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## Field Evaluation of Struvite as a Phosphorus Source

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Field Evaluation of Struvite as a Phosphorus Source

A dissertation submitted in partial fulfillment  
of the requirements for the degree of  
Doctor of Philosophy in Environmental Dynamics

by

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This dissertation is approved for recommendation to the Graduate Council.

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## Abstract

Phosphorus (P) is an element that is crucial in many biological processes in all forms of life and is not substitutable. Excess P in wastewaters leading to the degradation of receiving waters or eutrophication once released is a major environmental concern. Removal of excess P from wastewater as the mineral struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) could be a promising solution to reduce P discharge into receiving waters and can potentially provide a valuable fertilizer-P source for agricultural production. The objectives of this project were to evaluate the effects of: 1) chemically precipitated struvite (CPST), compared to triple superphosphate (TSP) and an unamended control (UC) treatment, and irrigation (irrigated and non-irrigated/dryland) on soybean [*Glycine Max* (L.) Merrill] and wheat (*Triticum aestivum* L.) response in a wheat-soybean, double-crop production system on a silt-loam soil (Aquic Fraglossudalf) in eastern Arkansas in 2018 and 2019 and 2) evaluate two struvite materials [electrochemically precipitated struvite (ECST) and CPST] relative to several other common fertilizer-P sources [TSP, monoammonium phosphate (MAP), diammonium phosphate (DAP), and RP] on the response of a pureline rice (*Oryza sativa* L.) cultivar grown under flood-irrigation in a P-deficient, silt-loam soil (Typic Glossaqualfs) and corn (*Zea mays* L.) and soybean grown in a P-deficient, silt-loam soil (Aquic Fraglossudalfs) in eastern Arkansas in 2019 and 2020. Aboveground dry matter, yield, and P, nitrogen, and magnesium tissue concentrations and uptake were measured. Results from the double-crop study showed that soybean seed yield were unaffected by fertilizer-P treatment and irrigation ( $P > 0.05$ ), but differed between years ( $P < 0.01$ ). Seed yield was 1.4 times greater in 2019 than in 2018 when averaged across irrigation and fertilizer-P treatments. In 2019, wheat yield did not differ ( $P > 0.05$ ) among fertilizer-P sources. In 2019, rice grain yield did not differ ( $P > 0.05$ ) among fertilizer-P sources. In 2020, rice yield was numerically largest

from TSP (9.8 Mg ha<sup>-1</sup>), which did not differ from that from DAP, MAP, RP, and the UC, and was numerically smallest from ECST (8.2 Mg ha<sup>-1</sup>), which did not differ ( $P > 0.05$ ) from that from CPST, and was lower ( $P < 0.05$ ) than that from TSP, DAP, MAP, RP, and the UC. Averaged across years, corn yield from ECST was at least 1.2 times greater ( $P < 0.05$ ) than that from CPST, TSP, MAP, DAP, RP, and UC. Soybean seed yield differed among fertilizer-P sources between years ( $P = 0.03$ ). In 2019, seed yield was numerically largest from ECST (4.1 Mg ha<sup>-1</sup>), which did not differ from CPST, TSP, MAP, DAP, or RP, and was 1.2 times greater than that from UC. In 2020, soybean seed yield was numerically greatest from CPST (2.8 Mg ha<sup>-1</sup>) and numerically smallest from ECST (2.2 Mg ha<sup>-1</sup>). Results showed that wastewater-recovered struvite materials have the potential to be a viable, alternative fertilizer-P source for wheat-soybean, double-crop production system, flood-irrigated rice, and furrow-irrigated corn and soybean in P-deficient, silt-loam soils in eastern Arkansas.

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## **Dedication**

I dedicate this dissertation to my mother who taught me to believe in myself and that a good name is better than gold or silver.

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## **Introduction**

Phosphorus (P) is an essential macronutrient for crop growth and is thus commonly applied as a fertilizer-P source to sustain agricultural production. However, P is mostly unavailable in soils as P is fixed by Fe/Al in acidic soil or by Ca in alkaline soil or bound to organic compounds (Sims & Pierzynski, 2005). Therefore, low P availability is a major limitation to plant growth and development. Additionally, rock phosphate (RP), from which most fertilizer-P sources are created, is limited and non-renewable (Cordell, Drangert, & White, 2009). Rock phosphate reserves have been declining globally and it is estimated that the amount of economically feasibly mined P will be exhausted within 100 to 250 years (Liu, Kumar, Kwag, & Ra, 2012).

In addition to future availability concern of the limited RP, P contamination in aquatic ecosystems is a major environmental concern. Phosphorus is largely a limiting nutrient in surface waters and, when P is in excess, can cause eutrophication and degradation of aquatic ecosystems. Agriculture and effluent from municipal wastewater treatment plants have been identified as major sources of P loading in water bodies (Liu et al., 2012). Eutrophication has been identified as a critical problem in water bodies listed as having impaired water quality in the United States, with agriculture the major source of nutrients in impaired lakes (50%) and rivers (60%; Daniel, Sharpley, & Lemunyon, 1998). Recovery of P as mineral struvite from wastewaters could be an alternative option for a sustainable future food security.

Struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) is considered to be a slow-release, efficient fertilizer-P source that can be recovered from both liquid and solid wastes. Struvite has been recovered from numerous wastewaters through biological, chemical, and in recent years, electrochemical precipitation techniques (Le Corre et al., 2009; Kékedy-Nagy, Teymouri, Herring, & Greenlee, 2020). The electrochemical precipitation technique for the recovery of struvite utilizes a Mg-

based anode that supplies Mg ions as the electrode corrodes in the presence of an electrical current (Kékedy-Nagy et al., 2019). In contrast to chemical precipitation, electrochemical precipitation eliminates the need for external chemical addition.

Soybean (*Glycine max* L.), wheat (*Triticum aestivum* L.), corn (*Zea mays* L.), and rice (*Oryza sativa* L.) are important food crops for a large human population around the world and are widely grown in Arkansas. One common denominator among soybean, wheat, corn, and rice is that P is essential for growth and development of each crop. Recent studies worldwide are currently investigating struvite's potential as a fertilizer-P source, however, applications of recovered struvite have not been well studied in the mid-southern United States, particularly in row crops under field settings.

The objectives of this dissertation were to 1) evaluate the effects of fertilizer-P source [chemically precipitated struvite (CPST) and triple superphosphate (TSP)] and water management (irrigated and non-irrigated) on above- (i.e., dry matter, yield, and N, P and Mg tissue concentrations and uptake) and belowground (i.e., root tissue N, P, and Mg concentrations) soybean and wheat properties over two years in a wheat-soybean, double-crop production system on a silt-loam soil in eastern Arkansas, 2) evaluate the effects of two wastewater-recovered struvite materials [i.e., electrochemically precipitated struvite (ECST) and CPST] relative to several other common fertilizer-P sources (i.e., TSP, monoammonium phosphate (MAP), diammonium phosphate (DAP), and rock phosphate (RP) (i.e., P-source study) on the response of a pureline rice cultivar grown under flood-irrigation in a P-deficient, silt-loam soil (Typic Glossaqualfs) in eastern Arkansas, and 3) evaluate the effects of two struvite materials (i.e., ECST and CPST) relative to several other common fertilizer-P sources (i.e., TSP, MAP, DAP, and RP) on corn and soybean response in two consecutive growing seasons in a P-deficient, silt-

loam soil (Aquic Fraglossudalfs) in eastern Arkansas.

For the wheat-soybean, double-crop study, it was hypothesized that soybean amended with CPST would have similar or greater aboveground dry matter and greater aboveground and belowground tissue P and N concentrations, aboveground P and N uptake, but greater aboveground and belowground tissue Mg concentrations than P fertilization with the TSP. Yield, aboveground dry matter, aboveground nutrient uptake, aboveground and belowground tissue P, N, and Mg concentrations of soybean were also expected to be greater under irrigated than under dryland production. In addition, it was hypothesized that soybean grown in year one would have similar yield, aboveground dry matter, aboveground nutrient uptake, aboveground and belowground tissue P, N, and Mg concentrations compared to that in year two. It was hypothesized that there would be no difference in aboveground and total wheat dry matter or wheat yield when amended with CPST compared to TSP. Aboveground and belowground wheat tissue P and N concentrations, and aboveground wheat P and N uptake, were also expected to be greater from TSP than from CPST fertilization, while aboveground and belowground wheat tissue Mg concentrations were expected to be greater from CPST than from TSP fertilization because of the greater Mg concentration in the chemical composition of CPST.

For rice, it was hypothesized that rice amended with either wastewater-recovered struvite source (i.e., ECST and CPST) would have at least similar aboveground dry matter and yield, above- and belowground tissue and grain P, N, and C concentrations, and aboveground and grain P, N, and C contents, but greater aboveground tissue and grain Mg concentration and uptake and belowground tissue Mg concentrations than other common, commercially available fertilizer-P sources (i.e., TSP, MAP, DAP, and RP) due to lower water solubility and greater initial Mg concentrations in ECST and CPST.

For corn, it was hypothesized that both wastewater-recovered struvite sources (i.e., ECST and CPST) would have at least similar total aboveground dry matter and yield, total aboveground tissue P and N uptake, and belowground P and N concentrations, but greater total aboveground tissue Mg uptake and belowground tissue Mg concentrations than other common, commercially available fertilizer-P sources (i.e., TSP, MAP, DAP, and RP) due to struvite's lower water solubility and greater initial Mg concentrations. In addition, it was hypothesized that corn grown in year two would have greater total aboveground dry matter, yield, total aboveground tissue P, N, and Mg uptake, and belowground tissue P, N, and Mg concentrations compared to that in year one due to a carry-over effect from fertilization in year one.

For soybean, it was hypothesized that both wastewater-recovered struvite sources (i.e., ECST and CPST) would also have at least similar aboveground dry matter and yield, above- and belowground tissue and seed P and N concentrations, and aboveground and seed P and N uptake, but greater aboveground tissue and seed Mg concentration and uptake and belowground tissue Mg concentrations than other common, commercially available fertilizer-P sources (i.e., TSP, MAP, DAP, and RP). In addition, it was hypothesized that soybean grown in year two would also have greater aboveground dry matter, yield, above- and belowground tissue and seed P, N, and Mg concentrations, and aboveground tissue and seed P, N, and Mg uptake compared to that in year one.

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## **Chapter 1**

Wastewater-recovered Struvite Effects on Plant and Soil Response and Implications for Society  
and Sustainability from a Global Perspective



## Abstract

Phosphorus (P) and nitrogen (N) loading in surface waters from wastewater discharges is a global environmental quality concern. Phosphorus is a limiting resource and the recovery and reuse of P as struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) is essential for the long-term sustainability of the agriculture and fertilizer industries as well as the global society. Struvite can be recovered from several types of wastewaters including sewage sludge, urine, fertilizer plant, anaerobic effluent, swine, landfill leachate, dairy, and agro-industrial wastes. The nutrient composition in recovered struvite varies according to the type of wastewater source and recovery process. The objective of this study is to review the effects of wastewater-recovered struvite on plant and soil response and implications for society and sustainability from a global perspective. There is a wide variation in fertilization effects of struvite on several plants such as corn (*Zea mays* L.), wheat (*Triticum aestivum* L.), tomato (*Solanum lycopersicum*), ryegrass (*Lolium perenne*), and potato (*Solanum tuberosum* L.), under different soil conditions with comparatively greater, similar, or lower growth to various conventional P fertilizers. The recovery of struvite from wastewaters can impact the society in different ways, such as restoring aquatic biodiversity, helping solve the problem of struvite scaling in pipes for the wastewater treatment industry, and promoting water quality. Wastewater-recovered struvite has been shown to be an effective, eco-friendly, sustainable alternative source of P fertilizer. The recovery of struvite from wastewater could provide a viable fertilizer-P source, help protect biodiversity through reduction of eutrophication in waterbodies, and create employment opportunities in the society.

## **Introduction**

There has been increased interest in removing P from waste streams because of future rock phosphate scarcity and potential water pollution. Rock phosphate (RP) is a non-renewable resource and existing reserves are projected to be depleted within the next 50 to 100 years (Cordell et al., 2009). The recovery of P and N as struvite has gained global interest over the years. Many advances in technology are available to recover P and N before wastewater is discharged into waterbodies, thereby reducing the threat to water quality (Burns and Moody, 2002; Zhang et al., 2010). In different parts of the world, recovered struvite from various sources have been tested on different crops/plants. It is important to know the implications of the value-added product on the global society and sustainability. This review paper focuses on the effects of wastewater-recovered struvite on plant and soil response and implications for society and sustainability from a global perspective.

## **Wastewaters**

Over the years, global environmental change has gained more interest because of the potential risk of surface water eutrophication and groundwater contamination by altering the Earth's biogeochemical cycles of phosphorus (P) and nitrogen (N) through various human activities, which include inefficient use of fertilizers and the discharge of untreated wastewaters. The most significant sources of water pollution include inefficient management of agricultural runoff, and discharge of municipal and industrial wastewaters. There is a 10 to 20% increase in nutrient pollution as a result of increases in fertilizer used in the agriculture industry (UNEP 2007). Additionally, only 20% of the globally produced wastewaters are treated before being discharged back into water bodies across the world (UNESCO, 2012).

Wastewaters are composed of a large amount of P, N, and organic matter, with a considerable quantity of magnesium (Mg), various macro- and micro-elements, and heavy metals (Deng et al., 2006; Suzuki et al., 2007; Liu et al., 2011a,b; Rahman et al., 2014). Therefore, wastewater is considered as one of the major polluting agents in the environment. However, wastewater is increasingly being recognized as a valuable resource in terms of nutrients, water, and energy. Wastewater composition varies according to the source of origin. A summary of wastewater composition from three different sources is shown in Table 1 which indicates swine and municipal wastewaters have a large total N and P concentrations.

#### Nutrient Recovery in Wastewaters

Nitrogen and P are essential nutrients for crop and animal production. Over 907 million kg of RP are consumed yearly as fertilizer and the amount of N fertilizer used is approximately three folds that of P consumption (Rahman et al., 2011). One of the largest inefficiencies in P conversion is associated with agricultural operations (Suh and Yee, 2011). In the environment, P can attach to soil particles and, thus, moves into surface water from erosion. Approximately 8 million Mg of P are lost from farms per year through soil erosion and leaching, in which much of the P ends up in streams and lakes (Cordell et al., 2009). In the global food system, the P cycle is inefficient and the majority of P derived from food wastes is lost to the environment via sewer systems, wastewater treatment, and landfills (Suh and Yee, 2011). Approximately 98% of P in the human diet is lost through sewage (Smil, 2000; FalkØgaard and Brod, 2016). In addition, P from industrial wastes and detergents ultimately ends up in sewage and wastewater (FalkØgaard and Brod, 2016).

Generally, wastewaters containing P and N result from the fertilizer industry, municipal wastewaters, swine production industry, dye industry, tannery industry, and agriculture and livestock farms (Tünay et al., 1997; Chimenos et al., 2003). Precipitation of phosphate ( $\text{PO}_4^{3-}$ ) and ammonium ions ( $\text{NH}_4^+$ ) as struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) is a way of removing P and N from wastewaters. Over the years, struvite has been recovered from various types of wastewaters, such as industrial wastewater (El Diwani et al., 2007), fertilizer plant wastewater (Yu et al., 2013), swine wastewater (Nelson et al., 2003; Deng et al., 2006; Suzuki et al., 2007; Rahman et al., 2011), municipal landfill leachate (Kim et al., 2007), calf manure wastewater (Schuiling and Andrade, 1999), anaerobic digester effluents (Celen and Turker, 2001), dairy wastewater (Massey et al., 2007), leather tanning wastewater (Tunay et al., 1997), urine (Etter et al., 2011), animal manure (Burns and Moody, 2002), sewage sludge (Munch and Barr, 2001), synthetic wastewater (Adnan et al., 2003), wasted sludge (Jaffer et al., 2002), agro-industrial wastes (Moerman et al., 2009), digester supernatant (Battistoni et al., 2000; Pastor et al., 2010), slaughterhouse wastewater (Kabdasli et al., 2003), lagoon wastewater (Westerman et al., 2009), slurry type swine wastewater (Kim et al., 2004), and poultry manure wastewater (Yetilmezsoy and Zengin, 2009). In domestic wastewater, urine contributes 50 to 80% of the P concentration and 75% of N concentration is excreted in the form of urea, making urine a potential source of struvite (Rubio-Rincon et al., 2014; Arredondo et al., 2015).

## Common Methods

In the past decade, struvite precipitation has gained interest as a way to recover P (Doyle et al., 2003). Great efforts have been made by researchers to remove P and N from wastewater through biological (Welander et al., 1998), chemical (Bonmati and Flotats, 2003; Cho et al.,

2009; Rahman et al., 2011), and electrochemical conversion (Kim et al., 2006; Liu et al., 2011a). The cheaper source of P and N in wastewater makes wastewater a potential raw material for the fertilizer industry, provided that the nucleation and quality of recovered struvite crystals can be controlled. Recently, struvite has been produced from wastewaters using microbial fuel cells (Ichihashi and Hirooka, 2012).

Precipitation of struvite is controlled by factors as the concentrations and molar mass of  $Mg^{2+}$ ,  $NH_4^+$  and  $PO_4^{3-}$  as well as pH, temperature, ionic strength, and impurities, such as calcium (Ca; Hao et al., 2008; Yetilmezsoy and Zengin, 2009). Of all the factors, pH is generally considered to be a key factor controlling formation of struvite, as struvite can be precipitated at a wide range of pH (7.0 to 11.0) (Le Corre et al., 2005; Kim et al., 2007; Pastor et al., 2008), but the suitable range is between 7.5 to 9.0 (Hao et al., 2008). The presence of  $Ca^{2+}$  in wastewater or urine can also inhibit struvite precipitation due to the more favorable reaction of phosphates to generate calcium phosphates, dicalcium phosphate, hydroxyapatite, and octacalcium phosphate (Kim et al., 2007; Le Corre et al., 2005). The disadvantage of using high pHs is the addition of alkalinity and a potential co-precipitation of carbonates (Wang et al. 2010).

Generally, wastewaters from livestock, or municipal leachates contains less Mg compared with P and N. Therefore, it is necessary to add a source of Mg to optimize the process of struvite precipitation (Moerman et al., 2009; Yetilmezsoy and Zengin, 2009). Several studies have reported different Mg sources, including  $MgCl_2$ ,  $MgSO_4$ ,  $MgO$ , and  $Mg(OH)_2$ , that, if added to wastewater or urine, can increase the reaction rate of struvite precipitation, but can increase the cost of production (Moerman et al., 2009; Yetilmezsoy and Zengin, 2009; Liu et al., 2011c; Rahman et al., 2014). Yetilmezsoy and Zengin (2009) reported no difference between  $MgCl_2$  and  $MgSO_4$  as Mg source; however, Yetilmezsoy and Zengin (2009) reported that  $MgCl_2$  showed greater

efficiencies of  $\text{NH}_4^+$ -N, chemical oxygen demand (COD) and color removal during struvite precipitation. Additionally, it has been reported that seawater can be used as a natural and free  $\text{MgCl}_2$  source to precipitate struvite from urine, which is an economical and effective technique for chemical struvite crystallization (Rubio-Rincon et al., 2014; Tang et al., 2015). Furthermore, the  $\text{PO}_4$ :Mg ratio is also important in struvite precipitation. The optimum  $\text{PO}_4$ :Mg ratios are between 1:1 and 1:1.2 because larger crystals and large efficiency can be obtained (Hutnik et al., 2013; Matynia et al., 2013). However, the concentration of  $\text{NH}_4^+$  in urine can also be affected by aeration since the air flow agitates the solution producing an increase in ammonia volatilization (Rahman et al., 2014). A decrease in the concentration of  $\text{NH}_4^+$  will result in a decrease in struvite recovery.

### Precipitation Techniques

The recovery of nutrients from wastewaters using a crystallization or chemical precipitation can provide a value-added product, called struvite. Traditionally, crystallization precipitation is the primary technique for struvite recovery, but there are challenges and disadvantages of chemical precipitation. The method requires external chemical addition and the kinetics are slow at near-neutral pH (Rahman et al., 2011).

Electrochemical precipitation is a newly adopted technique for the removal and recovery of struvite from wastewater to reduce the scarcity of RP resources and water eutrophication worldwide. Electrochemical precipitation technique is designed to permit energy recovery and utilizes a sacrificial Mg-based anode, which supplies Mg ions and corrodes in the presence of an electrical current (Moussa et al., 2006). The electrochemical precipitation method could be advantageously used to recover struvite from wastewaters of animal origin, without any use of

added chemicals (Moussa et al., 2006). Wang et al. (2010) used an electrochemical method as a means to improve the formation of pure struvite from waste streams at a neutral pH, using a lower voltage (Direct current: 3 to 12V). Moussa et al. (2006) also used electrochemical method to deposit pure struvite on the electrode surface from an aqueous solution containing  $\text{PO}_4^{3-}$ ,  $\text{NH}_4^+$ , and  $\text{Mg}^{2+}$ .

There has been demonstration of electrochemically precipitated struvite from wastewater both at the bench scale and at the pilot scale (Kruk et al., 2014). Usually, electrodes are made of metals, Mg salt is added, and the struvite precipitation reaction is driven by electrochemistry. In these electrochemical systems, struvite precipitation has been reported in the range of pH 7.5 to 11 (Moussa et al., 2006; Wang et al., 2010; Hao et al., 2013) with low energy consumption at  $70 \text{ W m}^{-3}$ . Electrochemically precipitated struvite has certain advantages over chemically precipitated forms, such as a reduction in added chemicals (Moussa et al., 2006). In addition, precipitation with adequate Mg addition recycles up to 95% of P in the treated streams and addresses at least 25% of the influent P load of a wastewater treatment plant (WWTP). Thus, there is a potential window of opportunity for a more energy-efficient struvite recovery technology.

## **Struvite**

Struvite, the common name for magnesium ammonium phosphate hexahydrate,  $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$  (Johnston and Richards, 2003), is a slow-release, fertilizer-P source that can be recovered from both solid wastes, such as food, animal, and human waste (Kataki et al., 2016b; Farrow et al., 2017), and wastewaters, such as municipal, industrial, and agricultural wastewaters (Westerman et al., 2009; Mayer et al., 2016). Equi-molar concentrations (1:1:1) of  $\text{Mg}^{2+}$ ,  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  with alkaline pH and appropriate mixing are necessary to precipitate

struvite (Rahaman et al., 2008). Struvite has a molecular weight of  $245.43 \text{ g mol}^{-1}$ , and is readily soluble in acidic media, sparingly soluble under neutral conditions, but insoluble in alkaline media (Chirmuley, 1994). The solubility of struvite is low in water:  $0.018 \text{ g } 100 \text{ mL}^{-1}$  at  $25 \text{ }^\circ\text{C}$  in water, large in acids:  $0.033 \text{ g } 100 \text{ mL}^{-1}$  at  $25 \text{ }^\circ\text{C}$  in  $0.001 \text{ N HCl}$ , and  $0.178 \text{ g } 100 \text{ mL}^{-1}$  at  $25 \text{ }^\circ\text{C}$  in  $0.01 \text{ N HCl}$  (Le Corre et al., 2009). Struvite fertilizer is granular, concentrated, non-sludgy, odorless, easy to handle, and free of traditional sludge-handling problems (Bouropoulos and Koutsoukos, 2000). Pure struvite as a fertilizer-P source has an average fertilizer grade of 5.7-28-0 for N,  $\text{P}_2\text{O}_5$ ,  $\text{K}_2\text{O}$  and an average of 9.9% Mg (Westerman, 2009).

Generally, recovered struvite contains between 11 to 26% total P (Johnston and Richards, 2003) depending on the initial source and method of production, yet only about 0.02% is water-soluble (Li and Zhao, 2002; Negrea et al., 2010), while the remaining P is acid soluble (Bridger et al., 1962), thus making struvite an ideal slow-release source of P for plants. Struvite is treated as fertilizer-P source, although it is also an effective source of N and Mg. Struvite has a lower seedling toxicity because of the slow-release characteristic, in which the struvite begins to dissolve once organic acids are released by plant roots into the rhizosphere (Katanda et al., 2016). The slow release is a better match in the timing of P availability with plant need compared to other conventional P fertilizers, such as triple superphosphate (TSP) and monoammonium phosphate (MAP), representing the most advantageous characteristic of struvite as a fertilizer-P source. However, a factor that could be considered is that struvite may require supplementation with potassium (K) to meet the NPK requirements of certain specific crops (Gaterell et al., 2000).



## Struvite Effects on Soil Response

The fertilizer effect of struvite varies with different soils because of variation in solubility and soil sorption characteristics of soil (Kataki et al., 2016a). The solubility of struvite changes with pH, temperature, and ionic strength (Le Corre et al., 2009). Under acidic conditions, the solubility of struvite is increased, which increases fertilizer-uptake efficiency. The solubility of struvite can range from 65 to 100% in acidic soils, which is similar to the solubility of TSP (Cabeza et al., 2011). Phosphorous adsorption increases in soil under acidic condition as well as P dissolution and availability (Bowden et al., 1980). At  $\text{pH} < 5$ , struvite solubility can range from  $1 \times 10^{-3}$  (Abonna et al., 1982) to  $1 \times 10^{-2} \text{ mol L}^{-1}$  (Borgerding, 1972) whereas within the pH range of 8.2 to 8.8, struvite reaches a minimum solubility of  $4 \times 10^{-5} \text{ mol L}^{-1}$  (Le Corre et al., 2009). In addition, the solubility of struvite changes with temperature from  $1 \times 10^{-3} \text{ mol L}^{-1}$  at 0 and  $50^\circ\text{C}$  and reaching  $2 \times 10^{-3} \text{ mol L}^{-1}$  at  $20^\circ\text{C}$  in the same solution (Borgerding, 1972). In soils under aerobic condition, the  $\text{NH}_4^+$  concentration is much lower than that of nitrate ( $\text{NO}_3^-$ ) in solution because bacteria rapidly convert  $\text{NH}_4^+$  to  $\text{NO}_3^-$ . This biological process of conversion depends on temperature, microbial composition, presence of oxygen, and other nutrients.

In soils with large P fixation, such as soils with large amount of calcium carbonate ( $\text{CaCO}_3$ ) and aluminum (Al) or Iron (Fe), the moderate solubility of struvite makes struvite an attractive fertilizer-P (Goto, 1998). Precipitation of various calcium phosphate minerals is common in calcareous, alkaline soil. Rock phosphate is ineffective in calcareous soil because of the alkaline pH and large Ca concentration inhibit RP dissolution (Chien & Menon, 1995); thus, a sustainable, effective alternative P fertilizer is needed for agriculture in semi-arid environments. Struvite is a potential fertilizer-P source in calcareous soils, as struvite is substantially more soluble than calcium phosphate at alkaline pH. Massey et al. (2007)

conducted a 90-day greenhouse trial in the US for spring wheat (*Triticum aestivum* L. 'Zeke') production to determine the effectiveness of recovered struvite in different soil pH levels. Two soils with different pH were used in the trial: a neutral to slightly acidic soil (pH 6.5), classified as fine-loamy over sandy or sandy-skeletal, mixed, mesic Aridic Argiustoll, and a calcareous soil (pH 7.6) of the same class. There were five treatments at two application rates (45 and 90 kg ha<sup>-1</sup>): recovered struvite from dairy wastewater, TSP, organic RP, dittmarite [(NH<sub>4</sub>)Mg(PO<sub>4</sub>).H<sub>2</sub>O], and 'Colorado product', which consisted of ground carbonate fluorapatite seed crystal, little amounts of recovered magnesium phosphate, and sand grains. Results showed that in the low pH soil (pH 6.5), struvite and TSP significantly increased plant P concentration (3.6 g P kg<sup>-1</sup> and 3.7 g P kg<sup>-1</sup>, respectively) at the large application rate compared to the control (3.4 g P kg<sup>-1</sup>). In the calcareous soil (pH 7.6), struvite applied at the large application rate significantly increased dry matter (DM) production by 1.31 times more than the control, but performed similar to other P sources. Massey et al. (2007) concluded that recovered struvite could be a useful alternative P fertilizer in arid and semi-arid environments.

In addition to P, struvite application could be a potential source of Mg to the soil (Gonzalez-Ponce et al., 2009). In a field trial in the Netherlands, Gell et al. (2011) reported a two-fold change in exchangeable Ca:Mg ratio of a sandy loam soil with pH of 4.5 (Typic Andisol) from roughly 4:1 to 2:1 over 146 days due to application of Mg in blackwater-derived struvite at an application rate of 200 Kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup>. At such an application rate over several years, there could be possibility of Mg accumulation in soil. Thus, to maintain a healthy Ca:Mg ratio over a long period of time, the amount of Ca in the soil must be replenished, perhaps by liming, at an equal molar rate that struvite is applied (Gell et al., 2011).

Potential P and N runoff and leaching is minimized due to the low water solubility of

struvite. Rahman et al. (2011) conducted a 5-week trial to examine nutrient (N and P) leaching loss from struvite recovered from swine wastewater and commercial P and N fertilizers in soil using soil columns (Plexiglas tube about 55 cm long with a diameter of 8 cm) in South Korea. The experimental soil contained 15% clay and 5% organic materials. Struvite was used as P and N source, while an equivalent weight of P was used from fused superphosphate (FSP) and N was from urea. Results showed that P leaching was negligible from both fertilizer treatments. The explanation was that both fertilizers are sparingly soluble in water and P molecules might have formed bonds with clay particles and other chemicals involved in P fixation, such as Al, Fe, and Ca. Results also revealed that less N was leached from struvite treated soils (1.9 and 2.0%) compared to FSP-urea treated soils (7.8 and 6.5%). The explanation was that the slowly releasing pattern of struvite provided a gradual nutrient supply for a longer period and hence improved the efficiency and reduced N leaching loss. Based on these results and reports, struvite would be an effective alternative fertilizer-P source in a wide range of soil environments.

## **Struvite Effects on Crop Response**

### **Greenhouse Studies**

Many studies have been conducted on the effect of recovered struvite on crop response in different parts of the world. Most of the studies are greenhouse pot experiments with many plant types (i.e., turf, tree seedlings, vegetables, orchards, ornamentals) and crops (i.e., corn (*Zea mays* L.), wheat).

Several plants have been grown with struvite application, such as corn (Barak and Stafford, 2006; Kern et al., 2008; Cabeza et al., 2011; Gell et al., 2011; Thompson, 2013, Uysal et al., 2014), ryegrass (*Lolium perenne*; Johnston and Richards 2003; Plaza et al., 2007;

Yetilmezsoy and Sapci-Zengin, 2009; Antonini et al., 2012), water spinach (*Ipomoea aquatica*), chinese flowering cabbage (*Brassica parachinensis*), water convolvulus (*Ipomoea aquatica*, I. reptans)], Chinese chard (*Brassica rapa* var. chinensis; Li and Zhao, 2003), lettuce (*Lactuca sativa* L.; Gonzalez-Ponce et al., 2009; Cerrillo et al., 2014), wheat (*Triticum aestivum* L.; Ganrot et al., 2007; Massey et al., 2009; Talboys et al., 2016), tomato (*Solanum lycopersicum*; Uysal et al., 2014), gram (*Cicer arietinum* L.; Ghosh et al., 1996), broad bean plant (*Vicia faba*; El Diwani et al., 2007), canola (*Brassicca napus* L.; Ackerman et al., 2013, Katanda et al., 2016), and potato (*Solanum tuberosum* L.; Collins et al., 2016).

Most of the studies reported similar effects of struvite with chemical fertilizer on different plants (Ghosh et al., 1996; Johnston and Richards, 2003; Li and Zhao, 2003; Plaza et al., 2007; Gonzalez-Ponce and Garcia-Lopez-de-Sa, 2008; Gonzalez-Ponce et al., 2009; Massey et al., 2009; Liu et al., 2011b; Cerillo et al., 2014; Uysal and Kuru, 2015; Katanda et al., 2016). Johnston and Richards (2003) compared a variety of recovered phosphate fertilizers in pot experiments with a sandy loam soil (pH 6.6) and a sandy clay loam soil (pH 7.1) using perennial ryegrass for 100 days in the UK. The treatments used were seven struvites recovered from different wastewater discharges (municipal sewage, corn steep liquor, and carmin red dye industry), two laboratory-synthesized struvites, a synthetic iron phosphate, and a recovered calcium phosphate applied at a rate equivalent to 0.087g P pot<sup>-1</sup>. Results showed no difference in total DM yield of ryegrass and P uptake (calculated by multiplying DM yield by % P per pot) between the different recovered and synthetic struvites and between any of the precipitated struvites and monocalcium phosphate applied at the equal rate in both soils. Johnston and Richards (2003) concluded that struvite's effect on crop yield is comparable to that of monocalcium phosphate.

Antonini et al. (2012) reported similar crop yield and P uptake when struvite derived from urine and commercial phosphate fertilizer were compared using corn and ryegrass. In a pot experiment in Germany, Perez et al. (2009) reported a similar P uptake in corn from Sattenhausen soil (pH 5.6) and a slightly greater P uptake in Gieboldehausen soil (pH 6.8) treated with struvite recovered from sewage sludge compared with TSP. There was no difference between P uptake of corn grown with struvite compared to TSP (Cabeza et al., 2011). According to Plaza et al. (2007), no difference in P uptake for ryegrass treated with struvite recovered from municipal wastewater in Spain compared with super phosphate, but with increasing struvite dose, P uptake was also increased and there was no effect on K uptake. Similarly, studies have shown no difference in N uptake between struvite and chemical fertilizers for corn (Uysal and Kuru, 2015), ryegrass (Plaza et al., 2007), or lettuce (Korchef et al., 2011).

In a 45-day greenhouse pot experiment in Egypt, El Diwani et al., (2007), El Diwani et al. (2007) evaluated the effect of struvite recovered from industrial wastewater on broad bean plant growth and nutrient uptake compared to chemical fertilizer [ $\text{NH}_4\text{NO}_3$ , super phosphate, potassium sulphate ( $\text{K}_2\text{SO}_4$ )] in a sandy soil of pH 8.01. Results showed greater fresh yield (54%) and dry yield (58%) of broad bean plant were observed with struvite compared with chemical fertilizer at the 45<sup>th</sup> day growth stage. In a study conducted by Ryu and Lee (2016), struvite was applied as a N fertilizer at  $110 \text{ kg ha}^{-1}$  and obtained an increase of 179% in plant height, 200% in fresh yield, and 275% in dry yield compared with a complex fertilizer.

Antonini et al. (2012) conducted two greenhouse pot experiments in Germany [Experiment A (82 days) and Experiment B (76 or 51 days)], and evaluated the effectiveness of human urine-derived struvite as compared to a mineral-P fertilizer (Cederan phosphate fertilizer). The test crops were Italian ryegrass (*Lolium multiflorum* Lam.) and corn grown in a

P-deficient 'Meckenheim' soil. In Experiment A, results showed a 134% and 244% increased DM yield for ryegrass and corn with struvite compared with the commercial mineral fertilizer. Results also showed that struvite treated pots had a 211 to 281% greater P uptake in ryegrass and corn in Experiment A and no difference in Experiment B compared with the mineral fertilizer. Greater P uptake by ryegrass and corn treated with struvite was due to the secretion mechanism of root exudates for mobilizing sparingly soluble P in the rhizosphere. The release of organic acid ions might have had an impact on soil pH and on mobilization of other soil and/or fertilizer constituents in Experiment A. In addition, greater growth in struvite-treated plants was due to the greater Mg concentration in struvite compared with the mineral fertilizer, which contained large concentrations of silicon and strontium concentrations that may have affected plant growth by inducing phytotoxic effects (Antonini et al., 2012).

In a greenhouse pot experiment in Turkey, Yetilmezsoy and Sapci-Zengin (2009) reported that struvite recovered from poultry manure wastewater significantly increased plant heights and fresh and dry weights. Results showed that struvite obtained an increase by 18% in growth rate, 150% increase in fresh yield and 207% increase in dry yield for purslane (*Purtulaca oleracea*) grown in sand (pH 6.47); 21% in growth rate, 28% in fresh yield, and 115% in dry yield for Garden cress (*Lepidum sativum*) grown in sand (pH 6.47); and 156% in growth rate, 257% in fresh yield and 402% in dry yield for ryegrass grown in garden soil (pH 7.5) compared with the control. The difference in rate of increase was probably due to plant species and type of growing media under study or N (Yetilmezsoy and Sapci- Zengin, 2009). According to Neumann and Römheld (2012), in some plant species, P deficiency may enhance the root secretion of organic acid anions (e.g., carboxylates such as citrate, oxalate, malate, and malonate), thereby increase the solubility of P forms by displacing phosphate anions from

anion sorption sites on the soil matrix.

Additionally, past studies reported that the P concentration was greater in vegetable plants grown with struvite than plants grown with other P fertilizers (Li and Zhao, 2003; Gonzalez-Ponce and Garcia-Lopez-de-Sa, 2007). Li and Zhao (2003) conducted a pot trial evaluating the fertility effect of recovered struvite from a Hong Kong landfill leachate on four vegetables (Water spinach, Chinese flowering cabbage, Chinese chard, and Water convolvulus) grown in a sandy clay (Red Earth). Struvite showed greater vegetable growth rate than the control. Results showed a 77 to 172% increased P uptake with struvite compared with chemical fertilizer [ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ) + calcium phosphate  $\text{Ca}_3(\text{PO}_4)_2$ ]. When one to eight times greater doses of struvite were applied, 98 to 186% more P was taken up by water spinach compared to that from chemical fertilizer. Over-dosing of about two to eight times of struvite in the soil did not cause any problems with the growth of water spinach due to struvite's limited solubility in water. Results also revealed a 205 to 245% increase in Mg uptake with struvite compared with chemical fertilizer.

Gonzalez-Ponce et al. (2009) conducted a three-month greenhouse pot experiment in Spain to compare the effectiveness of struvite recovered from anaerobically digested municipal sludge liquor with single superphosphate (SSP) using lettuce grown in a loamy sand soil. Nitrogen and K were uniformly supplied to all treatments. Results showed greater head fresh weight (approximately 12%) and greater P uptake (5.6%) in lettuce treated with struvite compared to chemical fertilizer at the same P rate. The significant increase in yield and P uptake with lettuce treated with struvite was probably attributed to large amount of Mg incorporated by struvite and a synergistic effect on P uptake (Gonzalez-Ponce et al., 2009). Magnesium is a building block of the chlorophyll molecule, helps in enzyme activation, which

is needed for plant growth, and plays a role in P transport within the plant (Mengel and Kirkby, 2004). There are studies that have reported increasing the Mg level may cause an increase in P absorption by the plants and vice versa (Saleque et al., 2001; Li et al., 2004). In a 53-day pot trial in Germany, Kern et al. (2008) reported 85.9% and 66.7% increase in P uptake for corn and wheat, respectfully, treated with sewage sludge-derived struvite (50 mg P/pot) compared with the control in a mixture of siliceous sand and perlite. Similarly, there have been greater N and K uptake in broad bean, 80.6% N and 95.2% K, with struvite compared with chemical fertilizer (El Diwani et al., 2007).

Compared with MAP, diammonium phosphate (DAP), and single superphosphate, struvite has been reported to produce at least comparable plant P uptake, aboveground dry weight, and yield (Barak and Stafford, 2006; Gonzalez-Ponce et al., 2009; Yetilmezsoy et al., 2013). Barak and Stafford (2006) reported struvite to have greater P uptake efficiency (117%) than chemical fertilizer. Barak and Stafford (2006) conducted a 6-week greenhouse pot experiment to compare the P uptake efficiency of corn amended with laboratory-synthesized struvite and DAP in a Plano silt loam soil in Wisconsin. One rate of struvite (36 mg struvite-P kg<sup>-1</sup>), two rates of DAP (50 and 100 mg DAP-P kg<sup>-1</sup>), and a control with uniform rate of N applied as urea were used. Results showed that corn DM from 36 mg struvite-P kg<sup>-1</sup> was similar to that from 100 mg DAP-P kg<sup>-1</sup> treatment and both outperformed corn DM from 50 mg DAP-P kg<sup>-1</sup> treatment and that from the control. Aboveground tissue P uptake from 36 mg struvite-P kg<sup>-1</sup> struvite did not differ from 42 mg DAP-P kg<sup>-1</sup> and the corn amended with struvite had a P uptake efficiency of 117% compared to DAP.

However, some studies reported lower yield from struvite-treated plants due to lower availability of nutrients compared to chemical-P fertilizer (Ganrot et al., 2007; Ackerman et al.,



2013). In a pot experiment, Ackerman et al. (2013) reported a lower growth of canola (*Brassica napus* L.) grown in a slightly alkaline soil (pH 7.7) with 28% lower biomass yield at a large application rate (47.5 mg P pot<sup>-1</sup>) from struvite recovered from swine manure compared to MAP. Ackerman et al. (2013) recommended a mixture of struvite with other chemical fertilizers, such as Map, to produce improved crop yield.

In a 21-day, climate-chamber pot trial in Sweden, Ganrot et al. (2007) evaluated plant availability of struvite derived from urine with addition of MgO compared with a NPK fertilizer (14-4-21) using spring wheat grown in quartz sand. Ganrot et al. (2007) reported lower nutrient availability in struvite derived from urine with addition of MgO than in NPK fertilizer as observed in the average dry weight of struvite-treated wheat, which was 50% lower than that of NPK-treated wheat. Bonvin et al. (2015) reported lower N uptake (10%) by Italian ryegrass treated with a synthetic, urine-derived struvite treatment compared to chemical fertilizer, which resulted in 34% N uptake.

Additionally, in 30-, 36-, and 90- day pot trials in the UK, Talboys et al. (2016) examined the P release properties of commercial struvite in a sandy loam Eutric Cambisol soil (pH 6.0) by spring wheat, a low organic acid exuder, and *Fagopyrum esculentum* (buckwheat, a high organic acid exuder), compared to more soluble chemical fertilizers (DAP and TSP). In the 30-day pot experiment comparing struvite and DAP for early growth of spring wheat, results revealed lower (70%) P uptake in spring wheat treated with struvite compared to DAP, due to less solubility of struvite in soil (26% of struvite dissolution), whereas no difference occurred between buckwheat treated with struvite compared to DAP. Buckwheat had greater P uptake from struvite due to greater root exudation of organic acids, which increased struvite solubilization and mobilized more struvite P compared to spring wheat, which produced less root exudation of organic acids.

In the 90-day pot experiment comparing struvite and TSP using spring wheat, results revealed that struvite produced similar yield, P uptake, and crop-P recovery at harvest by spring wheat compared to TSP.

In the 36-day pot experiment using several struvite:DAP fertilizer ratios (100:0, 30:70, 20:80, 10:90, and 0:100) in spring wheat, results showed a significant reduction (by 39%) in early plant-P uptake for spring wheat-treated struvite (100:0) compared to DAP (0:100). However, the struvite:DAP ratio of 20:80 had similar early plant-P uptake compared to the struvite:DAP ratio of 0:100 (Talboys et al., 2016).

## Field Studies

Apart from potted plants, orchards, and ornamental plants, struvite has been used for field crops. Few studies have been conducted across the world to determine the agronomic effectiveness of recovered struvite under field conditions. Some studies reported comparable effects of struvite with chemical fertilizers for different plants under field conditions (Pérez et al., 2009; Gell et al., 2011; Thompson, 2013).

In a field trial in Germany, Perez et al. (2009) evaluated the effects of two struvite fertilizers recovered from municipal sewage wastewater [struvite-11 (11% P) and struvite-9.6 (9.6% P)] compared to TSP-60 (60 kg P ha<sup>-1</sup>), TSP-100 (100 kg P ha<sup>-1</sup>), rock phosphate, a heavy-metal-depleted sewage sludge ash (sl-ash), an alkali sinter phosphate (sinter-P), and an unfertilized control using three crops in different P-deficient loamy soils: oilseed rape (*Brassica napus*) in Sattenhausen (pH 5.6), winter wheat in Gieboldehausen (pH 6.8), and winter barley (*Hordeum vulgare*) in Luttenbeck (pH 7.1). Total P concentration in all crops did not differ among fertilizer-P sources. Results also revealed no difference in oilseed rape grain yield and P

uptake between treatments in a Sattenhausen soil. No yield or P uptake data were reported for the Gieboldehausen soil. However, in the Luttenbeck soil, P uptake in winter barley treated with struvite-11 (32.9 kg P ha<sup>-1</sup>) was similar to that with TSP-100 (29.6 kg P ha<sup>-1</sup>), TSP-60 (26.6 kg P ha<sup>-1</sup>), and the unfertilized control (24.3 kg P ha<sup>-1</sup>), but greater than plots with struvite-9.6 (25.3 kg P ha<sup>-1</sup>), sinter-P (24.0 kg P ha<sup>-1</sup>), sl-ash (23.6 kg P ha<sup>-1</sup>), and RP (23.4 kg P ha<sup>-1</sup>). Perez et al. (2009) explained that similar P uptake in the TSP-100 treatment and the unfertilized control may be due to dissolution and availability of native soil P for plant use despite the low soil-test P. Perez et al. (2009) concluded that struvite was comparable to chemical fertilizer.

Similarly, Thompson (2013) conducted a three-year field trial with corn-soybean (*Glyxine max* L.) rotation to evaluate the P availability in corn fiber-processing-derived struvite compared to TSP on three soils [silt loam (Typic Endoaquolls), loam (Aquic Pachic Hapludolls), and silty clay (Typic Endoaquolls)] in Iowa. Treatments used were 0, 12, 24, 36, 48, 72, and 120 kg P ha<sup>-1</sup> of struvite and TSP. Results showed that P uptake was occasionally greater with struvite, but no difference in soybean and corn grown with struvite compared with TSP at similar applied-P rate. Thompson, (2013) concluded that recovered struvite had comparative crop-P availability to inorganic-P fertilizer.

In a 135-day field experiment in the Netherlands, Gell et al. (2011) evaluated the effectiveness of two struvites recovered from black water and human urine as P fertilizer compared with TSP in a sandy loam soil with pH of 4.5 (Typic Andisol). Each fertilizer was applied at 30 and 200 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup> and N was used from urea NH<sub>4</sub>NO<sub>3</sub> (UAN). Results showed that on day 66, halfway through the growth period, struvite recovered from black water and TSP, both at rate 200 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup>, had greater corn growth than the control based on visual inspection. Urine-derived struvite had no improvement on corn growth, compared to TSP and the

control. The explanation was that struvite particles from urine were clumped together and therefore had lower surface area than blackwater-derived struvite, which probably accounted for slower nutrient release from the urine-derived struvite than blackwater-derived struvite and TSP. However, at harvest, there was no difference between dry matter yield (averaging 23000 kg ha<sup>-1</sup>) and P uptake (averaging 70 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup>) across all treatments. Results also showed that blackwater-derived struvite (200 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup>) increased Mg concentrations by 28% in aboveground biomass and by 100% in the soil compared to TSP and the control. Gell et al. (2011) concluded that struvite had similar effectiveness to soluble-P fertilizer, such as TSP.

There are few reports on struvite studies under field conditions compared with the vast number of struvite studies conducted in pot experiments. Based on the available field reports, more research on the effectiveness of struvite under field conditions would contribute to agronomic data in different parts of the world.

### **Wastewater-recovered Struvite Implications for Society and Sustainability**

Wastewater management is a vital issue, with both sanitary and environmental concerns at local and global levels. In developed countries, the implementation of large wastewater treatment plants (WWTPs) has reached its limits since the construction of WWTPs is highly expensive, rigid to population growth, and makes wastewater valorization difficult (Breach and Simonovic, 2018). Conversely, in developing countries, there is a vital issue of unimproved sanitary facilities (Breach and Simonovic, 2018), which could be improved by designing and promoting new treatment technologies and concepts. Globally, 20% of wastewaters receive the right treatment (UNESCO, 2012), although wastewater discharges are projected to increase (Breach and Simonovic, 2018). In this regard, and for sustainable development in different parts

of the world, wastewater treatment must be reconsidered with the mind of adopting technologies that ensure the valorization of nutrients that are contained in wastewaters. Recovery of nutrients in wastewaters as struvite has much more to offer to our society than just the value-added product.

Over the years, struvite scaling or deposits are generally common in WWTP, causing reductions in flow capacities in piping, operational inefficiencies, and the removal of the material is difficult and costly (Doyle et al., 2003; Forrest et al., 2008). Therefore, nutrients recovered as struvite, before struvite forms and accumulates in WWTP infrastructure, is a big relief in the wastewater treatment industry. About 80 to 90% of struvite can be recovered from wastewaters through struvite precipitation processes (Shu et al., 2006; Forrest et al., 2008; Korchef et al., 2011; Petzet and Cornel 2011; Chanan et al., 2013; Xavier et al., 2014; Geerts et al., 2015).

Besides solving the problem of struvite scaling in pipes for the wastewater treatment industry, wastewater-recovered struvite also provides an environmentally sustainable, renewable nutrient source for the agriculture industry. Similar to N, P has no substitute in agricultural production but the diminishing quantities of mined RP is of great concern to future food security (Elser et al., 2007). Research and development into P recovery and recycling aims to solve this problem. Phosphorus recovery would reduce humanities' dependence on the finite, non-renewable resource of phosphate rock from which phosphate fertilizers are made, and ensure food security (Ashley et al., 2011). Development of P-recovery systems can reduce the reliance of crop production on limited remaining quantities of minable rock phosphate, improve the economies of local and limited-resource farmers, and ensure food security and community health (Cordell et al., 2009, 2011). The use of struvite as a fertilizer does not only involve struvite recovery, but also the reuse of nutrients, thus promoting sustainable WWTP management.

Eutrophication is a big environmental issue across the world today. Eutrophication can lead to severe economic, environmental, and human health problems. Reduction in visibility in water bodies and odor problems from eutrophic algal blooms decrease property values in the surrounding area (Dodds et al., 2009). Wastewater treatment plants thus play an important role, as WWTP is one of the main routes of non-diffuse P losses. Phosphorus recovery from WWTP effluent is a way to reduce eutrophication problems (Lee et al., 2007). Recovered struvite would be an eco-friendly, sustainable fertilizer source, which would be useful to lower P loading/runoff losses to surfacewaters to cease eutrophication due to the slow-release behavior of struvite (Rahman et al., 2014). Sustainable P management should therefore focus on P recovery from wastewater.

In addition to the potential benefit of reducing eutrophication, recovery of struvite from wastewaters can cause reduction in offensive odors, which may impact property values in the neighboring areas to eutrophic water bodies, and can reduce human health hazard, originating from toxins released by eutrophic blue-green algae (cyanobacteria). Furthermore, struvite recovery from domestic wastewater could be an opportunity to generate local supplies of P fertilizers for crop production, create job opportunities and improve the economic status of the members of the local communities, and increase biodiversity in waterbodies due to the reduction in the problem of eutrophication. The positive impacts of wastewater-recovered struvite in the society strengthen the economic, social and environmental aspects of sustainability.

Additionally, P loading in waterbodies can reduce the water quality. Water quality degradation can result in increased water scarcity, as polluted waters may be considered unfit for some specific types of human use (Davies and Simonovic, 2011; Liu et al., 2016). Water scarcity and pollution are closely connected to the global food production, population, energy and

economics and may limit the development of the society if left unchecked (Simonovic, 2002). Recovery of P and N as struvite from wastewaters can eliminate the problem of nutrient over-enrichment occurring on local and global scales, and can improve drinking water quality, thereby increasing use of water bodies for recreation. The treated wastewaters can also be used for irrigation in agriculture. The understanding and the optimization of the process of P recovery as struvite will enhance the sustainable development of society. Imagine a future where wastewaters are no longer hazardous materials, but a valuable resource when all wastewaters undergo a treatment process! This will create an eco- friendly, pollution-free environment and enhance a sustainable society.

## **Conclusions**

Struvite can be recovered from several types of wastewaters including sewage sludge, urine, fertilizer plant, anaerobic effluent, swine, landfill leachate, dairy, and agro-industrial wastes. The nutrient composition in recovered struvite varies according to the type of wastewater source and recovery process. Wastewater-recovered struvite, as a P source attractive to the fertilizer market with additional agronomic and environmental benefits has been reported to display excellent fertilizer qualities under specific conditions when compared with standard P fertilizers. Qualities of recovered struvite include the slow release of nutrients due to low solubility, providing available N and Mg, helping attenuate consumption of rock phosphate, low nutrient leaching, and reducing release of P from treated wastewaters to surface and groundwater systems. The recovery of struvite from wastewaters could reduce eutrophication, restore and promote aquatic biodiversity as well as a healthy and sustainable society. Several types of plants have been successfully grown under different soil conditions using struvite with comparatively

greater, similar, or lower growth to various conventional P fertilizers. Recovered struvite could be an effective, eco-friendly, sustainable alternative fertilizer-P source for future food security, though extensive field trials are yet to be undertaken using struvite as a fertilizer across the globe. Further research on struvite application across a wide range of soils and crops under field conditions is needed to determine the agronomic effectiveness of wastewater-recovered struvite in different parts of the world.



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## Tables

Table 1. Summary of different wastewater compositions.

Measured parameters (mg L <sup>-1</sup> )	Nylon industry wastewater (Huanget al., 2012)	Municipal wastewater (Kim et al., 2007)	Swine wastewater (Liu et al., 2011c)
TN <sup>†</sup>	781	800 – 2200	2623.5
TP	0.2	1 – 45	31.6
Mg	0.5	Not reported	Not reported
SO <sub>4</sub> <sup>2-</sup>	389	Not reported	Not reported
COD	1095	1500 – 2500	Not reported
TSS	Not reported	100 – 200	10.7
VSS	Not reported	Not reported	2.2
pH	7.9	7.2 - 8.7	8.3

<sup>†</sup> TN = total nitrogen, TP = total phosphorus, Mg = Magnesium, SO<sub>4</sub><sup>2-</sup> = sulphate, COD = chemical oxygen demand, TSS = total suspended solids, VSS = volatile suspended solids

## **Chapter 2**

# **Wastewater-recycled Struvite as a Phosphorus Source in a Wheat-soybean, Double-crop Production System in Eastern Arkansas**

## Abstract

Phosphorus (P) is a fundamental nutrient for plant growth and is thus commonly applied as a fertilizer to sustain crop production. Majority of fertilizer-P sources are derived from rock phosphate (RP), which is a finite, non-renewable resource, and is actively mined. With growing human populations globally, alternative P sources are vitally important to ensure future food security. Recovery of P as the mineral struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) has been an area of recent interest due to the potential food security risk of conventional RP-derived-fertilizer-P sources. The objective of this study was to evaluate the effects of chemically precipitated struvite (CPST), compared to triple superphosphate (TSP) and an unamended control (UC) treatment, and irrigation (irrigated and non-irrigated/dryland) on soybean [*Glycine max* (L.) Merr.] and wheat (*Triticum aestivum* L.) response in a wheat-soybean, double-crop production system on a silt-loam soil (Aquic Fraglossudalf) in eastern Arkansas. Soybean aboveground and wheat belowground tissue Mg concentrations were 1.1 and 1.2 times, respectively, greater ( $P < 0.05$ ) from CPST than from TSP, while soybean belowground tissue Mg and wheat stem P concentrations were similar between CPST and TSP. Wheat stem Mg and belowground tissue N concentrations were 1.1 and 1.1 times, respectively, greater ( $P < 0.05$ ) from TSP than from CPST. Soybean seed P and Mg concentrations were 1.2 and 1.1 times, respectively, greater ( $P < 0.05$ ) under irrigated than under dryland management. Results of this study substantiate the use of CPST material as a potential alternative fertilizer-P source on a silt-loam soil for food production in eastern Arkansas. Using nutrients in a row-crop agricultural setting that have been recovered from wastewater may offset the need to use energy-intensive commercial fertilizers to supply essential plant nutrients.

## Introduction

Phosphorus (P) is an essential nutrient for plant growth and agricultural production. Following nitrogen (N), P is the most-limiting nutrient in crop production. Rock phosphate (RP) is the most essential, finite resource for the production of synthetic fertilizer-P sources and the major portion of the known reserves are located in Morocco (Cordell, Drangert, & White, 2009). The gradual depletion of the limited RP reserves, which are only located in few countries, is expected to cause an increase in price of RP. (Talboys et al., 2015). The prospect of P depletion ultimately threatens a sustainable global food production. However, an alternative source of P comes from the recovery of the mineral struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) from various wastewaters, which contain P and N (Bouwman, Van Drecht, Knoop, Beusen, & Meinardi, 2005; Metson, Macdonald, Haberman, Nesme, & Bennett, 2016).

Struvite is a white crystalline material that is formed in equal molar concentrations of  $\text{Mg}^{2+}$ ,  $\text{NH}_4^+$ , and  $\text{PO}_4^{3-}$  combined with six water molecules (Johnston and Richards, 2003; Schoumans, Bouraoui, Kabbe, Oenema, & van Dijk, 2015). The recovery of struvite from wastewater is a new trend because of the reduction in the potential risk of surface water eutrophication of the receiving water bodies and meeting the stringent P removal requirements for wastewater disposal, which have become stricter in recent years (De-Bashan and Bashan, 2004). Over the years, the spontaneous deposition of struvite has been recorded in pipes of many wastewater treatment plants (WWTPs), which can be problematic (Doyle, Oldring, Churchley, Price, & Parsons, 2003; De-Bashan and Bashan, 2004). Therefore, the recovery of struvite at the right place in WWTP processes has become an attractive option and can reduce operating costs by improving sludge dewatering, reducing biosolids volume, and preventing unwanted deposits in pipes (Parsons, Wall, Doyle, Oldring, & Churchley, 2001).

Different methods have been used to recover nutrients from wastewater such as biological, chemical, and electrochemical precipitation (De-Bashan and Bashan, 2004; Huang et al., 2016). In particular, chemical P recovery technology can recover 10 to 80% of P in wastewaters (De-Bashan and Bashan, 2004). In addition to reducing the P load to surface receiving waters after being processed in a WWTP, struvite has been shown to be a slow-release fertilizer that could provide a longer-term source of P than P fertilizers derived from RP. The slow-release characteristic of struvite may be more beneficial for plant growth than more readily soluble fertilizer-P sources, such as triple superphosphate (TSP), monoammonium phosphate (MAP), and diammonium phosphate (DAP), thus limiting fixation on soil particles, more closely matching the timing of plants' P need later in the growing season, and improving P-uptake efficiency by plants (Withers, Sylvester-Bradley, Jones, Healey, & Talboys, 2014; Talboys et al., 2015).

Many studies have shown struvite to exhibit comparable plant growth or P uptake to other commercially available, fertilizer-P sources (Thompson, 2013; Massey, Davis, Ippolito, & Sheffield, 2009; Kern et al., 2008). Thompson (2013) conducted a three-year field trial with a corn (*Zea mays* L.)-soybean (*Glycine max* L.) rotation to evaluate P availability in corn-fiber-processing-derived struvite compared to TSP in three soils [silt loam (Typic Endoaquolls), loam (Aquic Pachic Hapludolls), and silty clay (Typic Endoaquolls)] in Iowa. Treatments used were 0, 12, 24, 36, 48, 72, and 120 kg P ha<sup>-1</sup> of struvite and TSP (Thompson, 2013). Results showed that P uptake was similar, and occasionally greater with struvite, and there was no difference in soybean or corn grown with struvite compared with TSP at a similar applied-P rate, thus it was concluded that recovered struvite had at least comparable crop-growth performance to TSP (Thompson, 2013). Similar to Thompson (2013), a 90-day greenhouse study conducted in the US



by Massey et al. (2009) evaluated struvite, TSP, organic rock phosphate, dittmarite ( $\text{MgNH}_4\text{PO}_4 \cdot \text{H}_2\text{O}$ ), and ‘Colorado product’, which consisted of ground carbonate fluorapatite seed crystals, small amounts of recovered magnesium phosphate, and sand grains, fertilization on hard red spring wheat (*Triticum aestivum* L.) grown in a loamy Argiustoll with different soil pH levels (6.5 and 7.6). Results showed struvite was comparable to other P sources for dry matter (DM) production and tissue-P concentration, indicating that struvite could be an effective source of P in both acidic and alkaline soils. Massey et al. (2009) concluded that struvite could be a useful alternative fertilizer-P source in arid and semi-arid environments. In a 53-day pot trial in Germany, Kern et al. (2008) reported 86 and 67% increase in P uptake for corn and wheat, respectively, treated with sewage-sludge-derived struvite compared with the unamended control in a mixture of siliceous sand and perlite.

In contrast, some studies reported a reduction in agronomic effectiveness as a result of struvite fertilization (Talboys et al., 2015; Everaert, Da Silva, Degryse, McLaughlin, & Smolders, 2017). Talboys et al. (2015) evaluated buckwheat (*Fagopyrum esculentum*) and spring wheat grown in a sandy loam (Eutric Cambisol) in a 30-day, potted-plant study with fertilizer applications of struvite (Crystal Green) and DAP. Result showed that struvite had 70% lower P uptake compared to DAP in spring wheat, whereas no difference occurred for buckwheat amended with either fertilizer-P source (Talboys et al., 2015). Additionally, Everaert et al. (2017) assessed granular struvite (Crystal Green), monoammonium phosphate (MAP), and P-exchanged, layered double hydroxides in spring wheat grown in acid and alkaline soils, where results of the pot experiment showed a lower plant-P uptake and dry matter yield, thus lower agronomic effectiveness, from granular struvite compared to granular MAP in both soils. However, despite the somewhat negative agronomic results, Everaert et al. (2017) conclude that

the environmental impacts and potential residual value could still make struvite an attractive fertilizer-P-source option for P fertilization of some row crops.

Globally, soybean is the leading oilseed crop produced and consumed (Wilcox, 2004). In the United States (US) alone, soybean accounted for about 90% of the total national oilseed production in 2019 (USDA-ERS, 2020). Over 36 million hectares of soybean were planted in the United States in 2018, making soybean the most planted crop in the US for the first time since 1983 (USDA-ERS, 2020). Although the majority of the land planted to soybean in the US is concentrated in the upper Midwest, a large concentration of soybean production occurs in the lower Mississippi River delta region, namely in Missouri, Tennessee, Arkansas, Louisiana, and Mississippi. In 2019, soybean ranked second among the top agricultural commodities in Arkansas and accounted for 14% of Arkansas' total farm receipts (USDA-ERS, 2020). Most of Arkansas' row-crop land is in the eastern part of the state, principally in the Mississippi River Alluvial Plain (USDA-NASS, 2020).

For soybean, large yields are directly related to large nutrient uptake (Tamagno et al., 2017), with the N demand provided by a combination of biological N fixation and soil-N supply (Tamagno et al., 2017). In addition, P is critical for root and seed development and growth and is a major component of a plant's energy supply in the form of adenosine triphosphate (ATP). Soybean plants need P during vegetative growth early in the season, but the demand for P is greatest during pod and seed development, where more than 60% of P ends up in the pods and seeds (Usherwood, 1998). For each 67 kg of soybean seed harvested per hectare, approximately 1.1 kg ha<sup>-1</sup> of P<sub>2</sub>O<sub>5</sub> are removed from the soil (Slaton, Roberts, & Ross, 2013), thus soil P must be replenished periodically. To assure adequate soybean yields, most producers also irrigate when needed during the growing season (Bajaj et al., 2008). In contrast, when water is

unavailable or the implementation of irrigation is too costly, producers will practice dryland production, in which the only source of water to the crop is rainfall. However, the lack of available water or water-stressed conditions can limit plant biomass production and productivity or reduce yield from extended dry conditions. Consequently, irrigation is essential to producing adequate yields to recoup economic investments, particularly in a wheat-soybean, double-crop system.

Similar to soybean, wheat is another major row crop grown in the US, and in Arkansas, where most of the wheat is winter wheat. Phosphorus increases wheat seedling vigor and critical for proper tiller formation and development. Phosphorus fertilizer is best applied shortly before or after wheat is planted and preferably no later than Feekes stage 3, before tiller formation (Roberts and Slaton, 2014). Wheat grain removes about 80% of the aboveground plant-P, while the remainder of the P is contained in the wheat straw (Roberts and Slaton, 2014).

Double-cropping wheat after soybean is a common production system in the lower Mississippi River delta region, particularly in Arkansas (USDA-NASS, 2020). In eastern Arkansas, the common agronomic management practices that producers adopt for the wheat-soybean, double-crop production system consist of N fertilization usually applied in the early spring for optimal wheat production followed by residue burning and conventional tillage after wheat harvest with furrow irrigation of the subsequent soybean crop as needed throughout the growing season (Brye, Quarta, Morrison, & Rothrock, 2018). One of the benefits of legume crops, such as soybean, in a double-crop production system is that soybean can provide additional soil N from N fixation (Salvagiotti et al., 2008), thus often reducing the necessary N fertilization rate for a successive crop. Double-crop systems also provide pest control benefits in addition to increased revenue from the winter cash crop, which could serve as a second annual

cash crop (Kyei-Boahen and Shang, 2006; Thomason et al., 2017). Another important agronomic characteristic of eastern Arkansas is the substantial nutrient, namely P, imbalance that exists throughout much of the region (Slaton et al., 2004), which further substantiates the importance of P fertilization for optimal crop productivity.

Although a few row-crop field trials have been conducted, most studies have focused on struvite application as a fertilizer-P source in greenhouse pot studies. Furthermore, no field studies in the mid-southern US have examined the agronomic effectiveness of wastewater-recovered struvite as a fertilizer-P source in any row crop. Therefore, the objective of this study was to evaluate the effects of fertilizer-P source [chemically precipitated struvite (CPST) and TSP] and irrigation (irrigated and non-irrigated) on above- (dry matter, yield, and N, P and Mg tissue concentrations and uptake) and belowground (root tissue N, P, and Mg concentrations) soybean and wheat properties over two years in a wheat-soybean, double-crop production system on a silt-loam soil in eastern Arkansas.

It was hypothesized that soybean amended with CPST would have similar or greater, aboveground dry matter and greater aboveground and belowground tissue P and N concentrations, aboveground P and N uptake, but greater aboveground and belowground tissue Mg concentrations than P fertilization with the TSP. Yield, aboveground dry matter, aboveground nutrient uptake, aboveground and belowground tissue P, N, and Mg concentrations of soybean were also expected to be greater under irrigated than under dryland production. In addition, it was hypothesized that soybean grown in year one would have similar yield, aboveground dry matter, aboveground nutrient uptake, aboveground and belowground tissue P, N, and Mg concentrations compared to that in year two. It was hypothesized that there would be no difference in aboveground and total wheat dry matter or wheat yield when amended with

CPST compared to TSP. Aboveground and belowground wheat tissue P and N concentrations, and aboveground wheat P and N uptake, were also expected to be greater from TSP than from CPST fertilization, while aboveground and belowground wheat tissue Mg concentrations were expected to be greater from CPST than from TSP fertilization because of the greater Mg concentration in the chemical composition of CPST.

## **Materials and Methods**

### **Site Description and History**

The current field study was initiated in June 2018 at the Lon Mann Cotton Branch Experiment Station (CBES) near Marianna, AR (34°44'1.40"N; 90°45'48.23"W). The study site was located in major land resource area (MLRA) 134, Southern Mississippi Valley Loess (Brye, Mersiovsky, Hernandez, & Ward, 2013). The soil throughout the 0.56-ha study area was mapped as a Calloway silt loam (fine-silty, mixed, active, thermic Aquic Fraglossudalfs; Soil Survey Staff, 2015). Calloway soils have a surface layer of dark brown to brown silt loam with a subsoil of light brownish-gray silt loam and are derived from loess parent materials (NRCS, 2020). The top 10 cm of the soil profile is silt-loam textured and comprised of 16% sand, 73% silt, and 11% clay (Brye, Cordell, Longer, & Gbur, 2006).

The current field study was established in two large, interior border areas of a long-term, wheat-soybean, double-crop field study that was initiated in Fall 2001 (Figure 1). For the six years prior to Fall 2001, the cropping system was a conventionally tilled soybean monoculture (Cordell, Brye, Longer, & Gbur, 2007). The long-term study was established to examine the effects of several field treatment combinations, including wheat residue levels (high and low, achieved with differential N fertilization), wheat residue burning and non-burning, and

conventional tillage and no-tillage, on long-term trends in soybean growth and productivity and near-surface soil properties.

Starting in November 2001, and for every fall thereafter usually between late October and mid-November, a ‘Coker’ wheat variety was drill-seeded throughout the study area with a 19-cm row spacing at a rate of 168 kg seed ha<sup>-1</sup> (Norman, Brye, Gbur, Chen, & Rupe, 2016; Brye et al., 2018). Each year, wheat was harvested in late May to early June. For safety purposes, four tiers of 12 plots each associated with the long-term study were separated with ~ 12-m-wide alley ways to prevent the fire from escaping control and burning unintended areas when imposing the residue-burning treatment each year. The burn alleys were always conventionally tilled by multiple passes with a tandem disc each year immediately after wheat harvest to serve as the fire break.

Between 2002 and 2013, a glyphosate-resistant, maturity group 4 to 5, soybean cultivar was drill-seeded throughout the whole study area with 19-cm row spacing by early to late-June each year. Between 2014 and 2016, a Liberty-Link, maturity group 4 to 5, ‘Armor’ soybean cultivar was drill-seeded throughout the whole study area to mitigate large weed pressure that had developed throughout the study area. The whole study area was uniformly tractor-sprayed with herbicides usually twice after soybean had been established each year to control weeds. For the first three soybean growing seasons, soybeans in the whole study area were grown with periodic furrow irrigation. From 2005 on, the study area was split into two halves, one half maintaining furrow irrigation, while the other half of the study area was converted to dryland soybean production with no additional irrigation (Brye et al., 2018).

The current field study, initiated in 2018, was consequently established in the large burn alleys of the long-term study, which had been consistently managed since 2001 with

conventional tillage, no residue burning, no N, P, or K fertilization, and with the same wheat and soybean cultivars planted, managed, and harvested each year as were used for the long-term study. The current study had 12 plots established in a burn alley that received furrow irrigation since 2002, while 12 plots were established in a burn alley that received furrow irrigation from 2002 through 2004, then no irrigation (i.e., dryland) from 2005 thereafter (Figure 1).

The climate in the region encompassing the study area is humid temperate, with the 30-year (i.e., 1981 to 2010) average annual air temperature and precipitation of 16.6°C and 128.4 cm, respectively (NOAA, 2020). The 30-year mean monthly minimum and maximum air temperatures in the area are 0.6°C in January and 32.3°C in July, respectively (NOAA, 2020).

### **Field Treatments and Experimental Layout**

The current field study consisted of two experimental factors, irrigation and fertilizer-P source. Out of practical necessity, each irrigation treatment (i.e., furrow-irrigated and dryland) was a strip across different parts of the study area, separated by a levee that was established each year, and was replicated twice with adjacent strips (Figure 1). The fertilizer-P source factor, which consisted of three treatments, CPST, TSP, and an unamended control (UC), was replicated twice within each adjacent irrigation strip (Figure 1). The CPST fertilizer-P source was created by chemical precipitation of raw wastewater from a wastewater treatment plant near Atlanta, GA, which is marketed and sold under the trade name Crystal Green by Ostara Nutrient Recovery Technologies, Inc. (Vancouver, Canada).

## Fertilizer-P Source Characterization

Chemical analyses were conducted on five replications of pelletized CPST and TSP. The pH of each fertilizer material was measured in a 1:2 (mass/volume) fertilizer-to-water-ratio paste. Total N concentration was determined by high-temperature combustion using a VarioMax CN analyzer (Elementar Americas, Inc., Mt. Laurel, NJ). A strong-acid digest was conducted (USEPA, 1996) and extracts were analyzed by inductively coupled, argon-plasma spectrometry (ICAPS) to determine total-recoverable P and Mg. Table 1 summarizes the measured chemical properties of the two fertilizer materials.

## Plot Management

In 2018, new plots were established in the burn alleys of the long-term, double-crop study, where P had not been applied since prior to Fall 2001. A total of 24 field plots (Figure 1), 6.1-m long by 3.1-m wide, were established after conventional tillage, which consisted of three passes with a tandem disc to a 5- to 10-cm depth followed by three passes with a field cultivator to break up soil clods and soften the seed bed. On 9 June, 2018, plots were drill-seeded with the Progeny 5414 LLS, a LibertyLink, maturity group 5.4, soybean variety at a rate of 101 kg seed ha<sup>-1</sup> (i.e., ~ 370,650 seed ha<sup>-1</sup>; Ross, Elkins, & Norton, 2021) with 19-cm row spacing and approximately 10 cm between plants in a row. Dual II Magnum (1.17 L ha<sup>-1</sup>; Syngenta, Greensboro, NC; 2-chloro-N-(2-ethyl-6-methylphenyl)-N-[(2S)-1-methoxypropan-2-yl] acetamide) and the Liberty (2.3 L ha<sup>-1</sup>; Bayer CropScience, Research Triangle Park, NC; azanium, 2-amino-4-[hydroxy(methyl)phosphoryl] butanoate) herbicides were tractor-sprayer-applied twice after soybean planting to control weeds, such as Palmer amaranth (*Amaranthus palmeri* S.) and perennial ryegrass (*Lolium perenne* L.). Four days after soybean planting in 2018



(13 June), fertilizer P in the form of CPST and TSP was manually applied at 44 kg P ha<sup>-1</sup> and incorporated by light, manual raking. Bifenthrin (0.37 L ha<sup>-1</sup>; Control Solutions Inc, Pasadena, TX; (2-methyl-3-phenylphenyl)methyl 3-[(Z)-2-chloro-3,3,3-trifluoroprop-1-enyl]-2,2-dimethylcyclopropane-1-carboxylate) was tractor-sprayer-applied once during the soybean growing season to control insects. Twelve plots were temporarily flood-irrigated on a flat surface as needed approximately three times each year for the soybean crop, while the other 12 plots were non-irrigated (i.e., dryland soybean production). Soybeans were harvested with a plot combine on 30 October, 2018. All soybean seed harvested from a 1.9-m width by 5.7-m length of plot was collected and bagged.

Following soybean harvest, without any additional field manipulations, winter wheat (USG 3895) was drill-seeded on 20 November, 2018 at 168.1 kg seed ha<sup>-1</sup> with 19-cm row spacing. On 23 November, 2018, Finesse (0.035 kg ha<sup>-1</sup>; DuPont, Wilmington, DE; 1-(2-chlorophenyl)sulfonyl-3-(4-methoxy-6-methyl-1,3,5-triazin-2-yl)-urea) and RoundUp (3.5 L ha<sup>-1</sup>; Bayer CropScience; 2-(phosphonomethylamino) acetate, propan-2-ylazanium) were sprayed once for weed control. The winter wheat crop was rain-fed only without irrigation. Fertilizer P in the form of CPST and TSP was manually surface-applied 8 days after planting (28 November) at 44 kg P ha<sup>-1</sup> and incorporated by light, manual raking. On 27 February, 2019, Axial (1.17 L ha<sup>-1</sup>; Syngenta, Greensboro, NC; [8-(2,6-diethyl-4-methylphenyl)-7-oxo-1,2,4,5-tetrahydropyrazolo [1,2-d] [1,4,5] oxadiazepin-9-yl] 2,2-dimethylpropanoate) and on 20 March, 2019 Harmony Extra (1.75 L ha<sup>-1</sup>; DuPont, Wilmington, DE; methyl 3-[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)carbamoylsulfamoyl]thiophene-2-carboxylate;methyl 2-[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)-methylcarbamoyl]sulfamoyl]benzoate) were sprayed twice for weed control. Nitrogen, in the form of uncoated urea (460 g N kg<sup>-1</sup>), was hand-applied at 56 kg N ha<sup>-1</sup> on 17 March, 2019

and an additional split application was hand-applied at 56 kg N ha<sup>-1</sup> approximately one month later on 12 April, 2019 to all 24 plots. Due to a severe herbicide-resistant ryegrass infestation, wheat could not be combine-harvested at maturity in 2019. After individual wheat plant samples were collected, as described below, wheat and ryegrass in all plots were mowed with a rotary mower and conventionally tilled with five passes of a tandem disk to a 5- to 10-cm depth followed by five passes with a field cultivator to prepare the plots for subsequent soybean planting.

Following wheat termination, mowing, and tillage, soybean (Pioneer 46A70L) were drill-seeded on 12 June, 2019 at a rate of 134.5 kg seed ha<sup>-1</sup> (~ 370,650 seed ha<sup>-1</sup>; Ross et al., 2021) with 19-cm row spacing and approximately 10 cm between plants in a row. Liberty (2.3 L ha<sup>-1</sup>) was tractor-sprayer-applied twice (14 June, and 13 July, 2019) and Dual II Magnum (1.17 L ha<sup>-1</sup>) was sprayed once to control weeds two days after soybean planting. Fertilizer P was manually applied to the same plots as in 2018 at 44 kg P ha<sup>-1</sup> again and incorporated by light, manual raking on 13 June, 2019. Intrepid Edge (0.37 L ha<sup>-1</sup>; Dow AgroSciences, Indianapolis, IN; N'-tert-butyl-N'-(3,5-dimethylbenzoyl)-3-methoxy-2-methylbenzohydrazide) and Acephate 97UP (1.12 kg ha<sup>-1</sup>; United Phosphorus Inc., King of Prussia, PA; O,S-dimethyl acetyl phosphoramidothioate) were tractor-sprayer-applied on 29 August, 2019 to control insects. Soybeans were harvested with a plot combine on 23 October, 2019. All soybean seed harvested from a 1.9-m width by 5.5-m length of plot was collected and bagged.

Soybean seed samples from 2018 and 2019 were air-dried for 14 days at approximately 25°C. A sub-sample of air-dried seed from each plot was oven-dried for 48 hr at 70°C to determine oven-dried soybean seed yield per plot, which was subsequently adjusted to 130 g kg<sup>-1</sup> (13%) moisture for yield reporting each year.

## **Soil Sampling, Processing, and Analyses**

On 20 April, 2018, prior to soybean planting, soil samples were collected in all plots from the top 10 cm. Samples were oven-dried at 70°C for 48 hours, mechanically crushed, and sieved through a 2-mm mesh screen. Soil pH and electrical conductivity (EC) were measured potentiometrically in a 1:2 (mass/volume) soil-to-water-ratio paste (Brye, West, & Gbur, 2004; Sikora and Kissel, 2014). Weight-loss-ignition was used to determine soil organic matter (SOM) concentration using a muffle furnace at 360°C for 2 hr (Zhang and Wang, 2014). Total nitrogen (TN) and total carbon (TC) concentrations were determined by high-temperature combustion using a VarioMax CN analyzer (Provin, 2014). A Mehlich-3 extraction (Zhang et al., 2014) was also conducted with a 1:10 (mass:volume) soil:extractant solution ratio to determine extractable nutrient (i.e., P, K, Ca, Mg, S, Na, Fe, Mn, Zn, Cu, and B) concentrations by ICAPS (Soltanpour, Johnson, Workman, Jones, & Miller, 1996).

## **Fertilizer-P Application Rate Determination**

The amount of fertilizer-P material applied to both soybean and wheat crops, in the forms of CPST and TSP, was based on: i) the initial Mehlich-3 soil-test-P concentration prior to planting the first of two consecutive, summer soybean crops, ii) the recommended fertilizer-P rate for soybean (44 kg P ha<sup>-1</sup>; Slaton et al., 2013) and wheat (44 kg P ha<sup>-1</sup>; Roberts and Slaton, 2014) production on a silt-loam soil in Arkansas, and the measured, total-recoverable P concentrations of the two fertilizer materials (Table 1). Based on soil sampling on 20 April, 2018, after plot establishment, but before soybean planting, Mehlich-3-extractable soil P averaged 24.8 mg kg<sup>-1</sup> [standard error (SE) = 0.7] in the top 10 cm, which was in the low

category for irrigated soybean production on a silt-loam soil in eastern Arkansas (Slaton et al., 2013; Roberts and Slaton, 2014), thus a plant response from added fertilizer P from both soybean and wheat was expected.

### **Plant Sampling, Processing, and Analyses**

On 15 October, 2018 and 13 October, 2019, when soybeans reached reproductive stage 7, initial maturity (Popp, Purcell, & Salmerón, 2016), three soybean plants were randomly collected per plot for above- and belowground nutrient assessment. Each plant was carefully dug out with a shovel to a depth of approximately 15 cm. The qualitatively sampled plant roots were vigorously shaken and rinsed in tap water to remove attached soil particles. Aboveground biomass was separated from the roots by cutting each plant at the point on the stem where the soil surface was at. The three above- and belowground replications were combined for one sample per plot. In 2018 and 2019, above- and belowground soybean tissue samples were oven-dried at approximately 55°C for 7 days. Dry matter per unit area was calculated only for the total aboveground plant biomass based on the combined area represented by the three individual sampled plants.

Sub-samples of above- and belowground dry matter were mechanically ground and passed through a 2-mm mesh screen for chemical analyses. A sub-sample of oven-dried seed from the 2019 soybean harvest was also mechanically pulverized for chemical analyses. Total N TC concentrations were measured by high-temperature combustion. A strong-acid digestion was conducted, and extracts were analyzed by ICAPS (Soltanpour et al., 1996) to determine total tissue P and Mg concentrations. Total P, N, and Mg concentrations and aboveground dry matter were used to calculate P, N, and Mg uptake ( $\text{kg ha}^{-1}$ ).

On 4 June, 2019, at physiological maturity, eight random wheat plants were manually collected per plot for above- and belowground nutrient assessment using the same sampling procedures as described above for soybean. The aboveground wheat samples were further separated into heads and stems. Wheat root, stem, and head samples were oven-dried at approximately 55°C for 7 days. Wheat heads and stems were weighed to determine head and stem dry matter, respectively. Total aboveground wheat dry matter was obtained by the addition of head and stem dry matter. Sub-samples of dried roots, stems, and heads were mechanically ground and passed through a 2-mm mesh screen for chemical analyses. Total TN and total P and Mg were determined using the same procedures as described above for soybean tissue samples. Head and stem P, N and Mg uptake were calculated from the head and stem dry matter and the respective total tissue nutrient (P, N and Mg) concentrations. Since a combine-harvest could not be performed in 2019 for wheat, an estimate of wheat yield on an area basis was calculated from the individual plant samples collected. To estimate wheat seed yield, seeds were assumed to constitute 90% (Dr. R.E. Mason, personal communication, 11 December 2019) of the wheat head mass. The wheat head dry mass of the eight wheat samples collected was multiplied by the fraction of the seed-to-wheat-head mass fraction (0.90) and divided by area represented by the eight individual wheat plants that were collected in the field, where the result was scaled to kg ha<sup>-1</sup> and reported at a grain moisture content of 120 g kg<sup>-1</sup> (12%) moisture.

### **Statistical Analyses**

Based on a split-plot experimental design, the effects of fertilizer-P treatment (CPST, TSP, and UC), irrigation (furrow-irrigated and dryland/non-irrigated), and their interaction on

initial soil properties (soil pH and EC; Mehlich-3 extractable soil P, K, Ca, Mg, S, Na, Fe, Mn, Zn, Cu, and B; and SOM, TC, and TN concentrations) prior to any fertilizer-P addition were evaluated by a two-factor analysis of variance (ANOVA) in SAS (version 9.4, SAS Institute, Inc., Cary, NC) using the PROC GLIMMIX procedure. The whole-plot factor was irrigation and the split-plot factor was fertilizer-P treatment. A gamma distribution was used for soil pH, EC, and Mehlich-3 extractable soil P, K, Ca, Mg, S, Na, Fe, Mn, Zn, Cu, and B concentration data, while a beta distribution was used for SOM, TC, and TN concentration data because these properties were expressed as percentages.

Based on a split-split-plot experimental design, a three-factor ANOVA was conducted using the PROC GLIMMIX procedure in SAS to evaluate the effects of year (2018 and 2019), irrigation, fertilizer-P treatment, and their interactions on soybean aboveground dry matter (DM), above- and belowground N, P, and Mg tissue concentrations, aboveground N, P, and Mg tissue uptake, and yield. The whole-plot factor was year, the split-plot factor was irrigation, and the split-split-plot factor was fertilizer-P treatment. A gamma distribution was used for the aboveground soybean DM, aboveground N, P, and Mg tissue uptake, and yield data, while a beta distribution was used for the above- and belowground N, P, and Mg tissue concentration data because these concentrations were expressed as percentages.

Based on a split-plot experimental design, a two-factor ANOVA was conducted in SAS using the PROC GLIMMIX procedure to evaluate the effects of irrigation, fertilizer-P treatment, and their interaction on seed N, P, and Mg concentration and uptake from the 2019 soybean seed samples. The whole-plot factor was irrigation and the split-plot factor was fertilizer-P treatment. A beta distribution was used for seed N, P, and Mg concentration data, while a gamma distribution was used for seed N, P, and Mg uptake data.

Based on a completely random design, the effects of fertilizer-P treatment on wheat head, stem, and total DM, seed-head and stem N, P, and Mg concentration and uptake, belowground N, P, and Mg tissue concentrations, and estimated wheat yield were evaluated by a one-factor ANOVA in SAS using the PROC GLIMMIX procedure. A gamma distribution was used for head, stem, and total DM, seed-head and stem N, P, and Mg uptake, and yield data, while a beta distribution was used for seed-head and stem N, P, and Mg concentration and belowground N, P, and Mg tissue concentration data. For all analyses, significance was judged at  $P < 0.05$ . When appropriate, means from were separated by least significant difference at the 0.05 level.

## **Results and Discussion**

### **Initial Soil Properties**

Since the current study was conducted in border areas of a long-term field study that began in 2001, but was consistently annually managed, it was still necessary to assess the degree of soil property uniformity prior to imposing any fertilizer-P treatments. It was expected that there was little to no inherent spatial soil property variation attributable to the pre-assigned fertilizer-P treatments on account of the consistent annual management of the larger studies' border areas. However, the annual irrigation treatment for the summer soybean crop had been imposed annually since 2005, thus some soil property variation due to the differential irrigation methods was expected.

As expected, no measured soil property in the top 10 cm differed among pre-assigned fertilizer-P treatments ( $P > 0.05$ ; Table 2). Consequently, the near-surface soil properties among all plots that received the fertilizer-P treatments were considered uniform in Year 1 of this study (2018). In contrast, initial soil pH, extractable soil K, Mg, Na, and B differed between irrigation

treatments ( $P < 0.01$ ), while EC, extractable soil P, Ca, S, Fe, Mn, Zn, and Cu, and TN, TC, and SOM concentrations were unaffected by irrigation treatment ( $P > 0.05$ ; Table 2). Initial soil pH was 1.1 times greater in the irrigated than in the slightly more acidic dryland area (Table 3). Similar to soil pH, initial extractable soil Mg, Na, and B were 1.2, 1.4, and 0.1 times, respectively, greater in the irrigated than in the dryland area (Table 3). However, initial soil K was 1.3 times lower in the irrigated than in the dryland area (Table 3). Alkaline groundwater (Amuri, Brye, Gbur, Popp, & Chen, 2008) was used for annual furrow-irrigation of the summer soybean crop, thus causing the soil became enriched with base-forming cations, such as Mg and Na, in the irrigated area, resulting in the more alkaline pH in the top 10 cm. Lower initial soil K under irrigated conditions was likely due to greater leaching as a result of increased available water from furrow-irrigation compared to only being rain-fed under dryland conditions. Table 3 also summarizes the mean magnitudes for all initial soil properties that were unaffected by irrigation and fertilizer-P treatments. With no differences in initial soil properties in the top 10 cm among pre-assigned fertilizer-P treatments, any subsequent measured plant response differences were assumed to have been the result of the actual fertilizer-P treatments rather than any inherent differences that existed prior to fertilizer-P additions.

### Soybean Response

Despite an expected yield response from fertilizer-P treatment because the initial soil-test P concentration was low for optimal soybean production (Slaton et al., 2013), soybean aboveground DM and seed yield were unaffected by fertilizer-P treatment and irrigation ( $P > 0.05$ ), but differed between years ( $P < 0.01$ ; Table 4). Soybean aboveground DM and seed yield were unaffected by fertilizer-P treatment likely due to the soil's large P-adsorption capacity such



that the P released from both fast-dissolving TSP and slow-dissolving CPST was similarly unavailable for plant uptake as a result of P adsorption to clays and/or secondary mineral formation by precipitation with Fe, which was also present in large concentrations in the top 10 cm (Table 3), at the slightly acidic soil pH throughout the study area (Table 3). Results of this study were similar to that reported by Ylagan, Brye, & Greenlee (2020) in a 79-day greenhouse pot study, where soybean aboveground DM and yield were reported to be unaffected by eight fertilizer-P treatments including CPST, TSP, and No P/-N (control) in a Captina silt loam (Typic Fragiudults) with an optimum soil-test P for soybean. In a similar study, but using corn, Ylagan et al. (2020) also reported corn aboveground DM from TSP to be similar to that from CPST and corn aboveground DM from both fertilizers was at least 1.6 times greater than that from control.

Averaged across irrigation and fertilizer-P treatments, soybean aboveground DM was 1.3 times greater in 2018 than in 2019 (Table 5). However, soybean seed yield was 1.4 times greater in 2019 than in 2018 when averaged across irrigation and fertilizer-P treatments (Table 5). Soybean seed yield response in 2019 was likely due to differences in precipitation, particularly earlier in the growing season in June and July 2019, which were more than 2.3 and 5.3 times, respectively, and 2.1 times greater in October 2019 than in similar months in 2018 (Figure 2A). Increased rainfall in 2019 likely reduced soil pH, which promoted nutrient dissolution and availability earlier to set yield limits. However, the decreased rainfall in August and September 2019, which represented 97 and 5% of the rainfall in the same months in 2018, likely caused reduction in soybean aboveground DM in 2019 (Figure 2A). In September 2019, soybeans were at the R6 growth stage with full seed and an already set yield limit, thus the dramatic relative reduction in rainfall may have only affected aboveground DM (Figure 2A). In addition, the cumulative effects of the three P fertilization events for soybean and wheat in 2018 and soybean

in 2019 cannot be underestimated, as there was likely carry-over P from 2018 that had not become plant available in 2018 or early 2019 that benefited soybean yield in 2019. However, differences in soybean yield may also be attributed to the different cultivars grown each year, as soybean yields are closely related to genetic potential (Scaboo, Chen, Sleper, & Clark, 2010).

Similar to DM and yield, soybean aboveground tissue P and N concentrations were unaffected by fertilizer-P treatment and irrigation ( $P > 0.05$ ), but differed between years ( $P < 0.03$ ; Table 4). Averaged across irrigation and fertilizer-P treatments, soybean aboveground tissue P and N concentrations were 1.3 and 1.1 times, respectively greater in 2019 than in 2018 (Table 5). Total rainfall during the five-month soybean growing season in 2019 was more than 1.3 and 1.8 times greater than that in 2018 and 30-year-mean rainfall, respectively (Figure 2A). In August and September 2018 and 2019, soybeans were at reproductive stage 3 (beginning pod) to reproductive stage 6 (full seed), the period which most of the P is directed into the seeds because the demand for P is greatest during pod and seed development (Usherwood, 1998). Mean air temperature in August and September 2019 were slightly greater than similar months in 2018 (Figure 2A). The warmer air temperature coupled with above-normal precipitation in 2019 likely created more ideal conditions promoting dissolution of adsorbed P, microbiological mineralization of soil organic matter, which released some plant-available P and N, and facilitated greater P diffusion, thus resulting in greater aboveground tissue P and N concentrations in 2019 than in 2018.

Similar to the current result, Ylagan et al. (2020) reported no difference in corn stem-plus-leaves tissue P concentration between CPST, TSP, and an unamended control. However, soybean stem-plus-leaves tissue P concentration was similar between CPST and TSP, in which both were more than 1.3 times greater than that from the unamended control (Ylagan et al.,

2020). Corn stem-plus-leaves tissue N concentration was also reported to be more than 1.2 and 3.2 times greater from CPST than from TSP and the unamended control, respectively (Ylagan et al., 2020).

In contrast to P and N, soybean aboveground tissue Mg concentration differed ( $P < 0.01$ ) among fertilizer-P treatments, but was unaffected by irrigation and year ( $P > 0.05$ ; Table 4). Averaged across irrigation treatments and years, soybean aboveground tissue Mg concentration was 1.1 times greater from CPST than from TSP or the unamended control, which did not differ (Table 6). The greater aboveground tissue Mg concentration from CPST was expected due to the composition and dissolution of the struvite, with an initial Mg concentration more than 13.8 times greater than that in TSP (Table 1). The slower dissolution of CPST likely kept the Mg closer to the actively growing soybean root zone longer for greater plant availability.

In a 135-day corn field experiment in the Netherlands, Gell, De Ruijter, Kuntke, De Graaff, & Smit (2011) evaluated the effectiveness of two struvite materials recovered from black water and human urine as P fertilizers compared with TSP and an unamended control in a sandy loam Andisol with a low soil-test P and pH of 4.5. Similar to soybean aboveground Mg concentration results of the current study, Gell et al. (2011) reported that blackwater-derived struvite increased Mg concentrations by 28% in corn aboveground tissue compared to TSP and the control. However, Ylagan et al. (2020) reported similar soybean stem-plus-leaves tissue Mg concentration from CPST and TSP, but both were more than 1.1 times greater than that from the control. Ylagan et al. (2020) also reported that corn stem-plus-leaves tissue Mg concentration was at least 1.1 and 1.8 times greater from CPST than from TSP and the control, respectively.

In contrast to aboveground tissue concentrations, soybean belowground tissue P, N, and Mg concentrations differed between irrigation treatments across years ( $P < 0.01$ ; Table 4).

Soybean belowground tissue P concentration was largest under irrigated management in 2018 (0.11% P) among all treatment combinations and 2.2 times greater than under irrigated management in 2019 (0.05% P), which was smallest among all treatment combinations (Figure 3A). Soybean belowground tissue P concentration was also more than 1.2 times greater under dryland management in 2018 (0.09% P) than under dryland management in 2019 (0.07% P), both of which were at least 1.4 times greater than under irrigated management in 2019 (Figure 3A).

Soybean belowground tissue N concentration was more than 2.2 times greater from the irrigated and dryland managements in 2018, which did not differ, than from dryland management in 2019 and were more than 2.5 times greater than from irrigated management in 2019 (Figure 3B). Similar to tissue P, soybean belowground tissue N concentration was more than 2.6 times greater under irrigated management (1.6% N) in 2018 than under irrigated management (0.6% N) in 2019 (Figure 3B), while that from dryland management (1.5% N) in 2018 was also more than 2.1 times greater than that from dryland management (0.7% N) in 2019 (Figure 3B).

Similar to tissue P and N, soybean belowground tissue Mg concentration was more than 1.5 times greater under irrigated management (0.14% Mg) in 2018 than under irrigated management (0.09% Mg) in 2019, which was lowest among all treatment combinations (Figure 3C). However, unlike tissue P and N, soybean belowground tissue Mg concentration under dryland management in 2019 did not differ from that under dryland managements in 2018 (Figure 3C).

In October 2019, rainfall was numerically largest (25.3 cm) compared to all other months comprising the 5-month soybean growing season in 2018, 2019, and 30-year monthly means (Figure 2A). Soybean already reached an initial maturity stage in October 2019, when the

demand for P, N, and Mg in the pods and seeds had been maximized, thus resulting in a reduction in the aboveground nutrient demand at this period. A possible explanation for lower belowground P, N, and Mg concentrations under irrigated and dryland managements in 2019 than in 2018 could be that some portion of P, N, and Mg in the soil solution was lost to leaching below the root zone from the above-normal precipitation, particularly in October 2019 (Figure 2A). Though the present study showed that soybean belowground tissue P concentration was unaffected by fertilizer-P treatment, Ylagan et al. (2020) reported that soybean belowground tissue P concentration was at least 1.1 and 1.3 times greater from TSP than from CPST and an unamended control, respectively.

Similar to aboveground tissue Mg, soybean belowground tissue Mg concentration differed among fertilizer-P treatments ( $P = 0.03$ ; Table 4). Averaged across irrigation treatments and years, belowground tissue Mg concentration was 1.2 times greater in the unamended control than from TSP, while that from CPST was intermediate and similar to both the unamended control and TSP (Table 6). Though soybean aboveground tissue Mg concentration did not differ between TSP and the unamended control, belowground tissue Mg concentration was greater from the unamended control than from TSP likely due to the large initial, background soil Mg concentration (Table 3) and the low Mg concentration in TSP (Table 1) that may not have become completely plant-available during plant nutrient uptake.

In contrast to aboveground tissue P concentration, soybean aboveground P uptake was unaffected ( $P > 0.05$ ) by fertilizer-P treatment, but differed between irrigation treatments across years ( $P = 0.03$ ; Table 4). Soybean aboveground P uptake was numerically largest under irrigated management in 2019 ( $51 \text{ kg ha}^{-1}$ ) and numerically lowest under dryland management in 2019 ( $35 \text{ kg ha}^{-1}$ ; Figure 3D). Unlike soybean belowground tissue P, N, and Mg, soybean

aboveground P uptake under irrigated management ( $42 \text{ kg ha}^{-1}$ ) in 2018 did not differ from that under irrigated management in 2019 (Figure 3D). Similarly, soybean aboveground P uptake under dryland management ( $43 \text{ kg ha}^{-1}$ ) in 2018 did not differ from that under dryland management in 2019 (Figure 3D). The alkaline groundwater used in the irrigated area likely increased soil pH at least slightly that enhanced P dissolution from secondary compounds and facilitated P diffusion and uptake into the plant when needed, causing greater soybean aboveground P uptake than under dryland conditions in 2019. Though results of the current study showed no fertilizer effect on soybean aboveground P uptake, in a two-year pot experiment, Cabeza, Steingrobe, Romer, & Claassen (2011) reported no difference between shoot P uptake of corn grown with sewage-treatment-plant-recovered struvite compared to TSP in an acidic sandy and pH-neutral loamy soil.

In contrast to tissue N and Mg concentrations and tissue P uptake, soybean aboveground N and Mg uptake were unaffected by fertilizer-P or irrigation treatments ( $P > 0.05$ ; Table 4) and averaged  $436$  and  $62.6 \text{ kg ha}^{-1}$ , respectively, across all treatments. The lack of a significant effect from the fertilizer or irrigation treatment on soybean aboveground N uptake was likely because soybean provided sufficient N through N fixation (Salvagiotti et al., 2008) uniformly across all treatments. Mastrodomenico and Purcell (2012) reported that N fixation could supply up to 90% of the total N required by the soybean plant. Nutrient uptake and partitioning are a function of aboveground tissue nutrient concentration and aboveground DM (Peoples, & Boddey, 2008; Bender, Haegele, & Below, 2015). A soybean with a large aboveground tissue Mg concentration will not always result in a large aboveground Mg uptake. Though soybean aboveground tissue Mg concentration differed among fertilizer-P treatments, there was no fertilizer effect on soybean aboveground DM to produce an effect on aboveground Mg uptake.

In 2019, neither soybean seed P, N, or Mg concentrations were affected by fertilizer-P treatments ( $P > 0.05$ ; Table 7). However, in 2019, soybean seed P and Mg concentrations differed between irrigation treatments ( $P < 0.01$ ), whereas seed N concentration was also unaffected by irrigation ( $P > 0.05$ ; Table 7) and averaged 6.7% N across all treatments. Averaged across fertilizer-P treatments, soybean seed P and Mg concentrations were 1.2 and 1.1 times, respectively, greater under irrigated (0.7% P and 0.31% Mg) than under dryland management (0.6% P and 0.29% Mg). The alkaline groundwater used for irrigation likely increased soil pH enough to enhanced P dissolution of iron phosphates and facilitated P diffusion into soybean roots, causing greater soybean seed P concentrations. Magnesium was present in groundwater used for irrigation (Amuri et al., 2008; Slaton et al., 2013), which was easily plant-available and taken up by soybean plants resulting in greater soybean seed Mg concentrations under irrigation than dryland conditions.

In 2019, neither soybean seed P, N, or Mg uptake were affected by fertilizer-P or irrigation treatments ( $P > 0.05$ ; Table 7) and averaged 20, 199, and 9 kg ha<sup>-1</sup>, respectively, across all treatments. Soybean seed P, N, or Mg uptake were likely unaffected by irrigation treatments due to the above-normal, growing-season rainfall in 2019 negated the effect of irrigation treatment as soybeans under both irrigated and dryland management likely received ample water from rainfall. However, the fertilizer-P sources have different solubilities and compositions, thus it stands to reason that the soybean's ability to fix N could have outperformed the slow-release mechanism of CPST, which had greater N and Mg in its chemical composition than TSP (Table 1). The lack of significant fertilizer treatment effect on soybean seed P uptake was likely that the soil had large P-adsorption ability such that, over a period of time, the P released from both fast-dissolving TSP and slow-dissolving CPST was unavailable for substantial plant uptake. A large

proportion of released P from dissolving P fertilizers was likely rendered unavailable for plant uptake due to P adsorption to clays and/or secondary mineral formation by precipitation with Fe.

### Wheat Response

In 2019, wheat head, stem, and total DM, yield, seed-head P, N, and Mg concentrations and uptake, stem N concentration, belowground P concentration, and stem P, N, and Mg uptake did not differ among fertilizer-P treatments ( $P > 0.05$ ; Table 8). Table 8 summarizes mean values of wheat head, stem, and total DM, yield, seed-head P, N, and Mg concentrations and uptake, stem N concentration, belowground P concentration, and stem P, N, and Mg uptake averaged across fertilizer-P treatments. Though a wheat yield response was expected because the initial soil-test P concentration was low for optimal wheat production (Roberts & Slaton, 2014), P likely still limited wheat growth and productivity.

The overall mean precipitation and air temperature during the 8-month wheat growing season across 2018 and 2019 was more than 1.5 and 1.0 times greater than the 30-year-mean (Figure 2B). Thus, there are two possible explanations for why fertilizer treatments did not affect aboveground wheat properties. First, greater air temperature and above-normal precipitation during the wheat growing season in 2018/2019 likely created a favorable condition for the mineralization of SOM, allowing increased concentrations of plant-available P and N to increase DM production in various plant parts and yield in all plots. Secondly, the entire field was affected by ryegrass, which likely robbed wheat plants of nutrients released from the fertilizer-P sources and any carryover N from the previous soybean crop. Soybean residue generally contains a low C:N ratio (15 to 41:1; Green & Blackmer, 1995), which allows rapid mineralization of soybean residue-N and enhanced mineralization of SOM. The mineralization of previous



soybean residue-N provides one of the rotational benefits for wheat following a soybean crop (Green & Blackmer, 1995). Similar to results of the current study, Gell et al. (2011) reported no difference between corn DM and P uptake across all treatments (i.e., struvite, TSP, and an unamended control).

In contrast to the above wheat properties, wheat stem P and Mg and belowground N and Mg concentrations differed among fertilizer-P treatments in 2019 ( $P < 0.04$ ; Table 8). Wheat stem P concentration was more than 1.4 times greater from the two fertilized treatments, which did not differ, than from the unamended control (Table 8). Though the two fertilizer-P sources were not expected to behave the same due to differential solubilities, it is plausible that the slow-release mechanism of CPST provided just enough P when wheat P demand occurred to match the available P from the faster-dissolving TSP. There could also have been some carry-over P from CPST applied that had not become plant available for prior soybean crop, but may have become available to benefit wheat-P demand. Similar results were confirmed by Ylagan et al. (2020) in soybean stem-plus-leaves tissue P concentration, which did not differ between CPST and TSP, and both fertilizers resulted in soybean stem-plus-leaves tissue P concentrations that were more than 1.3 times greater than that from the unamended control.

Unlike stem P, in 2019, wheat stem Mg concentration was 1.1 times greater from TSP than from CPST and the unamended control, which did not differ (Table 8). Similar to stem Mg, in 2019, wheat belowground N concentration was more than 1.1 times greater from TSP than from CPST and the unamended control, which did not differ (Table 8). Greater wheat stem Mg concentration from TSP was not expected due to the lower initial Mg concentration in the TSP fertilizer compared to that in CPST (Table 1). However, the greater stem Mg concentration from TSP was likely related to greater TSP dissolution, which would have increased cations in the soil

solution, including Mg, coupled with the slow-release CPST characteristics that likely limited plant-available Mg and N concentrations.

In contrast to belowground N, in 2019, wheat belowground Mg concentration was more than 1.2 times greater from CPST than both TSP and the unamended control, while that from TSP was also 1.1 times greater than from the unamended control (Table 8). A greater belowground tissue Mg concentration was expected from CPST due to the Mg-containing composition of the struvite material (Table 1). Since wheat stem Mg concentration was lower than that from TSP, but the root Mg concentration was greater than that from TSP, it is plausible that the form of Mg once in the plant from CPST was less translocatable in the plant than that from TSP, perhaps due to chelation from organic compounds. Results from the current study agreed with reports from Gell et al. (2011) that struvite application could be a source of Mg in crop production.

## **Implications**

Struvite recovery from different types of wastewaters, such as industrial wastewater (El Diwani, El Rafie, El Ibiari, & El-Aila, 2007), swine wastewater (Suzuki et al., 2007; Rahman, Liu, Kwag, & Ra, 2011), municipal landfill leachate (Kim, D., Ryu, Kim, M., Kim, J., & Lee, 2007), sewage sludge (Munch and Barr, 2001), and agro-industrial wastes (Moerman, Carballa, Vandekerckhove, Derycke, & Verstraete, 2009), has attracted interest from agribusiness, environmentalists, and the wastewater treatment industry. The slow-release nature of struvite helps to reduce fertilizer-P application rate and can maintain or improve crop yield, making struvite beneficial to agricultural producers (Talboys et al., 2016). Furthermore, recycling P from P-containing wastewaters to create struvite in the wastewater treatment industry may improve

energy use and reduce labor and other costs associated with struvite removal (Doyle & Parsons, 2002), while also generating a valuable product for use in agricultural production.

As a potentially attractive source of P in agriculture due to its slow-release characteristic (Talboys et al., 2016), wastewater-recovered struvite use in the environment could also help reduce the potential risk of surface water eutrophication and groundwater contamination (Jan, Zhang, Zhao, Zhang, & Huang, 2017) due to cleaner effluent from WWTP and from less surface P lost via runoff (Metson et al., 2016). Application of struvite in crop production is also a potential source of N and Mg, which could provide a potential reduction in cost of N fertilizers applied and help in soils deficient in Mg. However, when struvite is used in large amount over a long-term application, close monitoring of the soil Ca:Mg ratio would be important through soil testing to avoid any unintended, negative effects on plant growth.

Alternatively, as struvite may be marketed as “environmentally friendly” or “green”, this may allow producers to gain value by taking advantage of environmental premiums (Yetilmezsoy et al., 2017). As an alternative fertilizer-P source, struvite may help reduce the long-term threat to future food security from limited remaining quantities of minable rock phosphate from which phosphate fertilizers are produced (Hallas, Mackowiak, Wilkie, & Harris, 2019). Therefore, more field tests of struvite application in row-crop agriculture is warranted and may represent a major step towards sustainable food production system across the globe.

## **Conclusions**

Results from a 2-year field study in a wheat-soybean, double-crop production system on a silt-loam soil in eastern Arkansas showed that CPST provided similar soybean and wheat yields and aboveground DM to TSP, but neither fertilizer significantly increased yield compared to the

unamended control meaning that some other factor, or combination of factors, limited soybean and wheat growth. In addition, CPST provided similar wheat stem P and soybean belowground Mg concentration, lower N and Mg concentration in wheat, greater Mg concentration in wheat, and greater Mg concentration in soybean compared to TSP. Despite some lower tissue concentrations from CPST compared to TSP, differences were relatively small and likely had no major negative effects on soybean or wheat growth and productivity. Results also showed soybean seed P and Mg concentrations differed between irrigation treatments, which emphasized the significant role of the presence of sufficient water for soil nutrient distribution and plant nutrient uptake during crop production. This study demonstrated that wastewater-recovered struvite has the potential to serve as an alternative fertilizer-P source in upland, row-crop agricultural production. However, further research is still necessary to evaluate the application and performance of wastewater-recovered struvite under field conditions with a wider variation of management practices and crops in agricultural soils

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## Tables and Figures

Table 1. Summary of the pH, total nitrogen (N), phosphorus (P), and magnesium (Mg) concentrations, and resulting measured fertilizer grade for the chemically precipitated struvite (CPST) and triple super phosphate (TSP) fertilizer-P materials used in this wheat-soybean, double-crop study in eastern Arkansas. Means ( $\pm$  standard error) are reported (n = 5).

Fertilizer-P Source	pH	Nutrient Concentration			Measured Fertilizer Grade
		N	P	Mg	
CPST	8.77 (0.13)	5.7 (0.2)	11.7 (0.2)	8.3 (0.2)	6-27-0
TSP	2.42 (0.02)	0.0 (0.0)	18.2 (0.4)	0.6 (0.0)	0-42-0

Table 2. Analysis of variance summary of the effects of fertilizer-P source, irrigation, and their interaction on initial soil-test pH, electrical conductivity (EC), Mehlich-3 extractable nutrient concentrations (P, K, Ca, Mg, S, Na, Fe, Mn, Zn, Cu, and B), and total N (TN), total C (TC), and soil organic matter (SOM) concentrations in the top 10 cm in a wheat-soybean, double-crop production system in eastern Arkansas.

Soil Properties	Source of Variation		
	Fertilizer	Irrigation	Irrigation x Fertilizer
	<i>P</i>		
pH	0.61	< <b>0.01</b> <sup>†</sup>	0.69
EC	0.35	0.27	0.13
P	0.69	0.18	0.73
K	0.70	< <b>0.01</b>	0.84
Ca	0.11	0.22	0.93
Mg	0.13	< <b>0.01</b>	0.47
S	0.77	0.09	0.65
Na	0.79	< <b>0.01</b>	0.25
Fe	0.59	0.49	0.38
Mn	0.85	0.09	0.51
Zn	0.55	0.22	0.61
Cu	0.65	0.77	0.89
B	0.64	< <b>0.01</b>	0.64
TN	0.25	0.11	0.05
TC	0.31	0.06	0.12
SOM	0.71	0.94	0.90

<sup>†</sup>Bolded values were considered significant at  $P < 0.05$ .

Table 3. Summary of the effects of irrigation on initial soil-test pH, electrical conductivity (EC), Mehlich-3 extractable nutrient concentrations (P, K, Ca, Mg, S, Na, Fe, Mn, Zn, Cu, and B), and total N (TN), total C (TC), and soil organic matter (SOM) concentrations in the top 10 cm in a wheat-soybean, double-crop production system in eastern Arkansas.

Soil Properties	Irrigated	Dryland	Overall Mean
pH	6.9 a <sup>†</sup>	6.5 b	-
EC (dS m <sup>-1</sup> )	0.120 a	0.110 a	0.115
P (mg kg <sup>-1</sup> )	23.5 a	25.9 a	24.7
K (mg kg <sup>-1</sup> )	46.9 b	61.5 a	-
Ca (mg kg <sup>-1</sup> )	1353 a	1421 a	1387
Mg (mg kg <sup>-1</sup> )	385 a	311 b	-
S (mg kg <sup>-1</sup> )	9.7 a	8.9 a	9.3
Na (mg kg <sup>-1</sup> )	17.7 a	12.6 b	-
Fe (mg kg <sup>-1</sup> )	210 a	217 a	214
Mn (mg kg <sup>-1</sup> )	198 a	172 a	185
Zn (mg kg <sup>-1</sup> )	1.3 a	1.5 a	1.4
Cu (mg kg <sup>-1</sup> )	1.6 a	1.6 a	1.6
B (mg kg <sup>-1</sup> )	0.1 a	0.0 b	-
TN (%)	0.1 a	0.1 a	0.1
TC (%)	1.1 a	0.9 a	1.0
SOM (%)	2.3 a	2.3 a	2.3

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

Table 4. Summary of the effects of irrigation (I), fertilizer-P source (Fert), year (Yr), and their interactions on soybean aboveground dry matter (AGDM), yield, aboveground tissue P (AGPC), N (AGNC), and Mg (AGMgC) and belowground tissue P (BGPC), N (BGNC), and Mg (BGMgC) concentrations, and aboveground P (AGPU), N (AGNU), and Mg (AGMgU) uptake for 2018 and 2019 in a wheat-soybean, double-crop production system in eastern Arkansas.

Plant Properties	Source of Variation						
	I	Fert	I x Fert	Yr	I x Yr	Fert x Yr	I x Fert x Yr
	<i>P</i>						
AGDM	0.44	0.83	0.08	< <b>0.01</b>	0.16	0.53	0.95
Yield	0.80	0.73	0.72	< <b>0.01</b>	0.11	0.43	0.63
AGPC	0.17	0.19	0.20	< <b>0.01</b>	0.27	0.91	0.71
AGNC	0.97	0.54	0.60	<b>0.03</b>	0.63	0.67	0.69
AGMgC	0.69	< <b>0.01</b> <sup>†</sup>	0.39	0.31	0.49	0.55	0.84
BGPC	0.03	0.48	0.86	< 0.01	< <b>0.01</b>	0.36	0.72
BGNC	0.25	0.28	0.69	< 0.01	<b>0.01</b>	0.99	0.60
BGMgC	0.20	<b>0.03</b>	0.56	< 0.01	< <b>0.01</b>	0.32	0.55
AGPU	0.12	0.58	0.30	0.85	<b>0.03</b>	0.72	0.59
AGNU	0.39	0.84	0.28	0.09	0.07	0.92	0.61
AGMgU	0.76	0.26	0.07	0.08	0.15	0.49	0.99

<sup>†</sup> Bolded values were considered significant at  $P < 0.05$ .

Table 5. Summary of the effects of year on aboveground dry matter (AGDM), yield, and aboveground tissue phosphorus (P) and nitrogen (N) concentrations for soybean in 2018 and 2019 in this wheat-soybean, double-crop study in eastern Arkansas.

Year	AGDM	Yield	P	N
	kg ha <sup>-1</sup>		%	
2018	15439 a <sup>†</sup>	2119 b	0.3 b	3.1 b
2019	11619 b	2951 a	0.4 a	3.4 a

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

Table 6. Summary of the effects of fertilizer-P treatment [chemically precipitated struvite (CPST), triple superphosphate (TSP), and unamended control (UC)] on above- and belowground Mg tissue concentration for soybean in 2018 and 2019 in a wheat-soybean, double-crop production system in eastern Arkansas.

Fertilizer-P Treatment	Aboveground Mg	Belowground Mg
	%	
CPST	0.49 a <sup>†</sup>	0.11 ab
TSP	0.45 b	0.10 b
UC	0.43 b	0.12 a

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

Table 7. Summary of the effects of irrigation, fertilizer-P source, and their interactions on seed P, N, and Mg concentrations and seed P, N, and Mg uptake for soybean in 2019 in a wheat-soybean, double-crop production system in eastern Arkansas.

Plant Properties	Source of Variation		
	Irrigation	Fertilizer	Irrigation x Fertilizer
	<i>P</i>		
Seed P concentration	< <b>0.01</b> <sup>†</sup>	0.26	0.86
Seed N concentration	0.71	0.28	0.35
Seed Mg concentration	< <b>0.01</b>	0.22	0.45
Seed P uptake	0.57	0.52	0.49
Seed N uptake	0.52	0.82	0.90
Seed Mg uptake	0.43	0.68	0.54

<sup>†</sup> Bolded values were considered significant at  $P < 0.05$ .



Table 8. Summary of the effects of fertilizer-P source on seed head, stem and total dry matter, grain yield, seed-head P, N, and Mg concentrations, stem P, N, and Mg concentrations, belowground P, N, and Mg concentrations, seed-head P, N, and Mg uptake, and stem P, N, and Mg uptake for wheat in 2019 in a wheat-soybean, double-crop production system in eastern Arkansas.

Wheat Properties	Fertilizer-P			UC	Overall Mean
	Source	CPST	TSP		
	— <i>P</i> —				
Head dry matter (kg ha <sup>-1</sup> )	0.37	339 a	445 a	371 a	385
Stem dry matter (kg ha <sup>-1</sup> )	0.45	461 a	536 a	524 a	507
Total dry matter (kg ha <sup>-1</sup> )	0.41	800 a	979 a	893 a	891
Yield (kg ha <sup>-1</sup> )	0.38	2180 a	1926 a	1659 a	1922
Seed-head P concentration (%)	0.37	0.36 a	0.37 a	0.34 a	0.36
Seed-head N concentration (%)	0.08	1.68 a	1.81 a	1.69 a	1.73
Seed-head Mg concentration (%)	0.58	0.15 a	0.15 a	0.14 a	0.15
Stem P concentration (%)	<b>0.04</b> <sup>†</sup>	0.08 a	0.08 a	0.06 b	-
Stem N concentration (%)	0.21	0.55 a	0.61 a	0.53 a	0.56
Stem Mg concentration (%)	< <b>0.01</b>	0.10 b	0.11 a	0.10 b	-
Belowground P concentration (%)	0.07	0.13 a	0.12 a	0.10 a	0.12
Belowground N concentration (%)	<b>0.03</b>	0.77 b	0.88 a	0.76 b	-
Belowground Mg concentration (%)	< <b>0.01</b>	0.12 a	0.10 b	0.09 c	-
Seed-head P uptake (kg ha <sup>-1</sup> )	0.23	1.20 a	1.64 a	1.29 a	1.38
Seed-head N uptake (kg ha <sup>-1</sup> )	0.24	5.62 a	7.73 a	6.21 a	6.52
Seed-head Mg uptake (kg ha <sup>-1</sup> )	0.22	0.49 a	0.67 a	0.53 a	0.56
Stem P uptake (kg ha <sup>-1</sup> )	0.16	0.36 a	0.39 a	0.28 a	0.34
Stem N uptake (kg ha <sup>-1</sup> )	0.32	2.52 a	3.22 a	2.74 a	2.83
Stem Mg uptake (kg ha <sup>-1</sup> )	0.54	0.47 a	0.57 a	0.52 a	0.52

<sup>†</sup> Bolded values were considered significant at  $P < 0.05$ .



Fig. 1. Aerial view of the plot arrangement in the irrigated and dryland/non-irrigated portion of the study area for a wheat-soybean, double-crop production system in eastern Arkansas. The north direction is towards the top of the image. Individual plots dimensions are 6.1-m long by 3-m wide.

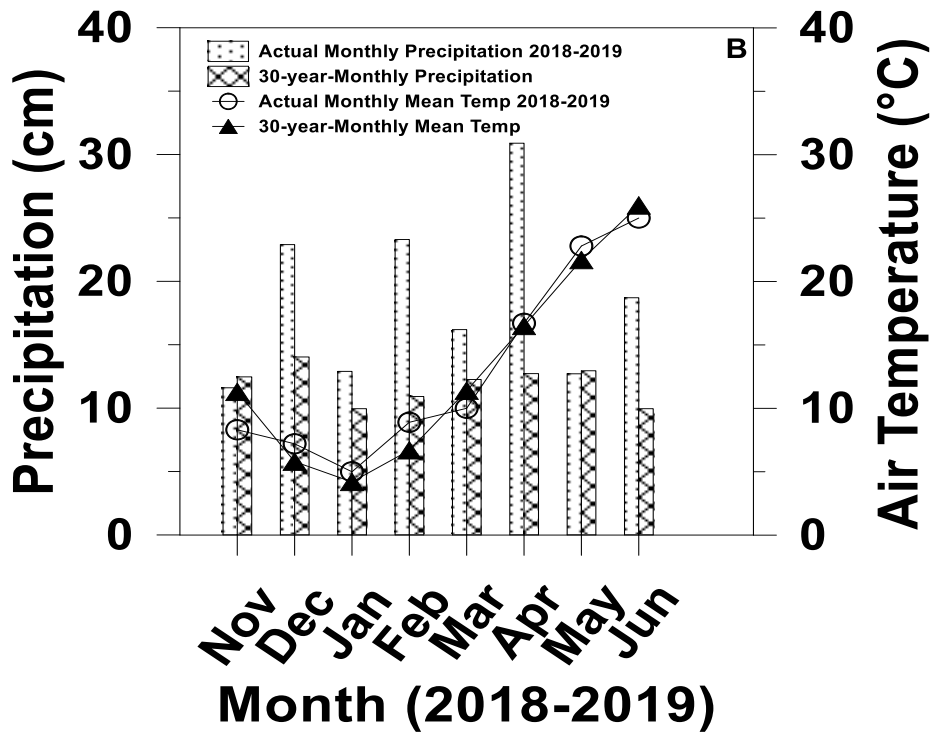
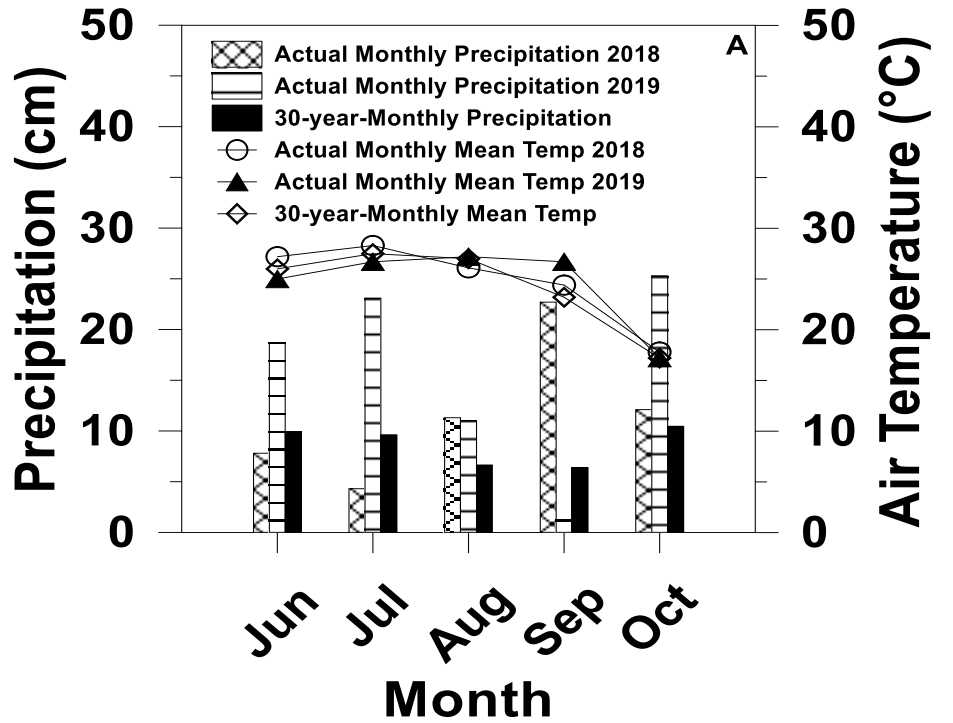


Fig. 2. Comparison among the 30-year (1981 to 2010), monthly precipitation and air temperature and actual monthly precipitation and air temperature during the five-month soybean growing season in 2018 and 2019 (A) and during the eight-month wheat growing season in 2019 (B) at the Cotton Branch Experiment Station near Marianna, Arkansas.

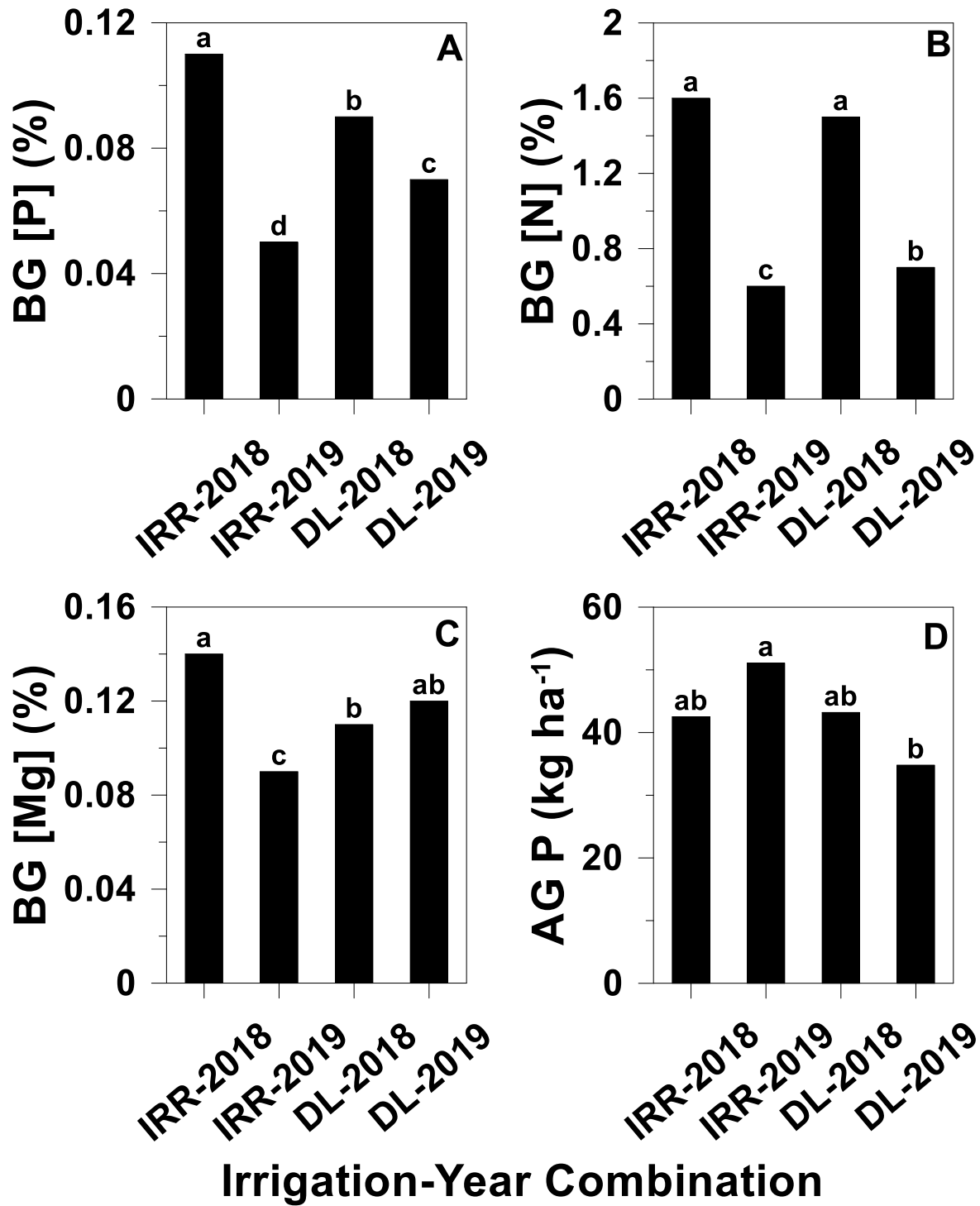


Fig. 3. Interaction effect between irrigation [irrigated (IRR) and dryland (DL)] and year (2018 and 2019) treatment combination on soybean belowground tissue phosphorus (BG [P]; A), nitrogen (BG [N]; B), and magnesium concentrations (BG [Mg]; C), and aboveground P uptake (AG P; D) for a wheat-soybean, double-crop production system in eastern Arkansas.

## **Chapter 3**

### **Field Evaluation of Wastewater-recovered Struvite as a Fertilizer-Phosphorus Source in Flood-irrigated Rice in Eastern Arkansas**

## Abstract

Phosphorus (P) is a major contaminant in many wastewater sources and has gained interest due to the role P has in eutrophication of receiving waters. Recycling P from wastewater as the mineral struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) could be a promising option to reduce P discharge into receiving waters. Since non-renewable rock phosphates are the only fossil resource to produce phosphate fertilizers, struvite could also potentially provide an alternative fertilizer-P source for crop production. The objective of this study was to evaluate the effects of two struvite materials [i.e., electrochemically precipitated struvite (ECST) and chemically precipitated struvite (CPST)] relative to several other common fertilizer-P sources [i.e., triple super phosphate (TSP), monoammonium phosphate (MAP), diammonium phosphate (DAP), and rock phosphate (RP)] on the response of a pureline rice (*Oryza sativa* L.) cultivar grown under flood-irrigation in a P-deficient, silt-loam soil (Typic Glossaqualfs) in eastern Arkansas. In 2019, rice grain yield did not differ ( $P > 0.05$ ) among fertilizer-P sources. In 2020, rice grain yield was numerically largest from TSP ( $9.8 \text{ Mg ha}^{-1}$ ), which did not differ from that from DAP, MAP, RP, and an unamended control (UC), and was numerically smallest from ECST ( $8.2 \text{ Mg ha}^{-1}$ ), which did not differ ( $P > 0.05$ ) from that from CPST, and was lower ( $P < 0.05$ ) than that from TSP, DAP, MAP, RP, and UC. In 2020, grain yield from CPST ( $8.9 \text{ Mg ha}^{-1}$ ) did not differ ( $P > 0.05$ ) from that from DAP, MAP, RP, or UC. However, rice aboveground dry matter, above- and belowground tissue and grain P, N, and C concentrations, aboveground and grain tissue P uptake, and aboveground tissue N and C contents from ECST and CPST did not differ ( $P > 0.05$ ) from that from TSP, MAP, DAP, RP, or UC. The many similar rice responses compared to other commonly used, commercially available fertilizer-P sources suggest that wastewater-recovered

struvite materials have the potential to be an alternative fertilizer-P-source option for flood-irrigated rice production.

## Introduction

Phosphorus (P) and nitrogen (N) are essential plant nutrients that often restrict crop growth and food production in many parts of the world (Elser et al., 2007). Some of the restrictions can be addressed through external fertilizer-N additions, where the capacity to produce fertilizer-N materials is essentially infinite, but P reserves are limited. The majority of fertilizer-P sources are obtained by mining non-renewable rock phosphate (RP), where existing global P reserves are projected to be depleted within the next 50 to 100 years (Cordell, Drangert, & White, 2009; Gilbert, 2009). Additionally, the price of RP, used in the production of most fertilizer-P, is expected to increase as the limited RP reserves, which are located in only a few countries, are depleted (Liu, Kumar, Kwag, & Ra, 2013; Cordell, & Neset, 2014; Talboys et al., 2015). Consequently, it is important to consider options for a sustainable source of P for future agricultural production.

One potential alternative to the diminishing quantities of mined RP that has received increased interest recently is recovering P as the mineral struvite from wastewaters, such as municipal (De-Bashan, Hernandez, Morey, & Bashan, 2004; Kataki, West, Clarke, & Baruah, 2016) and/or agricultural (Mayer et al., 2016) wastewaters (Le Corre, Valsami-Jones, Hobbs, & Parsons, 2009; Antonini, Arias, Eichert, & Clemens, 2012; Cid, Jasper, & Hoffmann, 2018; Li et al., 2019). Magnesium ammonium phosphate hexahydrate ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ), or struvite, is a white, crystalline mineral with an orthorhombic structure (Johnston & Richards, 2003; Le Corre, et al., 2009). The unintentional precipitation of struvite is well known to cause scaling and has been a major problem in wastewater treatment plants (WWTPs) (Doyle, Oldring, Churchley, Price, & Parsons, 2003). However, these problems have been addressed through struvite removal at strategic locations in WWTP processes within specialized reactors (Talboys et al., 2015), thus



preventing unwanted deposits in pipes, reducing operating costs by improving sludge dewatering, and reducing biosolid volume (Parsons, Wall, Doyle, Oldring, & Churchley, 2001).

Struvite recovery from wastewater has been achieved through biological, chemical, and, more recently, electrochemical precipitation methods (Le Corre et al., 2009; Cusick, & Logan, 2012; Manas, Sperandio, Decker, & Biscans, 2012; Huang et al., 2016; Kékedy-Nagy, Teymouri, Herring, & Greenlee, 2020). The formation of struvite crystals is controlled by the concentrations of magnesium ( $Mg^{2+}$ ), ammonium ( $NH_4^+$ ), and phosphate ( $PO_4^{3-}$ ) ions, pH, mixing energy, temperature, ionic strength, and the presence of foreign ions (Bouropoulos, & Koutsoukos, 2000; Le Corre, Valsami-Jones, Hobbs, & Parsons, 2005). Chemical precipitation is a commonly used method for intentional struvite production and removal from WWTPs. However, chemical precipitation often requires the addition of chemicals, such as Mg-containing salts and a base for pH adjustment (Jaffer, Clark, Pearce, & Parsons, 2002; Kékedy-Nagy et al., 2019). The optimal pH for struvite precipitation has been reported to range from 7 to 9 (Kofina, & Koutsoukos, 2005; Bhuiyan, Mavinic, & Beckie, 2007, 2009; Wang, C. C., Hao, Guo, & Van Loosdrecht, 2010; Uysal, & Kuru, 2013). In contrast to chemical precipitation, electrochemical precipitation of struvite from wastewater is a newly adopted technique that uses a sacrificial Mg-based anode that releases Mg ions as the electrode corrodes in the presence of an electrical current (Kruk, Elektorowicz, & Oleszkiewicz, 2014; Kékedy-Nagy et al., 2019). One benefit of electrochemical compared to chemical precipitation is the elimination of the need for external chemical additions.

Recovered struvite generally contains between 11 and 28% total P (Johnston and Richards, 2003; Ostara Nutrient Recovery Technologies Inc., 2020) depending on the initial source and method of production, yet only < 1 to 5% is water-soluble (Li & Zhao, 2002; Negrea, Lupa, Negrea, Ciopec, & Muntean, 2010), while the remaining P is organic-acid soluble

(Cabeza, Steingrobe, Römer, & Claassen, 2011; Antonini et al., 2012). The solubility of struvite in organic acids can differ depending on the source material and purity of the struvite produced (Hertzberger, Cusick, & Margenot, 2020).

One chemically precipitated struvite (CPST) material produced from municipal wastewater, marketed and sold as Crystal Green by Ostara Nutrient Recovery Technologies Inc. (Vancouver, Canada), is reported to be 4% water-soluble and 96% citrate-soluble (i.e., struvite dissolution driven by organic acids exuded by plant roots) (Ostara Nutrient Recovery Technologies Inc., 2020). Rech et al. (2019) reported the neutral ammonium citrate and water (NAC + H<sub>2</sub>O) soluble P to be 17.5, 28.9, and 28.8% from struvite produced from chicken manure, swine manure wastewater, and municipal wastewater (Crystal Green, CPST), respectively, while the water-soluble P in these struvite sources was 3.8, 2.0, and 2.5%, respectively.

In contrast to struvite, commercially available phosphate fertilizers commonly used in agricultural production, such as single superphosphate, triple superphosphate (TSP), and monoammonium phosphate (MAP), have a water-soluble P fraction of 80 to 90% of the total P concentration, where the remaining P fraction is citrate soluble (Chien, Prochnow, Tu, & Snyder, 2011), while the NAC + H<sub>2</sub>O and water-soluble P for TSP was reported as 97 and 82%, respectively (Prochnow, van Raij, & Kiehl, 2002). Thus, fertilizer-P sources commonly used in agriculture tend to be substantially more soluble than struvite. Consequently, the solubility characteristics make struvite a potentially ideal, slow-release P source for many types of plants, including agricultural crops, in upland, well-drained (Thompson, 2013; Achat et al., 2014; Anderson et al., 2021b) and lowland, poorly drained soils (Anderson, Brye, Greenlee, Roberts, & Gbur, 2021a; Anderson et al., 2021c).

Phosphorus deficiency is a major constraint to crop growth and production, and is one of the most important global soil fertility problems (Rashid, Awan, & Ryan, 2005). The P concentration and availability in soil depends on the degree of P adsorption on soil colloids and precipitation in secondary minerals (Dhillon, Dhesi, & Brar, 2004). Although P is a relatively abundant element in nature, crop yields are often limited by low soil-P availability, principally due to adsorption and precipitation reactions of both native soil-P and applied fertilizer-P with iron (Fe) and/or aluminum (Al) in acidic soil or calcium (Ca) in alkaline soil (Khiari & Parent, 2005; Sims & Pierzynski, 2005; Khademi, Jones, Malakouti, & Asadi, 2010). Consequently, limited soil-P availability often results in low P uptake efficiency in plants (Bhattacharyya et al., 2015). However, one plant response mechanism to increase inorganic-P availability in the soil solution is by rhizosphere acidification and the release of organic acid anions, such as citrate, oxalate, and malate, which can solubilize P from mineral surfaces by ligand exchange or by ligand-promoted dissolution of Fe- or Al-oxides (Hinsinger, 2001; Wouterlood, Cawthray, Scanlon, Lambers, & Veneklaas, 2004; Johnson & Loeppert, 2006).

To date, studies have reported variation in crop response to struvite compared with other common fertilizer-P sources. Many studies have shown struvite to exhibit comparable plant growth or P uptake to other commercially available, fertilizer-P sources (Kern et al., 2008; Massey, Davis, Ippolito, & Sheffield, 2009; Thompson, 2013). In contrast, other studies reported a reduction in agronomic effectiveness with a CPST material as the fertilizer-P source compared to other commercially available, fertilizer-P sources (Talboys et al., 2015; Everaert, Da Silva, Degryse, McLaughlin, & Smolders, 2017). Rech et al. (2019) evaluated the effects of struvite from multiple sources, including CPST and TSP on wheat (*Triticum aestivum* L.) and soybean (*Glycine max* L.) response in a greenhouse potted-plant study, concluding that struvite could be

used as a sustainable P source for crops despite struvite's low water solubility. More recently, Ylagan, Brye, & Greenlee (2020) evaluated the effects of a CPST material (i.e., Crystal Green) and an innovative electrochemically precipitated struvite (ECST) on corn (*Zea mays* L.) and soybean response in a greenhouse potted-plant study. Struvite materials (CPST and ECST) were also evaluated for their water-soluble and plant-available nutrient concentrations and soil pH changes over time relative to TSP, MAP, and diammonium phosphate (DAP) in plant-less soil incubations in various soil textures under moist- (Anderson et al., 2021b) and flooded-soil (Anderson et al., 2021a,c) conditions.

The relatively quick immobilization of P in many agricultural soils causes producers to compensate by applying large amounts of expensive, inorganic-P fertilizers to attain optimal crop yields (Ayaga, Todd, & Brookes, 2006; Metson, Macdonald, Haberman, Nesme, & Bennett, 2016). However, the accumulation of soil P from the excess fertilizer-P application can result in off-site transport in agricultural runoff, which, in turn, can cause eutrophication and the creation of hypoxic zones in freshwaters from P contamination (Correll, 1998; MacDonald et al., 2016). Thus, matching soil-P availability with the timing of plant-P need through innovative fertilizer technology would provide both agronomic benefits and environmental protection (Massey et al., 2009; Talboys et al., 2015), such as through the use of struvite (Rech et al., 2019; Anderson et al., 2021a,b,c).

One crop that responds positively to optimal P fertilization is rice (*Oryza sativa* L.). In addition to California and the gulf coast of Texas, the mid-southern region encompassed by southeast Missouri, eastern Arkansas, eastern Louisiana, and western Mississippi make up the main rice-producing regions in the United States (US), where Arkansas has consistently been the largest rice-producing state in recent decades, with nearly 50% of the total annual US rice

production (USDA-ERS, 2021). In Arkansas, more than 90% of rice is grown under flood-irrigated conditions and approximately 25% of all rice in Arkansas is grown in a continuous rice production system, while approximately 69% is grown in rotation with soybean (Hardke, 2020). In soils used for flood-irrigated rice production, soil-P availability tends to increase following imposition of the flood and the development of reducing conditions that solubilize precipitated Fe, thus releasing a fraction of the P that was co-precipitated with Fe, particularly in acidic soils (Krairapanond, Jugsujinda, & Patrick, 1993; Saleque, Abedin, & Bhuiyan, 1996).

Although a few row-crop field trials have been reported (Gell, De Ruijter, Kuntke, De Graaff, & Smit, 2011; Thompson, 2013; Collins, Kimura, Frear, & Kruger, 2016; Hilt et al., 2016), most studies have focused on struvite application as a fertilizer-P source in greenhouse pot studies (Ganrot et al., 2007; Kern et al., 2008; Massey et al., 2009; Cabeza et al., 2011; Uysal, Demir, Sayilgan, Eraslan, & Kucukyumuk, 2014; Talboys et al., 2015; Ylagan et al., 2020). Furthermore, no studies have examined the agronomic effectiveness of wastewater-recovered struvite as a fertilizer-P source in flooded-irrigated rice in the US. Therefore, the objectives of this field study were to: i) evaluate the effects of two wastewater-recovered struvite materials (i.e., ECST and CPST) relative to several other common fertilizer-P sources (i.e., TSP, MAP, DAP, and RP) (i.e., P-source study) and ii) evaluate the effect of TSP fertilizer rate (i.e., 0, 22, 44, and 66 kg P ha<sup>-1</sup>) (i.e., P-rate study) on the response of a pureline rice cultivar grown under flood-irrigation in a P-deficient, silt-loam soil (Typic Glossaqualfs) in eastern Arkansas.

It was hypothesized that rice amended with either wastewater-recovered struvite source (i.e., ECST and CPST) would have at least similar aboveground dry matter and yield, above- and belowground tissue and grain P, N, and C concentrations, and aboveground and grain P, N, and C contents, but greater aboveground tissue and grain Mg concentration and uptake and

belowground tissue Mg concentrations than other common, commercially available fertilizer-P sources (i.e., TSP, MAP, DAP, and RP) due to lower water solubility and greater initial Mg concentrations in ECST and CPST. In addition, it was hypothesized that aboveground P uptake would be greater from 44 kg TSP-P ha<sup>-1</sup> compared to fertilizer rates of 22 and 66 kg TSP-P ha<sup>-1</sup>.

## **Materials and Methods**

### **Site Description and Cropping History**

Research was conducted at the Pine Tree Research Station (PTRS) near Colt, AR in 2019 and 2020 (Figure 1) in a Calhoun silt loam (fine-silty, mixed, active, thermic Typic Glossaqualfs; Soil Survey Staff, 2015). The Calhoun series consists of poorly drained, slowly permeable, loamy-textured soils derived from loess parent material (NRCS, 2020) that is commonly used for rice production.

The study area was previously cropped in a rice-soybean rotation for at least five years. The previous soybean crops were conventionally tilled with one to two passes with a field cultivator to a depth of approximately 10 cm and a single pass with a bedder roller. A glufosinate-tolerant, maturity group 4.4 to 4.6, soybean cultivar was planted throughout the whole study area with 76.2-cm row spacing between early May and early June every other year. For the previous rice crops, tillage included one to two passes with a field cultivator to a depth of approximately 10 cm and multiple passes with a land plane to prepare a smooth seed bed. Rice was planted at a 19.1-cm row spacing between early April and mid-May every other year. The previous soybean and current rice crops were managed using University of Arkansas System Division of Agriculture recommendations (Roberts, Slaton, Wilson, & Norman, 2016).

The climate in the region encompassing the study area is humid temperate, with the 30-year (i.e., 1981 to 2010) average annual air temperature and precipitation of 16.1°C and 123.0 cm, respectively (NOAA, 2020). The 30-year mean monthly minimum and maximum air temperatures in the area are -1°C in January and 32.3°C in July, respectively (NOAA, 2020). Daily rainfall throughout the growing season each year was recorded approximately 2.5 km east from the field site that was obtained for comparison to the 30-year average rainfall data.

## **Field Treatments and Experimental Layout**

### **P-source Study**

The P-source study was conducted in 2019 and 2020 to evaluate the effects of fertilizer-P source [i.e., ECST, CPST, TSP, DAP, MAP, RP and an unamended control (UC)]. In both years, the P-source study was conducted in an area following soybeans, thus the exact study area shifted in 2020 from where the study took place in 2019 (Figure 1). Consequently, the P-source study consisted of two experimental factors, fertilizer-P source and site (or year). Each year, there were four blocks containing one randomized replication of each of the seven fertilizer-P-source treatments. Two sources of struvite were used, including ECST and CPST. The ECST material was produced from synthetic wastewater by researchers in the Department of Chemical Engineering at the University of Arkansas (Kékedy-Nagy et al., 2020). Two separate batches of ECST were generated, where the first batch was used in 2019 and the second batch was used in 2020. Each ECST batch was produced in a bench-top-scale, single-compartment reactor, with a pure Mg or AZ31 Mg alloy anode and a 5- x 5-cm, stainless-steel (316SS) plate as the cathode, that contained 0.85 L of a 0.077 M solution of synthetic wastewater containing 7.53 g L<sup>-1</sup> of ammonium dihydrogen phosphate (NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>; Kékedy-Nagy et al., 2020). At the end of each

batch, the precipitate from the anode and the cathode were collected and stored separately. The CPST material was generated from raw municipal wastewater from an active wastewater treatment plant near Atlanta, GA, which is marketed and sold under the trade name Crystal Green by Ostara Nutrient Recovery Technologies, Inc. The ECST and CPST materials have similar basic elemental compositions and morphology, as characterized by X-ray diffraction, to constitute the mineral struvite (Kékedy-Nagy et al., 2020). However, the CPST material had a more diverse composition, having been produced from raw, municipal wastewater, compared to the ECST material, which was generated from a synthetic, P- and N-containing wastewater.

#### P-rate Study

The P-rate study consisted of four blocks with each block containing one randomized replication of four TSP fertilizer rates (0, 22, 44, and 66 kg P ha<sup>-1</sup>). The P-rate study was conducted in the same field adjacent to the P-source study each year and was separated by a 9-row border of rice.

#### **Fertilizer-P Source Characterization**

Chemical analyses were conducted on five replications of each batch of ECST and the CPST, TSP, DAP, MAP, and RP fertilizers. The fertilizer-P sources had different particle sizes, varying from small pellets (i.e., TSP, MAP, DAP, and CPST), with an average diameter of 3.1 mm (Anderson, 2020), to crystals (ECST) to powder (i.e., RP). The ECST and pelletized fertilizers were mechanically crushed and chemically analyzed in powdered form to facilitate comparison among all fertilizer-P sources. The pH of each fertilizer material was measured in a 1:2 (mass/volume) fertilizer-to-water-ratio paste (Sikora & Kissel, 2014). Total N concentration



was determined by high-temperature combustion using a VarioMax CN analyzer (Elementar Americas, Inc., Mt. Laurel, NJ; Provin, 2014). A strong-acid digest was conducted (USEPA, 1996) and extracts were analyzed by inductively coupled, argon-plasma spectrometry (ICAPS) to determine total-recoverable P and Mg. All fertilizer analyses were conducted at the University of Arkansas, Division of Agriculture's Agricultural Diagnostic Lab in Fayetteville. Table 1 summarizes the measured chemical properties of the fertilizer-P materials.

## **Plot Management**

### **P-source Study**

In April 2019, 28 field plots, 4.9-m long by 1.8-m wide, were established for all fertilizer treatments except for ECST after conventional tillage as described previously. The ECST plots (5-m long by 5-m wide) were smaller sub-plots of the larger plot area due to the limited quantity of ECST material. The fertilizer-P quantity that was used per plot was based on the initial Mehlich-3 soil-test-P concentration and soil pH in the top 10-cm, the recommended P-fertilization rate for flood-irrigated rice on a silt-loam soil with soil pH  $\geq 6.5$  (29.4 kg P ha<sup>-1</sup>; Roberts et al., 2016), and the measured total-recoverable P concentrations of each fertilizer-P material (Table 1).

Each fertilizer-P material contained different N concentrations (Table 1). Consequently, the quantity of N needed to be added to balance the N rate across all fertilizer-P treatments, including the unamended control, was determined based on the amount of N added in DAP, which had the largest N concentration (Table 1). Extra N was added in the form of uncoated urea (460 g N kg<sup>-1</sup>). However, approximately 1.3 times more urea-N was mistakenly added to the

ECST-amended plots in 2019 than what should have been added. Despite the extra N, rice yields or performance were not expected to be significantly impacted.

On 30 April, 2019, fertilizer-P materials were manually applied separately to each plot at 29.4 kg P ha<sup>-1</sup>. In addition, all plots were mechanically broadcast-amended with 83.7 kg ha<sup>-1</sup> potassium (K) as muriate of potash. The original, solid form of all fertilizers were surface-applied and incorporated to a depth of approximately 10 cm with a rototiller prior to planting. On the same day, after fertilizer application and incorporation, plots were drill-seeded with the pureline rice cultivar “Diamond” at a rate of 80 kg seed ha<sup>-1</sup>, which resulted in each plot containing nine rows with 19-cm row spacing.

In 2019, weeds were managed with various herbicides at various times during the growing seasons. On 1 May, 2019, Glyphosate (2.8 L ha<sup>-1</sup>; Bayer CropScience, Research Triangle Park, NC; 2-(phosphonomethylamino) acetate, propan-2-ylazanium) was tractor-sprayer-applied. Prowl (5.6 L ha<sup>-1</sup>; BASF Corporation, Florham Park, NJ; Pendimethalin) and Facet (136 L ha<sup>-1</sup>; BASF Corporation, Florham Park, NJ; Quinclorac) were applied on 6 May, 2019. Ricebeux (8.4 L ha<sup>-1</sup>; RICECO LLC, Memphis, TN; Thibencarb) was sprayed on 29 May, 2019. PermitPlus (52.5 mL ha<sup>-1</sup>; Gowan Company LLC, USA; Halosulfuron-methyl) was sprayed on 1 June, 2019. On 3 June, 2019, 1.12 kg ha<sup>-1</sup> of Zn was tractor-sprayer-applied as Zn-ethylene diamine tetraacetate (Zn-EDTA). On 4 June, 2019, a single pre-flood application of uncoated urea was mechanically broadcast-applied at 145.7 kg N ha<sup>-1</sup>. The flood was established on 5 June, 2019, one day after urea application to minimize N loss in the form of ammonia volatilization, and was maintained at approximately a 10-cm depth throughout the remainder of the rice growing season. The flood was released on 25 August, 2019. Rice grain was harvested with a plot combine from a 1.3-m width by 3-m length of plot on 17 September, 2019.

In 2020, the P-source study was relocated to approximately 55 m away within the same field to maintain the rice-following-soybean rotation, where the soil was comparable to that of the 2019 study area. On 4 May, 2020, fertilizer-P materials were manually applied at 29.4 kg P ha<sup>-1</sup>. Similar to 2019, an equivalent amount of N, in the form of uncoated urea, was also applied to all fertilizer treatments including the unamended control. Additionally, all plots were mechanically broadcast-amended with 83.7 kg K ha<sup>-1</sup> as muriate of potash. All fertilizers were surface-applied in the original solid form and incorporated prior to planting. On the same day, after fertilization and incorporation, plots were drill-seeded with the pureline rice cultivar “Diamond” at a rate of 80 kg seed ha<sup>-1</sup>.

In 2020, weeds were again managed with various herbicides at different times during the growing seasons. On 5 May, 2019, Prowl (5.6 L ha<sup>-1</sup>) and Facet (136 L ha<sup>-1</sup>) were tractor-sprayer-applied. On 11 May, 2020, Facet (136 L ha<sup>-1</sup>) and Basagran (4.2 L ha<sup>-1</sup>; Dow AgroSciences, Indianapolis, IN; floryprauxifen-benzyl) were applied. Stam (8.4 L ha<sup>-1</sup>; Propanil) and PermitPlus (52.5 mL ha<sup>-1</sup>) was sprayed on 1 June, 2020. On 5 June, 2020, 1.1 kg ha<sup>-1</sup> of Zn was tractor-sprayer-applied as Zn-EDTA. On 12 June, 2020, a single pre-flood application of uncoated urea was mechanically broadcast-applied at 145.7 kg N ha<sup>-1</sup>. The flood was established on 13 June, 2020, one day after urea application to minimize N loss in the form of ammonia volatilization, and was maintained at approximately a 10-cm depth throughout the remainder of the rice growing season. The flood was released on 20 August, 2020. Rice grain was harvested with a plot combine from a 1.3-m width by 3-m length of plot on 10 September, 2020.

## P-rate Study

All establishment and management activities associated with the P-source study each year were used for the P-rate study each year. In 2019 and 2020, 16 field plots, 4.9-m long by 1.8-m wide, were established after conventional tillage. On 30 April, 2019 and 4 May, 2020, TSP was manually applied at 0, 22, 44, and 66 kg P ha<sup>-1</sup>. All plots were mechanically broadcast-amended with 83.7 kg K ha<sup>-1</sup> as muriate of potash. All fertilizers were surface-applied and incorporated prior to planting. On the same day, after fertilization and incorporation, plots were drill-seeded with the pureline rice cultivar “Diamond” at a rate of 80 kg seed ha<sup>-1</sup>. Weeds management, Zn application, single pre-flood N application, and flood establishment were the same as conducted for the P-source study each year. Rice grain was harvested with a plot combine from a 1.3-m width by 3-m length of plot on 17 September, 2019 and on 10 September, 2020.

In the P-source and P-rate studies, the mass of grain harvested per plot was recorded by a calibrated scale and the moisture content of the harvested grain mass was measured by a calibrated sensor on the combine. Based on the combine-measured grain moisture content, rice grain masses were adjusted to 120 g kg<sup>-1</sup> (12%) moisture content for yield reporting.

## Soil Sampling, Processing, and Analyses

On 30 April, 2019 and 4 May, 2020, prior to rice planting, soil samples were collected from six random locations from the top 10 cm in each plot, combined, and mixed for one composite sample per block. Samples were oven-dried at 70°C for 48 h, mechanically crushed, and sieved through a 2-mm mesh screen. Particle-size analyses were conducted using a modified

12-h hydrometer method (Gee & Or, 2002) to determine sand, silt, and clay fractions and confirm the soil textural class. Soil pH and electrical conductivity (EC) were measured potentiometrically in a 1:2 (mass/volume) soil-to-water-ratio paste (Brye, West, & Gbur, 2004; Sikora and Kissel, 2014). Weight-loss-ignition was used to determine soil organic matter (SOM) concentration using a muffle furnace at 360°C for 2 h (Zhang and Wang, 2014). Total nitrogen (TN) and total carbon (TC) concentrations were determined by high-temperature combustion using a VarioMax CN analyzer (Provin, 2014). A Mehlich-3 extraction (Zhang et al., 2014) was also conducted with a 1:10 (mass:volume) soil:extractant solution ratio to determine extractable nutrient (i.e., P, K, Ca, Mg, S, Na, Fe, Mn, Zn, Cu, and B) concentrations by ICAPS (Soltanpour, Johnson, Workman, Jones, & Miller, 1996). All soil analyses were conducted at the University of Arkansas, Division of Agriculture's Agricultural Diagnostic Laboratory in Fayetteville.

### **Plant Sampling, Processing, and Analyses**

On 2 August, 2019 and 5 August, 2020, when rice reached 50% heading, which is the growth stage when 50% of the panicles have partially emerged from the boot, in both studies, a 1-m row length of aboveground plants was cut at the soil surface and collected from the second or seventh rice row in each plot. In the P-source study in 2019 and 2020, three rice root masses of the cut aboveground plants in each plot were carefully dug out with a hand shovel to a depth of approximately 15 cm. The qualitatively sampled plant roots were vigorously shaken and immediately rinsed in the flooded water in the field and bagged. Prior to drying, the root material was further rinsed with tap water to remove attached soil particles. The above- and belowground rice tissue samples were oven-dried at approximately 55°C for 7 days and weighed. Dry matter

per unit area was calculated only for the aboveground plant biomass. Sub-samples of above- and belowground dry matter were mechanically ground and passed through a 2-mm mesh screen for chemical analyses.

Total C and TN concentrations in sub-samples of above- and belowground dry matter were determined separately by high-temperature combustion using a VarioMax CN analyzer (Provin, 2014). A concentrated nitric acid digestion was conducted and extracts were analyzed by ICAPS (Soltanpour et al., 1996) to determine above- and belowground tissue P and Mg concentrations. Aboveground N, C, P, and Mg concentrations and oven-dried aboveground dry matter were used to calculate aboveground N, C, P, and Mg uptake ( $\text{kg ha}^{-1}$ ) in the P-source study, while only aboveground P uptake ( $\text{kg ha}^{-1}$ ) was calculated from the aboveground P concentration and oven-dried aboveground dry matter in the P-rate study.

All rice grain from both studies harvested with the plot combine was collected and bagged. Rice grain samples were air-dried for 14 days at approximately 25°C. A sub-sample of air-dried grain from each plot was oven-dried for 48 h at 70°C. A sub-sample of oven-dried rice grain harvested from the P-source study only in 2019 and 2020 was mechanically pulverized for measurement of TN, TC, P, and Mg concentration as previously described. Total N, C, P, and Mg concentrations and oven-dried yield were used to calculate total N, C, P, and Mg accumulation ( $\text{kg ha}^{-1}$ ) in the grain. All plant analyses were conducted at the University of Arkansas, Division of Agriculture's Agricultural Diagnostic Laboratory in Fayetteville.

### **Statistical Analyses**

Based on a randomized complete block design, the effect of site (i.e., year, 2019 and 2020) on sand, silt, and clay content, and initial soil properties (i.e., soil pH and EC; Mehlich-3

extractable soil P, K, Ca, Mg, S, Na, Fe, Mn, Zn, Cu, and B; and SOM, TC, and TN concentrations) prior to any fertilizer-P addition was evaluated by a one-factor analysis of variance (ANOVA) in SAS (SAS, 2013) using the PROC GLIMMIX procedure. A gamma distribution was used for sand, silt, and clay content, and soil pH, EC, and extractable soil nutrient concentration data, while a beta distribution was used for SOM, TC, and TN concentration data because these properties were reported as percentages.

For the P-source study, the experimental design was a randomized complete block for each year, with fertilizer-P treatment as the single factor. Since the actual location of the plots changed each year, for the 2-year study, site was treated as the whole-plot factor and fertilizer-P treatment was treated as the split-plot factor. A two-factor ANOVA was conducted in SAS (SAS, 2013) using the PROC GLIMMIX procedure to evaluate the effects of site (2019 and 2020), fertilizer-P treatment (ECST, CPST, TSP, MAP, DAP, RP, and UC), and their interactions on rice aboveground dry matter (DM), above- and belowground and grain N, P, Mg and C tissue concentrations, aboveground and grain N, P, Mg, and C tissue content, and yield. A beta distribution was used for above- and belowground and grain N, P, Mg, and C concentration data because these properties were reported as percentages, while a gamma distribution was used for aboveground DM, yield, aboveground and grain N, P, Mg, and C content data.

For the P-rate study, the effect of fertilizer rate on rice yield and aboveground P uptake were evaluated with a one-factor ANOVA in SAS (SAS, 2013) using the PROC GLIMMIX procedure based on a randomized complete block design. Each site was analyzed separately because the objective of the study was to determine the yield-maximizing P rate by site among the four TSP fertilizer rates. A gamma distribution was used for rice yield and aboveground P

uptake data. For all statistical analyses, significance was judged at  $P < 0.05$ . When appropriate, means from were separated by least significant difference at the 0.05 level.

## **Results and Discussion**

### **Initial Soil Properties**

Since both field studies were conducted in different areas within the same field each year, it was necessary to assess the difference in initial soil properties between the two sites for better understanding of potential fertilizer-P-source effects on rice properties. Initial soil pH, EC, extractable soil Ca, Mg, S, Na, Fe, Mn, B, and SOM concentrations in the P-source study and initial soil pH, EC, extractable soil Ca, S, Na, Mn, B, and SOM concentrations in the P-rate study differed between sites ( $P < 0.05$ ). Sand, silt, clay, and extractable soil P, K, Zn, and Cu, and TN and TC concentrations in both studies and extractable soil Mg and Fe concentrations in the P-rate study only were unaffected by site ( $P > 0.05$ ; Table 2).

For the P-source study, initial soil pH was 0.4 units greater in 2020 than in 2019 (Table 3). Similar to soil pH, EC was 1.4 times greater in 2020 than in 2019 (Table 3). Initial extractable soil Ca, S, Na, and Fe concentrations were 19, 30, 47, and 6 %, respectively, greater in 2020 than in 2019 (Table 3). In contrast, initial soil Mg, Mn, B, and SOM concentrations were 9, 18, 29, and 12 %, respectively, greater in 2019 than in 2020 (Table 3). The initial extractable soil Ca and Mg levels in both years were above the optimum category ( $> 400 \text{ mg kg}^{-1}$  for Ca and  $> 30 \text{ mg kg}^{-1}$  for Mg) based on the interpretation of soil nutrient concentration ranges and soil test levels of surface soil samples for most row crops in Arkansas (Espinosa, Slaton, & Mozaffari, 2021). Although some of the soil-test factors were different, neither rice growth nor yield were expected to be negatively impacted.



For the P-rate study, initial soil pH and EC were 0.8 units and 1.3 times, respectively, greater in 2020 than in 2019 (Table 3). Initial extractable soil Ca, S, and Na concentrations were 17, 39, and 55 %, respectively, greater in 2020 than in 2019 (Table 3). In contrast, initial soil Mn, B, and SOM concentrations were 14, 29, and 12 %, respectively, greater in 2019 than in 2020 (Table 3). Table 3 also reports means for all initial soil properties that were unaffected by site.

Based on soil sampling in April 2019 and May 2020, after plot establishment, but before rice planting, Mehlich-3-extractable soil P averaged 19.0 mg kg<sup>-1</sup> [standard error (SE) = 0.3] in 2019 and 19.9 mg kg<sup>-1</sup> (SE = 0.9) in 2020 in the top 10 cm, which showed that both sites were in on the low end of the medium category (17 to 25 mg kg<sup>-1</sup>) for flood-irrigated rice production based on the measured soil-test-P concentration and soil pH (Roberts et al., 2016), thus a plant response from added fertilizer-P in both years was expected.

#### Rice Response in the P-source Study

Rice response to the various fertilizer-P sources evaluated was variable, where 12 of the 22 total rice properties evaluated were affected ( $P < 0.05$ ) by fertilizer-P source, site, or both, while 10 rice properties were unaffected ( $P > 0.05$ ) by fertilizer-P source or site (Table 4). Contrary to expectations, aboveground rice DM was unaffected ( $P > 0.05$ ) by fertilizer-P source or site (Table 4), despite the soil-test-P levels in both years being below optimum, such that a plant response was expected, at least from the P-fertilized treatments relative to the unamended control. Aboveground rice DM ranged from 11.8 Mg ha<sup>-1</sup> from CPST in 2020 to 16.1 Mg ha<sup>-1</sup> from RP in 2020 and averaged 14.1 Mg ha<sup>-1</sup> across all fertilizer-P sources and both years (Table 5).

The alkaline soil in 2019 and 2020, with a large concentration of initial extractable soil Ca, likely precipitated and fixed more available P as Ca-phosphate (Ca-P) from the dissolution of fertilizer-P, thus resulting in no difference in aboveground DM among fertilizer-P sources or sites. The lack of difference in aboveground DM among fertilizer-P sources or sites could have, in part, been due to rice-root-induced acidification and exudation of organic acid anions, such as citrate (Kirk, George, Courtois, & Senadhira, 1998; Kirk, Santos, & Findenegg, 1999; Aulakh et al., 2001), which may have decreased the soil pH, causing dissolution of fixed Ca-P (Hoffland, Boogaard, Nelemans, & Findenegg 1992; Kirk et al., 1999). The precipitation of Ca-citrate (Dinkelaker, Römheld, & Marschner, 1989) at a lower soil pH and/or the chelation of metal ions, particularly Fe, Al, and Mn, that would otherwise immobilize P, could have also contributed increased P availability for plant uptake among all treatments (Kirk et al., 1999). It is plausible that some P was released from soil organic matter through mineralization during flooding (Willet, 1989) to have contributed to the lack of difference in aboveground DM among fertilizer-P sources or sites. Based on direct visual observations in the field, uniform P availability among treatments to result in a lack of difference in aboveground biomass may have also occurred as rice roots grew past the 10-cm soil depth, which was the lower depth of soil sampling used to determine the initial soil-test P and fertilizer-P rate. Furthermore, upon application of the flood and anaerobic conditions develop in the soil, some of the adsorbed, oxidized Fe becomes reduced, releasing some of the previously adsorbed P (Roberts et al., 2016). The mechanism could have provided additional plant-available P that was not measured initially and that masked potential and expected differences in plant response among fertilizer-P sources.

Though no field studies have been previously conducted evaluating struvite as a potential fertilizer-P source for rice, similar results to the current study have been reported for other crops

(Gell et al., 2011; Ylagan et al., 2020). In an 11-week greenhouse pot study, Ylagan et al. (2020) reported that soybean aboveground DM was unaffected by eight fertilizer-P treatments, including ECST, CPST, TSP, MAP, DAP, RP, and no P/+N and no P/-N controls in a silt loam (Typic Fragiudults) with a soil pH of 7.3. Additionally, in a 135-day corn field experiment, Gell et al. (2011) evaluated the effectiveness of two struvite materials recovered from human urine and black water as fertilizer-P sources compared with TSP and an unamended control in a sandy loam Andisol with a low soil-test P and pH of 4.5. Gell et al. (2011) reported no difference between corn DM across all treatments (i.e., struvite, TSP, and an unamended control). In contrast, other studies have reported differences in aboveground DM among fertilizer-P sources (Rech et al., 2019; Thompson, 2013). Rech et al. (2019) reported wheat shoot DM from TSP to be 1.3 and 1.7 times greater than that from each of three struvite sources and an unamended control, respectively, while soybean shoot DM from TSP was similar to that from three struvite sources and was 1.6 times greater than that from the control.

Similar to aboveground DM, aboveground tissue P, N, and Mg concentrations were unaffected ( $P > 0.05$ ) by fertilizer-P source or site (i.e., year; Table 4). In addition, aboveground tissue C concentration was unaffected ( $P > 0.05$ ) by fertilizer-P source. However, averaged across fertilizer-P sources, aboveground tissue C concentration was 1.1 times greater ( $P < 0.01$ ) in 2020 than in 2019 (Table 6). Table 5 summarizes the overall range and average aboveground tissue P, N, and Mg concentrations across all fertilizer-P-source-site combinations.

The lack of aboveground tissue P concentration difference among fertilizer-P treatments was likely due to the soil's large P-adsorption capacity such that the P released from fertilizer-P was unavailable for plant uptake as a result of P adsorption to clays and/or secondary mineral formation by precipitation with Ca, which was also present in large concentrations in the top 10

cm (Table 3), in the alkaline soil throughout the study area (Khiari & Parent, 2005; Sims & Pierzynski, 2005; Khademi et al., 2010; Roberts et al., 2016; Table 3). Phosphorus availability to rice is optimum when the soil pH is below 6.5, but when the soil pH is greater than 6.5, the P is associated with Ca and Mg and the availability of P from Ca-P complexes could be low for several weeks after flood establishment in pH > 6.5 (Roberts et al., 2016). Rice roots may have also extracted sufficient soil P from below the top 10 cm to mask potential differences among fertilizer-P sources. In addition, the release of previously unavailable P from the reduction of Fe after imposition of the flood may have counteracted variations in solubilities to conceal differences in aboveground tissue P concentration among fertilizer-P sources.

In a plant-less soil incubation study, Anderson et al. (2021a) reported similar total Mehlich-3 soil P concentrations among CPST, TSP, and MAP in a silty clay loam and two silt-loam soils under flooded conditions, which suggests a comparative agronomic potential of struvite to traditional fertilizer-P sources under flooded-soil condition. Ylagan et al. (2020) reported that corn stem-plus-leaves tissue P concentration was numerically largest from ECST, which differed from all other treatments. In addition, Ylagan et al. (2020) reported that soybean stem-plus-leaves tissue P concentration was similar between ECST, CPST, MAP, TSP, and DAP treatments, which differed from RP and no P/+N and no P/-N control treatments.

The preplant N application as uncoated urea to unify the N rate across all fertilizer-P treatments, including the unamended control, and the single pre-flood N applied to all treatments as uncoated urea to the dry soil surface, which was flooded the following day after application to minimize ammonia volatilization loss, was likely uniformly available for plant uptake, thus resulting in the lack of aboveground N tissue concentration difference relative to the unamended control. Ylagan et al. (2020) confirmed similar results to the current study in soybean stem-plus-

leaves and pod tissue N concentrations. However, Ylagan et al. (2020) reported fertilizer-source differences in other plant properties, where corn stem-plus-leaves tissue N concentration from ECST and CPST, which did not differ from that from DAP and RP, was at least 1.2 times greater from both struvite materials than that from TSP and both control treatments. Corn stem-plus-leaves tissue N concentration from ECST did not differ from that from MAP, but was 1.1 times greater from CPST than from MAP.

The lack of aboveground Mg tissue concentration difference relative to the unamended control was likely due to the inherent large concentrations of initial extractable soil Mg ( $> 300 \text{ mg kg}^{-1}$ ) in the top 10 cm of the soil (Table 3), despite the two struvite sources containing greater Mg concentrations than the other fertilizer-P sources (Table 1). Furthermore, rice roots may have extracted sufficient soil Mg from below the top 10 cm to mask potential differences among fertilizer-P sources. Similar results were reported by Ylagan et al. (2020) for corn cob-plus-husk and soybean pod tissue Mg concentrations. However, soybean and corn stem-plus-leaves tissue Mg concentrations differed among fertilizer-P treatments, which was likely due to the low concentration of initial extractable soil Mg (mean =  $33 \text{ mg kg}^{-1}$ ; Ylagan et al., 2020).

Similar to aboveground tissue C concentration, aboveground tissue C content was unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 4). However, averaged across fertilizer-P sources, aboveground tissue C content was 1.1 times greater ( $P < 0.05$ ) in 2020 than in 2019 (Table 6). Similar to aboveground DM and aboveground tissue P, N, and Mg concentrations, aboveground tissue P and N uptake were unaffected ( $P > 0.05$ ) by fertilizer-P source or site (i.e., year; Table 4). Aboveground P uptake ranged from  $25 \text{ kg ha}^{-1}$  from CPST in 2020 to  $38 \text{ kg ha}^{-1}$  from RP in 2020 and averaged  $32 \text{ kg ha}^{-1}$  across all fertilizer-P sources and both years (Table 5).

Aboveground N uptake ranged from 94 kg ha<sup>-1</sup> from CPST in 2020 to 160 kg ha<sup>-1</sup> from RP in 2020 and averaged 128 kg ha<sup>-1</sup> across all fertilizer-P sources in both years (Table 5).

Similar to results of the current study, Gell et al. (2011) reported no difference between corn P uptake across all treatments (i.e., struvite, TSP, and an unamended control). However, studies have reported a difference in aboveground tissue P uptake among fertilizer-P treatments in other crops (Cabeza, Steingrobe, Romer, & Claassen 2011; Rech et al., 2019). Rech et al. (2019) reported that wheat total P uptake from TSP was similar to that from two struvite materials and was 1.3 and 2.1 times greater than that from CPST and an unamended control, respectively. Soybean total P uptake from TSP was similar to that from the three struvite materials and was 1.7 times greater than that from the control (Rech et al., 2019). In a 2-year pot experiment, Cabeza et al. (2011) reported that corn shoot P uptake from sewage-treatment-plant-recovered struvite and TSP, which did not differ, was twice as large as that from an unamended control. Furthermore, in two soils, corn shoot P uptake from RP did not differ from that from the control in the first year, but in the second year, corn shoot P uptake from RP was greater than that from the control (Cabeza et al., 2011).

In contrast to aboveground tissue P and N uptake, aboveground tissue Mg uptake differed among fertilizer-P sources between sites (i.e., years) ( $P = 0.03$ ; Table 4). In 2019, aboveground tissue Mg uptake did not differ among fertilizer-P sources, ranging numerically from 30.7 kg ha<sup>-1</sup> from RP to 35.5 kg ha<sup>-1</sup> from ECST (Table 7). However, in 2020, aboveground tissue Mg uptake was numerically largest from RP (42 kg ha<sup>-1</sup>), which did not differ from that from ECST, DAP, TSP, and the unamended control, and was numerically smallest from CPST (24.8 kg ha<sup>-1</sup>), which differed from all other treatments (Table 7). With the exception of RP and CPST, all aboveground tissue Mg uptake were similar ( $P > 0.05$ ) in 2019 and 2020 from their respective

fertilizer-P source (Table 7). Aboveground tissue Mg uptake was at least 1.3 times greater from RP in 2020 than in 2019 (Table 7), while aboveground tissue Mg uptake was 1.4 times greater from CPST in 2019 than in 2020 (Table 7).

The greater amount of rainfall in summer 2019 (June and July) compared to summer 2020 (Figure 2) likely reduced the soil pH, which may have facilitated quicker dissolution of CPST granules and the release of  $Mg^{2+}$  from CPST in 2019 than in 2020, thus resulting in the greater aboveground tissue Mg uptake. In addition, the initial extractable soil Mg concentration was 1.1 times greater in 2019 than in 2020 (Table 3). The greater aboveground tissue Mg uptake from RP in 2020 than in 2019 was unexpected due to the greater concentrations of initial extractable soil Mg in 2019, RP's low solubility (Kumari & Phogat, 2008), and RP's low Mg concentration (Table 1). However, it is plausible that the greater rainfall in May 2020 than in May 2019 (Figure 2) could have facilitated greater RP solubility and release of Mg that became available for plant uptake.

Similar to aboveground tissue C concentration and uptake, belowground P, N, and C tissue concentrations were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 4). However, averaged across fertilizer-P sources, belowground P tissue concentration was 1.3 times greater ( $P < 0.01$ ) in 2020 than in 2019 (Table 6), while belowground N and C tissue concentrations were 1.3 and 1.2 times, respectively, greater ( $P < 0.05$ ) in 2019 than in 2020 (Table 6).

Total rainfall during May 2020, before flooding the rice, was greater than in May 2019 and the 30-year mean rainfall (Figure 2). The initial extractable soil P concentration was also numerically greater in 2020 than in 2019, but did not significantly differ. The above-normal rainfall in 2020 likely created more ideal conditions promoting dissolution of adsorbed P and microbial mineralization of soil organic matter, which facilitated greater P diffusion, thus

resulting in greater belowground P tissue concentration in 2020 than in 2019, as P is critical for root development. The lack of belowground tissue P concentration difference among fertilizer-P sources could at least partially be due to sufficient sub-soil P below the top 10 cm that rice roots were able to extract to mask potential differences among fertilizer-P sources.

Similar results to the current study were reported by Rech et al. 2019, where soybean root P concentration did not differ among fertilizer-P sources. However, previous studies have reported significant differences in belowground tissue P concentration among fertilizer-P treatments from wheat (Rech et al., 2019), soybean, and corn (Ylagan et al., 2020). Wheat root P concentration did not differ between TSP and three struvite materials, but was greater than that from the unamended control (Rech et al., 2019). Additionally, soybean belowground tissue P concentrations from ECST and CPST were similar to each other and lower than that from MAP and greater than that from unamended controls (Ylagan et al., 2020). Corn belowground tissue P concentrations from ECST did not differ from that from RP and the unamended controls, but were lower than that from CPST, TSP, MAP, and DAP (Ylagan et al., 2020).

The uniform total N rate applied to all treatments likely caused the lack of a fertilizer-P treatment effect on belowground tissue N concentration. The greater belowground tissue N concentration in 2019 could have been the result of increased microbial activity, therefore facilitating greater N mineralization for plant uptake in 2019 than in 2020 as the rice plots were in different locations each year. Similar results to the current study were reported by Ylagan et al. (2020), where soybean belowground tissue N concentration did not differ among fertilizer-P treatments. However, corn belowground tissue N concentration from CPST, which was similar to that from TSP, MAP, DAP, RP, and the no P/+N control, was at least 1.4 times greater than that from ECST and the no P/-N control (Ylagan et al., 2020).



Similar to aboveground DM, aboveground tissue P, N, and Mg concentrations, and aboveground tissue P and N uptake, belowground tissue Mg concentration was unaffected ( $P > 0.05$ ) by fertilizer-P source or site (Table 4). Belowground tissue Mg concentration ranged from 0.17 % from MAP in 2020 to 0.20 % from CPST in 2020 and averaged 0.19 % across all fertilizer-P sources and both years (Table 5). The lack of belowground tissue Mg concentration difference relative to the unamended control was likely due to the inherent large concentration of initial extractable soil Mg throughout the study area (Table 3). Similar results were reported by Ylagan et al. (2020), where soybean and corn belowground tissue Mg concentrations did not differ among fertilizer-P treatments.

In contrast to aboveground dry matter, rice grain yield differed among fertilizer-P sources between sites (i.e., years) ( $P = 0.04$ ; Table 4). In 2019, grain yields did not differ among fertilizer-P sources, ranging numerically from 10.8 Mg ha<sup>-1</sup> from the unamended control to 11.7 Mg ha<sup>-1</sup> from TSP (Table 7), despite the ECST treatment receiving 1.3 times more urea-N than intended. However, in 2020, grain yield was numerically largest from TSP (9.8 Mg ha<sup>-1</sup>), which did not differ from that from DAP, MAP, RP, and the UC, and was numerically smallest from ECST (8.2 Mg ha<sup>-1</sup>), which did not differ from CPST and was lower than that from TSP, DAP, MAP, RP, or the UC (Table 7). In 2020, grain yield from CPST (8.9 Mg ha<sup>-1</sup>) did not differ from that from DAP, MAP, RP, or the UC (Table 7). Rice grain yield was at least 1.1 times lower in 2020 from the two struvite materials (ECST and CPST) than from TSP (Table 7).

Rice yield response was expected from added fertilizer-P in both years because the initial soil-test P concentration in both years was in the medium category for optimal flood-irrigated rice production (Roberts et al., 2016), indicating P would likely limit rice growth and productivity. Though grain yield from both struvite sources (ECST and CPST) used in the

current study were similar within each site (i.e., year), the lower grain yield from ECST and CPST than TSP in 2020 was potentially due to differential P transport once in the rice plants, as belowground tissue P concentrations were similar among all fertilizer-P sources each year (Table 4), but different batches of laboratory-made ECST were used between years. Though not the focus of this study, hence not evaluated, it is possible that the exact forms of P taken up by the rice plants from the soil differed somewhat among fertilizer-P sources and/or there were slight variations in chemical transformations that occurred to the P once in the rice plant (Ylagan et al., 2020) that resulted in differential P transport in the rice plants that affected grain yield. However, grain tissue P concentration did not differ among fertilizer-P sources or between years (Table 4).

With the exception of TSP, rice grain yields from all other fertilizer-P treatments were lower ( $P < 0.05$ ) in 2020 than those in 2019 (Table 7) despite belowground tissue P concentrations, averaged across all fertilizer-P sources, being greater in 2020 than in 2019 (Table 8). Rice grain yield was at least 1.1 times greater from ECST, CPST, DAP, MAP, RP, TSP, and UC treatments in 2019 than their respective treatment in 2020, while the grain yield from the UC ( $10.8 \text{ Mg ha}^{-1}$ ) in 2019 did not differ from that from TSP ( $9.8 \text{ Mg ha}^{-1}$ ) in 2020 (Table 7). Though aboveground DM did not differ between years, aboveground tissue C and grain N content were greater in 2020 than in 2019 (Table 6), indicating the rice plants actually grew well in 2020 compared to 2019, but that improved growth did not translate into greater grain yield, which may have been limited by the lower grain P uptake in 2020 compared to 2019 (Table 6) from potentially differential P transport within the rice plants.

Averaged across all fertilizer-P sources, rice grain yields from the current study were similar (2020) to slightly greater (2019) than those from the Arkansas Rice Performance Trial yield results in 2019 ( $9.0 \text{ Mg ha}^{-1}$ ) for the same pureline rice cultivar ('Diamond') grown at

PTRS (Moldenhauer, Scott, & Hardke, 2020). In 2019, grain yield from the current study ranged from 10.8 Mg ha<sup>-1</sup> from the UC to 11.7 Mg ha<sup>-1</sup> from TSP and averaged 11.4 Mg ha<sup>-1</sup> across all fertilizer-P sources (Table 7). In 2020, grain yield from the current study ranged from 8.2 Mg ha<sup>-1</sup> from ECST to 9.8 Mg ha<sup>-1</sup> from TSP and averaged 9.2 Mg ha<sup>-1</sup> across all fertilizer-P sources (Table 7). These results confirm that the rice grown in 2019 and 2020 did not under-perform compared to that from the rice grown under optimal conditions according to University of Arkansas recommendations in the 2019 Arkansas Rice Performance Trials.

Similar to aboveground DM, grain tissue N concentration was unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 4). However, averaged across fertilizer-P sources, grain tissue N concentration was 1.1 times greater ( $P < 0.01$ ) in 2019 than in 2020 (Table 6). Application of a uniform total N rate to all treatments was likely why grain tissue N concentration was unaffected by fertilizer-P treatments. Similar to the results of the current study, Ylagan et al. (2020) reported soybean pod tissue N concentration was unaffected by fertilizer-P treatments.

Similar to aboveground tissue Mg uptake and grain yield, grain tissue C concentration differed among fertilizer-P sources between sites (i.e., years) ( $P < 0.01$ ; Table 4). In 2019, grain tissue C concentration was numerically largest from RP (43.4%), which did not differ from that of ECST, CPST, TSP, MAP, and the UC, and was numerically smallest from DAP (43.0%), which was lower than from RP (Table 7). In 2020, grain tissue C concentration from ECST did not differ from that from CPST, MAP, RP, and the UC (Table 7). Grain tissue C concentration from DAP and TSP, which did not differ, was greater than that in the ECST treatment in 2020 (Table 7). Grain tissue C concentration from CPST did not differ from that of DAP and TSP, but was 1.1 times greater than that of MAP, RP, and the UC (Table 7).

Similar to aboveground DM, aboveground tissue P and N concentrations and uptake, and above- and belowground Mg tissue concentrations, grain tissue P and Mg concentrations were unaffected ( $P > 0.05$ ) by fertilizer-P source or site (Table 4). Grain tissue P concentration ranged from 0.25% from MAP and CPST in 2020 to 0.31% from RP in 2019 and averaged 0.27% across all fertilizer-P sources (Table 5). Grain tissue Mg concentration ranged from 0.11% from TSP in 2019 to 0.13% from RP in 2019 and averaged 0.12% across all fertilizer-P sources (Table 5). The lack of difference in grain tissue P concentration among fertilizer sources may have at least been partially related to the additional plant-available P created upon Fe reduction following flood establishment. Despite the lack of grain tissue P concentration difference among fertilizer-P sources, it is possible that the form of P that was translocated to the rice grain differed somewhat among fertilizer-P sources to have resulted in differences in rice yield among fertilizer-P sources between years (Table 4). The lack of grain tissue Mg concentration difference among fertilizer-P treatments, despite variations in Mg concentrations among the fertilizer-P materials (Table 1), was likely due to the large concentrations of initial extractable soil Mg (Table 3). Similar to the results of the current study, Ylagan et al. (2020) reported no difference in soybean pod tissue Mg concentration among fertilizer-P treatments. However, soybean pod tissue P concentration from ECST, which did not differ from that from CPST, TSP, MAP, and DAP, differed from that from RP and the control treatments, which did not differ (Ylagan et al., 2020).

In contrast to above- and belowground and grain tissue concentrations and aboveground tissue uptake, only rice grain N uptake differed ( $P < 0.01$ ; Table 4) among fertilizer-P sources. Averaged across sites (i.e., years), rice grain N uptake was numerically largest from TSP (144 kg ha<sup>-1</sup>), which did not differ from that from RP, MAP, or DAP, and was numerically smallest from CPST (130 kg ha<sup>-1</sup>), which did not differ from that from ECST, DAP, and the UC (Table 8).

Grain N uptake from TSP, RP, and MAP was 1.1 times greater than that from both struvite materials, which averaged 131 kg ha<sup>-1</sup> (Table 8). The greater grain N uptake from TSP, RP, and MAP than both struvite materials support the lower solubility and slow-release characteristic of struvite materials relative to other fertilizer-P sources (Massey et al., 2009; Chauhan, Vyas, & Joshi, 2011; Talboys et al., 2015). The consistent similarities in plant response from the two struvite materials (ECST and CPST) suggest that difference in physical form applied (i.e., crystalline for ECST and prill for CPST) between the struvite sources had little to no effect on P release and behavior of the materials. Anderson et al. (2021c) reported a similar incremental increase from the initial value of water-soluble and Mehlich-3 soil P concentrations between ECST- and CPST-treated soil in a silty clay loam and two silt-loam soils under flooded condition after 1, 2, 3, and 4 months of a plant-less soil incubation study. Averaged across fertilizer-P sources, grain tissue N uptake was at least 1.3 times greater ( $P < 0.01$ ) in 2019 than in 2020 (Table 6).

Similar to aboveground tissue Mg uptake, grain yield, and grain tissue C concentration, grain tissue C content differed among fertilizer-P sources between sites (i.e., years) ( $P = 0.02$ ; Table 4). In 2019, grain tissue C content did not differ among fertilizer-P sources, ranging numerically from 6253 kg ha<sup>-1</sup> from the UC to 6753 kg ha<sup>-1</sup> from RP (Table 7). However, in 2020, grain tissue C content was numerically largest from TSP (4881 kg ha<sup>-1</sup>), which did not differ from that from DAP, MAP, and the UC, and was numerically smallest from ECST (4029 kg ha<sup>-1</sup>), which differed from CPST, TSP, DAP, MAP, RP, or the UC (Table 7). In 2020, grain tissue C content from CPST (4399 kg ha<sup>-1</sup>), which did not differ from that from DAP, MAP, RP, or the UC, was 1.1 times greater than that from ECST (Table 7). Grain tissue C content,

averaged across all fertilizer-P sources, was 1.5 times greater in 2019 than in their respective treatments in 2020 (Table 7).

Similar to grain tissue C concentrations, grain tissue P uptake was unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 4). However, averaged across fertilizer-P sources, grain tissue P uptake was at least 1.2 times greater ( $P = 0.01$ ) in 2019 than in 2020 (Table 6). The larger P-adsorption characteristic of the more alkaline soil, with greater initial extractable Ca, in 2020 than 2019 (Table 3) likely contributed to the interannual variation in grain tissue P uptake.

Similar to aboveground DM, aboveground tissue P and N concentrations and uptake, above- and belowground and grain Mg tissue concentrations, and grain P tissue concentration, grain tissue Mg uptake was unaffected ( $P > 0.05$ ) by fertilizer-P source or site (Table 4). Grain tissue Mg uptake ranged from 13 kg ha<sup>-1</sup> from CPST in 2020 to 20 kg ha<sup>-1</sup> from RP in 2019 and averaged 15.8 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 5). The large initial extractable soil Mg concentration in both years (Table 3) likely contributed to the lack of grain tissue Mg uptake differences among fertilizer-P treatments.

#### Rice Response in the P-rate Study

The P-rate study was conducted to determine the yield-maximizing P rate from four TSP fertilizer rates (0, 22, 44, and 66 kg P ha<sup>-1</sup>) applied to the same P-deficient, silt-loam soil each year as used for the P-source study. However, rice response to the various TSP rates evaluated was variable, where only one of the four rice properties evaluated (i.e., 2019 grain yield) was affected ( $P < 0.05$ ), while three rice properties were unaffected ( $P > 0.05$ ) by fertilizer rate (Table 9).

Contrary to the expected plant response due to sub-optimal initial extractable soil P in the top 10 cm (Table 3), aboveground P uptake was unaffected ( $P > 0.05$ ) by fertilizer rate in 2019 (Table 9). In 2019, aboveground P uptake ranged from 23.2 kg P ha<sup>-1</sup> from the 22 kg P ha<sup>-1</sup> rate to 29.5 kg P ha<sup>-1</sup> from the 44 kg ha<sup>-1</sup> rate and averaged 26.6 kg P ha<sup>-1</sup> across all fertilizer rates (Table 9). However, in 2019, grain yield from the 22, 44, and 66 kg P ha<sup>-1</sup> rates, which did not differ, were 1.1 times greater than that from the 0 kg P ha<sup>-1</sup> rate (Table 9). Among the four fertilizer rates evaluated in the P-rate study in 2019, the 22 kg ha<sup>-1</sup> rate, which produced the numerically largest grain yield (Table 9), was the closest to the recommended rate of 29.4 kg P ha<sup>-1</sup> used in the P-source study. From the perspective of a producer, it would be reasonable to choose the 22 kg P ha<sup>-1</sup> rate over 66 kg P ha<sup>-1</sup> rate due to economic reasons, though grain yields from these two rates did not differ ( $P > 0.05$ ). This result supports that the recommended rate 29.4 kg P ha<sup>-1</sup> used in the P-source study was optimum for maximizing rice grain yield in 2019.

Similar to aboveground P uptake in 2019, aboveground P uptake and grain yield in 2020 were unaffected ( $P > 0.05$ ) by fertilizer rate (Table 9). In 2020, aboveground P uptake ranged from 26.3 kg ha<sup>-1</sup> from the 0 kg P ha<sup>-1</sup> rate to 36.2 kg ha<sup>-1</sup> from the 66 kg P ha<sup>-1</sup> rate and averaged 31.5 kg P ha<sup>-1</sup> across all fertilizer rates (Table 9). In 2020, grain yield ranged from 9.1 Mg ha<sup>-1</sup> from the 0 kg P ha<sup>-1</sup> rate to 9.6 Mg ha<sup>-1</sup> from the 66 kg P ha<sup>-1</sup> rate and averaged 9.4 Mg ha<sup>-1</sup> across all fertilizer rates (Table 9). Though a rice yield response was expected from the added fertilizer P each year, there was no grain yield difference among fertilizer rates in 2020, suggesting that P did not limit grain yield in 2020. Following grain yield from the 66 kg P ha<sup>-1</sup> rate, which was numerically largest, grain yield from the 22 kg P ha<sup>-1</sup> rate was next numerically largest in 2020 (Table 9).

The lack of plant growth benefit, as determined by similar aboveground tissue P uptake, both years from fertilizer-P additions may have been due to the ability of the rice roots to explore the soil for sufficient soil P below the top 10 cm, resulting in similar P availability for root uptake and masking potential differences among P-rate treatments. Plants are known to increase their root hair density and/or change their root architecture to improve soil- and/or fertilizer-P uptake (Gahoonia & Nielsen, 1992). In addition, the P released from the highly water-soluble TSP may have migrated by diffusion to a greater distance from the source, thereby encouraging plant roots to spread laterally (Rech et al., 2019), exposing the root to a potentially greater source of available soil P. Furthermore, potential differences in P uptake could have been masked from the increase in available P as previously unavailable P was released when Fe reduced as anaerobic soil conditions developed after imposing the flood.

## **Implications**

Recovering excess P as struvite can improve cost, energy, and treatment efficiencies of WWTPs and reduce P concentrations in WWTP effluent and P loading to receiving waters (Parsons et al., 2001; Tansel, Lunn, & Monje, 2018). Additionally, due to its N concentration, wastewater-recovered struvite can reduce fertilizer-N needs and can maintain or, in some instances, improve yields of various crops, including rice, making struvite a viable alternative blended fertilizer material from agricultural producers (Talboys et al., 2015).

Specific to the current study, a generally similar plant response was observed for aboveground DM, above- and belowground tissue P, N, Mg, and C concentrations, and aboveground tissue P, N, and C contents, grain tissue P, N, and Mg concentrations, and grain tissue P and Mg uptake among struvite materials (ECST and CPST) and TSP, which is the most



common fertilizer-P source used in rice production, which further supports the potential utilization of wastewater-recovered struvite as an alternative fertilizer-P source for various row crops. However, the similar grain tissue P uptake from the highly soluble TSP and the less soluble ECST and CPST materials suggests that the previously reported slow-release nature of struvite may not be entirely accurate for flood-irrigated rice, as the reducing conditions that develop following flood establishment for rice production may conceal variations in solubilities among various fertilizer-P sources.

With further research, struvite may become justifiably marketable as “environmentally friendly” or “green” in the future, which may allow producers to gain value from potential environmental premiums (Yetilmezsoy et al., 2017). As an alternative fertilizer-P source, struvite may help reduce the long-term threat to future food security from limited remaining quantities of minable rock phosphate from which current phosphate fertilizers are produced (Hallas, Mackowiak, Wilkie, & Harris, 2019).

## **Conclusions**

Previous research has demonstrated struvite’s agronomic benefits in several ornamental and vegetable crops and a few row crops. However, to our knowledge, no studies to date have examined struvite effects on flood-irrigated rice relative to other commercially available, commonly used fertilizer-P sources. As hypothesized, results demonstrated similar rice aboveground DM, above- and belowground tissue and grain P, N, and C concentrations, aboveground and grain tissue P uptake, and aboveground tissue N and C contents from wastewater-recovered struvite sources (i.e., ECST and CPST) compared to other common,

commercially available fertilizer-P sources (i.e., TSP, MAP, DAP, and RP). However, the hypothesis was only partially supported because grain yield and grain tissue N and C contents differed among ECST, CPST, TSP, MAP, DAP, and RP treatments.

Results did not support the hypothesis that aboveground tissue and grain Mg concentrations and uptake and belowground tissue Mg concentration would be greater for the struvite materials (ECST and CPST) treatment due to greater initial Mg concentrations. Similarly, results did not support the hypothesis that aboveground tissue P uptake would be greater from the 44 kg TSP-P ha<sup>-1</sup> rate compared to fertilizer rates of 22 and 66 kg TSP-P ha<sup>-1</sup>.

Results from this study overall provide valuable information about the response of rice to crystalline ECST and pelletized CPST compared with other conventional fertilizer-P source in a delayed-flood production system on a silt-loam soil in eastern Arkansas. Despite a large P adsorption capacity with large initial extractable Ca and alkaline soil pH, results clearly showed that wastewater-recovered struvite, from either chemical and/or electrochemical precipitation techniques, has potential to be a viable, alternative fertilizer-P source for flood-irrigated rice. Considering the ECST used in this study was produced from synthetic wastewater, further research is still required to evaluate the behavior and effectiveness of ECST produced from natural wastewater as a fertilizer-P source in various soil physiochemical conditions and row crops, particularly in flood-irrigated rice, under field conditions.

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## Tables and Figures

Table 1. Summary of the pH, total nitrogen (N), phosphorus (P), and magnesium (Mg) concentrations and resulting measured fertilizer grade for the two batches of electrochemically precipitated struvite (ECST) used each year and the chemically precipitated struvite (CPST), triple superphosphate (TSP), monoammonium phosphate (MAP), diammonium phosphate (DAP), and rock phosphate (RP) fertilizer-P materials used for the rice P-source study in a P-deficient, silt-loam soil in eastern Arkansas. Means ( $\pm$  standard error) are reported (n = 5).

Fertilizer-P Source	pH	Nutrient Concentration			Measured Fertilizer Grade <sup>†</sup>
		N	P	Mg	
		%			
ECST 2019	- <sup>††</sup>	3.3 (0.2)	18.5 (0.1)	13.3 (0.1)	3-42-0
ECST 2020	-	5.1 (0.2)	16.1 (0.3)	12.7 (0.3)	5-37-0
CPST	8.77 (0.13)	5.7 (0.2)	11.7 (0.2)	8.3 (0.2)	6-27-0
TSP	2.42 (0.02)	< 0.1 (< 0.1)	18.2 (0.4)	0.6 (< 0.1)	0-42-0
MAP	4.37 (0.02)	10.7 (0.1)	20.9 (0.2)	1.5 (< 0.1)	11-48-0
DAP	7.32 (0.03)	18.1 (0.1)	18.3 (0.1)	0.7 (< 0.1)	18-42-0
RP	6.67 (0.04)	< 0.1 (< 0.1)	7.6 (0.1)	0.3 (< 0.1)	0-17-0

<sup>†</sup> Measured fertilizer grade is reported as N-P<sub>2</sub>O<sub>5</sub>-K<sub>2</sub>O

<sup>††</sup> Limited supply of ECST prohibited pH determinations

Table 2. Analysis of variance summary of the effect of site (2019 and 2020) on sand, silt, and clay content, initial soil-test pH, electrical conductivity, Mehlich-3 extractable nutrient concentrations, and total nitrogen, total carbon, and soil organic matter concentrations in the top 10 cm for the rice phosphorus (P)-source and P-rate studies in a P-deficient, silt-loam soil in eastern Arkansas.

Soil Property	Effect of Site	
	P-source Study	P-rate Study
	<i>P</i>	
Sand	0.84	0.81
Silt	0.18	0.45
Clay	0.20	0.41
pH	< <b>0.01</b> <sup>†</sup>	< <b>0.01</b>
Electrical conductivity	< <b>0.01</b>	< <b>0.01</b>
Phosphorus	0.15	0.37
Potassium	0.16	0.63
Calcium	< <b>0.01</b>	< <b>0.01</b>
Magnesium	<b>0.02</b>	0.79
Sulfur	< <b>0.01</b>	< <b>0.01</b>
Sodium	< <b>0.01</b>	< <b>0.01</b>
Iron	<b>0.04</b>	0.37
Manganese	<b>0.02</b>	<b>0.03</b>
Zinc	0.18	0.11
Copper	0.18	0.23
Boron	< <b>0.01</b>	<b>0.02</b>
Total nitrogen	0.72	0.49
Total carbon	0.93	0.16
Soil organic matter	< <b>0.01</b>	< <b>0.01</b>

<sup>†</sup> Bolded values were considered significant at  $P < 0.05$

Table 3. Summary of initial soil properties in the top 10 cm for both sites (2019 and 2020) for the rice phosphorus (P)-source and P-rate studies in a P-deficient, silt-loam soil in eastern Arkansas.

Soil Property <sup>†</sup>	P-source Study			P-rate Study		
	2019	2020	Overall Mean	2019	2020	Overall Mean
Sand (kg kg <sup>-1</sup> )	11.8 a <sup>††</sup>	12.1 a	12.0	12.1 a	11.8 a	12.0
Silt (kg kg <sup>-1</sup> )	72.6 a	71.1 a	71.9	71.5 a	72.3 a	71.9
Clay (kg kg <sup>-1</sup> )	15.5 a	16.9 a	16.2	16.4 a	15.7 a	16.1
pH	7.4 b	7.8 a	-	7.2 b	8.0 a	-
EC (dS m <sup>-1</sup> )	0.20 b	0.28 a	-	0.21 b	0.28 a	-
P (mg kg <sup>-1</sup> )	19.0 a	19.9 a	19.5	18.9 a	19.7 a	19.3
K (mg kg <sup>-1</sup> )	81.1 a	73.2 a	77.2	72.5 a	74.4 a	73.5
Ca (mg kg <sup>-1</sup> )	1977 b	2427 a	-	2034 b	2443 a	-
Mg (mg kg <sup>-1</sup> )	337 a	305 b	-	319 a	316 a	318
S (mg kg <sup>-1</sup> )	9.1 b	13.0 a	-	8.3 b	13.6 a	-
Na (mg kg <sup>-1</sup> )	30.3 b	57.5 a	-	27.4 b	60.8 a	-
Fe (mg kg <sup>-1</sup> )	373 b	398 a	-	375 a	390 a	383
Mn (mg kg <sup>-1</sup> )	287 a	235 b	-	286 a	247 b	-
Zn (mg kg <sup>-1</sup> )	3.3 a	4.6 a	3.9	3.2 a	4.1 a	3.7
Cu (mg kg <sup>-1</sup> )	1.5 a	1.4 a	1.5	1.5 a	1.6 a	1.6
B (mg kg <sup>-1</sup> )	0.7 a	0.5 b	-	0.7 a	0.5 b	-
TN (%)	0.1 a	0.1 a	0.1	0.1 a	0.1 a	0.1
TC (%)	1.0 a	1.0 a	1.0	1.0 a	1.0 a	1.0
SOM (%)	2.5 a	2.2 b	-	2.5 a	2.2 b	-

<sup>†</sup> Electrical conductivity (EC), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), sulfur (S), sodium (Na), iron (Fe), manganese (Mn), zinc (Zn), copper (Cu), boron (B), total nitrogen (TN) and carbon (TC), soil organic matter (SOM)

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

Table 4. Summary of the effects of fertilizer-phosphorus (P) source, site, and their interactions on rice plant properties across 2019 and 2020 data in a P-deficient, silt-loam soil in eastern Arkansas.

Plant Property	Source of Variation		
	Source	Site	Fertilizer x Site
	<i>P</i>		
Aboveground			
Dry Matter	0.16	0.28	0.13
Phosphorus Concentration	0.35	0.06	0.20
Nitrogen Concentration	0.13	0.81	0.27
Magnesium Concentration	0.06	0.79	0.13
Carbon Concentration	0.79	< <b>0.01</b> <sup>†</sup>	0.18
Phosphorus Uptake	0.10	0.06	0.07
Nitrogen Uptake	0.13	0.25	0.07
Magnesium Uptake	0.07	0.25	<b>0.03</b>
Carbon Content	0.14	<b>0.04</b>	0.12
Belowground			
Phosphorus Concentration	0.09	< <b>0.01</b>	0.79
Nitrogen Concentration	0.54	< <b>0.01</b>	0.45
Magnesium Concentration	0.15	0.42	0.76
Carbon Concentration	0.55	<b>0.04</b>	0.36
Grain			
Yield	0.01	< 0.01	<b>0.04</b>
Phosphorus Concentration	0.54	0.51	0.42
Nitrogen Concentration	0.17	< <b>0.01</b>	0.40
Magnesium Concentration	0.57	0.68	0.59
Carbon Concentration	0.02	< 0.01	< <b>0.01</b>
Phosphorus Uptake	0.21	<b>0.01</b>	0.55
Nitrogen Uptake	< <b>0.01</b>	< <b>0.01</b>	0.33
Magnesium Uptake	0.28	0.06	0.67
Carbon Content	< 0.01	< 0.01	<b>0.03</b>

<sup>†</sup> Bolded values were considered significant at  $P < 0.05$

Table 5. Summary of rice plant properties that did not differ ( $P > 0.05$ ) among fertilizer-phosphorus (P)-source-site combinations in the P-source study in a P-deficient, silt-loam soil in eastern Arkansas.

<b>Plant Property</b>	<b>Minimum</b>	<b>Maximum</b>	<b>Mean</b>
<b>Aboveground</b>			
Dry Matter (Mg ha <sup>-1</sup> )	11.8	16.1	14.1
Phosphorus Concentration (%)	0.21	0.24	0.22
Nitrogen Concentration (%)	0.8	1.0	0.9
Magnesium Concentration (%)	0.21	0.26	0.24
Phosphorus Uptake (kg ha <sup>-1</sup> )	25.0	38.0	32.0
Nitrogen Uptake (kg ha <sup>-1</sup> )	94.0	160	128
<b>Belowground</b>			
Magnesium Concentration (%)	0.17	0.20	0.19
<b>Grain</b>			
Phosphorus Concentration (%)	0.25	0.31	0.27
Magnesium Concentration (%)	0.11	0.13	0.12
Magnesium Uptake (kg ha <sup>-1</sup> )	13.0	20.0	15.8



Table 6. Summary of mean rice plant properties for both sites (2019 and 2020) in the phosphorus (P)-source study in a P-deficient, silt-loam soil in eastern Arkansas.

<b>Plant Properties</b>	<b>Site</b>	
	<b>2019</b>	<b>2020</b>
Aboveground		
Carbon Concentration (%)	38 b <sup>†</sup>	40 a
Carbon Content (kg ha <sup>-1</sup> )	5248 b	5779 a
Belowground		
Phosphorus Concentration (%)	0.07 b	0.09 a
Nitrogen Concentration (%)	0.6 a	0.4 b
Carbon Concentration (%)	41.6 a	34.6 b
Grain		
Nitrogen Concentration (%)	1.04 a	0.97 b
Phosphorus Uptake (kg ha <sup>-1</sup> )	42 a	33 b
Nitrogen Uptake (kg ha <sup>-1</sup> )	157 a	119 b

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

Table 7. Summary of mean rice plant properties among fertilizer-phosphorus (P)-source-site combinations in the P-source study in a P-deficient, silt-loam soil in eastern Arkansas.

Fertilizer <sup>†</sup>	Site	Yield	kg ha <sup>-1</sup>		
			AGMgU <sup>†</sup>	GCCnt <sup>†</sup>	GCC <sup>†</sup>
					— % —
ECST	2019	11467 a <sup>††</sup>	35.5 ab	6618 a	43.3 ab
CPST	2019	10928 a	32.4 b	6283 a	43.2 ab
DAP	2019	11409 a	31.8 b	6557 a	43.0 b
MAP	2019	11672 a	34.1 ab	6745 a	43.3 ab
RP	2019	11658 a	30.7 bc	6753 a	43.4 a
TSP	2019	11689 a	30.8 bc	6749 a	43.3 ab
UC	2019	10778 ab	33.2 ab	6253 a	43.3 ab
ECST	2020	8223 e	37.8 ab	4029 d	36.8 de
CPST	2020	8903 de	24.8 c	4399 c	37.1 cd
DAP	2020	9246 dc	34.6 ab	4581 bc	37.3 c
MAP	2020	9533 dc	31.7 b	4634 bc	36.5 e
RP	2020	9144 dc	42.0 a	4463 c	36.5 e
TSP	2020	9842 bc	37.6 ab	4881 b	37.2 c
UC	2020	9521 dc	35.5 ab	4651 bc	36.7 e

<sup>†</sup> Electrochemically precipitated struvite (ECST), chemically precipitated struvite (CPST), diammonium phosphate (DAP), monoammonium phosphate (MAP), rock phosphate (RP), triple superphosphate (TSP), unamended control (UC), aboveground Mg (AGMgU) uptake, and grain C (GCCnt) content, and grain C (GCC) concentration

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

Table 8. Summary of mean rice grain nitrogen (N), phosphorus (P), and magnesium (Mg) uptake among fertilizer-P sources, averaged across 2019 and 2020, for the P-source study in a P-deficient, silt-loam soil in eastern Arkansas.

Fertilizer-P Source	Grain N	Grain P	Grain Mg
	Uptake	Uptake	Uptake
	kg ha <sup>-1</sup>		
Electrochemically precipitated struvite (ECST)	132 b <sup>†</sup>	34.9 a	14.9 a
Chemically precipitated struvite (CPST)	130 b	33.7 a	14.2 a
Diammonium phosphate (DAP)	136 ab	38.5 a	16.4 a
Monoammonium phosphate (MAP)	141 a	37.5 a	15.9 a
Rock phosphate (RP)	142 a	39.5 a	17.0 a
Triple superphosphate (TSP)	144 a	38.2 a	16.2 a
Unamended control (UC)	132 b	37.2 a	15.9 a

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

Table 9. Summary of the effect of fertilizer-phosphorus (P) rate on rice yield and aboveground P uptake (AGPU) for both sites (2019 and 2020) in a P-deficient, silt-loam soil in eastern Arkansas.

P Rate (kg ha <sup>-1</sup> )	Yield 2019	Yield 2020	AGPU 2019	AGPU 2020
0	10805 b <sup>†</sup>	9146 a	28.7 a	26.3 a
22	11478 a	9510 a	23.2 a	32.8 a
44	11280 a	9270 a	29.5 a	30.8 a
66	11423 a	9606 a	25.0 a	36.2 a
<i>P</i> -value	0.03	0.66	0.07	0.47

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

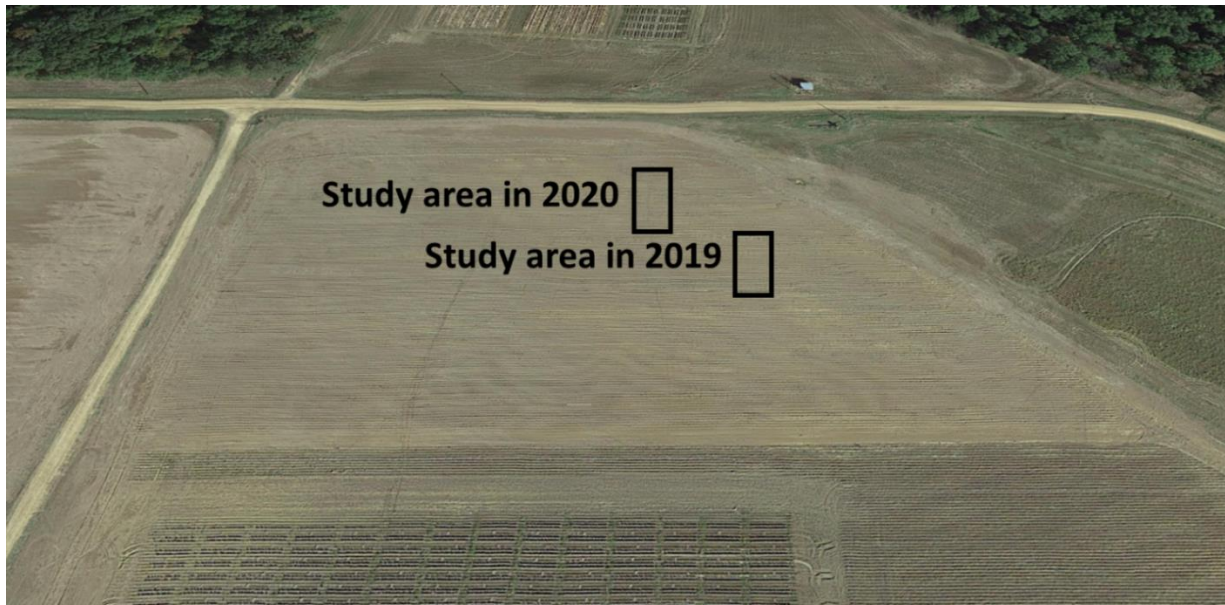


Fig. 1. Aerial view of the approximate plot arrangement for the phosphorus (P)-source and P-rate studies within the study area at the Pine Tree Research Station near Colt, AR. North is towards the top of the image. Individual plot dimensions were 4.9-m long by 1.8-m wide except for the electrochemically precipitated struvite plots (5-m long by 5-m wide). Rectangles marking study areas are not drawn to scale.

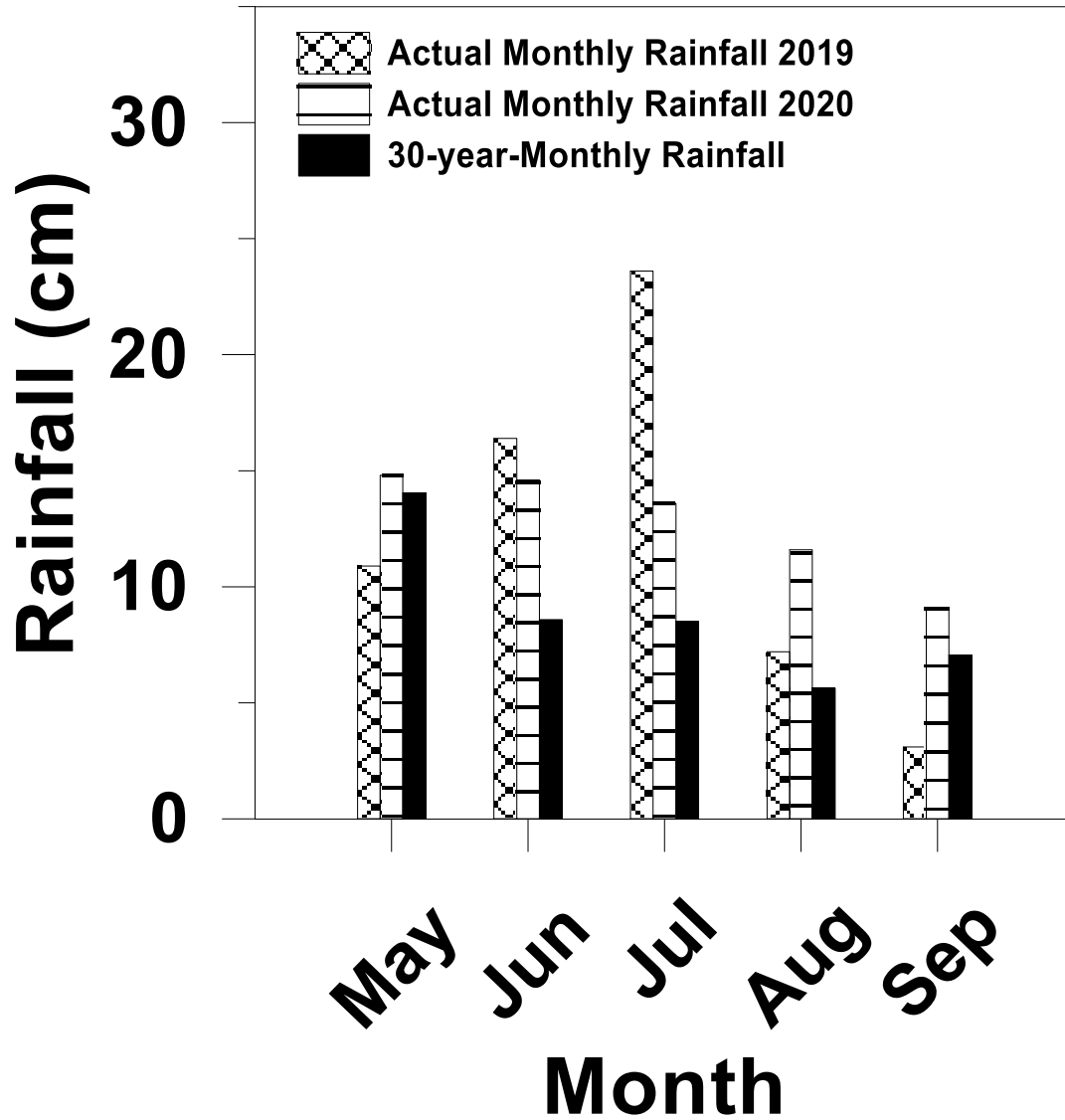


Fig. 2. Comparison among the 30-year (1981 to 2010) monthly rainfall and actual monthly rainfall during the five-month rice growing season in 2019 and 2020 at the Pine Tree Research Station near Colt, AR.

## **Chapter 4**

# **Evaluation of Wastewater-recovered Struvite as a Fertilizer-phosphorus Source in Corn and Soybean in Eastern Arkansas**

## Abstract

Recycling phosphorus (P) from wastewaters as the mineral struvite ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) has gained global attention because of potential P reductions discharged into receiving waters and as an alternative fertilizer-P source for crop production. The objective of this field study was to evaluate the effects of two struvite materials [electrochemically precipitated struvite (ECST) and chemically precipitated struvite (CPST)] relative to several other common fertilizer-P sources [triple superphosphate (TSP), monoammonium phosphate (MAP), diammonium phosphate (DAP), and rock phosphate (RP)] on corn (*Zea mays* L.) and soybean [*Glycine max* (L.) Merr.] response in two consecutive growing seasons in a P-deficient, silt-loam soil (Aquic Fraglossudalfs) in eastern Arkansas. Results showed that corn yield and soybean aboveground P uptake differed ( $P < 0.05$ ) among fertilizer-P sources. Averaged across years, corn yield was numerically largest from ECST ( $12.9 \text{ Mg ha}^{-1}$ ), which differed from all other treatments, and was numerically smallest from DAP ( $10.1 \text{ Mg ha}^{-1}$ ), which did not differ from CPST ( $10.3 \text{ Mg ha}^{-1}$ ), MAP ( $10.7 \text{ Mg ha}^{-1}$ ), RP ( $10.3 \text{ Mg ha}^{-1}$ ), and UC ( $10.3 \text{ Mg ha}^{-1}$ ). Corn yield from ECST was at least 1.2 times greater than that from CPST, TSP, MAP, DAP, RP, and UC. Averaged across years, soybean aboveground tissue P uptake was numerically largest from ECST ( $28.4 \text{ kg ha}^{-1}$ ), which did not differ from CPST ( $26.7 \text{ kg ha}^{-1}$ ) or TSP ( $25.9 \text{ kg ha}^{-1}$ ), and was numerically smallest from RP ( $21.4 \text{ kg ha}^{-1}$ ), which did not differ from the UC ( $22.0 \text{ kg ha}^{-1}$ ). Soybean aboveground tissue P uptake from ECST was at least 1.2 times greater than that from DAP or MAP and at least 1.3 times greater than that from RP or UC. Soybean aboveground tissue P uptake from CPST, which did not differ from DAP or TSP, was at least 1.1 times greater than that from MAP and at least 1.2 times greater than that from RP or UC. Results clearly showed that wastewater-recovered struvite materials have the potential to be a viable, alternative

fertilizer-P source for corn and soybean production in a P-deficient, silt-loam soil.



## Introduction

Phosphorus (P) is one of the three primary macronutrients that are essential for plant growth and development. Phosphorus is an integral component of a plant's energy supply in the form of adenosine triphosphate (ATP) and is critical for nucleic acid replication, as well as root and seed development (Shen et al., 2011). Although P is a relatively abundant element in nature, P is one of the most unavailable macronutrients required by plants due to generally low solubility and inaccessibility in soils (Le Corre, Valsami-Jones, Hobbs, & Parsons, 2009). Phosphorus can either form insoluble complexes with iron (Fe) and/or aluminum (Al) in acidic soil or calcium (Ca) in alkaline soil, be adsorbed to soil particles, or be bound to organic compounds, which must be enzymatically cleaved before plant uptake (Horst et al., 2001; Sims & Pierzynski, 2005; Khademi, Jones, Malakouti, & Asadi, 2010).

The global reserves of rock phosphate (RP), from which most fertilizer-P sources are created, are limited, non-renewable, and unevenly distributed, with the largest portion of mined RP located in Morocco (Stewart et al., 2005; Liu, Kumar, Kwag, & Ra, 2013). Additionally, existing world RP reserves will be halved by 2060, but the demand for fertilizer-P is projected to increase as the human population is expected to reach 9 billion by 2050 (Vance, Uhde-Stone, & Allan, 2003; Cordell, Drangert, & White, 2009). As the limited RP reserves are depleted, the cost of RP-derived, fertilizer-P sources is also expected to increase (Cordell & Neset, 2014).

A potential alternative to the fast-depleting RP reserves is the recovery of P as mineral struvite from wastewaters due to the relatively large P and nitrogen (N) concentrations in many wastewater effluent streams (Latifian, Liu, & Mattiasson, 2012; Uysal, & Kuru, 2015; Kataki, West, Clarke, & Baruah, 2016; Rahman et al., 2014; Ryu, & Lee, 2016; Li et al., 2019). Struvite, magnesium ammonium phosphate hexahydrate ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ), is a white crystalline

material that has been shown to be useful as fertilizer-P source (Johnston & Richards, 2003; Le Corre et al., 2009). On average, struvite contains 5.7% N, 12.6% P, and 9.9% Mg (Latifian et al., 2012; Nongqwenga, Muchaonyerwa, Hughes, Odindo, & Bame, 2017), but the final concentrations vary depending on the source material and recovery process used.

Struvite has been recovered from a variety of waste products through chemical, biological, and in recent years, electrochemical precipitation techniques (De-Bashan & Bashan 2004; Le Corre et al., 2009; Latifian et al., 2012; Manas, Sperandio, Decker, & Biscans, 2012; Huang et al., 2016; Kékedy-Nagy, Teymouri, Herring, & Greenlee, 2020). Unlike chemical precipitation, where external chemical additions are needed, electrochemical precipitation of struvite from wastewater is a newly adopted technique designed to also permit energy recovery and utilizes a sacrificial Mg-based anode that supplies Mg ions through corrosion in the presence of an electrical current (Kruk, Elektorowicz, & Oleszkiewicz, 2014; Kékedy-Nagy et al., 2019).

Struvite is considered a slow-release P source due to its reported lower solubility compared to common fertilizer-P sources used in crop production, such as triple superphosphate (TSP) and monoammonium phosphate (MAP; Chien, Prochnow, Tu, & Snyder, 2011; Prochnow, van Raij, & Kiehl, 2002), making struvite a potentially ideal, slow-release, fertilizer-P source for a variety of plants, including row crops in lowland, poorly drained soils (Anderson, Brye, Greenlee, Roberts, & Gbur, 2021a; Anderson et al., 2021c) and upland, well-drained soils (Thompson, 2013; Achat et al., 2014; Anderson et al., 2021b).

Corn (*Zea mays* L.) is a major crop grown throughout the world and is an increasingly important crop in the southern Mississippi River Basin region, which includes Arkansas (USDA-NASS 2020). In 2019, nearly 300,000 ha of corn were harvested in Arkansas (Kelly, & Capps, 2020; USDA-NASS 2020) and, in 2020, the total planted-corn area in Arkansas was 250,911 ha

(Kelly, & Capps, 2021; USDA-NASS, 2021). In 2020, corn ranked fifth among the top agricultural commodities in Arkansas and accounted for 5.9% of Arkansas' total farm receipts (USDA-ERS, 2021). Considering P's immobility in soil, plant response mechanisms to enhance P acquisition from soil/fertilizer include increasing root hair density and changing root architecture, formation of mycorrhizal associations, up-regulating P transport systems, and secretion of rhizosphere acidification (Gahoonia & Nielsen, 1992; Lambers, Shane, Cramer, Pearse, & Veneklaas, 2006). In Arkansas, recommended fertilizer-P rates range of 56 to 112 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup> for a corn yield goal of 14.1 Mg ha<sup>-1</sup> across soil textures (Espinoza and Ross, 2008), making corn a large-P-demanding crop for optimal production. Previous studies have reported that corn has a greater P efficiency expressed as root system size, greater P influx than wheat (*Triticum aestivum* L.; Gill et al., 2005). However, soybean (*Glycine max* L.) has been reported to have greater P uptake per unit of root mass than corn (Fernández, Belinque, Boem, & Rubio, 2009). The amount of P<sub>2</sub>O<sub>5</sub> required to produce 27.2 kg of soybean is 1.5 to 3.0 times greater than the amount needed to produce 25.4 kg of corn (Slaton, Roberts, & Ross, 2013).

Soybean is one of the most important economic grain crops for a large human population and is grown in many areas around the globe. In contrast to cereal grains, such as corn, wheat, and rice (*Oryza sativa* L.), soybean is rich in proteins and lipids. In 2019, soybean supplied close to 90% of the total national oilseed produced in the United States (US; USDA-ERS, 2021). In Arkansas, the harvested soybean area was 1.03 million ha in 2019 (USDA-NASS 2020; Ross, Elkins, & Norton, 2020) and 1.13 million ha in 2020 (Ross, Elkins, & Norton, 2021; USDA-NASS 2021). In 2020, soybean ranked second among the top agricultural commodities in Arkansas and accounted for 17.5% of Arkansas' total farm receipts (USDA-ERS, 2021). The demand for P during soybean vegetative growth is relatively low, but is greatest during pod and

seed development, where greater than 60% of P ends up in the pods and seeds (Usherwood, 1998). As a source of protein, soybean is nutrient intensive, with approximately 2.5 kg P ha<sup>-1</sup> removed from the soil for each 67 kg of soybean seed harvested per hectare (Slaton et al., 2013), thus periodic fertilizer-P additions are needed for soybean production to replenish soil P.

Several studies have examined crop response to struvite as the fertilizer-P source compared with other common fertilizer-P sources (i.e., TSP and MAP). Many studies have reported struvite to show comparable plant growth or P uptake to other commercially available, fertilizer-P sources (Li & Zhao, 2003; Kern et al., 2008; Massey, Davis, Ippolito, & Sheffield, 2009; Antonini, Arias, Eichert, & Clemens, 2012; Thompson, 2013). In contrast, other studies have reported a reduction in agronomic effectiveness with struvite fertilization compared to other commercially available, fertilizer-P sources (Ganrot, Dave, Nilsson, & Li, 2007; Ackerman, Zvomuya, Cicek, & Flaten, 2013; Talboys et al., 2015; Everaert, Da Silva, Degryse, McLaughlin, & Smolders, 2017).

Recently, in a 2-year field study, an electrochemically precipitated struvite (ECST) and a chemically precipitated struvite material (CPST; i.e., Crystal Green) were evaluated in flood-irrigated rice compared to TSP, diammonium phosphate (DAP), MAP, and RP in a P-deficient, silt-loam soil (Typic Glossaqualfs) in eastern Arkansas (Omidire et al., 2021). Additionally, corn and soybean response to struvite materials (ECST and CPST) were evaluated in a 79-day, greenhouse pot study in a silt-loam soil (Typic Fragiudults; Ylagan, Brye, & Greenlee 2020). In a series of plant-less soil incubation studies, struvite materials were also evaluated for their nutrient concentration and soil pH changes over time compared to DAP, MAP, and TSP in several different soils under flooded- (Anderson et al., 2021a,c) and moist-soil (Anderson et al., 2021b) conditions.

Few row-crop field trials have been conducted to evaluate the agronomic effectiveness of struvite materials (Gell, De Ruijter, Kuntke, De Graaff, & Smit, 2011; Thompson, 2013; Collins, Kimura, Frear, & Kruger, 2016; Hilt et al., 2016; Omidire et al., 2021). However, most studies have focused on struvite as a potential alternative fertilizer-P source in greenhouse pot studies (Ganrot et al., 2007; Kern et al., 2008; Massey et al., 2009; Cabeza, Steingrobe, Romer, & Claassen 2011; Uysal, Demir, Sayilgan, Eraslan, & Kucukyumuk, 2014; Talboys et al., 2015; Ylagan et al., 2020). Furthermore, no field studies have been conducted in the mid-southern United States to examine the agronomic effectiveness of wastewater-recovered struvite as a fertilizer-P source in any upland, row crop, particularly in corn or soybean production. Therefore, the objective of this field study was to evaluate the effects of two struvite materials (i.e., ECST and CPST) relative to several other common fertilizer-P sources (i.e., TSP, MAP, DAP, and RP) on corn and soybean response in two consecutive growing seasons in a P-deficient, silt-loam soil in eastern Arkansas.

For corn, it was hypothesized that both wastewater-recovered struvite sources (i.e., ECST and CPST) would have at least similar total aboveground dry matter (DM) and yield, total aboveground tissue P and N uptake, and belowground P and N concentrations, but greater total aboveground tissue Mg uptake and belowground tissue Mg concentrations than other common, commercially available fertilizer-P sources (i.e., TSP, MAP, DAP, and RP) due to struvite's lower water solubility and greater initial Mg concentrations. In addition, it was hypothesized that corn grown in year two would have greater total aboveground DM, yield, total aboveground tissue P, N, and Mg uptake, and belowground tissue P, N, and Mg concentrations compared to that in year one due to a carry-over effect from fertilization in year one. For soybean, it was hypothesized that both wastewater-recovered struvite sources (i.e., ECST and CPST) would also

have at least similar aboveground DM and yield, above- and belowground tissue and seed P and N concentrations, and aboveground and seed P and N uptake, but greater aboveground tissue and seed Mg concentration and uptake and belowground tissue Mg concentrations than other common, commercially available fertilizer-P sources (i.e., TSP, MAP, DAP, and RP). In addition, it was hypothesized that soybean grown in year two would also have greater aboveground DM, yield, above- and belowground tissue and seed P, N, and Mg concentrations, and aboveground tissue and seed P, N, and Mg uptake compared to that in year one.

## **Materials and Methods**

### **Site Description and Cropping History**

Research was conducted at the Lon Mann Cotton Branch Experiment Station (CBES) near Marianna, AR in 2019 and 2020 (34°44'01"N; 90°45'51"W). The study site was located in major land resource area (MLRA) 134, Southern Mississippi Valley Loess (Brye, Mersiovsky, Hernandez, & Ward, 2013). The soil throughout the 0.3-ha study area was mapped as a Calloway silt loam (fine-silty, mixed, active, thermic Aquic Fraglossudalfs; Soil Survey Staff, 2015). Calloway soils have a surface layer of dark brown to brown silt loam with a subsoil of light brownish-gray silt loam and are derived from loess parent materials (NRCS, 2020). The corn and soybean studies were established adjacent to one another (Figure 1). The top 10 cm of the soil profile is silt-loam textured and comprised of 7% sand, 80% silt, and 14% clay for the corn study area and 10% sand, 75% silt, and 15% clay for the soybean study area (Table 1).

The study area had been cultivated for the past 15 years with various crops including wheat, soybean, and corn, and occasionally left fallow for no more than one growing season at a time. No consistent crop rotation had been followed.

The climate in the region encompassing the study area is humid temperate, with the 30-year (i.e., 1981 to 2010) average annual air temperature and precipitation of 16.6°C and 128.4 cm, respectively (NOAA, 2020). The 30-year mean monthly minimum and maximum air temperatures in the area are 0.6°C in January and 32.3°C in July, respectively (NOAA, 2020).

### **Field Treatments and Experimental Layout**

The corn and soybean studies were conducted to evaluate the effects of fertilizer-P source [i.e., ECST, CPST, TSP, DAP, MAP, RP and an unamended control (UC)] on crop and soil response. Corn and soybean plots established in 2019 were the exact same plots used in 2020. Consequently, the corn and soybean studies consisted of two experimental factors, fertilizer-P source and year. Each year, there were four blocks containing one randomized replication of each of the seven fertilizer-P-source treatments.

### **Fertilizer-P Sources and Characterization**

The ECST material represents the result of relatively new technology used to recover nutrients from a waste stream. The ECST used in this field study was a synthetic-wastewater-derived struvite that was generated by researchers in the Department of Chemical Engineering at the University of Arkansas (Kékedy-Nagy et al., 2020). Two separate batches of ECST were produced for this study. The first batch was used in 2019 and the second batch was used in 2020. A stainless-steel (316SS) plate used as the cathode and a pure Mg or AZ31 Mg alloy anode were installed in a bench-top-scale, single-compartment reactor, where each ECST batch was produced (Kékedy-Nagy et al., 2020). The reactor contained 0.85 L of a 0.077 M solution of synthetic wastewater containing 7.53 g L<sup>-1</sup> of ammonium dihydrogen phosphate (NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>;

Kékedy-Nagy et al., 2020). The precipitate from the suspension and that which coated the electrodes was collected. Unlike ECST, the CPST material was derived from raw municipal wastewater, marketed and sold under the trade name Crystal Green by Ostara Nutrient Recovery Technologies, Inc. near Atlanta, GA. Though elemental compositions and morphology from X-ray diffraction indicated that the CPST and ECST materials are comparable (Kékedy-Nagy et al., 2020), the CPST derived from raw municipal wastewater had a more diverse composition compared to the ECST material, which was derived from a synthetic wastewater.

Chemical analyses were conducted on five replications of each batch of ECST and the CPST, MAP, DAP, TSP, and RP fertilizers. The particle sizes of the fertilizer-P sources varied from powder (i.e., RP) to crystals (ECST) to small pellets (i.e., CPST, MAP, DAP, and TSP), with an average diameter of 3.1 mm (Anderson, 2020). The ECST and pelletized fertilizers were mechanically crushed and chemically analyzed in powdered form to facilitate comparison among all fertilizer-P sources. Fertilizer pH was measured potentiometrically in a 1:2 (mass/volume) fertilizer-to-water-ratio paste (Sikora & Kissel, 2014) with the exception of the ECST material for which pH was not measured directly due to limited quantity that needed to be used for application in field studies. Total N concentration was determined by high-temperature combustion using a VarioMax CN analyzer (Elementar Americas, Inc., Mt. Laurel, NJ; Provin, 2014). A nitric acid digest was conducted (USEPA, 1996) and extracts were analyzed by inductively coupled, argon-plasma spectrometry (ICAPS) to determine total-recoverable Mg and P. The University of Arkansas, Division of Agriculture's Agricultural Diagnostic Lab in Fayetteville conducted all fertilizer analyses. Table 2 summarizes the measured chemical properties of the fertilizer-P materials.



## Plot Management

### Corn Study

The soil was conventionally tilled, consisting of three passes with a tandem disc to a 5- to 10-cm depth followed by three passes with a field cultivator to break up soil clods and soften the seed bed followed by creation of raised beds that were leveled at the top with a roller. In May 2019, 28 field plots, 6.1-m long by 3.1-m wide, were established for all fertilizer treatments except for ECST. The ECST plots were smaller sub-plots (1.5-m long by 1.5-m wide) of the larger plot area due to the limited quantity of ECST material available to apply to field plots. Beds were approximately 7.6-cm tall and 50-cm wide at the top after leveling and the spacing between beds was 90 cm. The fertilizer-P rate applied to each plot was based on the initial Mehlich-3 soil-test-P concentration in the top 10 cm measured in Fall 2018 (24.8 mg P kg<sup>-1</sup>), the recommended P-fertilization rate for corn (Espinoza & Ross, 2008), and the measured total-recoverable P concentrations of each fertilizer-P material (Table 2) such that an equivalent P-application rate of 29.4 kg P ha<sup>-1</sup> was used for all six fertilizer-P treatments.

Since each fertilizer-P material contained different N concentrations (Table 2), the quantity of N needed to be added to balance the N rate across all fertilizer-P treatments, including the unamended control, was determined based on the amount of N added in DAP, which had the largest N concentration (Table 2). Extra N was added in the form of uncoated urea (460 g N kg<sup>-1</sup>).

On 18 May, 2019, plots were drill-seeded with the Pioneer 1870YHR corn variety at a rate of 81500 seed ha<sup>-1</sup>, which resulted in each plot containing four corn rows. Dual II Magnum (1.4 L ha<sup>-1</sup>; Syngenta, Greensboro, NC; 2-chloro-N-(2-ethyl-6-methylphenyl)-N-[(2S)-1-methoxypropan-2-yl] acetamide) herbicide was tractor-sprayer applied once after corn planting

to control weeds. Two days after corn planting in 2019 (20 May), fertilizer-P materials and extra uncoated urea to balance the N were manually applied separately to each plot. The original, solid form of all fertilizers were surface-applied after planting because the beds, upon which corn and soybean were planted, had already been created, thus even light incorporation would have greatly disrupted the integrity of the beds. On 5 June, 2019, uncoated urea was manually broadcast-applied at 235.4 kg N ha<sup>-1</sup> for a corn yield goal of 13.8 Mg ha<sup>-1</sup> grown on a silt-loam soil (Espinosa & Ross, 2008). Acuron (7.0 L ha<sup>-1</sup>; Syngenta, Greensboro, NC; S-metolachlor), Atrazine (5.6 L ha<sup>-1</sup>; Syngenta, Greensboro, NC; 2-chloro-4-ethylamino-6-isopropylamino-s-triazine), and PermitPlus (52.5 mL ha<sup>-1</sup>; Gowan Company LLC, USA; Halosulfuron-methyl) were sprayed on 13 June, 2019. The corn crop was irrigated on 27 July, 13 August, and 21 August, 2019. Corn grain was harvested with a plot combine from a 1.7-m width by 4.6-m length of the two middle rows in each plot on 1 October, 2019.

On 6 April, 2020, Glyphosate (2.8 L ha<sup>-1</sup>; Bayer CropScience; 2-(phosphonomethylamino) acetate, propan-2-ylazanium) was sprayed once for weed control. In 2020, the exact same corn plots were used as were established in 2019. On 6 May, 2020, plots were drill-seeded with the Pioneer 1870YHR corn variety at a rate of 81500 seed ha<sup>-1</sup>. On 13 May, 2020, fertilizer-P materials were manually applied separately to each plot. Similar to 2019, N-(n-butyl) thiophosphoric triamide (NBPT)-coated urea was applied to balance the N across all fertilizer treatments, including the unamended control. All fertilizers were surface-applied in the original solid form. On 1 June, 2020, NBPT-coated urea was manually broadcast-applied at 235.4 kg N ha<sup>-1</sup> for a corn yield goal of 13.8 Mg ha<sup>-1</sup> grown on a silt-loam soil (Espinosa & Ross, 2008). On 3 June, 2020, Halex GT (5.6 L ha<sup>-1</sup>; Syngenta, Greensboro, NC; S-metolachlor) and Atrazine (2.8 L ha<sup>-1</sup>) were sprayed to control weeds. The corn crop was irrigated on 18 June, 1

July, 20 July, 23 July, 29 July, and 7 August, 2020. Corn grain was harvested with a plot combine from a 1.7-m width by 4.6-m length of the two middle rows in each plot on 21 August, 2020.

In both years, the mass of grain harvested per plot was recorded by a calibrated scale and the moisture content of the harvested grain mass was measured by a calibrated sensor on the combine. Based on the combine-measured grain moisture content, corn grain masses were adjusted to 155 g kg<sup>-1</sup> (15.5%) moisture content for yield reporting.

### Soybean Study

The soybean study was established adjacent to the corn study separated by four border rows of soybeans. Similar to the corn study, in May 2019, 28 field plots, 6.1-m long by 3.1-m wide for all fertilizer treatments except ECST, were established after conventional tillage previously described for the corn study. The ECST plots were smaller sub-plots (1.5-m long by 1.5-m wide) of the larger plot area due to the limited quantity of ECST material. The fertilizer-P rate applied per plot was based on the initial Mehlich-3 soil-test-P concentration in the top 10 cm measured in Fall 2018 (24.8 mg P kg<sup>-1</sup>), the recommended P-fertilization rate for soybean (Slaton et al., 2013), and the measured total-recoverable P concentration of each fertilizer-P material (Table 2) such that an equivalent P-application rate of 29.4 kg P ha<sup>-1</sup> was used for all six fertilizer-P treatments. Uncoated urea was applied to balance the N among the fertilizer-P treatments.

On 17 May, 2019, plots were drill-seeded with the Pioneer 46A70L SU26, a Liberty Link, maturity group 4.6, soybean variety, at a rate of 321230 seed ha<sup>-1</sup>, which resulted in each plot containing four soybean rows. Three days after soybean planting in 2019 (20 May),

fertilizer-P materials and extra uncoated urea to balance the N were manually applied separately to each plot. The original, solid form of all fertilizers were applied. On 13 July, 2019, the Liberty (2.8 L ha<sup>-1</sup>; Bayer CropScience, Research Triangle Park, NC; azanium, 2-amino-4-[hydroxy(methyl)phosphoryl] butanoate) herbicide was tractor-sprayer applied. On 19 July, 2019, Dual II Magnum (2.8 L ha<sup>-1</sup>) and Liberty (2.8 L ha<sup>-1</sup>) herbicides were sprayed once to control weeds, such as Palmer amaranth (*Amaranthus palmeri* S.) and perennial ryegrass (*Lolium perenne* L.). The soybean crop was irrigated on 27 July, 13 August, and 21 August, 2019. Intrepid Edge (0.35 L ha<sup>-1</sup>; Dow AgroSciences, Indianapolis, IN; N'-tert-butyl-N'-(3,5-dimethylbenzoyl)-3-methoxy-2-methylbenzohydrazide) and Acephate 97UP (1.12 kg ha<sup>-1</sup>; United Phosphorus Inc., King of Prussia, PA; O,S-dimethyl acetyl phosphoramidothioate<sub>2</sub>) were tractor-sprayer applied on 29 August, 2019 to control insects. Soybean grain was harvested with a plot combine from a 1.7-m width by 4.6-m length of the two middle rows in each plot on 23 October, 2019.

On 6 April, 2020, glyphosate (2.8 L ha<sup>-1</sup>) was sprayed for weed control. In 2020, the exact same soybean plots were used as were established in 2019. On 12 May, 2020, plots were drill-seeded with the USG 7469 GTL, a Liberty Link, maturity group 4.6, soybean variety at a rate of 321230 seed ha<sup>-1</sup>. On 13 May, 2020, fertilizer-P materials and extra NBPT-coated urea to balance the N were manually applied. All fertilizers were surface-applied in the original solid form. On 4 and 15 June, 2020, glyphosate (2.8 L ha<sup>-1</sup>) and Liberty (2.8 L ha<sup>-1</sup>) herbicides were tractor-sprayer applied to control weeds. Liberty (2.8 L ha<sup>-1</sup>) and Dual II Magnum (1.4 L ha<sup>-1</sup>) herbicides were sprayed for weed control on 18 July, 2020. The soybean crop was irrigated on 18 June, 1 July, 20 July, 23 July, 29 July, and 7 August, 2020. Soybean grain was harvested from the two middle rows of each plot with a plot combine on 1 October, 2020. The mass of soybean

grain harvested and the grain moisture content per plot were recorded on the combine. Based on the combine-measured grain moisture content, soybean grain masses were adjusted to 130 g kg<sup>-1</sup> (13%) moisture content for yield reporting each year.

### **Soil Sampling, Processing, and Analyses**

On 20 May, 2019, soil samples were collected from 10 random locations in the second and third rows from the top 10 cm on the top of the beds in each plot, combined, and mixed for one composite sample per block in the corn and soybean studies. Since the location for both studies did not change the following year after, on 13 May, 2020, soil samples were again collected from 10 random locations in the second and third rows from the top 10 cm on the top of the beds in each plot, combined, and mixed for one composite sample per plot in each study area. On 5 October, 2020, at the end of the corn and soybean growing seasons in the second consecutive year, soil samples were again collected from 10 random locations in the second and third rows from the top 10 cm on the top of the beds in each plot, combined, and mixed for one composite sample per plot in each study area.

All soil samples collected were oven-dried at 70°C for 48 h, mechanically crushed, and sieved through a 2-mm mesh screen. Particle-size analyses were conducted on the initial soil samples collected from the corn and soybean studies in 2019 using a modified 12-h hydrometer method (Gee & Or, 2002) to determine sand, silt, and clay fractions and confirm the soil textural class. Soil pH and electrical conductivity (EC) were measured potentiometrically in a 1:2 (mass/volume) soil-to-water-ratio paste (Sikora & Kissel, 2014). Soil organic matter (SOM) concentration was determined using weight-loss-ignition in a muffle furnace at 360°C for 2 h (Zhang & Wang, 2014). Total carbon (TC) and total nitrogen (TN) concentrations were

determined by high-temperature combustion using a VarioMax CN analyzer (Provin, 2014). The soil C:N ratio was calculated from the measured TC and TN concentrations. Soil did not effervesce upon treatment with dilute hydrochloric acid, thus all measured soil C was assumed to be organic C. A Mehlich-3 extraction (Zhang et al., 2014) was also conducted with a 1:10 (mass:volume) soil:extractant solution ratio to determine extractable nutrient (i.e., P, K, Ca, Mg, S, Na, Fe, Mn, Zn, and Cu) concentrations by ICAPS (Soltanpour, Johnson, Workman, Jones, & Miller, 1996). The University of Arkansas, Division of Agriculture's Agricultural Diagnostic Laboratory in Fayetteville conducted all soil analyses. The change in soil properties over time was determined by subtracting the single initial soil property value per study block from the respective soil property on a plot-by-plot basis after one complete year (i.e., May 2020 – May 2019) and then again after the second consecutive growing season (i.e., October 2020 – May 2019).

### **Plant Sampling, Processing, and Analyses**

On 2 September, 2019 and 20 August, 2020, when corn reached physiological maturity, and on 29 September, 2019 and 20 August, 2020, when soybeans reached reproductive stage 6.5, which is the growth stage when dry weight and nutrient accumulation is maximized in soybean (Popp, Purcell, & Salmerón, 2016), a 1-m row length of aboveground plants was cut at the soil surface and collected from the second corn and soybean row in each plot. Two corn root masses of the cut aboveground plants in each plot were carefully dug out with a shovel to a depth of approximately 20 cm. Five soybean root masses of the cut aboveground plants in each plot were carefully dug out with a shovel to a depth of approximately 15 cm. Plant samples were collected

from within the smaller sub-plots for only the ECST treatment in both corn and soybean studies. All sampled plant roots were vigorously shaken and rinsed using tap water to remove attached soil particles.

In the corn study in 2019 and 2020, the total aboveground DM was separated into three parts: stalk + leaves, cob + husk, and kernels. Kernels were removed from cobs using a hand-operated corn sheller. For soybean, the aboveground plant samples were not separated into any individual plant parts. The above- and belowground corn and soybean tissue samples were oven-dried at approximately 55°C for 7 days and weighed. Dry matter per unit area was calculated for all aboveground plant tissue samples. Sub-samples of corn stalk + leaves, cob + husk, kernels, and belowground DM and soybean above- and belowground DM samples were mechanically ground and passed through a 2-mm mesh screen for chemical analyses.

Above- and belowground soybean DM and the various parts of corn aboveground and belowground DM were analyzed separately for total N concentration by high-temperature combustion using a VarioMax CN analyzer (Provin, 2014). After digestion in concentrated nitric acid with heating, extracts were analyzed by ICAPS (Soltanpour et al., 1996) to determine above- and belowground tissue P and Mg concentrations. Corn stalk + leaves, cob + husk, and kernel and soybean aboveground P, N, and Mg concentrations and their respective DMs were used to calculate corn stalk + leaves, cob + husk, and kernel and soybean aboveground P, N, and Mg uptake ( $\text{kg ha}^{-1}$ ). Corn stalk + leaves, cob + husk, and kernel DM and nutrient uptake were summed to evaluate total aboveground DM and nutrient uptake.

All corn kernels and soybean seeds harvested with the combine were collected. Since plant samples were collected from one of the two middle rows that were combine-harvested in the corn study, the combine grain mass at 15.5% moisture was added to the grain mass obtained

during corn sampling at 15.5% moisture, resulting in the total corn grain yield reported each year.

Soybean seed samples from the combine harvest were air-dried for 14 days at approximately 25°C. A sub-sample of air-dried soybean seeds from each plot was oven-dried for 48 h at 70°C and was mechanically pulverized for P, N, and Mg concentration measurement as previously described. Phosphorus, N, and Mg concentrations and oven-dried yield were used to calculate P, N, and Mg accumulation ( $\text{kg ha}^{-1}$ ) in the soybean seeds. The University of Arkansas, Division of Agriculture's Agricultural Diagnostic Laboratory in Fayetteville conducted all plant analyses.

In addition to assessing annual DM and nutrient uptake data, two-year cumulative DM and nutrient uptake were calculated for both corn and soybean. Corn stalk + leaves, cob + husk, kernel, and total aboveground DM and their respective P, N and Mg uptakes and yield and soybean total aboveground DM, total aboveground and seed P, N and Mg uptake, and yield were summed for the two individual years to represent the two-year cumulative data.

### **Statistical Analyses**

Based on a randomized complete block design, the effect of fertilizer-P treatment (ECST, CPST, TSP, MAP, DAP, RP, and UC) on the change in soil properties (i.e., soil pH and EC, extractable soil P, K, Ca, Mg, S, Na, Fe, Mn, Zn, and Cu, and SOM, TC, and TN concentrations, and C:N ratio) from their initial magnitudes prior to any fertilizer-P addition to the beginning of the second year before fertilizer-P addition and from their initial magnitudes to the end of the second growing season after harvest were evaluated by a one-factor analysis of variance (ANOVA) in SAS (SAS, 2013) using the PROC GLIMMIX procedure. A normal distribution was used for all soil properties.



For corn, based on a split-plot experimental design, a two-factor ANOVA was conducted using the PROC GLIMMIX procedure in SAS (SAS, 2013) to evaluate the effects of fertilizer-P treatment, year (2019 and 2020), and their interaction on stalk + leaves, cob + husk, kernel, and total aboveground DM; stalk + leaves, cob + husk, and kernel P, N, and Mg tissue concentrations; stalk + leaves, cob + husk, kernel, and total aboveground P, N, and Mg uptake; belowground P, N, and Mg tissue concentrations; and yield. Since the actual location of the plots did not change each year, the whole-plot factor was year and the split-plot factor was fertilizer-P treatment. In addition, a one-factor ANOVA was conducted using the PROC GLIMMIX procedure in SAS (SAS, 2013) to evaluate the effects of fertilizer-P treatment on the two-year cumulative stalk + leaves, cob + husk, kernel, and total aboveground DM; stalk + leaves, cob + husk, kernel, and total aboveground P, N, and Mg uptake; and yield. A gamma distribution was used for the all DM, nutrient uptake, and yield data, while a beta distribution was used for all nutrient concentration data.

For soybean, similar to corn, based on a split-plot experimental design, a two-factor ANOVA was conducted using the PROC GLIMMIX procedure in SAS (SAS, 2013) to evaluate the effects of fertilizer-P treatment (ECST, CPST, TSP, MAP, DAP, RP, and UC), year (2019 and 2020), and their interaction on soybean total aboveground DM; above- and belowground and seed P, N, and Mg tissue concentrations; aboveground and seed P, N, and Mg tissue uptake; and yield. The whole-plot factor was year and the split-plot factor was fertilizer-P treatment. In addition, a one-factor ANOVA was conducted using the PROC GLIMMIX procedure in SAS (SAS, 2013) to evaluate the effects of fertilizer-P treatment on the two-year cumulative total aboveground DM, total aboveground and seed P, N, and Mg uptake, and yield. A gamma distribution was used for all DM, nutrient uptake, and yield data, while a beta distribution was

used for all nutrient concentration data. For all analyses, significance was judged at  $P < 0.05$  and least square means were reported. When appropriate, means from were separated by least significant difference at the 0.05 level.

## **Results and Discussion**

### **Corn Study**

#### *Initial Soil Properties*

Since field studies were conducted in the same area within the same field each year, it was important to assess the initial soil properties, which was a reference, for further determination of any change in soil properties after one year and two growing seasons. Initial soil pH was slightly alkaline (pH = 7.16) and initial extractable soil Ca and Mg levels were above the optimum category ( $> 400 \text{ mg kg}^{-1}$  for Ca and  $> 30 \text{ mg kg}^{-1}$  for Mg) for most row crops in Arkansas (Espinosa, Slaton, & Mozaffari, 2021). Since 5.8 to 7.0 is the desirable soil pH range for corn (Espinosa & Ross, 2008), the slightly alkaline soil pH, coupled with the large extractable soil Ca concentration, could render some fraction of fertilizer-applied P unavailable to plant roots due to binding with soil Ca to form insoluble complexes (Espinosa & Ross, 2008). Based on soil sampling in May 2019, after plot establishment, but before fertilizer application in the corn study, Mehlich-3-extractable soil P averaged  $33.9 \text{ mg kg}^{-1}$  [standard error (SE) = 4.7] in the top 10 cm, which showed that the study area was below optimum for corn production in a silt-loam soil (Espinosa & Ross, 2008), thus a plant response from the added fertilizer-P in both years was expected. Table 1 summarizes means for all initial soil properties in the corn-study site.

### *Corn Response*

Corn response to the various fertilizer-P sources evaluated in a given year was variable. Seven of the 29 corn properties evaluated were affected ( $P < 0.05$ ) by fertilizer-P source, either as a main effect or interaction, while 10 corn properties were completely unaffected ( $P > 0.05$ ) by fertilizer-P source or year (Table 3).

Corn stalk + leaves DM was unaffected ( $P > 0.05$ ) by fertilizer-P source or year (Table 3). Stalk + leaves DM ranged from 7.0 Mg ha<sup>-1</sup> from DAP in 2020 to 8.7 Mg ha<sup>-1</sup> from the UC in 2019 and averaged 7.6 Mg ha<sup>-1</sup> across all fertilizer-P sources and both years (Table 4). In contrast to the current results, Ylagan et al. (2020) reported differences in corn stalk + leaves DM among similar fertilizer-P sources, where stalk + leaves DM was numerically largest from TSP, which did not differ from DAP, MAP, ECST, RP, or the no P/+N control and was numerically smallest from the no P/-N control. Stalk + leaves DM from the two struvite materials, CPST and ECST, did not differ and both were similar to that from MAP, RP, and no P/+N control (Ylagan et al., 2020).

Similar to stalk + leaves DM, stalk + leaves P concentration was unaffected ( $P > 0.05$ ) by fertilizer-P source or year (Table 3). Stalk + leaves P concentration ranged from 0.07 % from RP in 2019 to 0.18 % from CPST in 2020 and averaged 0.11 % across all fertilizer-P sources and both years (Table 4). In contrast to the current results, Ylagan et al. (2020) reported corn stalk + leaves P concentration to be numerically greatest from ECST, which differed from all other treatments, and was numerically smallest from the no P/+N control. Stalk + leaves P concentration from CPST, which did not differ from TSP, MAP, DAP, or the no P/-N control, was 1.6 times greater than that from the no P/+N control (Ylagan et al., 2020).

Similar to stalk + leaves DM and P concentration, stalk + leaves N and Mg

concentrations were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 3). However, averaged across fertilizer-P sources, stalk + leaves N and Mg concentrations were 3.3 and 1.7 times, respectively, greater ( $P < 0.05$ ) in 2020 than in 2019 (Table 5). In contrast to the current results, Ylagan et al. (2020) reported differences in corn stalk + leaves N and Mg concentrations among fertilizer-P sources. Stalk + leaves N concentration was numerically largest from CPST, which did not differ from ECST, DAP, or RP, and was numerically smallest from the no P/-N control, which differed from all other treatments (Ylagan et al., 2020). Stalk + leaves N concentration from ECST, which did not differ from MAP, DAP, or RP, was 1.2, 1.1, and 3.1 times greater than that from TSP, the no P/+N, and the no P/-N controls, respectively (Ylagan et al., 2020). Stalk + leaves Mg concentration was numerically largest from ECST and CPST, which did not differ from RP, and numerically smallest from the no P/-N control, which differed from all other treatments (Ylagan et al., 2020).

Similar to stalk + leaves DM, P, N, and Mg concentrations, stalk + leaves P, N, and Mg uptake were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 3). However, averaged across fertilizer-P sources, stalk + leaves P, N, and Mg uptake were 1.8, 2.8, and 1.6 times, respectively, greater ( $P < 0.05$ ) in 2020 than in 2019 (Table 5). In contrast to the current results, Cabeza et al. (2011) in a 2-year pot experiment reported differences in corn shoot P uptake among fertilizer-P sources. Cabeza et al. (2011) reported that shoot P uptake from struvite derived from sewage treatment plant and TSP, which did not differ, was 2.0 times greater than that from the UC. Furthermore, in two soils in the first year, shoot P uptake from RP did not differ from that from the UC, but in the second year, shoot P uptake from RP was greater than that from the control (Cabeza et al., 2011).

Similar to stalk + leaves DM, P, N, and Mg concentrations and uptake, cob + husk DM

was unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 3). However, averaged across fertilizer-P sources, cob + husk DM was at least 1.1 times, greater ( $P = 0.02$ ) in 2019 than in 2020 (Table 5). In contrast to the current results, Ylagan et al. (2020) reported differences in corn cob + husk DM among fertilizer-P sources. Cob + husk DM was numerically largest from CPST, which did not differ from ECST, TSP, MAP, DAP, RP, or the no P/+N control, and was numerically smallest from the no P/-N control, which differed from all other treatments (Ylagan et al., 2020).

Similar to stalk + leaves DM and P concentration, cob + husk P concentration was unaffected ( $P > 0.05$ ) by fertilizer-P source or year (Table 3). Cob + husk P concentration ranged from 0.05 % from MAP in 2019 to 0.09 % from DAP in 2020 and averaged 0.07 % across all fertilizer-P sources and both years (Table 4). In contrast to the current results, Ylagan et al. (2020) reported differences in corn cob + husk P concentration among fertilizer-P sources. Cob + husk P concentration was numerically largest from ECST, which did not differ from MAP, DAP, or the no P/-N control, while cob + husk P concentration was numerically smallest from the no P/+N control, which did not differ from CPST, TSP, or RP (Ylagan et al., 2020). Cob + husk P concentration from ECST was 1.2 times greater than that from CPST and TSP, and at least 1.3 times greater than that from RP and the no P/+N control (Ylagan et al., 2020). It is important to point out that the Ylagan et al. (2020) study was conducted in space-limited pots in the greenhouse, where roots had no access to deeper soil nutrients and had to completely rely on applied nutrients. However, in the field, such as in the current study, roots had ample access to deeper soil and more nutrients than those that were applied to potentially mask the effects of the various fertilizer-P sources evaluated.

Similar to stalk + leaves DM, P, N, and Mg concentrations and uptake and cob + husk DM and P concentration, cob + husk N and Mg concentrations were unaffected ( $P > 0.05$ ) by

fertilizer-P source (Table 3). However, averaged across fertilizer-P sources, cob + husk N and Mg concentrations were 2.0 and 1.7 times, respectively, greater ( $P < 0.05$ ) in 2020 than in 2019 (Table 5). In contrast to the current results, Ylagan et al. (2020) reported corn cob + husk N concentration was numerically largest from MAP, which did not differ from ECST, CPST, TSP, RP, DAP, or the no P/+N control, and was numerically smallest from the no P/-N control, which differed from all other treatments. However, similar results to the current study were reported by Ylagan et al. (2020) where corn cob + husk Mg concentration did not differ among fertilizer-P sources.

Similar to stalk + leaves DM, nutrient concentrations and uptake, cob + husk DM and nutrient concentrations, cob + husk P, N, and Mg uptake were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 3). However, averaged across fertilizer-P sources, cob + husk P, N, and Mg uptake were 1.5, 1.8, and 1.4 times, respectively, greater ( $P < 0.01$ ) in 2020 than in 2019 (Table 5).

In contrast to stalk + leaves DM, nutrient concentrations and uptake and cob + husk DM, nutrient concentrations, and uptake, kernel DM (i.e., from 1-m row samples) differed ( $P < 0.05$ ; Table 3) among fertilizer-P sources. Averaged across year, kernel DM was numerically largest from ECST (11.5 Mg ha<sup>-1</sup>), which did not differ from that from MAP or UC, and was numerically smallest from DAP (9.2 Mg ha<sup>-1</sup>), which did not differ from CPST, RP, TSP, or UC (Table 6). Kernel DM from ECST was at least 1.1 times greater than that from TSP, RP, CPST, or DAP. Kernel DM from MAP was at least 1.1 times greater than that from DAP (Table 6).

Similar to stalk + leaves DM and P concentration and cob + husk P concentration, kernel P, N, and Mg concentrations were unaffected ( $P > 0.05$ ) by fertilizer-P source or year (Table 3). Kernel P concentration ranged from 0.27 % from RP in 2020 to 0.33 % from ECST in 2020 and

averaged 0.29 % across all fertilizer-P sources and both years (Table 4). Kernel N concentration ranged from 1.1 % from TSP in 2019 to 1.4 % from ECST in 2020 and averaged 1.2 % across all fertilizer-P sources and both years (Table 4). Kernel Mg concentration ranged from 0.09 % from MAP in 2019 to 0.12 % from ECST in 2020 and averaged 0.10 % across all fertilizer-P sources and both years (Table 4).

Similar to kernel DM, kernel P and Mg uptake differed ( $P < 0.05$ ; Table 3) among fertilizer-P sources. Averaged across year, kernel P uptake was numerically largest from ECST (35 kg ha<sup>-1</sup>), which did not differ from MAP, and was numerically smallest from DAP (26 kg ha<sup>-1</sup>), which did not differ from CPST, MAP, RP, TSP, or UC (Table 6). Kernel P uptake from ECST was at least 1.2 times greater than that from CPST, TSP, RP, DAP, or UC (Table 6). Averaged across year, kernel Mg uptake was numerically largest from ECST (12.8 kg ha<sup>-1</sup>), which did not differ from MAP or UC, and was numerically smallest from DAP (9.5 kg ha<sup>-1</sup>), which did not differ from CPST, RP, MAP, TSP, or UC (Table 6). Kernel Mg uptake from ECST was at least 1.2 times greater than that from CPST, DAP, RP, or TSP (Table 6).

Greater kernel Mg uptake from ECST was expected due to the composition and dissolution of the struvite, with an initial Mg concentration greater than that in other fertilizer-P sources (Table 2). The slower dissolution of ECST likely kept the Mg closer to the actively growing corn root zone longer for greater plant availability. However, kernel Mg uptake from CPST was expected to be greater than that from TSP, DAP, MAP, RP, and the UC because of greater initial Mg concentration in the CPST material (Table 2).

In contrast to stalk + leaves DM, nutrient concentrations and uptake, cob + husk DM, nutrient concentrations and uptake, kernel DM, nutrient concentrations and P and Mg uptake, only kernel N uptake differed among fertilizer-P sources between years ( $P = 0.04$ ; Table 3). In

2019, kernel N uptake was numerically largest from ECST (131 kg ha<sup>-1</sup>), which did not differ from DAP, MAP, TSP, or the UC, and was numerically smallest from RP (97 kg ha<sup>-1</sup>), which did not differ from CPST, DAP, MAP, or TSP (Table 7). In 2019, kernel N uptake from ECST was 1.3 and 1.4 times greater than that from CPST and RP, respectively (Table 7). In 2020, kernel N uptake was numerically greatest from ECST (170 kg ha<sup>-1</sup>), which did not differ from MAP or RP, and numerically smallest from DAP (122 kg ha<sup>-1</sup>), which did not differ from CPST, TSP, or UC (Table 7). In 2020, kernel N uptake from ECST was 1.2, 1.4, 1.2, and 1.3 times greater than that from CPST, DAP, TSP, and the UC, respectively (Table 7). Kernel N uptake from ECST, CPST, MAP, RP, and TSP in 2020 were greater than those in 2019 (Table 7). However, kernel N uptake from DAP and the UC in 2020 did not differ from those in 2019 (Table 7).

Similar to stalk + leaves DM, but contrary to expectations and in contrast to kernel DM, total aboveground corn DM was unaffected ( $P > 0.05$ ) by fertilizer-P source or year (Table 3), despite the initial soil-test-P levels being below optimum, such that a plant response was expected, at least from the P-fertilized treatments relative to the unamended control. Total aboveground corn DM ranged from 18.1 Mg ha<sup>-1</sup> from DAP in 2020 to 21.5 Mg ha<sup>-1</sup> from UC in 2019 and averaged 19.9 Mg ha<sup>-1</sup> across all fertilizer-P sources and both years (Table 4).

The lack of difference in stalk + leaves DM, P, N, and Mg concentrations and uptake, cob + husk DM, P, N, and Mg concentrations and uptake, kernel tissue P, N, and Mg concentrations, and total aboveground DM among fertilizer-P sources could have been due to the alkaline initial soil in 2019 and large concentration of initial extractable soil Ca (Table 1) that facilitated P fixation as Ca-phosphate (Ca-P) minerals as the fertilizer materials dissolved. Although soil pH after two growing seasons had generally decreased from the initial pH level across all fertilizer-P treatments (Figure 2; Table 8), soil pH in most treatments were still alkaline (Figure 2). After



fixation of P released from the added fertilizers, rendering at least some of the fertilizer-P unavailable for plant uptake, corn roots may have also explored soil below the top 10 cm, where the soil pH was likely lower than at the surface. Consequently, native soil P likely became the more reliable P source for root uptake resulting in no difference in aforementioned corn properties among fertilizer-P treatments. Furthermore, the fertilizer-P materials were surface-applied without incorporation, which likely rendered much of the fertilizer-P applied vertically stratified near the soil surface.

Similar results to the current study have been reported in a 135-day corn field experiment by Gell et al. (2011) who evaluated the effectiveness of two struvite materials recovered from human urine and black water as fertilizer-P sources compared with TSP and UC in a sandy loam Andisol with a low soil-test P and pH of 4.5. Gell et al. (2011) reported no difference between corn DM across all treatments (i.e., struvite, TSP, and UC).

In contrast, other studies have reported differences in aboveground DM among fertilizer-P sources (Rech et al., 2019; Thompson, 2013; Ylagan et al., 2020). Rech et al. (2019) reported wheat shoot DM from TSP to be 1.3 and 1.7 times greater than that from each of three struvite sources and the UC, respectively, while soybean shoot DM from TSP was similar to that from three struvite sources and was 1.6 times greater than that from the UC. Additionally, Ylagan et al. (2020) reported that corn aboveground DM was numerically largest from TSP, which did not differ from CPST, MAP, DAP, and RP, and was 1.1, 1.1, and 1.7 times greater than that from ECST and no P/+N and no P/-N controls, respectively, in a greenhouse study. Corn aboveground DM from the no P/-N control was numerically smallest among all treatments. Aboveground DM from the two struvite materials, ECST and CPST, did not differ from one other, and aboveground DM from ECST also did not differ from that from RP, DAP, MAP, and the no P/+N control.

Similar to stalk + leaves, cob + husk, and kernel tissue P concentrations and total aboveground DM, total aboveground tissue P uptake was unaffected ( $P > 0.05$ ) by fertilizer-P source or year (Table 3). Total aboveground tissue P uptake ranged from 30.5 kg ha<sup>-1</sup> from RP in 2019 to 50.9 kg ha<sup>-1</sup> from ECST in 2020 and averaged 39.2 Mg ha<sup>-1</sup> across all fertilizer-P sources and both years (Table 4). The lack of total aboveground tissue P uptake difference among fertilizer-P treatments was likely due to the soil's large P-adsorption capacity such that at least some of the P released from the fertilizer-P materials was unavailable for plant uptake as a result of P adsorption to clays and/or secondary mineral formation by precipitation with Ca, which was also present in large concentrations in the top 10 cm (Table 1), in the alkaline soil throughout the study area (Khiari & Parent, 2005; Sims & Pierzynski, 2005; Espinosa & Ross, 2008; Khademi et al., 2010; Roberts et al., 2016; Table 1). The desirable soil pH for corn ranges from 5.8 to 7.0 (Espinosa & Ross, 2008). When the soil pH is 7.0 or greater, P binds with Ca forming insoluble Ca-P that are largely unavailable to plant roots (Espinosa & Ross, 2008). Corn roots may have also extracted sufficient soil P from below the top 10 cm to mask potential differences among fertilizer-P sources.

Similar to results of the current study, Gell et al. (2011) reported no difference between corn P uptake across all treatments. However, other studies have reported a difference in aboveground tissue P uptake among fertilizer-P treatments in other crops (Rech et al., 2019). Rech et al. (2019) reported that wheat total P uptake from TSP was similar to that from two struvite materials and was 1.3 and 2.1 times greater than that from CPST and an unamended control, respectively.

In contrast to P uptake, total aboveground tissue N uptake was affected ( $P < 0.05$ ) by fertilizer-P source or year (Table 3). Averaged across years, total aboveground tissue N uptake

was numerically largest from ECST (228 kg ha<sup>-1</sup>), which did not differ from MAP, RP, or the UC, and was numerically smallest from DAP (179 kg ha<sup>-1</sup>), which did not differ from CPST, TSP or the UC (Table 6). Total aboveground tissue N uptake from ECST was at least 1.2 times greater than that from CPST, DAP, and TSP, which did not differ (Table 6). Total aboveground tissue N uptake from MAP and RP, which averaged 205 kg ha<sup>-1</sup>, was 1.1 times greater than that from DAP (Table 6). Similar to stalk + leaves and cob + husk N and Mg concentrations and uptake, averaged across fertilizer-P sources, total aboveground tissue N uptake was 1.7 times greater ( $P < 0.05$ ) in 2020 than in 2019 (Table 5).

Similar to stalk + leaves, cob + husk, and kernel tissue P concentrations, total aboveground DM, and P uptake, total aboveground tissue Mg uptake was unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 3), despite greater initial Mg concentration in the struvite materials, ECST and CPST, such that a plant response was expected, at least from the two struvite treatments relative to that from other fertilizer-P sources. Similar to total aboveground tissue N uptake, averaged across fertilizer-P sources, total aboveground tissue Mg uptake was 1.5 times greater ( $P < 0.05$ ) in 2020 than in 2019 (Table 5).

The differences in total aboveground tissue N uptake between the two struvite materials, ECST and CPST, was at least partially due to the N in the ECST material being derived from a synthetic rather than an actual wastewater like the N in the CPST material were. It is likely that the presence of additional compounds in the CPST material that made the N somewhat less mobile once in the plant than the relatively chemically cleaner N in the ECST (Ylagan et al., 2020).

Greater stalk + leaves and cob + husk tissue P uptake in 2020 than in 2019 was likely due to reduction in initial soil pH in the second growing season (Table 8, Figure 2), thus releasing

some P from precipitated/fixed Ca-P compounds. Greater total aboveground tissue N uptake in 2020 than in 2019 could in part be due to a reduction in ammonia volatilization from the use of NBPT-coated urea to balance the N rate across all fertilizer-P treatments and as mid-season N fertilizer in 2020, whereas uncoated urea was used in 2019. Additionally, it is plausible that total aboveground tissue N uptake in 2019 was lower than that in 2020 due to runoff of the surface-applied uncoated urea from the top of the beds into the furrows and/or leaching of N after 0.1 (later in the same day, but after mid-season N application), 1.0 (the following day after mid-season N application), and 9.2 cm (two days after mid-season N application) of rainfall fell on the study field in 2019. Greater total aboveground tissue Mg uptake in 2020 than in 2019 was likely due to a carry-over effect from fertilization in 2019 since the same plots were used in both years.

Similar to stalk + leaves DM, P concentration, cob + husk P concentration, and kernel P, N, and Mg concentrations, belowground P, N, and Mg concentrations were unaffected ( $P > 0.05$ ) by fertilizer-P source or year (Table 3). Belowground P concentration ranged from 0.05 % from the UC in 2020 to 0.12 % from TSP in 2019 and averaged 0.08 % across all fertilizer-P sources and both years (Table 4). Belowground N concentration ranged from 0.43 % from TSP in 2019 to 0.77 % from ECST in 2020 and averaged 0.57 % across all fertilizer-P sources and both years (Table 4). Belowground Mg concentration ranged from 0.12 % from MAP in 2019 to 0.21 % from ECST in 2020 and averaged 0.16 % across all fertilizer-P sources and both years (Table 4).

Application of a uniform total N rate to all treatments, which was expected to be uniformly available for plant uptake, was likely why stalk + leaves, cob + husk, kernel, and belowground tissue N concentrations were unaffected by fertilizer-P treatments. Despite no effect of fertilizer-P source on stalk + leaves, cob + husk, kernel, and belowground tissue Mg

concentration in the current study, and even though the Mg concentration of fertilizer-P materials varied, Gell et al. (2011) reported that blackwater-derived struvite increased Mg concentrations by 28% in corn aboveground tissue compared to TSP and the UC.

Similar to total aboveground tissue N and Mg uptake, combine yield differed ( $P < 0.01$ ) among fertilizer-P sources (Table 3). Averaged across years, yield was numerically largest from ECST (12.9 Mg ha<sup>-1</sup>), which differed from all other treatments, and was numerically smallest from DAP (10.1 Mg ha<sup>-1</sup>), which did not differ from CPST, MAP, RP, and the UC (Table 6). Yield from ECST was at least 1.2 times greater than that from CPST, DAP, MAP, RP, and the UC, which did not differ (Table 6). Yield from ECST was 1.2 times greater than that from TSP, which did not differ from CPST, MAP, RP, and the UC (Table 6). Yield from TSP was 1.1 times greater than that from DAP (Table 6). Similar to total aboveground tissue N and Mg uptake, but contrary to expectations and in contrast to total aboveground tissue P uptake, averaged across fertilizer-P sources, yield was at least 1.1 times greater ( $P = 0.02$ ) in 2019 than in 2020 (Table 5). Since the actual monthly air temperature during the two corn growing seasons were similar, greater corn yields in 2019 may be attributed to greater rainfall in 2019 than in 2020 (Figure 3). Rainfall was 1.3, 1.4, and 2.5 times greater in May, June, and July 2019, respectfully, than similar months in 2020 (Figure 3). Despite being furrow-irrigated, corn growth and productivity would have benefitted from more uniform, well-watered soil moisture conditions in 2019 with lower magnitudes of soil moisture fluctuations than in 2020 with less timely rainfall events.

Corn yield response was expected from added fertilizer-P because the initial soil-test P concentration was below optimum for a corn yield goal of 13.8 Mg ha<sup>-1</sup> grown on a silt-loam soil (Espinosa & Ross, 2008), indicating corn growth and productivity would likely be limited by P. Similar to kernel DM and P uptake, corn yield from ECST was numerically largest and differed

from all other treatments likely because the reduction in soil pH from the initial soil pH by 0.35 units was numerically largest in the ECST (Figure 2), making the soil pH fall within the desirable soil pH range for corn (5.8 to 7.0; Espinosa & Ross, 2008), thus greater dissolution and availability of P from precipitated/fixed Ca-P. The acidification effect of the ECST material in the soil may have also changed the available P species and ratio between  $\text{HPO}_4^{2-}$  and  $\text{H}_2\text{PO}_4^-$  towards comparatively more  $\text{H}_2\text{PO}_4^-$  in the soil solution, thus increasing dissolution and mobility of P from the ECST material and Ca-P (Gahoonia, Claassen, & Jungk, 1992). The difference in kernel P uptake and combine yield from both struvite sources (ECST and CPST) used in the current study was at least partially due to the presence of additional associated complexes in the actual wastewater-recovered CPST material, which likely made P less mobile in plant than the P taken up from the synthetic-wastewater-recovered ECST material that was purer in composition than CPST (Ylagan et al., 2020).

Averaged across all fertilizer-P sources, corn yields from the current study were lower in 2019 than those from the Arkansas Corn Performance Trial yield results in 2019 ( $13.5 \text{ Mg ha}^{-1}$ ) for the same Pioneer 1870YHR corn variety grown at CBES (Carlin, Bond, & Still, 2020a). In 2019, yield from the current study ranged from  $10.4 \text{ Mg ha}^{-1}$  from RP to  $13.2 \text{ Mg ha}^{-1}$  from ECST and averaged  $11.4 \text{ Mg ha}^{-1}$  across all fertilizer-P sources. There was no report for the Pioneer 1870YHR from the Arkansas Corn Performance Trial yield results in 2020 at CBES, however the closest corn variety to that of the current study was Pioneer 1847VYHR ( $15.1 \text{ Mg ha}^{-1}$ ; Carlin, Bond, & Morgan, 2021a). Similar to the current study, irrigated corn in the Arkansas Corn Performance Trial was grown on a Calloway silt loam in both years. Averaged across all fertilizer-P sources, corn yields from the current study were also lower in 2020 than those from the Arkansas Corn Performance Trial yield results in 2020 at CBES. In 2020, yield from the

current study ranged from 9.3 Mg ha<sup>-1</sup> from CPST to 12.7 Mg ha<sup>-1</sup> from ECST and averaged 10.3 Mg ha<sup>-1</sup> across all fertilizer-P sources. These results differ somewhat from the corn grown in 2019 and 2020 compared to that from the irrigated corn grown in the Arkansas Corn Performance Trial at CBES, which had earlier planting dates and 1.14 times more applied-N than what was used in the current study. A different corn variety used in the 2020 Arkansas Corn Performance Trial could also have been a contributing factor to greater yields than that from the current study.

#### Fertilizer-P source Effects on 2-yr Cumulative Corn Properties

To integrate over the two consecutive growing seasons, 2-yr cumulative corn properties were evaluated. The effects of the various fertilizer-P sources on 2-yr cumulative corn properties were variable. Two of the 17 corn properties evaluated were affected ( $P < 0.05$ ) by fertilizer-P source, while 15 corn properties were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 9).

Corn stalk + leaves DM, P, N, and Mg uptake were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 9). Stalk + leaves DM ranged from 14.6 Mg ha<sup>-1</sup> from DAP to 16.4 Mg ha<sup>-1</sup> from the UC and averaged 15.3 Mg ha<sup>-1</sup> across all fertilizer-P sources (Table 10). Stalk + leaves P uptake ranged from 15 kg ha<sup>-1</sup> from the UC to 19 kg ha<sup>-1</sup> from CPST and averaged 17 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 10). Stalk + leaves N uptake ranged from 119 kg ha<sup>-1</sup> from DAP to 142 kg ha<sup>-1</sup> from ECST and averaged 131 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 10). Stalk + leaves Mg uptake ranged from 60 kg ha<sup>-1</sup> from DAP to 71 kg ha<sup>-1</sup> from ECST and averaged 66 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 10).

Similar to corn stalk + leaves DM and nutrient uptake, cob + husk DM, P, N, and Mg uptake were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 9). Cob + husk DM ranged from

3.9 Mg ha<sup>-1</sup> from DAP to 4.9 Mg ha<sup>-1</sup> from RP and averaged 4.2 Mg ha<sup>-1</sup> across all fertilizer-P sources (Table 10). Cob + husk P uptake ranged from 2.5 kg ha<sup>-1</sup> from TSP to 2.9 kg ha<sup>-1</sup> from RP and averaged 2.7 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 10). Cob + husk N uptake ranged from 22 kg ha<sup>-1</sup> from DAP to 29 kg ha<sup>-1</sup> from RP and averaged 25 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 10). Cob + husk Mg uptake ranged from 2.8 kg ha<sup>-1</sup> from DAP to 3.9 kg ha<sup>-1</sup> from ECST and averaged 3.3 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 10).

Similar to corn stalk + leaves and cob + husk DM and nutrient uptake, kernel DM, P and Mg uptake were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 9). Kernel DM ranged from 18.5 Mg ha<sup>-1</sup> from DAP to 23.1 Mg ha<sup>-1</sup> from ECST and averaged 20.5 Mg ha<sup>-1</sup> across all fertilizer-P sources (Table 10). Kernel P uptake ranged from 53 kg ha<sup>-1</sup> from DAP to 71 kg ha<sup>-1</sup> from ECST and averaged 59 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 10). Kernel Mg uptake ranged from 19 kg ha<sup>-1</sup> from DAP to 26 kg ha<sup>-1</sup> from ECST and averaged 22 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 10). However, kernel N uptake differed ( $P = 0.03$ ) among fertilizer-P sources (Table 9). Kernel N uptake was numerically largest from ECST (301 kg ha<sup>-1</sup>), which did not differ from MAP or RP, and was numerically smallest from DAP (226 kg ha<sup>-1</sup>), which did not differ from CPST, TSP, or the UC (Table 11). Kernel N uptake from ECST was at least 1.2 times greater than that from CPST, TSP, or the UC, which did not differ (Table 11). Kernel N uptake from ECST was at least 1.3 times greater than that from DAP (Table 11). Kernel N uptake from CPST did not differ from DAP, TSP, MAP, RP, or the UC (Table 11). Kernel N uptake from MAP and RP, which averaged 265.5 kg ha<sup>-1</sup>, was 1.2 times greater than that from DAP (Table 11). Fertilizer-induced soil acidification from the initial magnitude to after two growing seasons (Figure 2) in the ECST, MAP, and RP treatments may have enhanced P dissolution and availability that had been precipitated as Ca-P. It is plausible that there was a



positive interaction between available P with N (Espinoza and Ross, 2008), promoting N uptake, thus resulting in greater kernel N from ECST, MAP, and RP than that from CPST, DAP, TSP, and the UC.

Similar to corn stalk + leaves and cob + husk DM and nutrient uptake and kernel DM, P and Mg uptake, total aboveground DM, P, N, and Mg uptake were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 9). Total aboveground DM ranged from 37.0 Mg ha<sup>-1</sup> from DAP to 42.6 Mg ha<sup>-1</sup> from ECST and averaged 40.0 Mg ha<sup>-1</sup> across all fertilizer-P sources (Table 10). Total aboveground P uptake ranged from 72.6 kg ha<sup>-1</sup> from DAP to 89.5 kg ha<sup>-1</sup> from ECST and averaged 78.9 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 10). Total aboveground N uptake ranged from 369 kg ha<sup>-1</sup> from DAP to 470 kg ha<sup>-1</sup> from ECST and averaged 413 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 10). Total aboveground Mg uptake ranged from 82 kg ha<sup>-1</sup> from DAP to 101 kg ha<sup>-1</sup> from ECST and averaged 90 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 10).

In contrast to corn stalk + leaves, cob + husk, and total aboveground DM and nutrient uptake, and kernel DM, P and Mg uptake, 2-yr cumulative corn yield differed ( $P < 0.01$ ) among fertilizer-P sources (Table 9). Corn yield was numerically largest from ECST (25.9 Mg ha<sup>-1</sup>), which differed from all other treatments (Table 11). Yield from ECST was at least 1.2 times greater than from CPST, TSP, MAP, DAP, RP, and the UC, which did not differ and averaged 21.0 Mg ha<sup>-1</sup> (Table 11). Greater 2-yr cumulative corn yield from ECST was likely due to a greater acidification effect of the ECST material on the change in soil pH from the initial pH to the end of the two growing seasons (Figure 2). Soil pH reduction brought the soil pH closer to 6.5, the pH of minimum P adsorption, thus enhancing P dissolution and availability of Ca-precipitated P in the soil for plant root uptake.

### *Change in Soil Properties*

The effects of various fertilizer-P sources on the change in soil properties in the top 10 cm from initial values after one year and after two growing seasons for the corn study were variable. The change in all 14 soil properties after one year were unaffected ( $P > 0.05$ ; Table 8) by fertilizer-P source. Two of the 12 soil property changes from the initial evaluated after two growing seasons were affected ( $P < 0.05$ ) by fertilizer-P source, while 10 soil property changes from the initial were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 8).

After two-growing seasons, soil pH generally decreased ( $P < 0.05$ ; Table 8) from the initial values in all treatments (Figure 2). Soil pH decreased from the initial soil values more for ECST (-0.35 pH units) than MAP, RP, and the UC (-0.19, -0.13, and -0.14 pH units, respectively), which all differed from a change of zero. However, the change in soil pH from the initial pH did not differ from zero for CPST, DAP, and TSP (Figure 2). The general acidifying effect that was present in ECST, MAP, RP, and the UC treatments after two corn growing seasons was likely due to differences in fertilizer compositions and forms of P present in the fertilizers (i.e.,  $\text{HPO}_4^{2-}$  and  $\text{H}_2\text{PO}_4^-$ ; FTRC, 2015). The dissolution of accumulated fertilizer-P and the influx of various cations (i.e.,  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ , and  $\text{NH}_4^+$ ), which displaced  $\text{H}^+$  on soil exchange sites at potentially different concentrations (Nascimento, Pagliari, Faria, & Vitti, 2018; Anderson, Brye, Greenlee, & Gbur, 2020) likely reduced the soil pH among fertilizer-P sources after two growing seasons. Fertilizers containing P in the phosphate form ( $\text{H}_2\text{PO}_4^-$ ), such as MAP and TSP, can cause an acidifying effect in alkaline soils due to the dissociation of  $\text{H}^+$  (FTRC, 2015), which did not happen for TSP. The greater reduction in soil pH in the ECST treatment than in other fertilizer treatments was at least partly caused by greater initial Mg concentrations from both batches of ECST in 2019 and 2020 than all other fertilizer-P sources, which likely

affected exchange-site equilibria with the soil solution as a greater concentration of  $Mg^{2+}$  from the dissolving ECST material could have replaced  $H^+$  ions on soil exchange sites to increase the  $H^+$  concentration in the soil solution.

After two growing seasons, the change in soil P concentration from the initial differed among fertilizer-P sources ( $P < 0.05$ ; Table 8). Though the change in soil P concentration did not differ from zero for all treatments, soil P concentration decreased the most in the UC (-17.4 mg  $kg^{-1}$ ), which did not differ from ECST and RP (-13.2 and -15.4 mg  $kg^{-1}$ ; Figure 2). Soil P concentration decreased least in the CPST treatment (-9.5 mg  $kg^{-1}$ ), which did not differ from ECST, DAP, MAP, and TSP (Figure 2). The decrease in soil P concentration in CPST differed from RP and the UC (Figure 2). The soil-P concentration decrease in RP did not differ from ECST, DAP, MAP, and TSP (Figure 2). The soil-P concentration decrease in ECST did not differ from any other treatment (Figure 2).

The greater reduction in soil pH from the initial in the ECST treatment likely enhanced nutrient dissolution and availability for corn uptake, which was demonstrated in greater kernel DM, tissue P and Mg uptake, total aboveground tissue N and Mg uptake, and yield in 2019 and 2020 and in the greater the 2-yr cumulative kernel tissue N uptake and yield than the most commonly used and more readily soluble fertilizer-P sources in Arkansas (i.e., TSP; Chien et al., 2011; Slaton et al., 2013). The large P uptake in the ECST treatment correlated with the reduction in soil-P concentration from the initial in the ECST treatment.

## Soybean Study

### *Initial Soil Properties*

Similar to the corn study, since the soybean field study was conducted in the same area

within the same field each year, it was necessary to assess the initial soil properties, which was used as a reference, for further determination of any change in soil properties after one year and two soybean growing seasons. Initial soil pH was slightly alkaline (pH = 7.02) and initial extractable soil Mg and Ca levels were above the optimum category ( $> 30 \text{ mg kg}^{-1}$  for Mg and  $> 400 \text{ mg kg}^{-1}$  for Ca) for most row crops in Arkansas (Espinosa, Slaton, & Mozaffari, 2021). Soybeans generally grow well in a soil pH range of 6.0 to 7.0, though the optimal range is between 6.3 and 6.5 (Staton, 2012). However, some fraction of fertilizer-applied P could be unavailable to plant roots due to the slightly alkaline soil pH, coupled with the large extractable soil Ca concentration, enabling P binding with soil Ca to form insoluble complexes (Espinosa & Ross, 2008). Based on soil sampling conducted in May 2019, after plots were established, but before fertilizer application occurred, Mehlich-3-extractable soil P averaged  $28.3 \text{ mg kg}^{-1}$  [standard error (SE) = 3.1] in the top 10 cm, which was below optimum for soybean production in a silt-loam soil (Slaton et al., 2013), thus a plant response from the added fertilizer-P in both years was expected. Table 1 summarizes means for all initial soil properties for the soybean study site.

### *Soybean Response*

Soybean response to the various fertilizer-P sources evaluated in a given year was variable. Thirteen of the 17 soybean properties evaluated were affected ( $P < 0.05$ ) by fertilizer-P source, either as a main effect or as an interaction with year, while four soybean properties were unaffected ( $P > 0.05$ ) by fertilizer-P source or year (Table 12).

Contrary to expectations, soybean aboveground DM was unaffected ( $P > 0.05$ ) by fertilizer-P source or year (Table 12), despite the soil-test-P levels being below optimum, such

that a plant response was expected, at least from the P-fertilized treatments relative to the unamended control. Aboveground DM ranged from 7.5 Mg ha<sup>-1</sup> from the UC in 2019 to 8.7 Mg ha<sup>-1</sup> from TSP in 2020 and averaged 8.2 Mg ha<sup>-1</sup> across all fertilizer-P sources and both years (Table 13). Similar to the current results, Ylagan et al. (2020) reported no differences in soybean aboveground DM among similar fertilizer-P sources.

In contrast to aboveground DM, aboveground tissue P concentration differed ( $P < 0.01$ ) among fertilizer-P sources (Table 12). Averaged across years, aboveground tissue P concentration was numerically largest from ECST (0.33 %), which did not differ from CPST and TSP, and was numerically smallest from RP (0.28 %), which did not differ from DAP, MAP, and the UC (Table 14). Aboveground tissue P concentration from ECST was at least 1.1 times greater than that from DAP, MAP, RP, or the UC (Table 14). Aboveground tissue P concentration from CPST was at least 1.1 times greater than that from MAP, RP, or the UC (Table 14). Aboveground tissue P concentration from TSP, which did not differ from DAP, MAP, or the UC, was 1.1 times greater than that from RP (Table 14). Similar to the current results, Ylagan et al. (2020) reported differences in soybean stem-plus-leaves tissue P concentration among fertilizer-P sources, where stem-plus-leaves tissue P concentration was similar between ECST, CPST, TSP, DAP, and MAP, which differed from RP and the no P/+N and no P/-N control treatments.

Similar to aboveground DM, aboveground tissue N concentration was unaffected ( $P > 0.05$ ) by fertilizer-P source or year (Table 12). Aboveground N concentration ranged from 2.6 % from the UC in 2020 to 3.8 % from ECST in 2019 and averaged 3.2 % across all fertilizer-P sources and both years (Table 13). Ylagan et al. (2020) documented similar results to the current study in for soybean stem-plus-leaves N concentration in a greenhouse pot study.

Similar to aboveground DM and tissue N concentration, aboveground tissue Mg concentration was unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 12). However, averaged across fertilizer-P sources, aboveground tissue Mg concentration was 1.2 times greater ( $P = 0.01$ ) in 2020 than in 2019 (Table 15). In contrast to the results of the current study, Ylagan et al. (2020) reported differences in soybean stem-plus-leaves tissue Mg concentration among fertilizer-P sources, where stem-plus-leaves tissue Mg concentration was numerically largest from ECST, which did not differ from CPST, TSP, or MAP, and was numerically smallest from RP, which did not differ from DAP and the no P/+N and no P/-N control treatments. Stem-plus-leaves tissue Mg concentration from ECST was at least 1.2 times greater than that from DAP, RP, no P/+N, or the no P/-N control treatments (Ylagan et al., 2020). Stem-plus-leaves tissue Mg concentration from CPST or MAP, which did not differ, was at least 1.2 times greater than that from RP or the no P/-N control treatment (Ylagan et al., 2020). Stem-plus-leaves tissue Mg concentration from TSP was at least 1.1 times greater than that from RP (Ylagan et al., 2020).

In contrast to aboveground DM and tissue N concentration, aboveground tissue P and N uptake differed ( $P < 0.05$ ) among fertilizer-P sources or years (Table 12). Averaged across years, aboveground tissue P uptake was numerically largest from ECST (28.4 kg ha<sup>-1</sup>), which did not differ from CPST or TSP, and was numerically smallest from RP (21.4 kg ha<sup>-1</sup>), which did not differ from the UC (Table 14). Aboveground tissue P uptake from ECST was at least 1.2 times greater than that from DAP or MAP (Table 14). Aboveground tissue P uptake from ECST was at least 1.3 times greater than that from RP or the UC (Table 14). Aboveground tissue P uptake from CPST, which did not differ from DAP or TSP, was at least 1.1 times greater than that from MAP and at least 1.2 times greater than that from RP or the UC (Table 14). Aboveground tissue P uptake from DAP, which did not differ from MAP or TSP, was at least 1.1 times greater than

that from RP or the UC (Table 14). Aboveground tissue P uptake from TSP, which did not differ from MAP, was at least 1.2 times greater than that from RP or the UC (Table 14). Aboveground tissue P uptake from MAP, which did not differ from the UC, was at least 1.1 times greater than that from RP (Table 14).

The differences in aboveground P concentration and uptake may have related to varying effects on the rhizosphere pH by the fertilizer-P sources. Greater aboveground P concentration and uptake from ECST could be due to rapid dissolution of the ECST material, which was more water-soluble than DAP, MAP, and RP (Anderson et al., 2021c). Similar aboveground P concentration and uptake between the two struvite materials (ECST and CPST) and the highly water-soluble TSP contradicts the slow-release characteristic of struvite previously reported (Rahman et al., 2014; Talboys et al., 2015). The struvite materials used in this study demonstrated a more rapid-release characteristic, which has also been reported in plant-less soil incubation experiments under moist- and flooded-soil conditions (Anderson et al., 2021a,c). In addition, soybean root exudation of carboxylate compounds could have also contributed to greater aboveground P concentration and uptake from the struvite materials, ECST and CPST, as struvite dissolution likely increased in the presence of organic acids exuded by plant roots (Tang, Qiao, Han, & Zheng, 2007; Cabeza et al., 2011; Antonini et al., 2012).

Averaged across years, aboveground N uptake was numerically largest from ECST (277 kg ha<sup>-1</sup>), which did not differ from CPST, DAP, or TSP, and was numerically smallest from RP (239 kg ha<sup>-1</sup>), which did not differ from MAP, TSP, or the UC (Table 14). Aboveground N uptake from ECST or CPST was at least 1.1 times greater than that from MAP, RP, or the UC (Table 14). Though the N rate was balanced across all fertilizer-P sources using urea, greater aboveground tissue N uptake from ECST and CPST than MAP, RP, and the UC could have been

due to rapid dissolution of the struvite materials, which met the N demand of the soybean plants. In addition, the  $\text{NH}_4^+$  form in MAP could have been less translocatable in the soybean plant or did not dissolve as rapidly as the struvite materials to meet the N and P needs of the soybean plant, which was in contrast to previous report of rapid dissolution of MAP (Chien et al., 2011). Aboveground N uptake from DAP, which did not differ from MAP or TSP, was at least 1.1 times greater than that from RP or the UC (Table 14).

Averaged across fertilizer-P sources, aboveground tissue P and N uptake were at least 1.3 times greater ( $P < 0.01$ ) in 2019 than in 2020 (Table 15). Similar to the results of the current study, Rech et al. (2019) reported that soybean total P uptake from TSP, which did not differ from three struvite materials, was 1.7 times greater than that from the unamended control.

Contrary to expectations, but similar to aboveground tissue Mg concentration, aboveground tissue Mg uptake was unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 12). However, averaged across fertilizer-P sources, aboveground tissue Mg uptake was at least 1.1 times greater ( $P = 0.02$ ) in 2020 than in 2019 (Table 15).

Similar to aboveground tissue Mg concentration and uptake, seed P and N concentrations were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 12). However, averaged across fertilizer-P sources, seed P concentration was 1.1 times greater ( $P < 0.01$ ) in 2019 than in 2020 (Table 15), while seed N concentration was also greater ( $P < 0.01$ ) in 2020 than in 2019 (Table 15). In contrast to the results of the current study, Ylagan et al. (2020) reported differences in soybean pod P concentration among fertilizer-P sources, where pod P concentration was numerically largest from MAP, which did not differ from ECST or DAP, and was numerically smallest from RP, which did not differ from the no P/+N or no P/-N control treatments. Pod P concentration from MAP was at least 1.1 times greater than that from CPST or TSP and at least 1.3 times



greater than that from RP and the no P/+N or no P/-N control treatments (Ylagan et al., 2020). Pod P concentration from ECST, which did not differ from CPST, DAP, or TSP, was at least 1.2 times greater than that from RP and the no P/+N or no P/-N control treatments (Ylagan et al., 2020). Pod P concentration from CPST and TSP, which did not differ, was at least 1.1 times greater than that from RP and the no P/+N or no P/-N control treatments (Ylagan et al., 2020).

Similar to aboveground DM and tissue N concentration, seed Mg concentration was unaffected ( $P > 0.05$ ) by fertilizer-P source or year (Table 12). Seed Mg concentration ranged from 0.32 % from DAP in 2020 to 0.34 % from MAP in 2020 and averaged 0.33 % across all fertilizer-P sources and both years (Table 13). Similar to the results of the current study, Ylagan et al. (2020) reported no differences in soybean pod N and Mg concentrations among fertilizer-P sources in a greenhouse pot study.

In contrast to aboveground DM, tissue nutrient concentrations and uptake, and seed nutrient concentrations, seed P, N, and Mg uptake differed among fertilizer-P sources between years ( $P = 0.03$ ; Table 12). In 2019, seed P uptake was numerically largest from ECST (25.2 kg ha<sup>-1</sup>), which did not differ from CPST, DAP, MAP, TSP, or RP, and was numerically smallest from the UC (21.0 kg ha<sup>-1</sup>), which did not differ from CPST, DAP, MAP, TSP, or RP (Table 16). In 2019, seed P uptake from ECST was 1.2 times greater than that from the UC (Table 16). In 2020, seed P uptake was numerically greatest from CPST (15.5 kg ha<sup>-1</sup>), which did not differ from DAP, MAP, TSP, or RP, and was numerically smallest from ECST (11.9 kg ha<sup>-1</sup>), which did not differ from the UC (Table 16). In 2020, seed P uptake from CPST was at least 1.3 times greater than that from ECST and the UC (Table 16). In 2020, seed P uptake from MAP, RP, or TSP, which did not differ, was at least 1.2 times greater than that from ECST and the UC (Table 16). In 2020, seed P uptake from DAP, which did not differ from the UC, was at least 1.2 times

greater than that from ECST (Table 16). Seed P uptake from all fertilizer-P sources in 2019 were greater than those in 2020 (Table 16).

In 2019, seed N uptake was numerically largest from ECST (254 kg ha<sup>-1</sup>), which did not differ from CPST, DAP, TSP, MAP, or RP, and was numerically smallest from the UC (215 kg ha<sup>-1</sup>), which did not differ from CPST, DAP, TSP, MAP, or RP (Table 16). In 2019, seed N uptake from ECST was 1.2 times greater than that from the UC (Table 16). In 2020, seed N uptake was numerically greatest from RP (168 kg ha<sup>-1</sup>), which did not differ from CPST, DAP, MAP, TSP, or the UC, and was numerically smallest from ECST (129 kg ha<sup>-1</sup>), which did not differ from the UC (Table 16). In 2020, seed N uptake from RP and CPST was at least 1.3 times greater than that from ECST (Table 16). In 2020, seed N uptake from MAP, DAP, and TSP was at least 1.2 times greater than that from ECST (Table 16). Seed N uptake from all fertilizer-P treatments in 2019 was greater than the corresponding treatments in 2020 (Table 16). Seed N uptake from the UC in 2019 was similar to that from CPST and RP in 2020 (Table 16).

In 2019, seed Mg uptake was numerically largest from ECST (13.2 kg ha<sup>-1</sup>), which did not differ from all other treatments (Table 16). In 2020, seed Mg uptake was numerically greatest from RP (8.7 kg ha<sup>-1</sup>), which did not differ from CPST, DAP, MAP, or TSP, and was numerically smallest from ECST (6.6 kg ha<sup>-1</sup>), which did not differ from the UC (Table 16). In 2020, seed Mg uptake from RP and CPST was at least 1.2 times greater than that from ECST and the UC (Table 16). In 2020, seed Mg uptake from MAP, DAP, and TSP was at least 1.2 times greater than that from ECST (Table 16). Seed Mg uptake from all fertilizer-P sources in 2019 were greater than those in 2020 (Table 16).

Similar to aboveground tissue Mg concentration and uptake and seed P and N concentrations, belowground tissue P and Mg concentrations were unaffected ( $P > 0.05$ ) by

fertilizer-P source (Table 12). However, averaged across fertilizer-P sources, belowground P and Mg concentrations were 3.3 and 2.9 times, respectively, greater ( $P < 0.05$ ) in 2020 than in 2019 (Table 15), likely due to a carry-over effect from the 2019 fertilization.

The lack of fertilizer-P-source differences in aboveground DM, seed P concentration and uptake, and belowground P concentration may have occurred from relatively uniform precipitation/fixation of dissolved-P from the fertilizer materials by the large concentration of initial extractable soil Ca coupled with the slight alkaline soil. It is also plausible that N-fixing legumes, such as soybean, may have taken up more cations than anions, thus releasing protons from their roots to balance their charge (Hinsinger, 2001; Tang et al., 2007). Consequently, the rhizosphere acidification, which occurred as a result of net export of protons to the rhizosphere, could have, at least in part, increased P availability through the dissolution of precipitated/fixed Ca-P in the alkaline soil (Hinsinger, 2001; Tang et al., 2007; Richardson, Barea, McNeill, & Prigent-Combaret, 2009; Gao et al., 2019), creating relatively uniform P availability from all fertilizer-P sources for the soybean plants to experience. Though a low concentration of carboxylate compounds are produced from soybean roots relative to other legumes (Tang et al., 2007), the effect of carboxylate root exudation could have, at least in part, reduced the rhizosphere pH, thus releasing some Ca-bound P. The lack of difference could have also been due to soil organic P mineralization related to increased phosphatase activity in the soybean rhizosphere (Nuruzzaman, Lambers, Bolland, & Veneklaas, 2006, Wang et al., 2009; Kong, Li, Wang, Li, Du, & Zhang, 2018). Plants are known to synthesize various enzymes that exist in soils, which may remain active for some time after synthesis. Soybean roots can exude acidic phosphatase (Wang et al., 2009, Li et al., 2012, Kong et al., 2018), which mineralizes organic P by catalyzing hydrolytic cleavage of inorganic P from organic-P compounds.

Similar to the results of the current study, Ylagan et al. (2020) reported differences in soybean belowground P concentration among similar fertilizer-P sources in a greenhouse pot study. Ylagan et al. (2020) reported that belowground P concentration was numerically largest from MAP, which did not differ from TSP, and was numerically smallest from the no P/-N control treatment, which did not differ from the no P/+N control treatment. Belowground P concentration from MAP was at least 1.2 times greater than that from ECST, CPST, or RP. Belowground P concentration from ECST and CPST was at least 1.1 times greater than that from the no P/+N and no P/-N control treatments. (Ylagan et al., 2020).

The lack of difference in aboveground and seed Mg concentrations and uptake and belowground Mg concentration among fertilizer-P sources was likely due to the inherent large concentrations of initial extractable soil Mg ( $> 300 \text{ mg kg}^{-1}$ ) in the top 10 cm of the soil (Table 1) and more available soil Mg below the top 10 cm, despite the two struvite sources containing greater initial Mg concentrations than the other fertilizer-P sources (Table 2). In addition, soybean plants may have uniformly accumulated Mg from P fertilization, which increased the concentration and uptake of Mg. Tang et al. (1998) reported that specific acid production in legumes was correlated with concentrations of excess cations, such as Mg. Similar to the results of the current study, Ylagan et al. (2020) reported no difference in soybean belowground Mg concentration among fertilizer-P sources in a greenhouse pot study.

Similar to aboveground DM and tissue N concentration and seed Mg concentration, belowground N concentration was unaffected ( $P > 0.05$ ) by fertilizer-P source or year (Table 12). Belowground N concentration ranged from 0.7 % from ECST in 2019 to 1.4 % from RP in 2020 and averaged 1.0 % across all fertilizer-P sources and both years (Table 13). Similar to other legumes, soybean has the ability to increase N availability by biological  $\text{N}_2$  fixation (Tamagno et

al., 2017; Santachiara, Salvagiotti, & Rotundo, 2019; Wang et al., 2019). The mechanism could have provided additional plant-available N that masked potential and expected differences in aboveground N concentration, seed N concentration and uptake, and belowground N concentration among fertilizer-P sources. Similar to the results of the current study, Ylagan et al. (2020) reported no differences in soybean belowground N concentration among fertilizer-P sources in a greenhouse pot study.

As expected and similar to seed P, N, and Mg uptake, seed yield, as measured with the plot combine, differed among fertilizer-P sources between years ( $P = 0.03$ ; Table 12). In 2019, seed yield was numerically largest from ECST ( $4.1 \text{ Mg ha}^{-1}$ ), which did not differ from CPST, TSP, MAP, DAP, or RP, and was numerically smallest from the UC ( $3.6 \text{ Mg ha}^{-1}$ ), which did not differ from CPST, TSP, MAP, DAP, or RP (Table 16). In 2019, seed yield from ECST was 1.2 times greater than that from the UC (Table 16) likely due to quick dissolution of the ECST material releasing P for root uptake, which is contrary to the reported slow-release characteristics of struvite (Nascimento et al., 2018; Anderson et al., 2020). In 2020, seed yield was numerically greatest from CPST ( $2.8 \text{ Mg ha}^{-1}$ ), which did not differ from DAP, MAP, RP, TSP, or the UC, and was numerically smallest from ECST ( $2.2 \text{ Mg ha}^{-1}$ ), which did not differ from the UC (Table 16). In 2020, seed yield from CPST, TSP, MAP, DAP, or RP was at least 1.2 times greater than that from ECST (Table 16). Since two batches of the ECST materials were generated, it appears that the second batch of the ECST was slower to dissolve than the first batch, resulting in lower yield and seed P, N, and Mg uptake than that from CPST, TSP, DAP, MAP, and RP in 2020.

Seed yield from all fertilizer-P sources in 2019 were greater than those in 2020 (Table 16), which was likely due to greater monthly rainfall during the growing season in 2019 than in 2020 (Figure 3). Though all plots were furrow-irrigated in both years, soybean growth and

productivity would have benefitted from more uniform, well-watered soil moisture conditions in 2019, with lower magnitudes of soil moisture fluctuations than in 2020, which had less timely rainfall events. It is also plausible that well-watered soil conditions in 2019 promoted P diffusion from the fertilizer-P source, which resulted in greater yield, aboveground tissue P uptake, and seed P concentration and uptake in 2019 than in 2020. However, it remains unclear why there was a lower belowground tissue P concentration in 2019 than in 2020. Additionally, differences in soybean yields between years may also be at least partially attributed to the different cultivars grown each year, as soybean yields are closely related to genetic potential (Scaboo, Chen, Sleper, & Clark, 2010).

Since there were no reports for Pioneer 46A70L SU26 grown in 2019 or USG 7469 GTL grown in 2020 at CBES from the Arkansas Soybean Performance Trial, yield results in the same years at CBES for the closest soybean varieties (i.e., 4.6 maturity group) were Pioneer 46A57BX in 2019 (4.5 Mg ha<sup>-1</sup>; Carlin, Bond, & Still, 2020b) and USG 7461 XT in 2020 (4.2 Mg ha<sup>-1</sup>; Carlin, Bond, & Morgan, 2021b). Averaged across all fertilizer-P sources, soybean yields from the current study were lower in 2019 and 2020 than those from the Arkansas Soybean Performance Trial yield results in 2019 and 2020 at CBES. In 2019, yield from the current study ranged from 3.6 Mg ha<sup>-1</sup> from the UC to 4.1 Mg ha<sup>-1</sup> from ECST and averaged 3.8 Mg ha<sup>-1</sup> across all fertilizer-P sources. In 2020, yield from the current study ranged from 2.2 Mg ha<sup>-1</sup> from ECST to 2.8 Mg ha<sup>-1</sup> from CPST and averaged 2.6 Mg ha<sup>-1</sup> across all fertilizer-P sources. Similar to the current study, irrigated soybean in the Arkansas Soybean Performance Trial in 2019 was grown on a Calloway silt loam, but the irrigated soybean in the Arkansas Soybean Performance Trial in 2020 was grown on a different soil series (Loring silt loam). Similar to corn, soybean yield results from the current study were somewhat different than the soybean grown under optimal

management according to University of Arkansas recommendations in the 2019 and 2020 Arkansas Soybean Performance Trials. Earlier planting dates in the 2019 and 2020 Arkansas Soybean Performance Trials could have been a contributing factor to greater yields from the Arkansas Soybean Performance Trials than from the current study each year.

#### Fertilizer-P source Effects on 2-yr Cumulative Soybean Properties

Similar to corn properties, to integrate over the two consecutive growing seasons, 2-yr cumulative soybean properties were evaluated. The 2-year cumulative soybean response to the various fertilizer-P sources was variable. Two of the eight soybean properties evaluated were affected ( $P < 0.05$ ) by fertilizer-P source, while six soybean properties were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 17).

Two-year cumulative soybean aboveground DM and Mg uptake were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 17). Aboveground DM ranged from 15.4 Mg ha<sup>-1</sup> from the UC to 17.2 Mg ha<sup>-1</sup> from ECST and averaged 16.3 Mg ha<sup>-1</sup> across all fertilizer-P sources (Table 18). Aboveground Mg uptake ranged from 74 kg ha<sup>-1</sup> from the UC to 84 kg ha<sup>-1</sup> from ECST and averaged 79 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 18).

In contrast to aboveground DM and Mg uptake, the 2-yr cumulative aboveground P and N uptake differed ( $P < 0.05$ ) among fertilizer-P sources (Table 17). Aboveground P uptake was numerically largest from ECST (58 kg ha<sup>-1</sup>), which did not differ from CPST, and was numerically smallest from RP (43 kg ha<sup>-1</sup>), which did not differ from the UC (Table 19). Aboveground P uptake from ECST was at least 1.2 times greater than that from DAP or MAP, which did not differ (Table 19). Aboveground P uptake from ECST was at least 1.3 times greater

than that from RP or the UC, which did not differ (Table 19). Aboveground P uptake from ECST was at least 1.1 times greater than that from TSP (Table 19). Aboveground P uptake from CPST, which did not differ from TSP, was at least 1.1 times greater than that from DAP, MAP, RP, or the UC (Table 19). Aboveground P uptake from TST, which did not differ from DAP, was at least 1.1 times greater than that from MAP, RP, or the UC (Table 19). Aboveground P uptake from DAP, which did not differ from MAP, was at least 1.1 times greater than that from RP or the UC (Table 19). Aboveground P uptake from MAP, which did not differ from the UC, was at least 1.1 times greater than that from RP (Table 19).

Two-year cumulative aboveground N uptake was numerically largest from ECST (566 kg ha<sup>-1</sup>), which did not differ from CPST, DAP, or TSP, and was numerically smallest from RP (483 kg ha<sup>-1</sup>), which did not differ from MAP, TSP, and the UC (Table 19). Aboveground N uptake from ECST and CPST, which averaged 562 kg ha<sup>-1</sup>, was at least 1.1 times greater than that from MAP, RP, or the UC (Table 19). Aboveground N uptake from DAP was at least 1.1 times greater than that from RP (Table 19).

Similar to soybean aboveground DM and Mg uptake, seed P, N, and Mg uptake were also unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 17). Seed P uptake ranged from 33 kg ha<sup>-1</sup> from the UC to 39 kg ha<sup>-1</sup> from CPST and averaged 37 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 18). Seed N uptake ranged from 358 kg ha<sup>-1</sup> from the UC to 394 kg ha<sup>-1</sup> from CPST and averaged 385 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 18). Seed Mg uptake ranged from 18.9 kg ha<sup>-1</sup> from the UC to 20.8 kg ha<sup>-1</sup> from CPST and averaged 20.2 kg ha<sup>-1</sup> across all fertilizer-P sources (Table 18).

Similar to soybean aboveground DM and Mg uptake and seed P, N, and Mg uptake, 2-yr cumulative soybean yield (i.e., combine measured) was unaffected ( $P > 0.05$ ) by fertilizer-P



source (Table 17). Two-year cumulative soybean yield ranged from 6.0 Mg ha<sup>-1</sup> from the UC to 6.6 Mg ha<sup>-1</sup> from CPST and averaged 6.4 Mg ha<sup>-1</sup> across all fertilizer-P sources (Table 18).

### *Change in Soil Response*

Similar to the corn study, the effects of various fertilizer-P sources on the change in soil properties from initial values after one year and after two-growing seasons in the top 10 cm for the soybean study were variable. After one year, all 14 soil property changes from the initial were unaffected ( $P > 0.05$ ; Table 8) by fertilizer-P source. However, after two growing seasons, two of the 12 soil property changes from the initial were affected ( $P < 0.05$ ) by fertilizer-P source, while 10 soil property changes from the initial were unaffected ( $P > 0.05$ ) by fertilizer-P source (Table 8).

After two growing seasons, the change in soil pH from the initial pH differed among fertilizer-P sources ( $P < 0.05$ ; Table 8), where soil pH decreased from the initial pH in MAP (-0.20 pH units) and DAP (-0.06 pH units), but increased in ECST (0.01 pH units), CPST (0.12 pH units), RP (0.03 pH units), and TSP (0.02 pH units). The soil pH minimally changed from the initial value in the UC (< -0.01 pH units; Figure 2). In all treatments, the change in soil pH differed from a change of zero only in MAP (Figure 2). The change in soil pH for MAP (-0.20 pH units), which was similar to DAP (-0.06 pH units), differed from ECST, CPST, RP, TSP, and the UC. The change in soil pH for CPST, which was similar to ECST, RP, TSP, and the UC, differed from DAP (Figure 2).

After two growing seasons, the change in soil P concentration from the initial soil concentration differed among fertilizer-P sources ( $P < 0.05$ ; Table 8). Though soil P

concentration did not differ from zero in all treatments, soil P concentration decreased the most in the RP treatment ( $-10.25 \text{ mg kg}^{-1}$ ), which did not differ from the UC ( $-9.91 \text{ mg kg}^{-1}$ ; Figure 2). The change in soil P concentration from the initial value increased in ECST, CPST, MAP, DAP, and TSP, which did not differ (Figure 2). Similar trends were observed in the change in soil pH and soil P concentration among ECST, CPST, and TSP, which suggests that the struvite materials used in this study behaved similar to the highly water-soluble TSP, which was also evident in the aboveground tissue P concentration and uptake and N uptake.

## **Implications**

All crop responses evaluated in both the corn and soybean study showed that struvite (ECST and CPST) had no substantially adverse effects on crop growth and production. Specific to the soybean study, struvite showed comparable aboveground tissue P concentration and P and N uptake to commonly used TSP. However, in some cases, the ECST treatment had a larger positive effect on corn properties than most of the other fertilizer-P sources. The greater reduction in soil pH from the initial value in the ECST treatment likely enhanced nutrient dissolution and availability for corn uptake, which was demonstrated in greater kernel DM, tissue P and Mg uptake, total aboveground tissue N uptake, yield in 2019 and 2020, and the 2-yr cumulative kernel tissue N uptake and yield than the more commonly used and more readily soluble fertilizer-P sources used in Arkansas (i.e., TSP or DAP). Though several studies have reported the slow-release nature for struvite materials (Bhuiyan, Mavinic, & Beckie, 2007; Achat et al., 2014; Rahman et al., 2014; Talboys et al., 2015; Nascimento et al., 2018; Anderson et al., 2020), the innovative ECST did not exhibit slow-release characteristics under field conditions, particularly in the corn study. Whether slow-release or not, considering the many similar corn

and soybean growth and yield responses from ECST and CPST compared to other commonly used fertilizer-P sources, struvite materials appear to be viable alternative fertilizer material for upland, row-crop production on a silt-loam in eastern Arkansas.

Apart from the P supply from wastewater-recovered struvite, producers get N and Mg basically for free in struvite compared to TSP. Additionally, the prices of TSP have almost doubled in the last two years (Fernández, 2021), therefore struvite, as an alternative fertilizer-P source, could be a more sustainable and economical nutrient source to help offset some of the supply and demand issues of RP. Furthermore, the primary energy demand associated with fertilizer creation from RP is with the mining process of RP itself (Lee, Assi, Daher, Mengoub, & Mohtar, 2020; Jing, Hou, Wang, Yao, & Liu, 2021). Compared to the large energy inputs needed for mining RP, struvite could be considered a more energy-efficient, fertilizer alternative.

Struvite recovery is beneficial not only to the agricultural industry as a viable fertilizer-P product to maintain or improve crop yields and reduce global dependency on dwindling RP reserves for long-term sustainability of crop production, but also to the wastewater treatment industry to reduce P concentrations in effluents and improve treatment efficiencies, cost, and time (Parsons et al., 2001; Talboys et al., 2015; Tansel, Lunn, & Monje, 2018; Hallas, Mackowiak, Wilkie, & Harris, 2019). Additionally, increasing public concern about environmental quality could be addressed through precipitation and recovery of nutrients from wastewater as struvite, thereby reducing eutrophication, which is a major threat to the quality, integrity, and biodiversity of surface water bodies.

## Conclusions

Agronomic benefits of wastewater-recovered struvite had been documented in previous studies particularly in the greenhouse pot experiments. However, to our knowledge, no studies to date have examined ECST effects on corn and soybean growth and productivity relative to other conventional fertilizer-P sources under field conditions.

The hypothesis that corn total aboveground DM and yield, total aboveground tissue P and N uptake, and belowground P and N concentrations from wastewater-recovered struvite sources (i.e., ECST and CPST) would be similar to other common, commercially available fertilizer-P sources (i.e., TSP, MAP, DAP, and RP) was only partially supported because total aboveground tissue N uptake and yield differed among ECST, CPST, TSP, MAP, DAP, and RP treatments. Similarly, results did not support the hypothesis that corn total aboveground tissue Mg uptake and belowground tissue Mg concentrations would be greater for the struvite materials (ECST and CPST) due to greater initial Mg concentrations. In addition, results partially supported the hypothesis that corn grown in year two would have greater total aboveground, DM, yield, total aboveground tissue P, N, and Mg uptake, and belowground tissue P, N, and Mg concentrations compared to that in year one due to a carry-over effect from year-one fertilization, where corn yield from year one (2019) was greater than that from year two (2020) and total aboveground DM and tissue P uptake and belowground tissue P, N, and Mg concentrations did not differ between years.

The hypothesis that soybean aboveground DM and yield, above- and belowground tissue and seed P and N concentrations, and aboveground and seed P and N uptake from wastewater-recovered struvite sources (i.e., ECST and CPST) would be similar to other common, commercially available fertilizer-P sources (i.e., TSP, MAP, DAP, and RP) was only partially

supported because aboveground tissue P concentration and P and N uptake differed among ECST, CPST, TSP, MAP, DAP, and RP treatments. Additionally, results partially supported the hypothesis that soybean aboveground tissue and seed Mg concentration and uptake and belowground tissue Mg concentrations would be greater for the struvite materials (ECST and CPST) treatment because above- and belowground tissue and seed Mg concentrations and aboveground Mg uptake did not differ among ECST, CPST, TSP, MAP, DAP, and RP treatments. Similarly, results partially supported the hypothesis that soybean grown in year two would also have greater aboveground DM, yield, above- and belowground tissue and seed P, N, and Mg concentrations, and aboveground tissue and seed P, N, and Mg uptake compared to that in year one, where soybean aboveground tissue P and N uptake and seed P concentration from year one (2019) was greater than that from year two (2020) and aboveground DM and tissue N concentration, belowground tissue N concentration, and seed Mg concentration did not differ between years.

Although the soil had a large P-adsorption capacity from the large initial extractable Ca concentration and slightly alkaline soil pH, results clearly showed that wastewater-recovered struvite, from either chemical or electrochemical precipitation techniques, has potential to be a viable, alternative fertilizer-P source for corn and soybean. All corn properties affected by fertilizer-P source (i.e., kernel DM, tissue P and Mg uptake, total aboveground tissue N and Mg uptake, and yield in 2019 and 2020, and the 2-yr cumulative kernel tissue N uptake and yield) had at least similar (from CPST) and even greater (from ECST) response than several other commonly used fertilizer-P sources (i.e., TSP, DAP, and RP). In addition, all soybean properties affected by fertilizer-P source (i.e., aboveground tissue P concentration and uptake and N uptake in 2019 and 2020 and the 2-year cumulative aboveground tissue P and N uptake) had similar

response among ECST, CPST, and TSP, and in some cases, greater response from both struvite treatments than from MAP, DAP, or RP. Results from the current study overall provide valuable information about the response of corn and soybean to crystalline ECST and pelletized CPST compared with other commercially available, commonly used fertilizer-P source on a P-deficient silt-loam soil in eastern Arkansas. Though the ECST used in this study was obtained from synthetic wastewater, further research is still required to evaluate the effectiveness of ECST produced from a real wastewater source (i.e., either municipal or animal agriculture source) as a sustainable source of fertilizer-P in various soil physiochemical conditions and other upland, row-crop production settings under field conditions.

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## Tables and Figures

Table 1. Summary of initial soil properties in the top 10 cm for the corn and soybean study areas in 2019 in a phosphorus (P)-deficient, silt-loam soil in eastern Arkansas. Means ( $\pm$  standard error) are reported (n = 4).

<b>Soil Properties</b>	<b>Corn</b>	<b>Soybean</b>
Sand (kg kg <sup>-1</sup> )	0.07 (0.02)	0.10 (0.04)
Silt (kg kg <sup>-1</sup> )	0.80 (0.02)	0.75 (0.03)
Clay (kg kg <sup>-1</sup> )	0.14 (< 0.01)	0.15 (0.02)
pH	7.16 (0.05)	7.02 (0.04)
Electrical conductivity (dS m <sup>-1</sup> )	0.127 (0.01)	0.147 (0.01)
Phosphorus (mg kg <sup>-1</sup> )	33.9 (4.7)	28.3 (3.1)
Potassium (mg kg <sup>-1</sup> )	110.5 (6.1)	119.9 (9.1)
Calcium (mg kg <sup>-1</sup> )	1081 (27.6)	1171 (43.5)
Magnesium (mg kg <sup>-1</sup> )	284 (18)	337 (33.4)
Sulfur (mg kg <sup>-1</sup> )	8.4 (0.8)	7.8 (0.1)
Sodium (mg kg <sup>-1</sup> )	13.9 (0.9)	17.1 (1.0)
Iron (mg kg <sup>-1</sup> )	202.7 (16.2)	195.7 (5.6)
Manganese (mg kg <sup>-1</sup> )	162 (12)	159 (2.2)
Zinc (mg kg <sup>-1</sup> )	1.9 (0.3)	1.8 (0.2)
Copper (mg kg <sup>-1</sup> )	1.4 (0.04)	1.7 (0.2)
Total nitrogen (g kg <sup>-1</sup> )	1.0 (0.03)	1.0 (0.01)
Total carbon (g kg <sup>-1</sup> )	6.0 (0.3)	6.0 (0.1)
Carbon:nitrogen ratio	9.5 (0.2)	8.9 (0.4)
Soil organic matter (g kg <sup>-1</sup> )	17.0 (0.3)	18.0 (0.6)

Table 2. Summary of the pH, total nitrogen (N), phosphorus (P), and magnesium (Mg) concentrations and resulting measured fertilizer grade for the two batches of electrochemically precipitated struvite (ECST) used each year and the chemically precipitated struvite (CPST), triple superphosphate (TSP), monoammonium phosphate (MAP), diammonium phosphate (DAP), and rock phosphate (RP) fertilizer-P materials used for the corn and soybean studies in a P-deficient, silt-loam soil in eastern Arkansas. Means ( $\pm$  standard error) are reported (n = 5).

Fertilizer-P Source	pH	Nutrient Concentration			Measured Fertilizer Grade <sup>†</sup>
		N	P	Mg	
		%			
ECST 2019	- <sup>††</sup>	3.3 (0.2)	18.5 (0.1)	13.3 (0.1)	3-42-0
ECST 2020	-	5.1 (0.2)	16.1 (0.3)	12.7 (0.3)	5-37-0
CPST	8.77 (0.13)	5.7 (0.2)	11.7 (0.2)	8.3 (0.2)	6-27-0
TSP	2.42 (0.02)	< 0.1 (< 0.1)	18.2 (0.4)	0.6 (< 0.1)	0-42-0
MAP	4.37 (0.02)	10.7 (0.1)	20.9 (0.2)	1.5 (< 0.1)	11-48-0
DAP	7.32 (0.03)	18.1 (0.1)	18.3 (0.1)	0.7 (< 0.1)	18-42-0
RP	6.67 (0.04)	< 0.1 (< 0.1)	7.6 (0.1)	0.3 (< 0.1)	0-17-0

<sup>†</sup> Measured fertilizer grade is reported as N-P<sub>2</sub>O<sub>5</sub>-K<sub>2</sub>O

<sup>††</sup> Limited supply of ECST prohibited pH determinations in 2019 and 2020

Table 3. Summary of the effects of fertilizer-phosphorus (P) source, year, and their interaction on corn properties for the 2019 and 2020 data in a P-deficient, silt-loam soil in eastern Arkansas.

Plant Property	Source of Variation		
	Source	Year	Source x Year
	<i>P</i>		
Stalk + leaves			
Dry matter	0.59	0.60	0.53
P concentration	0.27	0.20	0.39
N concentration	0.29	<b>0.04</b>	0.17
Mg concentration	0.06	< <b>0.01</b>	0.76
P uptake	0.78	<b>0.04</b>	0.16
N uptake	0.25	< <b>0.01</b>	0.06
Mg uptake	0.34	< <b>0.01</b>	0.72
Cob + husk			
Dry matter	0.09	<b>0.02</b>	0.28
P concentration	0.88	0.06	0.93
N concentration	0.82	<b>0.03</b>	0.69
Mg concentration	0.06	< <b>0.01</b>	0.57
P uptake	0.75	< <b>0.01</b>	0.40
N uptake	0.06	< <b>0.01</b>	0.46
Mg uptake	0.13	< <b>0.01</b>	0.79
Kernel			
Dry matter	<b>0.04</b>	0.22	0.21
P concentration	0.31	0.85	0.31
N concentration	0.11	0.13	0.23
Mg concentration	0.67	0.26	0.59
P uptake	<b>0.04</b>	0.24	0.52
N uptake	< 0.01	0.02	<b>0.04</b>
Mg uptake	<b>0.03</b>	0.13	0.38
Total aboveground			
Dry matter	0.13	0.69	0.46
P uptake	0.14	0.06	0.25
N uptake	<b>0.01</b>	< <b>0.01</b>	0.06
Mg uptake	0.14	< <b>0.01</b>	0.72
Belowground			
P concentration	0.30	0.39	0.36
N concentration	0.06	0.17	0.80
Mg concentration	0.16	0.07	0.09
Yield	< <b>0.01</b>	<b>0.02</b>	0.40

† Bolded values were considered significant at  $P < 0.05$

†† Nitrogen (N) and magnesium (Mg)

Table 4. Summary of corn properties that were unaffected ( $P > 0.05$ ) by fertilizer-phosphorus (P) source or year in a P-deficient, silt-loam soil in eastern Arkansas.

<b>Plant Property</b>	<b>Minimum</b>	<b>Maximum</b>	<b>Mean</b>
Stalk + leaves dry matter (Mg ha <sup>-1</sup> )	7.0	8.7	7.6
Stalk + leaves tissue P concentration (%)	0.07	0.18	0.11
Cob + husk tissue P concentration (%)	0.05	0.09	0.07
Kernel tissue P concentration (%)	0.27	0.33	0.29
Kernel tissue N <sup>†</sup> concentration (%)	1.1	1.4	1.2
Kernel tissue Mg <sup>†</sup> concentration (%)	0.09	0.12	0.10
Total aboveground dry matter (Mg ha <sup>-1</sup> )	18.1	21.5	19.9
Total aboveground tissue P uptake (kg ha <sup>-1</sup> )	30.5	50.9	39.2
Belowground tissue P concentration (%)	0.05	0.12	0.08
Belowground tissue N concentration (%)	0.43	0.77	0.57
Belowground tissue Mg concentration (%)	0.12	0.21	0.16

<sup>†</sup> Nitrogen (N) and magnesium (Mg)

Table 5. Summary of corn properties that differed between years (2019 and 2020), averaged across fertilizer-phosphorus (P) sources, in a P-deficient, silt-loam soil in eastern Arkansas.

Plant Properties	Year	
	2019	2020
Stalk + leaves tissue N <sup>††</sup> concentration (%)	0.4 b <sup>†</sup>	1.3 a
Stalk + leaves tissue Mg <sup>††</sup> concentration (%)	0.3 b	0.5 a
Stalk + leaves tissue P uptake (kg ha <sup>-1</sup> )	6 b	11 a
Stalk + leaves tissue N uptake (kg ha <sup>-1</sup> )	34 b	96 a
Stalk + leaves tissue Mg uptake (kg ha <sup>-1</sup> )	25 b	40 a
Cob + husk dry matter (kg ha <sup>-1</sup> )	2213 a	1953 b
Cob + husk tissue N concentration (%)	0.4 b	0.8 a
Cob + husk tissue Mg concentration (%)	0.06 b	0.10 a
Cob + husk tissue P uptake (kg ha <sup>-1</sup> )	1.1 b	1.6 a
Cob + husk tissue N uptake (kg ha <sup>-1</sup> )	9 b	16 a
Cob + husk tissue Mg uptake (kg ha <sup>-1</sup> )	1.4 b	1.9 a
Total aboveground tissue N uptake (kg ha <sup>-1</sup> )	153 b	257 a
Total aboveground tissue Mg uptake (kg ha <sup>-1</sup> )	36 b	54 a
Yield (kg ha <sup>-1</sup> )	11372 a	10265 b

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

<sup>††</sup> Nitrogen (N) and magnesium (Mg)

Table 6. Summary of corn total aboveground N (TANU) uptake, kernel dry matter (KDM), kernel tissue P (KPU) and Mg (KMgU) uptake, and grain yield among fertilizer-P sources, averaged across years, in a P-deficient, silt-loam soil in eastern Arkansas.

Fertilizer-P Source <sup>†</sup>	TANU	KDM	KPU	KMgU	Yield
	kg ha <sup>-1</sup>				
ECST	228 a <sup>††</sup>	11530 a	35 a	12.8 a	12940 a
CPST	187 bc	9657 bc	28 b	9.9 b	10344 bc
DAP	179 c	9246 c	26 b	9.5 b	10165 c
MAP	205 ab	10556 ab	30 ab	10.9 ab	10782 bc
RP	205 ab	10054 bc	27 b	10.0 b	10251 bc
TSP	187 bc	10096 bc	29 b	10.3 b	11128 b
UC	201 abc	10313 abc	29 b	10.9 ab	10270 bc

<sup>†</sup> Electrochemically precipitated struvite (ECST), chemically precipitated struvite (CPST), diammonium phosphate (DAP), monoammonium phosphate (MAP), rock phosphate (RP), triple superphosphate (TSP), and unamended control (UC)

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

Table 7. Summary of corn kernel tissue nitrogen (N) uptake among fertilizer-phosphorus (P)-source-year combinations in a P-deficient, silt-loam soil in eastern Arkansas.

<b>Fertilizer-P Source</b>	<b>Kernel N Uptake (kg ha<sup>-1</sup>)</b>	
	<b>2019</b>	<b>2020</b>
Electrochemically precipitated struvite	131 bcde <sup>†</sup>	170 a
Chemically precipitated struvite	102 f	139 bcd
Diammonium phosphate	103 ef	122 def
Monoammonium phosphate	110 def	155 abc
Rock phosphate	97 f	166 ab
Triple superphosphate	103 ef	139 bcd
Unamended control	124 cde	132 bcde

<sup>†</sup> Means across both columns with different letters are different at  $P < 0.05$

Table 8. Summary of the effects of fertilizer-phosphorus (P) source on the change in soil properties in the top 10 cm for the corn and soybean studies after one year and after two growing seasons in a phosphorus (P)-deficient, silt-loam soil in eastern Arkansas.

Soil Properties	Change After One Year		Change After Two Growing Seasons	
	Corn	Soybean	Corn	Soybean
	<i>P</i>			
pH	0.42	0.47	<b>0.01<sup>†</sup></b>	<b>0.03</b>
Electrical conductivity	0.82	0.38	0.14	0.98
Phosphorus	0.40	0.67	<b>0.02</b>	<b>&lt; 0.01</b>
Potassium	0.88	0.66	0.82	0.78
Calcium	0.60	0.91	0.91	0.85
Magnesium	0.45	0.95	0.85	0.58
Sulfur	0.06	0.51	0.06	0.69
Sodium	0.65	0.67	0.30	0.81
Iron	0.66	0.39	0.46	0.79
Manganese	0.84	0.53	0.63	0.80
Zinc	0.86	0.57	0.26	0.71
Copper	0.66	0.27	0.81	0.61
Total nitrogen	0.76	0.54	-	-
Total carbon	0.95	0.67	-	-

<sup>†</sup> Bolded values were considered significant at  $P < 0.05$



Table 9. Summary of the effects of fertilizer-phosphorus (P) source on the two-year cumulative corn properties in a P-deficient, silt-loam soil in eastern Arkansas.

Plant Property	Source
	— P —
Stalk + leaves	
Dry matter	0.63
P uptake	0.63
N <sup>†</sup> uptake	0.55
Mg <sup>†</sup> uptake	0.72
Cob + husk	
Dry matter	0.19
P uptake	0.88
N uptake	0.16
Mg uptake	0.15
Kernel	
Dry matter	0.15
P uptake	0.13
N uptake	<b>0.03<sup>††</sup></b>
Mg uptake	0.10
Total aboveground	
Dry matter	0.29
P uptake	0.38
N uptake	0.07
Mg uptake	0.42
Yield	<b>&lt; 0.01</b>

<sup>†</sup> Nitrogen (N) and magnesium (Mg)

<sup>††</sup> Bolded values were considered significant at  $P < 0.05$

Table 10. Summary of the two-year cumulative corn properties that were unaffected ( $P > 0.05$ ) by fertilizer-phosphorus (P) source or year in a P-deficient, silt-loam soil in eastern Arkansas.

<b>Plant Property</b>	<b>Minimum</b>	<b>Maximum</b>	<b>Mean</b>
Stalk + leaves dry matter (Mg ha <sup>-1</sup> )	14.6	16.4	15.3
Stalk + leaves P uptake (kg ha <sup>-1</sup> )	15	19	17
Stalk + leaves N <sup>†</sup> uptake (kg ha <sup>-1</sup> )	119	142	131
Stalk + leaves Mg <sup>†</sup> uptake (kg ha <sup>-1</sup> )	60	71	66
Cob + husk dry matter (Mg ha <sup>-1</sup> )	3.9	4.9	4.2
Cob + husk P uptake (kg ha <sup>-1</sup> )	2.5	2.9	2.7
Cob + husk N uptake (kg ha <sup>-1</sup> )	22	29	25
Cob + husk Mg uptake (kg ha <sup>-1</sup> )	2.8	3.9	3.3
Kernel dry matter (Mg ha <sup>-1</sup> )	18.5	23.1	20.5
Kernel P uptake (kg ha <sup>-1</sup> )	53	71	59
Kernel Mg uptake (kg ha <sup>-1</sup> )	19	26	22
Total aboveground dry matter (Mg ha <sup>-1</sup> )	37.0	42.6	40.0
Total aboveground P uptake (kg ha <sup>-1</sup> )	72.6	89.5	78.9
Total aboveground N uptake (kg ha <sup>-1</sup> )	369	470	413
Total aboveground Mg uptake (kg ha <sup>-1</sup> )	82	101	90

<sup>†</sup> Nitrogen (N) and magnesium (Mg)

Table 11. Summary of the two-year cumulative corn kernel nitrogen (N) uptake and yield among fertilizer-phosphorus (P) sources, averaged across years, in a P-deficient, silt-loam soil in eastern Arkansas.

Fertilizer-P Source	Kernel N	
	Uptake	Yield
	—— kg ha <sup>-1</sup> ——	
Electrochemically precipitated struvite	301 a <sup>†</sup>	25899 a
Chemically precipitated struvite	243 bc	20800 b
Diammonium phosphate	226 c	20367 b
Monoammonium phosphate	267 ab	21579 b
Rock phosphate	264 ab	20510 b
Triple superphosphate	242 bc	22285 b
Unamended control	257 bc	20609 b

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

Table 12. Summary of the effects of fertilizer-phosphorus (P) source, year, and their interaction on soybean properties in a P-deficient, silt-loam soil in eastern Arkansas.

Plant Property	Source of Variation		
	Source	Year	Source x Year
	<i>P</i>		
Aboveground			
Dry matter	0.15	0.87	0.89
P concentration	< <b>0.01</b> <sup>†</sup>	0.06	0.70
N <sup>††</sup> concentration	0.30	0.06	0.36
Mg <sup>††</sup> concentration	0.89	<b>0.01</b>	0.46
P uptake	< <b>0.01</b>	< <b>0.01</b>	0.81
N uptake	<b>0.02</b>	< <b>0.01</b>	0.39
Mg uptake	0.15	<b>0.02</b>	0.53
Seed			
P concentration	0.37	< <b>0.01</b>	0.63
N concentration	0.62	< <b>0.01</b>	0.20
Mg concentration	0.98	0.96	0.33
P uptake	0.13	< 0.01	<b>0.03</b>
N uptake	0.46	< 0.01	<b>0.03</b>
Mg uptake	0.38	< 0.01	<b>0.03</b>
Belowground			
P concentration	0.30	< <b>0.01</b>	0.73
N concentration	0.67	0.06	0.81
Mg concentration	0.91	<b>0.02</b>	0.70
Yield	0.39	< 0.01	<b>0.03</b>

<sup>†</sup> Bolded values were considered significant at  $P < 0.05$

<sup>††</sup> Nitrogen (N) and magnesium (Mg)

Table 13. Summary of soybean properties were unaffected ( $P > 0.05$ ) by fertilizer-phosphorus (P) source or year in a P-deficient, silt-loam soil in eastern Arkansas.

<b>Plant Property</b>	<b>Minimum</b>	<b>Maximum</b>	<b>Mean</b>
Aboveground dry matter ( $\text{Mg ha}^{-1}$ )	7.5	8.7	8.2
Aboveground tissue N <sup>†</sup> concentration (%)	2.6	3.8	3.2
Seed Mg <sup>†</sup> concentration (%)	0.32	0.34	0.33
Belowground tissue N concentration (%)	0.7	1.4	1.0

<sup>†</sup> Nitrogen (N) and magnesium (Mg)

Table 14. Summary of soybean aboveground tissue phosphorus (P) concentration (APC) and P (APU) and N (ANU) uptake among fertilizer-P sources, averaged across years, in a P-deficient, silt-loam soil in eastern Arkansas.

<b>Fertilizer-P Source</b>	<b>APC</b>	<b>APU</b>	<b>ANU</b>
	— % —	— $\text{kg ha}^{-1}$ —	
Electrochemically precipitated struvite	0.33 a <sup>†</sup>	28.4 a	277 a
Chemically precipitated struvite	0.32 ab	26.7 ab	276 a
Diammonium phosphate	0.30 bcd	24.3 bc	270 ab
Monoammonium phosphate	0.29 cd	23.6 cd	244 bc
Rock phosphate	0.28 d	21.4 e	239 c
Triple superphosphate	0.31 abc	25.9 abc	268 abc
Unamended control	0.29 cd	22.0 de	240 c

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

Table 15. Summary of soybean properties that differed between years (2019 and 2020), averaged across fertilizer-phosphorus (P) sources, in a P-deficient, silt-loam soil in eastern Arkansas.

<b>Plant Properties</b>	<b>Year</b>	
	<b>2019</b>	<b>2020</b>
Aboveground tissue Mg <sup>†</sup> concentration (%)	0.45 b <sup>††</sup>	0.52 a
Aboveground tissue P uptake (kg ha <sup>-1</sup> )	28.2 a	21.3 b
Aboveground tissue N <sup>†</sup> uptake (kg ha <sup>-1</sup> )	294 a	228 b
Aboveground tissue Mg uptake (kg ha <sup>-1</sup> )	37 b	42 a
Seed P concentration (%)	0.64 a	0.59 b
Seed N concentration (%)	6.4 b	6.6 a
Belowground tissue P concentration (%)	0.06 b	0.20 a
Belowground tissue Mg concentration (%)	0.14 b	0.40 a

<sup>†</sup> Magnesium (Mg) and nitrogen (N)

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

Table 16. Summary of soybean yield, seed phosphorus (P), nitrogen (N), and magnesium (Mg) uptake among fertilizer-P-source-year combinations in a P-deficient, silt-loam soil in eastern Arkansas.

Fertilizer-P Source <sup>†</sup>	Year	Yield	Seed P	Seed N	Seed Mg
			Uptake	Uptake	Uptake
			kg ha <sup>-1</sup>		
ECST	2019	4148 a <sup>††</sup>	25.2 a	254 a	13.2 a
CPST	2019	3761 ab	23.0 ab	225 ab	12.0 a
DAP	2019	3721 ab	22.1 ab	224 ab	12.0 a
MAP	2019	3807 ab	22.8 ab	229 ab	11.9 a
RP	2019	3735 ab	21.8 ab	224 ab	11.9 a
TSP	2019	3776 ab	23.0 ab	229 ab	12.3 a
UC	2019	3559 b	21.0 b	215 bc	11.6 a
ECST	2020	2168 d	11.9 e	129 e	6.6 d
CPST	2020	2756 c	15.5 c	167 cd	8.7 b
DAP	2020	2650 c	14.3 cd	158 d	8.0 bc
MAP	2020	2642 c	14.9 c	161 d	8.5 bc
RP	2020	2722 c	14.8 c	168 cd	8.7 b
TSP	2020	2690 c	14.9 c	163 d	8.3 bc
UC	2020	2386 cd	12.4 de	143 de	7.3 cd

<sup>†</sup> Electrochemically precipitated struvite (ECST), chemically precipitated struvite (CPST), diammonium phosphate (DAP), monoammonium phosphate (MAP), rock phosphate (RP), triple superphosphate (TSP), and unamended control (UC)

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

Table 17. Summary of the effects of fertilizer-phosphorus (P) source on the two-year cumulative soybean properties in a P-deficient, silt-loam soil in eastern Arkansas.

Plant Property	Source
	— <i>P</i> —
Aboveground	
Dry matter	0.13
P uptake	< <b>0.01</b> <sup>††</sup>
N <sup>†</sup> uptake	<b>0.02</b>
Mg <sup>†</sup> uptake	0.15
Seed	
P uptake	0.46
N uptake	0.83
Mg uptake	0.81
Yield	0.79

<sup>†</sup> Nitrogen (N) and magnesium (Mg)

<sup>††</sup> Bolded values were considered significant at  $P < 0.05$

Table 18. Summary of the two-year cumulative soybean properties that were unaffected ( $P > 0.05$ ) by fertilizer-phosphorus (P) source or year in a P-deficient, silt-loam soil in eastern Arkansas.

Plant Property	Minimum	Maximum	Mean
Aboveground dry matter (Mg ha <sup>-1</sup> )	15.4	17.2	16.3
Aboveground Mg <sup>†</sup> uptake (kg ha <sup>-1</sup> )	74	84	79
Seed P uptake (kg ha <sup>-1</sup> )	33	39	37
Seed N <sup>†</sup> uptake (kg ha <sup>-1</sup> )	358	394	385
Seed Mg uptake (kg ha <sup>-1</sup> )	18.9	20.8	20.2
Yield (Mg ha <sup>-1</sup> )	6.0	6.6	6.4

<sup>†</sup> Magnesium (Mg) and nitrogen (N)



Table 19. Summary of the two-year cumulative soybean aboveground phosphorus (P) and nitrogen (N) uptake among fertilizer-P sources, averaged across years, in a P-deficient, silt-loam soil in eastern Arkansas.

Fertilizer-P Source	Aboveground P	Aboveground N
	Uptake	Uptake
	————— kg ha <sup>-1</sup> —————	
Electrochemically precipitated struvite	58 a <sup>†</sup>	566 a
Chemically precipitated struvite	54 ab	558 a
Diammonium phosphate	49 cd	542 ab
Monoammonium phosphate	48 de	491 bc
Rock phosphate	43 f	483 c
Triple superphosphate	52 bc	539 abc
Unamended control	44 ef	484 bc

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



Fig. 1. Aerial view of the approximate plot arrangement for the corn and soybean studies within the study area at the Lon Mann Cotton Branch Experiment Station near Marianna, AR. North is towards the top of the image. Individual plot dimensions were 6.1-m long by 3.1-m wide, except for the electrochemically precipitated struvite plots which were 1.5-m long by 1.5-m wide. Rectangles marking study areas are not drawn to scale.

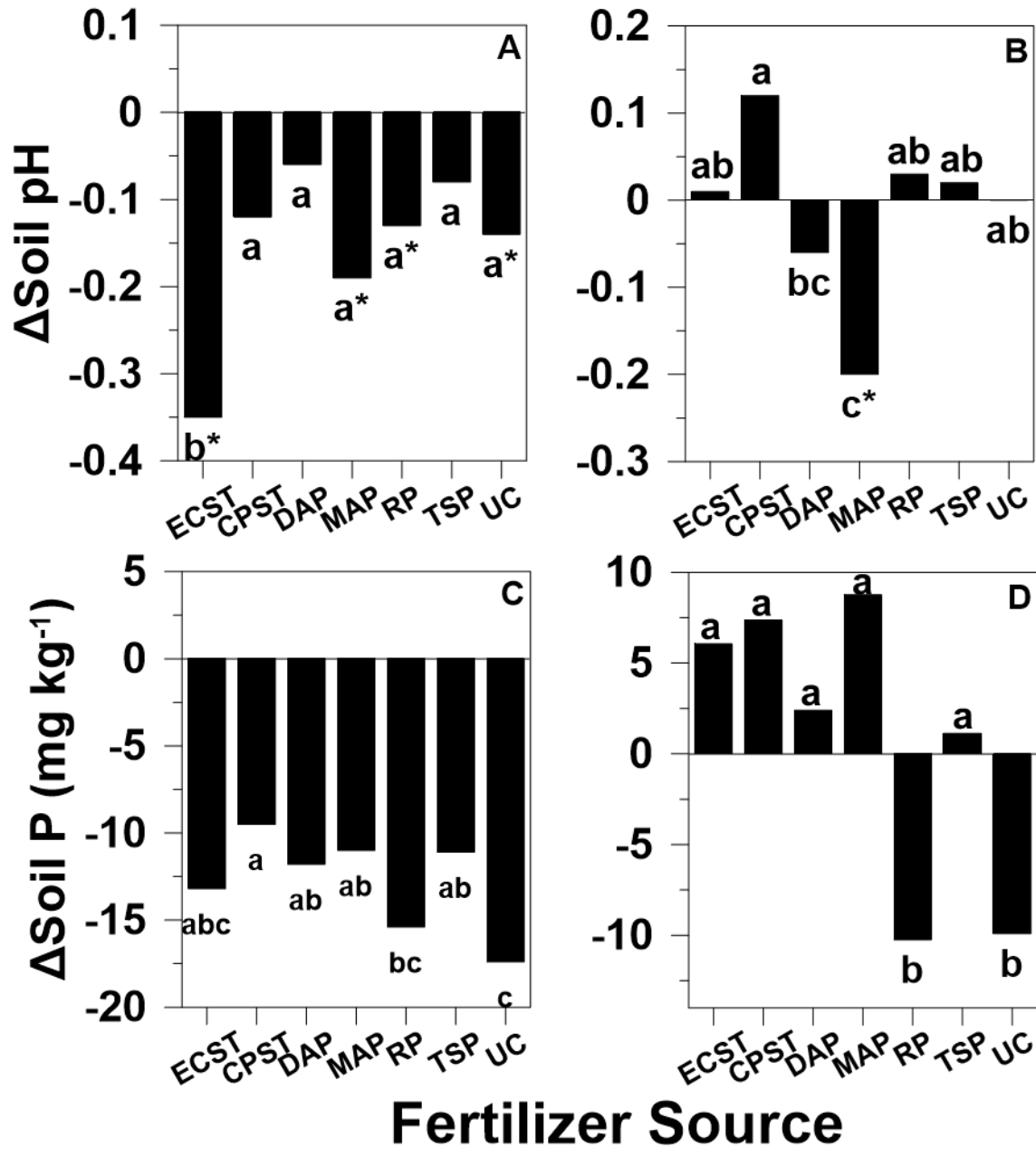


Fig. 2. Summary of the change in soil-test pH for the corn study (A) and the soybean study (B) and the change in extractable soil phosphorus (P) concentration for the corn study (C) and the soybean study (D) in the top 10 cm among fertilizer-P sources after two growing seasons on a P-deficient, silt-loam soil in eastern Arkansas. An asterisk (\*) indicates mean value is different than zero ( $P < 0.05$ ).

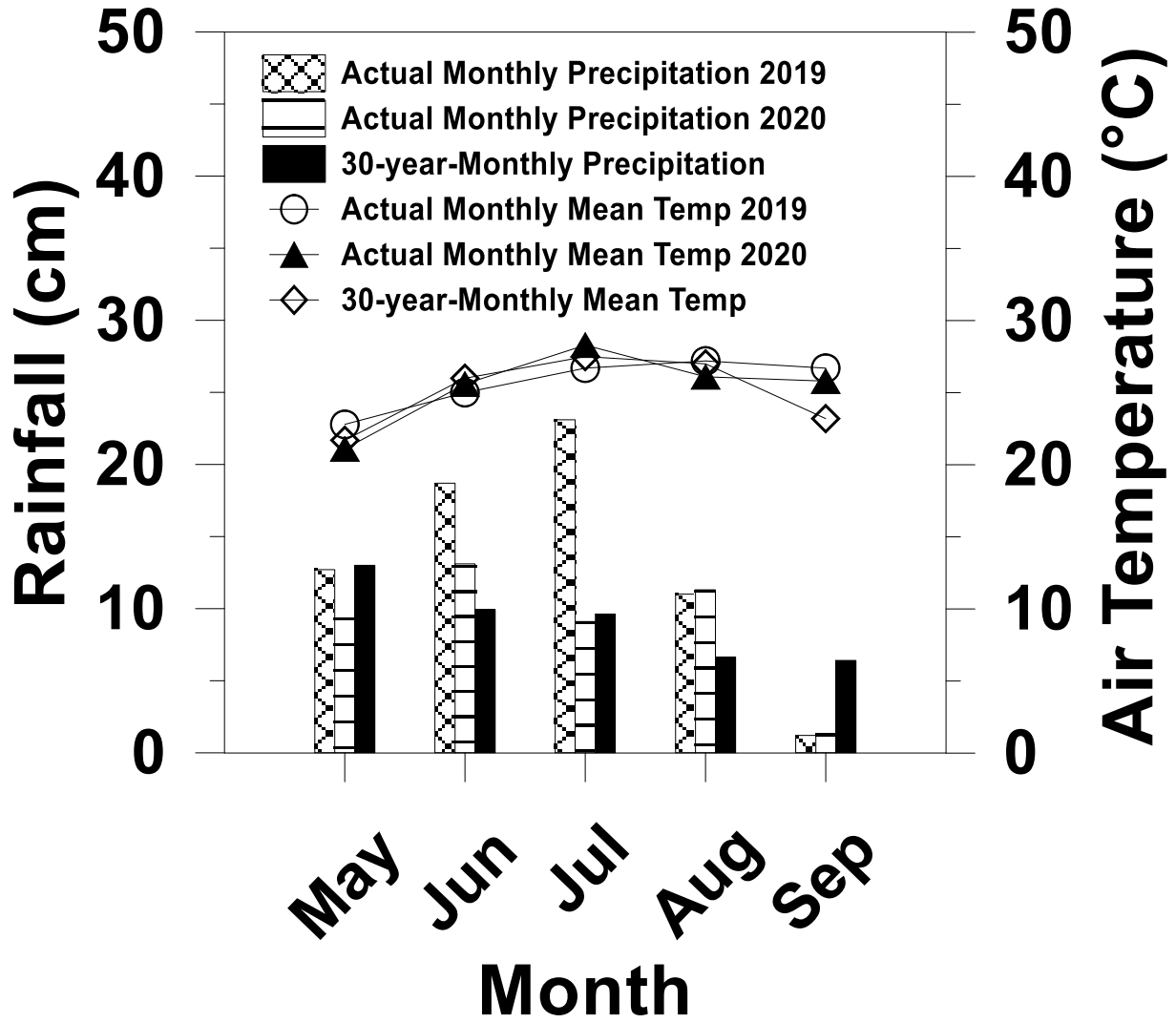


Fig. 3. Summary of the 30-year (1981 to 2010) mean monthly rainfall and air temperature and actual monthly rainfall and air temperature during the five-month corn and soybean growing seasons in 2019 and 2020 at the Cotton Branch Experiment Station near Marianna, AR.

## **Overall Conclusions**

Previous research has demonstrated struvite's agronomic benefits in several ornamental and vegetable crops and a few row crops. However, to our knowledge, no field studies to date have examined struvite effects in a wheat-soybean, double-crop production system, flood-irrigated rice, or furrow-irrigated corn and soybean relative to other commercially available, commonly used fertilizer-P sources in the mid-southern United States.

Results from the 2-year field study in a wheat-soybean, double-crop production system on a silt-loam soil in eastern Arkansas showed that CPST provided similar soybean and wheat yields and aboveground DM to TSP, but neither CPST nor TSP significantly increased yield compared to the unamended control meaning that some other factor, or combination of factors, limited soybean and wheat growth. In addition, CPST provided similar wheat stem P and soybean belowground Mg concentration, lower N and Mg concentration in wheat, greater Mg concentration in wheat, and greater Mg concentration in soybean compared to TSP. Despite some lower tissue concentrations from CPST compared to TSP, differences were relatively small and likely had no major negative effects on soybean or wheat growth and productivity. Results also showed soybean seed P and Mg concentrations differed between irrigation treatments, which emphasized the significant role of the presence of sufficient water for soil nutrient distribution and plant nutrient uptake during crop production. This study demonstrated that wastewater-recovered struvite has the potential to serve as an alternative fertilizer-P source in upland, row-crop agricultural production.

As hypothesized, results from the flood-irrigated rice demonstrated similar rice aboveground DM, above- and belowground tissue and grain P, N, and C concentrations, aboveground and grain tissue P uptake, and aboveground tissue N and C contents from wastewater-recovered struvite sources (i.e., ECST and CPST) compared to other common,

commercially available fertilizer-P sources (i.e., TSP, MAP, DAP, and RP). However, the hypothesis was only partially supported because grain yield and grain tissue N and C contents differed among ECST, CPST, TSP, MAP, DAP, and RP treatments.

Results from flood-irrigated rice did not support the hypothesis that aboveground tissue and grain Mg concentrations and uptake and belowground tissue Mg concentration would be greater for the struvite materials (ECST and CPST) treatment due to greater initial Mg concentrations. Similarly, results did not support the hypothesis that aboveground tissue P uptake would be greater from the 44 kg TSP-P ha<sup>-1</sup> rate compared to fertilizer rates of 22 and 66 kg TSP-P ha<sup>-1</sup>. Despite a large P adsorption capacity with large initial extractable Ca and alkaline soil pH, comparable results clearly showed that wastewater-recovered struvite, from either chemical and/or electrochemical precipitation techniques, and other commercial fertilizer-P source further support evidence from previous studies that have shown struvite to be a potentially viable alternative to traditional RP-derived fertilizer-P source for flood-irrigated rice.

In the corn study, the hypothesis that corn total aboveground DM and yield, total aboveground tissue P and N uptake, and belowground P and N concentrations from wastewater-recovered struvite sources (i.e., ECST and CPST) would be similar to other common, commercially available fertilizer-P sources (i.e., TSP, MAP, DAP, and RP) was only partially supported because total aboveground tissue N uptake and yield differed among ECST, CPST, TSP, MAP, DAP, and RP treatments. Similarly, results did not support the hypothesis that corn total aboveground tissue Mg uptake and belowground tissue Mg concentrations would be greater for the struvite materials (ECST and CPST) due to greater initial Mg concentrations. In addition, results partially supported the hypothesis that corn grown in year two would have greater total aboveground DM and tissue P uptake, yield, total aboveground tissue P, N, and Mg uptake, and

belowground tissue P, N, and Mg concentrations compared to that in year one due to a carry-over effect from year-one fertilization, where corn yield from year one (2019) was greater than that from year two (2020) and total aboveground DM and belowground tissue P, N, and Mg concentrations did not differ between years.

In the soybean study, the hypothesis that soybean aboveground DM and yield, above- and belowground tissue and seed P and N concentrations, and aboveground and seed P and N uptake from wastewater-recovered struvite sources (i.e., ECST and CPST) would be similar to other common, commercially available fertilizer-P sources (i.e., TSP, MAP, DAP, and RP) was only partially supported because aboveground tissue P concentration and P and N uptake differed among ECST, CPST, TSP, MAP, DAP, and RP treatments.

Results from soybean study partially supported the hypothesis that soybean aboveground tissue and seed Mg concentration and uptake and belowground tissue Mg concentrations would be greater for the struvite materials (ECST and CPST) treatment because above- and belowground tissue and seed Mg concentrations and aboveground Mg uptake did not differ among ECST, CPST, TSP, MAP, DAP, and RP treatments. Similarly, results partially supported the hypothesis that soybean grown in year two would also have greater aboveground DM, yield, above- and belowground tissue and seed P, N, and Mg concentrations, and aboveground tissue and seed P, N, and Mg uptake compared to that in year one, where soybean aboveground tissue P and N uptake and seed P concentration from year one (2019) was greater than that from year two (2020) and aboveground DM and tissue N concentration, belowground tissue N concentration, and seed Mg concentration did not differ between years.

Although the soil had a large P-adsorption capacity from the large initial extractable Ca concentration and slightly alkaline soil pH, results clearly showed that wastewater-recovered



struvite, from either chemical or electrochemical precipitation techniques, has potential to be a viable, alternative fertilizer-P source for corn and soybean. All corn properties affected by fertilizer-P source (i.e., kernel DM, tissue P and Mg uptake, total aboveground tissue N and Mg uptake, and yield in 2019 and 2020, and the 2-yr cumulative kernel tissue N uptake and yield) had at least similar (from CPST) and even greater (from ECST) response than several other commonly used fertilizer-P sources (i.e., TSP, DAP, and RP). In addition, all soybean properties affected by fertilizer-P source (i.e., aboveground tissue P concentration and uptake and N uptake in 2019 and 2020 and the 2-year cumulative aboveground tissue P and N uptake) had similar response among ECST, CPST, and TSP, and in some cases, greater response from both struvite treatments than from MAP, DAP, or RP. Results from corn and soybean study overall provide valuable information about the response of corn and soybean to crystalline ECST and pelletized CPST compared with other commercially available, commonly used fertilizer-P source on a P-deficient silt-loam soil in eastern Arkansas.

Although there were some differences in plant and soil response, it appears that there is ample evidence to suggest that both chemically and electrochemically precipitated struvite are viable, alternative fertilizer-P sources for several row crops. Considering the ECST used in this study was produced from synthetic wastewater, more in-depth research is still required to evaluate the behavior and effectiveness of ECST produced from natural wastewater (i.e., either municipal or an animal agriculture source) as a fertilizer-P source in various soil conditions and row crops under field conditions.

The importance of wastewater-recovered struvite has not only extended beyond serving as a potential solution to environmental issues, such as eutrophication, but also could be a potential alternative for a sustainable source of P for future food security. Rice, soybean, corn,

and wheat are major food crops directly or indirectly consumed by a large part of human population. Struvite can serve as a nutrient source to the soil system to enrich the soil with P, N, and Mg, which can be taken up by plants or microbes, thus can promote soil health.

Although human activities have been known to introduce P to freshwater sources at rates that exceed natural levels, such behavior could gradually change as agronomic benefits from struvite application become better known, thus could encourage and stimulate many societies across the globe to ensure the recovery of P from wastewaters as the mineral struvite.

Additionally, recovery of struvite could improve cost, labor, and treatment efficiency in wastewater treatment plants, thus providing solution to the historic struvite scaling in wastewater treatment pipes. Clean water is a crucial resource for drinking, fishing, irrigation, recreation, and supporting many biologically diverse and threatened aquatic species. Recovery of struvite could potentially be an integral part of humanity, providing food, energy, and ensuring clean water resources for human consumption. Recovery of struvite could also be beneficial to humanity through the increased employment opportunities, both in the creation process and evaluation of struvite as a fertilizer-P source in the field and greenhouse.