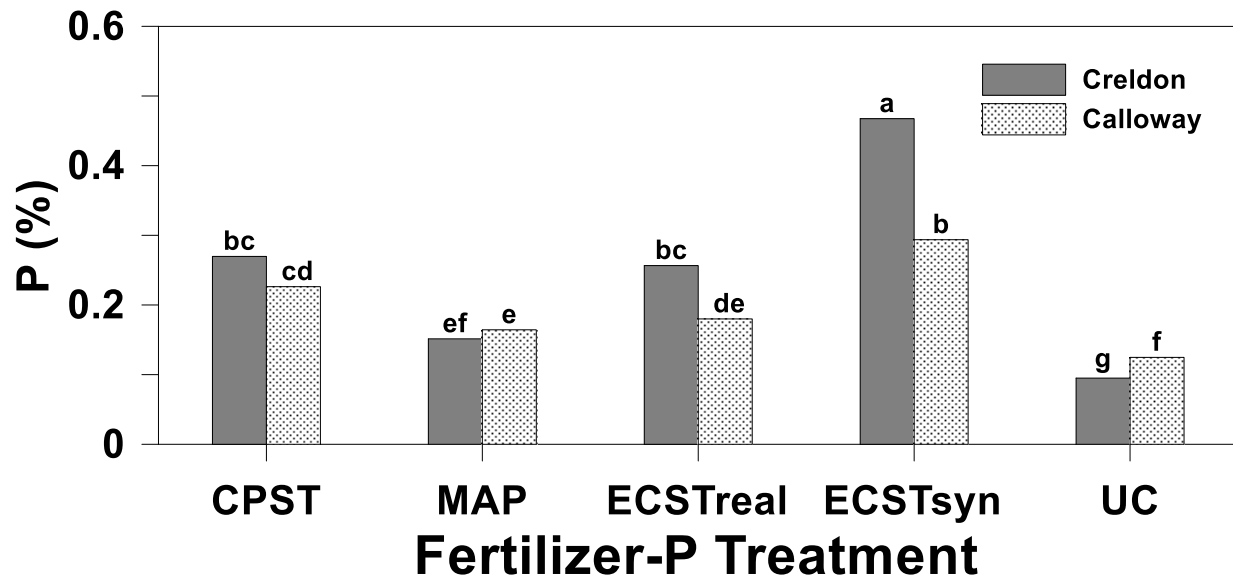




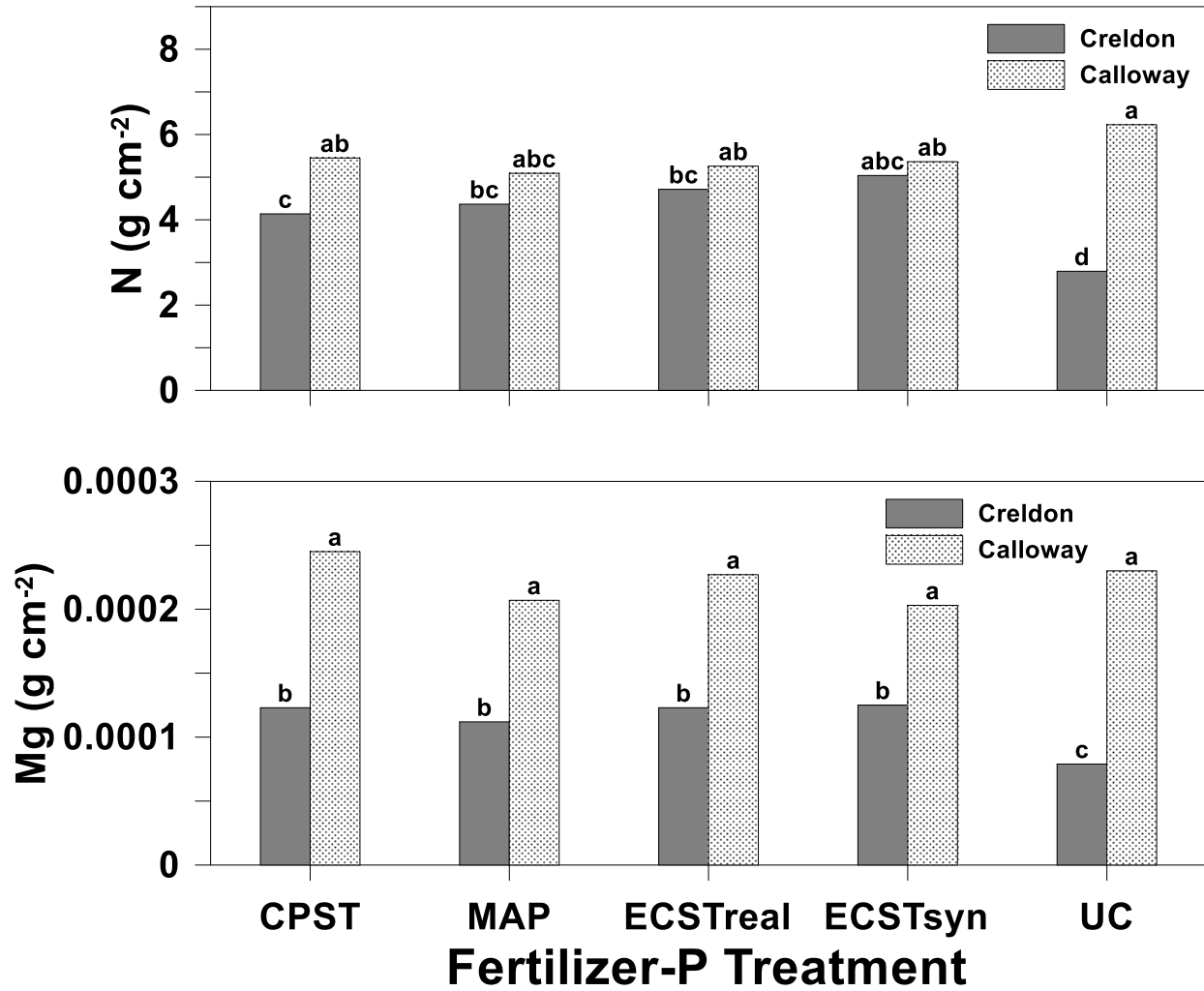
**Figure 6.** Total corn nitrogen (N) and phosphorus (P) uptakes among soil-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations for the 60-day greenhouse potted-plant study. Within a panel, bars with different lower-case letters are different at  $P < 0.05$ .



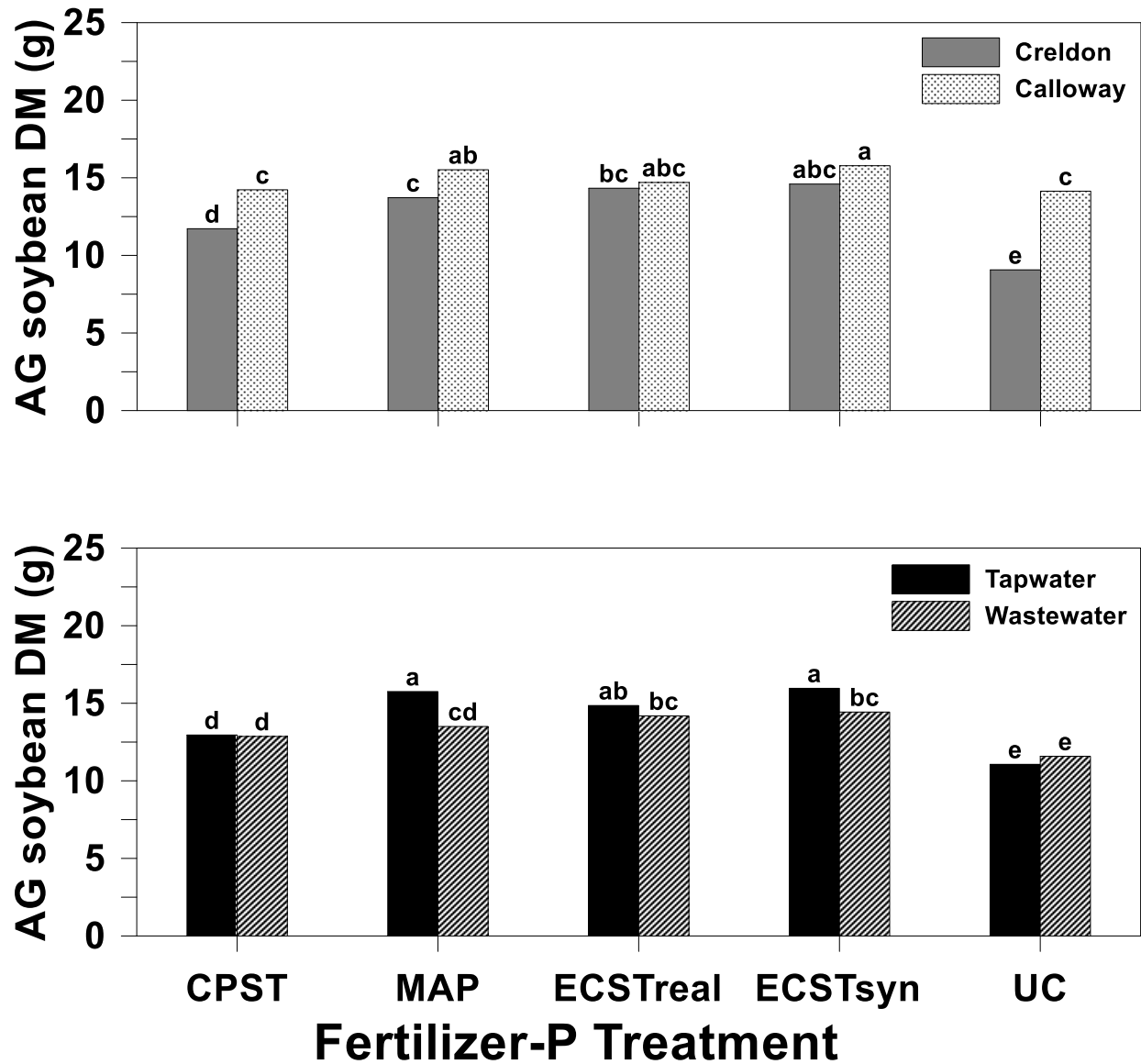
**Figure 7.** Belowground and total soybean dry matter (DM) among soil-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations and total soybean DM among water-fertilizer-P source combinations for the 60-day greenhouse potted-plant study. Within a panel, bars with different lower-case letters are different at  $P < 0.05$ .



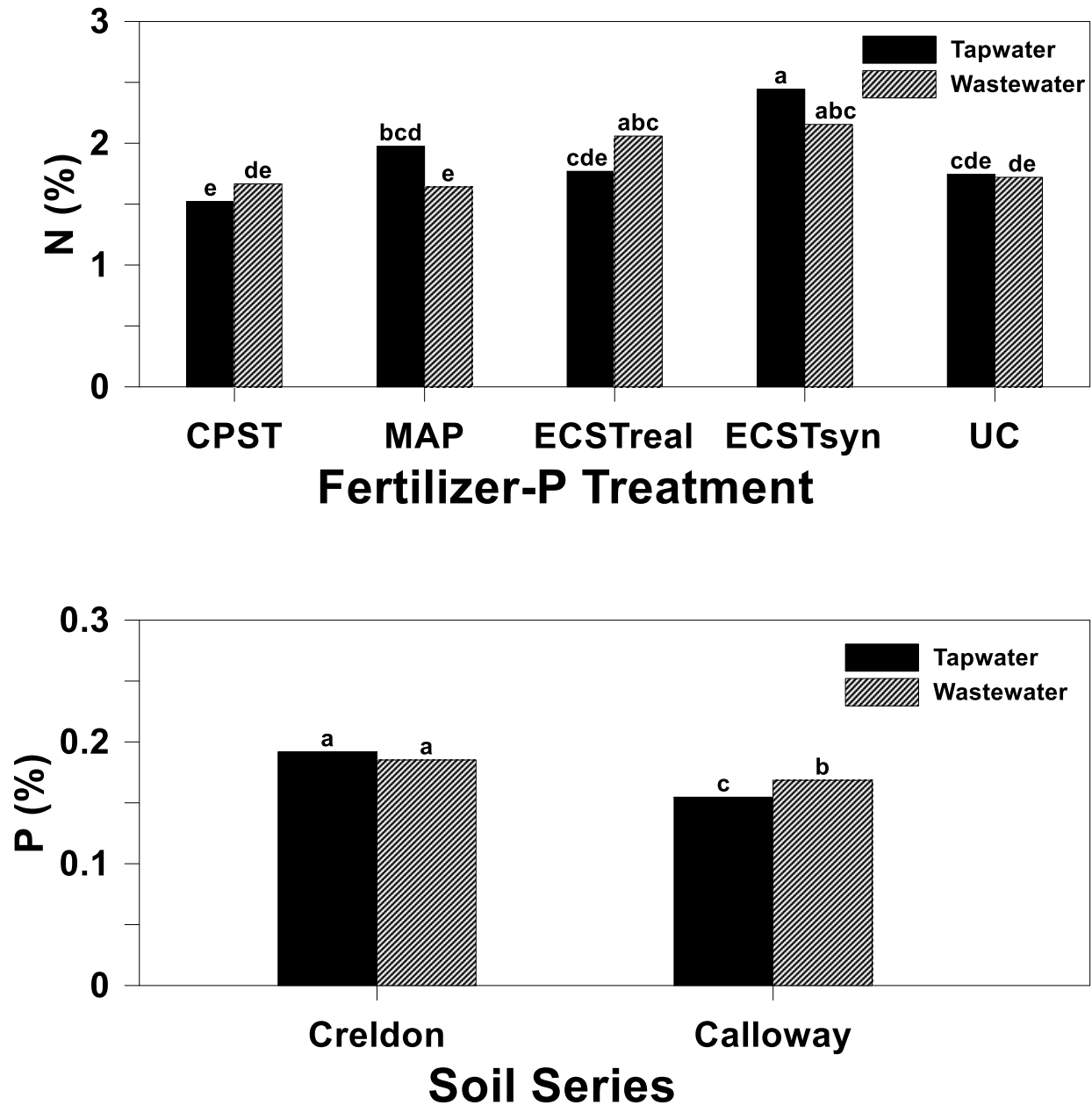
**Figure 8.** Belowground soybean phosphorus (P) concentration among soil-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations for the 60-day greenhouse potted-plant study. Bars with different lower-case letters are different at  $P < 0.05$ .



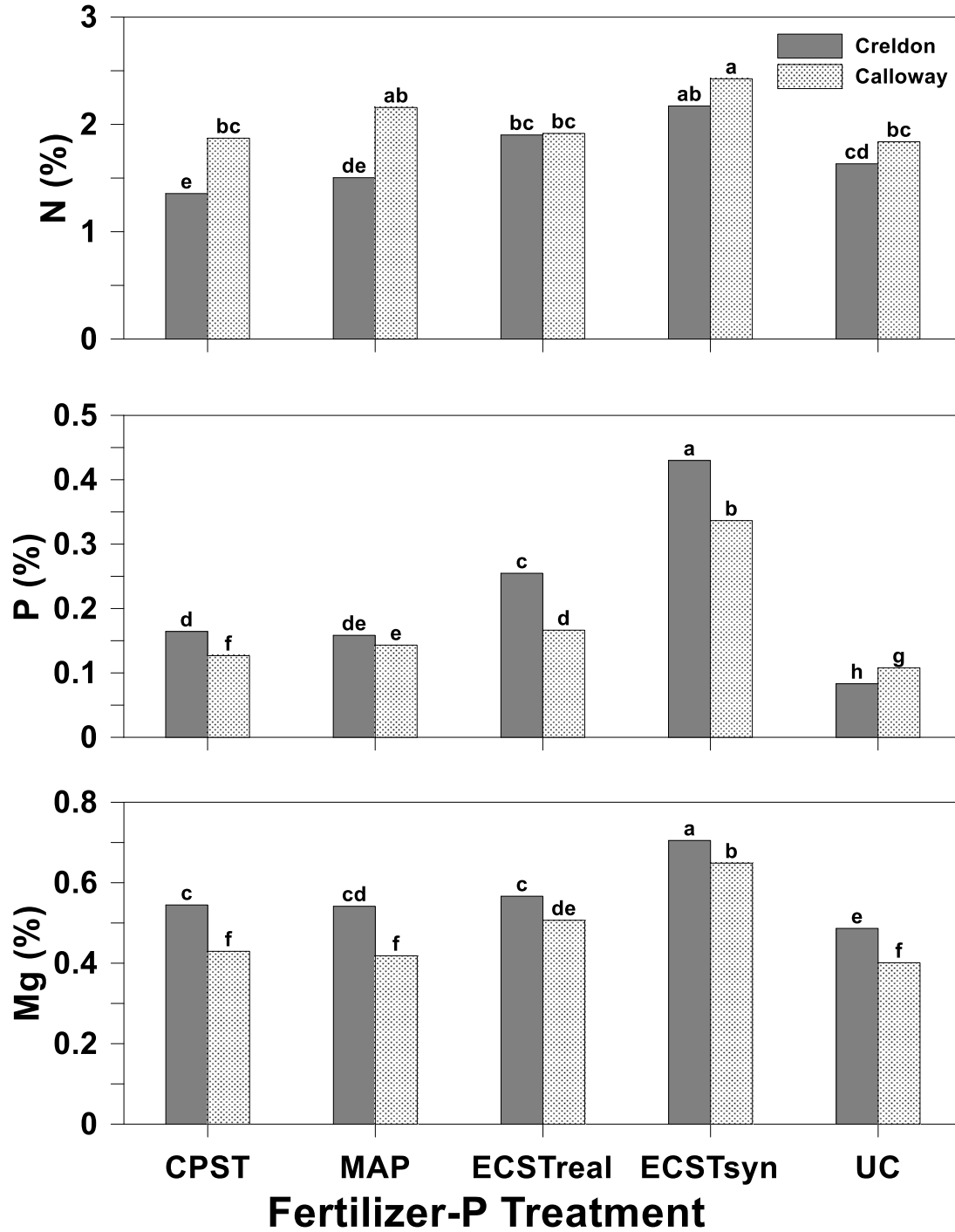
**Figure 9.** Belowground soybean nitrogen (N) and magnesium (Mg) uptakes among soil-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations for the 60-day greenhouse potted-plant study. Within a panel, bars with different lower-case letters are different at  $P < 0.05$ .



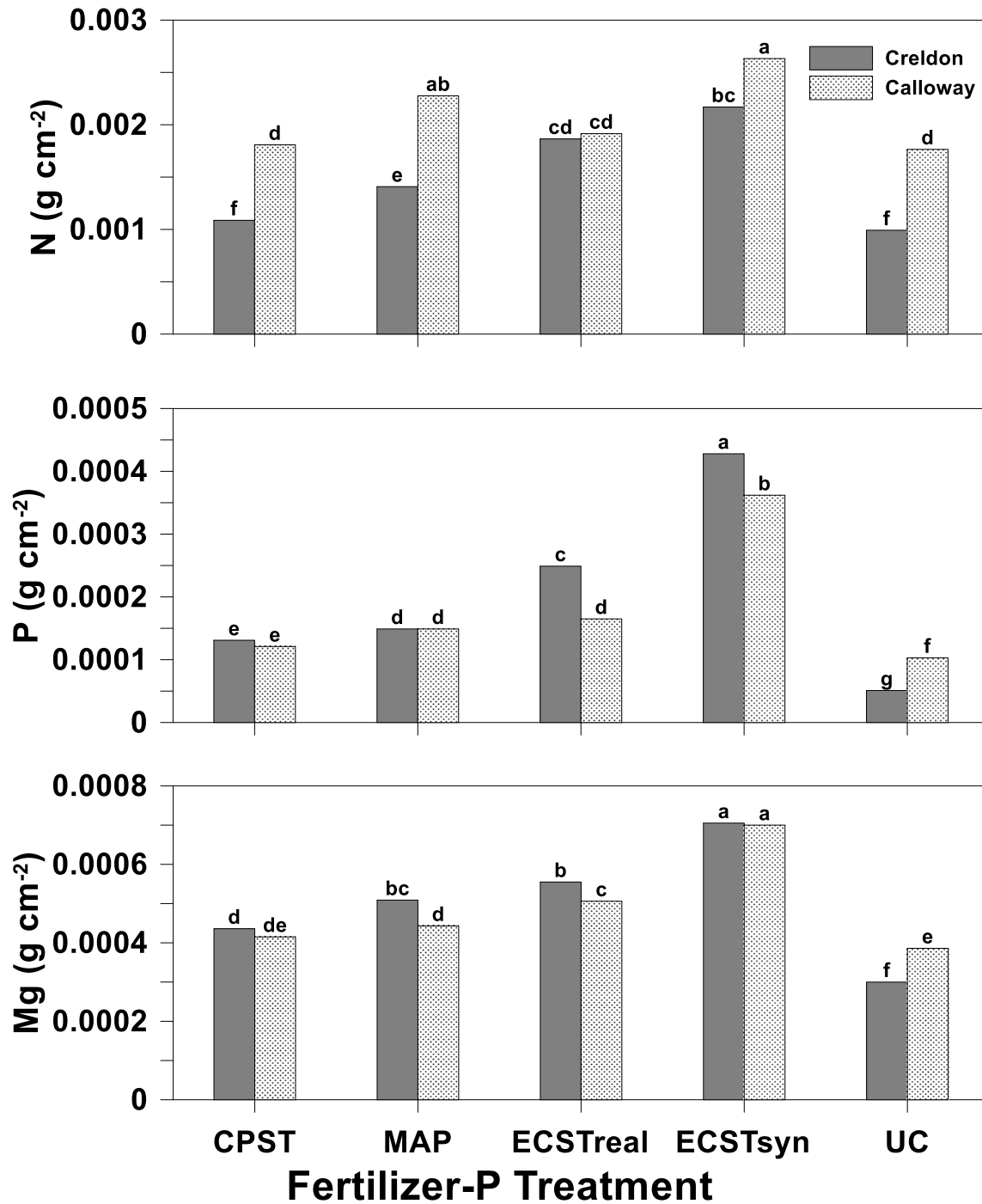
**Figure 10.** Aboveground (AG) soybean dry matter (DM) among soil-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations and aboveground soybean DM among water type-fertilizer-P source treatment combinations for the 60-day greenhouse potted-plant study. Within a panel, bars with different lower-case letters are different at  $P < 0.05$ .



**Figure 11.** Aboveground soybean nitrogen (N) concentration among water type-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations and aboveground soybean phosphorus (P) concentration among water type-soil combinations for the 60-day greenhouse potted-plant study. Within a panel, bars with different lower-case letters are different at  $P < 0.05$ .

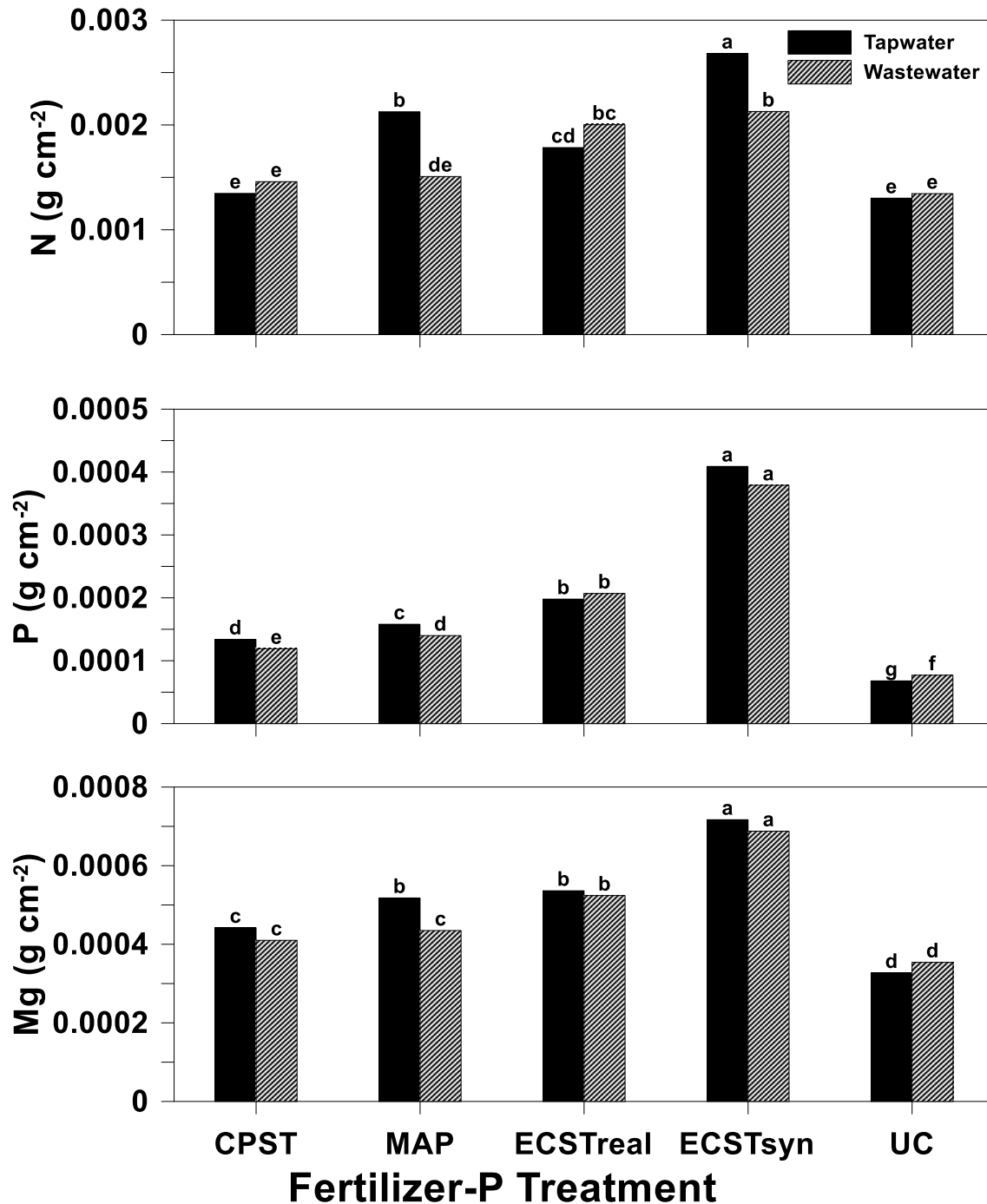


**Figure 12.** Aboveground soybean nitrogen (N), phosphorus (P), and magnesium (Mg) concentration among soil-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations for the 60-day greenhouse potted-plant study. Within a panel, bars with different lower-case letters are different at  $P < 0.05$ .

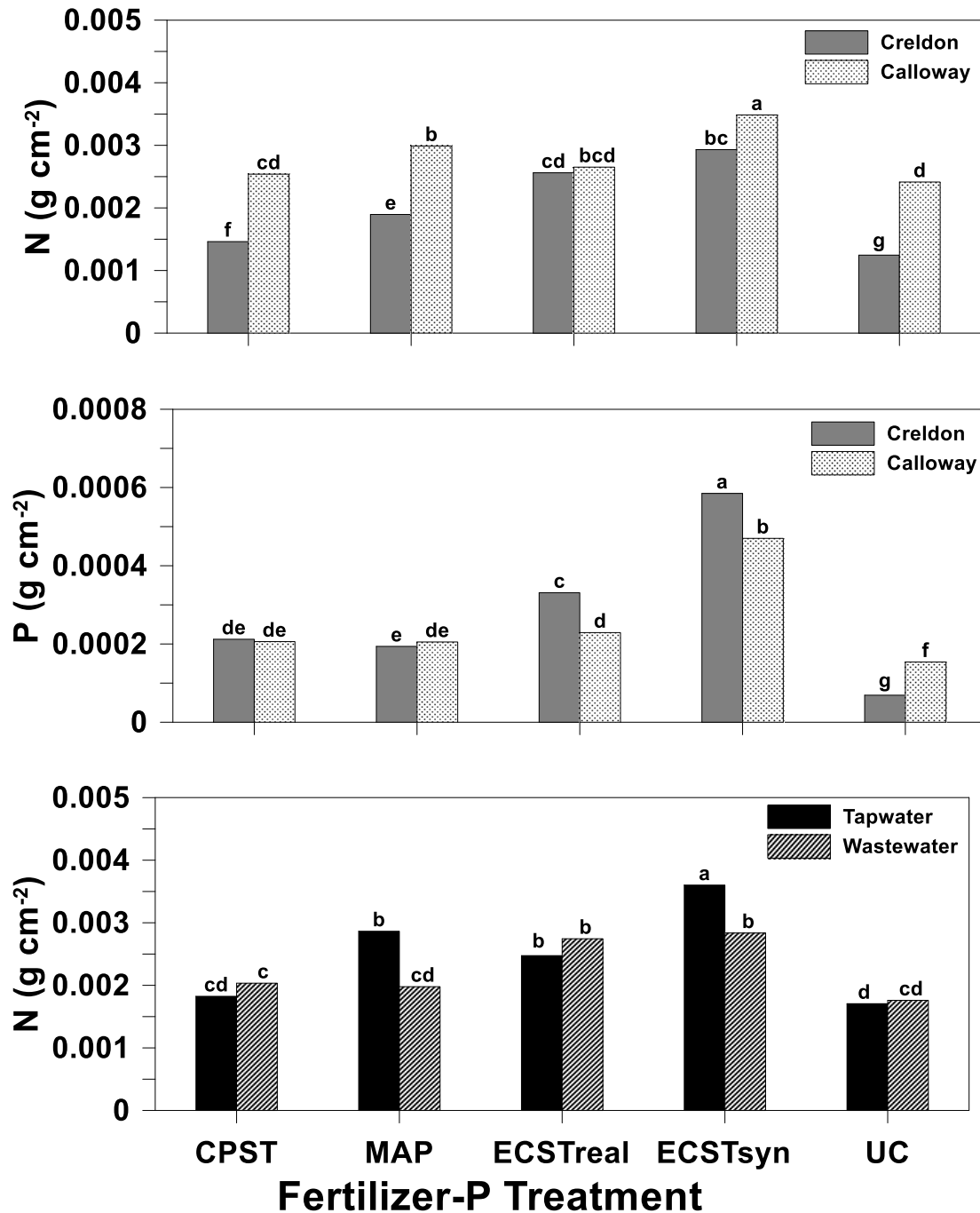


**Figure 13.** Aboveground soybean nitrogen (N), phosphorus (P), and magnesium (Mg) uptakes between soil series (i.e., Crelton and Dapue)-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations for the 60-day greenhouse potted-plant study. Within a panel, bars with different lower-case letters are different at  $P < 0.05$ .





**Figure 14.** Aboveground soybean nitrogen (N), phosphorus (P), and magnesium (Mg) uptakes among water type-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations for the 60-day greenhouse potted-plant study. Within a panel, bars with different lower-case letters are different at  $P < 0.05$ .



**Figure 15.** Total soybean nitrogen (N) and phosphorus (P) uptakes among soil-fertilizer-P source [i.e., chemically precipitated struvite (CPST), monoammonium phosphate (MAP), real-wastewater-derived electrochemically precipitated struvite (ECSTreal), synthetic electrochemically precipitated struvite (ECSTsyn) and an unamended control (UC)] combinations and total soybean nitrogen (N) uptake among water type-fertilizer-P source combinations for the 60-day greenhouse potted-plant study. Within a panel, bars with different lower-case letters are different at  $P < 0.05$ .

## Conclusions

The first objective of this thesis was a laboratory study evaluating the effects of soil, fertilizer-P source, and water source over a six-month period on runoff-water quality parameters from laboratory-conducted, rainfall-runoff simulations. Numerous runoff-water-quality parameters responded similarly across multiple soils. Most significantly, the struvite fertilizers (i.e., CPST, ECSTreal, and ECSTsyn) responded similarly to MAP in most treatment combinations. When struvite did not respond similarly to MAP, the differences in interactions were likely due to the slow-release nature of struvite and/or the unpredictable elemental composition of the wastewater from which ECSTreal was derived.

The second objective of this thesis was a greenhouse potted-plant study evaluating the combined effects of two silt-loam soils, two water types, and five fertilizer-P sources on corn and soybean properties over a 60-day period. Along with below- and aboveground dry matter, many below- and aboveground N, P, and Mg concentrations and uptakes responded similarly across the various soil-water-fertilizer-P source combinations. The hypothesis that the struvite materials (i.e., CPST, ECSTreal, and ECSTsyn) would have the greater corn and soybean response in the lower-pH soil (i.e., the Creldon soil) was partially supported, as only a sub-set of measured plant properties were larger in the Creldon than in the Calloway soil. Struvite-removed wastewater was initially studied to investigate its potential as a nutrient-supplying, irrigation-water source, but after watering corn and soybean with the wastewater only a few times a week throughout the 60-day period, wastewater often negatively affected corn and soybean properties, where tap water resulted in larger corn and soybean N, P, and Mg concentrations and uptakes, despite the wastewater having consistently greater initial nutrient concentrations. Therefore, the hypothesis that plant properties would remain unaffected by water source was not supported. Most

significantly, the hypothesis that corn and soybean properties would be greater in the struvite-P sources than in MAP was also only partially supported. In almost every fertilizer-P source treatment interaction, struvite-P sources behaved similarly to MAP, the traditional, commercially available fertilizer-P source. More specifically, ECSTsyn consistently had greater corn and soybean N, P, and Mg concentrations and uptakes than any other fertilizer-P source, likely due to ECST's large initial P and Mg concentration and crystalline application form providing greater surface area for reactions than pellets.

To date, the environmental impacts and crop growth potential of struvite, as a relatively new, potential replacement for traditional, commercially available fertilizer-P sources has been under-studied. The similar water-quality responses of the struvite fertilizers among the various soils and water types compared to MAP suggest that struvite has similar runoff-water-quality implications as at least one widely used, commercially available fertilizer-P sources. Similarly, the corn and soybean responses to struvite compared to MAP suggest that struvite has similar crop response to at least one commercially available, fertilizer-P source. However, more long-term soil and field studies are needed to fully understand the environmental and agronomic implications of using struvite as an alternative fertilizer-P source to commonly used, commercially available, fertilizer-P sources.