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Corn response to wastewater-recycled phosphorus fertilizers

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Cover Page Footnote

Shane R. Ylagan is a December 2020 honors program graduate with a major in Environmental, Soil, and Water Science and a minor in sustainability. Kristofor R. Brye, the faculty co-mentor, is a Professor in the Department of Crop, Soil, and Environmental Sciences.

Corn response to wastewater-recycled phosphorus fertilizers

Meet the Student-Author

Shane Ylagan

I am from The Woodlands, Texas, and I graduated from The Woodlands High School in 2017. I will graduate from the Dale Bumpers College of Agriculture, Food, and Life Science with honors and a B.S. in Environmental, Soil, and Water Science with a minor in Sustainability. This project was funded by the University of Arkansas Honors College with an Honors College research grant not only in order to conduct this study but also to present the results at the 2019 Arkansas Water Resources Center Annual Water Conference.

It was not until my sophomore year when I took Introduction to Soil Science and Soil Profile Description with Dr. David Miller and Dr. Kristofor Brye, where I found my passion for Soil Science. I have been a part of the University of Arkansas soil profile description team ever since my sophomore year, where the team placed first in the 2018 Region IV Collegiate Soils Contest. Additionally, I have held two hourly research positions in the Crop, Soil, and Environmental Sciences Department dealing with soil incubation and phytoremediation studies.

I would like to thank my mentor and professor, Dr. Kristofor Brye, whose help and teachings have been not only been essential in the success of this research, but also in my success as an individual. I would also like to state my appreciation to Dr. David Miller, Dr. Lisa Wood, and Ryder Anderson for their continuous help and judgment.

Shane overturning and mixing the field soil to air-dry at the University of Arkansas System Division of Agriculture's Rosen Alternative Pest Control Center.

Research at a Glance

- How soil or plants respond to electrochemically precipitated struvite (ECST) has not been evaluated.
- Data are needed to determine how ECST compares to common phosphorus (P) and other struvite fertilizers.
- ECST had at least similar, if not larger, plant and soil responses in corn to other common P and struvite fertilizers.

Corn response to wastewater-recycled phosphorus fertilizers

Shane R. Ylagan and Kristofor R. Brye†*

Abstract

The ability to recycle phosphorus (P) from wastewaters could provide a sustainable, continuous source of P that might also help protect surface water quality from P enrichment. The mineral struvite ($MgNH_4PO_4 \cdot 6H_2O$) is an understudied material that can be created from P- and nitrogen (N)-containing wastewater and has been shown to have agricultural fertilizer value. The objective of this study was to evaluate the effects of electrochemically precipitated struvite (ECST), chemically precipitated struvite (Crystal Green; CG), diammonium phosphate (DAP), monoammonium phosphate (MAP), rock phosphate (RP), and triple superphosphate (TSP) on corn (*Zea mays*) response in a greenhouse pot study. The effects of fertilizer treatment on select plant properties were evaluated. Corn plant properties and elemental tissue concentrations differed ($P < 0.05$) among fertilizer amendments. Belowground dry matter from ECST was 1.9 times greater than that from CG, TSP, DAP, and the No P/+N, and No P/-N control treatments. Corn cob-plus-husk tissue P concentration from ECST was similar to that from MAP and DAP and was 1.2 times larger than that from CG. Corn stem-plus-leaves tissue P concentration from ECST differed from that from all other treatments and was 1.8 times greater than that from the No P/+N control. Results generated from this study not only provide information on the new, thus understudied, electrochemically precipitated struvite material, but also further demonstrate why more research should be conducted on the implementation of struvite as an alternative fertilizer-P source and struvite's potential impact on sustainable food production and the preservation of water resources.

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† Kristofor R. Brye, the faculty co-mentor, is a Professor in the Department of Crop, Soil, and Environmental Sciences.

Introduction

Phosphorus (P) has been historically considered a nonrenewable resource that is a crucial nutrient for all life and sustains worldwide food production (Ashley et al., 2011). Phosphorus is obtained by mining phosphate-containing rock, also called rock phosphate (RP), where peak production has the potential to be reached in the next 50 years (Cordell et al., 2009; Filippelli, 2011). The combination of increasing preference for meat diets, global population growth, P demand, and P-fertilizer price with diminished quantity and quality of RP sources has the potential to severely affect the world's food supply and the world's economic, political, and social relations (Jarvie et al., 2015; Talboys et al., 2015).

Another complicating factor is that P has become a contaminant in many natural surface water sources from excessive fertilizer application, agricultural runoff containing excessive amounts of soluble and sediment-bound P from agriculturally dominated watersheds, and also from wastewater treatment plant (WWTP) effluent that contains excessive quantities of nutrients, namely P. Excess P in surface waters has been linked to eutrophication and the creation of hypoxic zones in freshwater and coastal marine environments (Hallas et al., 2019; Liu et al., 2012). The accelerating decline of RP reserves and the degradation of aquatic ecosystem health are both daunting issues that are only going to continue to grow, but there could be a solution.

The mineral struvite ($MgNH_4PO_4 \cdot 6H_2O$) is currently being studied as a potential P fertilizer. Struvite is an efficient, slow-release P source that can be recovered as a crystalline precipitate through recycling P from a variety of wastewater sources (Rahman et al., 2014). Consequently, struvite is an example of a wastewater-recycled P fertilizer that has the potential to be multi-beneficial. Crop producers who apply struvite as a fertilizer P source have the potential to not only maintain, or even increase, optimal crop yields while reducing fertilizer application rates, but also decrease the quantity of P that is lost in runoff due to struvite's slow-release characteristic (Massey et al., 2009; Talboys et al., 2015).

In order to provide more information on the possible benefits of struvite as a fertilizer-P source, a greenhouse potted-plant experiment was conducted. The objective of the study was to assess corn response to P fertilization with two wastewater-recovered struvite sources (i.e., chemically precipitated and electrochemically precipitated) and to compare corn response to that produced by other commonly used P fertilizers in an agriculturally managed silt-loam soil. It was hypothesized that corn plants amended with either struvite source would have an equal or even greater response to P fertilization than the plants that were treated with the conventional P fertilizers.

Materials and Methods

Soil Collection, Processing, and Initial Characterization

The soil used in this greenhouse study was a Captina silt-loam (Typic Fragiudults; Soil Survey Staff, 2017) that was collected from a field (36°05'47"N 94°09'58"W) at the Milo J. Shult Agricultural Research and Extension Center in Fayetteville, Arkansas, that had been under cultivated soybean production for at least several years prior. Ten, 18.9-L buckets of soil were manually collected on 18 February 2019 from the top 10 to 15 cm, transported to a greenhouse, and air-dried.

Five random subsamples of soil were collected while air-drying, oven-dried at 70 °C for 48 hours, mechanically ground, and sieved through a 2-mm mesh screen prior to soil physical and chemical property determinations, including percent sand, silt, and clay; soil pH; electrical conductivity (EC); soil organic matter (SOM); total soil N and C; and water-soluble and Mehlich-3 extractable (i.e., P, K, Ca, Mg, S, Na, Fe, Mn, Zn, Cu, and B) concentrations.

Fertilizer Treatments

Eight treatments were evaluated in this study, which included: 1) an electrochemically precipitated struvite (ECST), 2) Crystal Green (CG), a chemically precipitated struvite, 3) triple superphosphate (TSP), 4) monoammonium phosphate (MAP), 5) diammonium phosphate (DAP), 6) rock phosphate (RP; Fig. 1), 7) an unamended control that did not have added P, but had added N (No P/+N), and 8) an unamended control without added P or N (No P/-N).

This study included two different types of struvite material. Crystal Green is a chemically precipitated struvite material that was produced from a large municipal wastewater treatment plant near Atlanta, Georgia, and is commercially produced and sold by Ostara Nutrient Recovery Technologies Inc. (Fig. 1). The second struvite source was produced by researchers in the Department of Chemical Engineering at the University of Arkansas, Fayetteville, via electrochemical precipitation from synthetically made wastewater (Fig. 1).

The initial Mehlich-3 soil-test-P concentration corresponded to a P_2O5 recommendation for corn of 84.1 kg P_2O_5/ha, which equated to 36.6 kg P/ha for a yield goal of 11 Mg/ha (Espinoza and Ross, 2008). Since each P fertilizer also had a different N concentration, the corresponding amount of N was added to all treatments (except the No P/-N control) in the form of urea (46% N) in order to match the N concentration of DAP (Table 1).

Pot Preparation

Plastic, 6,435 cm^3, injection-molded nursery containers (Item # CN-NCIM/600 series, Pro Cal, South Gate, Cali-

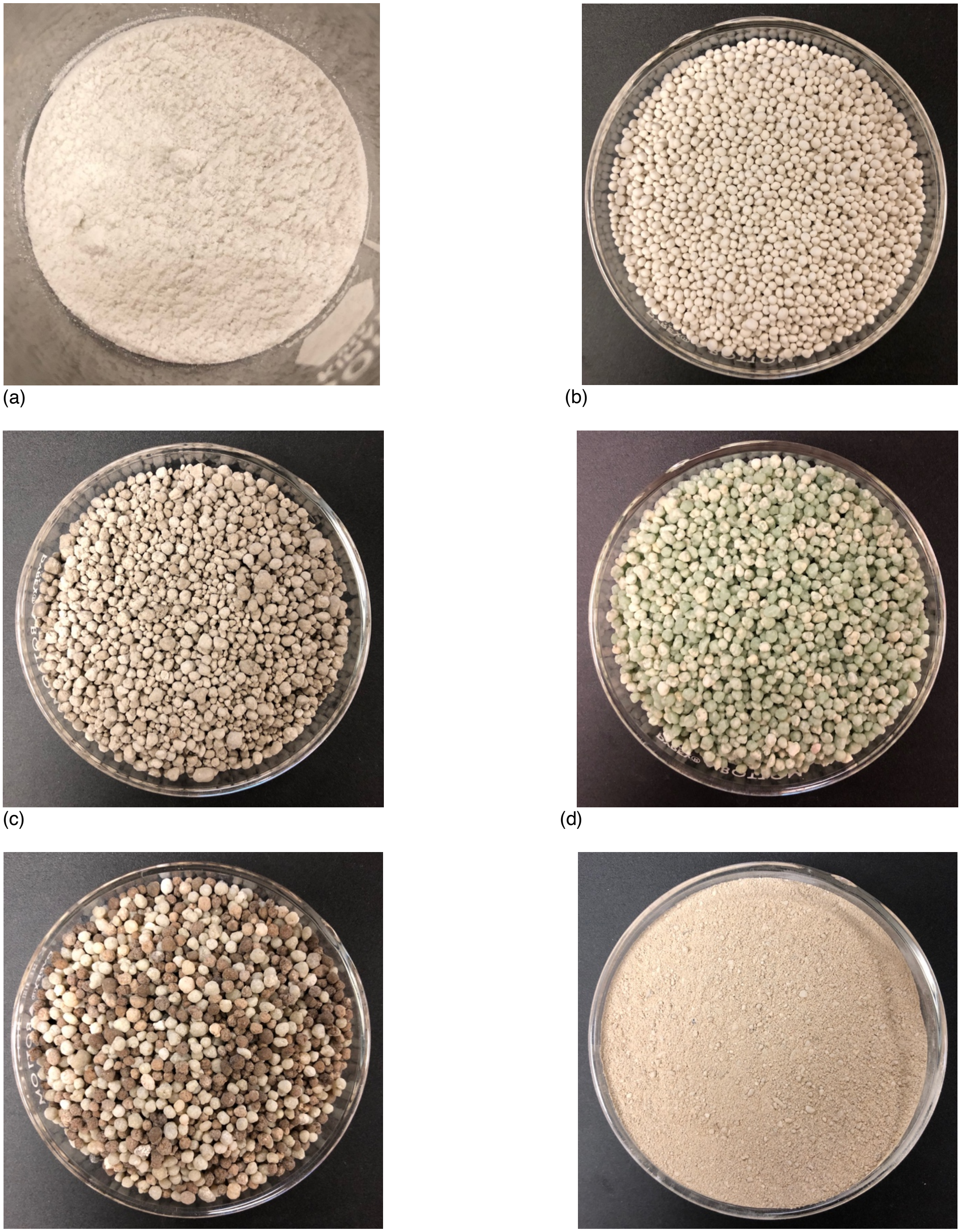

Fig. 1. The physical appearance of electrochemically precipitated struvite (a), Crystal Green struvite (b), triple superphosphate (c), monoammonium phosphate (d), diammonium phosphate (e), and rock phosphate (f).

fornia) that were 21.3 cm tall and 22.9 cm in diameter were used for this study. The pots were prepared by premixing air-dried soil and the fertilizers before the soil was added to the pots to simulate the common field practice of fertilizer incorporation by tillage.

In order to bring the air-dried soil in each pot to field moisture capacity (~26.1% v/v) initially, 684 mL of water was required. However, the target volumetric water content (VWC) range was chosen between 24% and 26% (v/v). The following day, three seeds were planted in all 24 corn pots, and the pots were randomized on a greenhouse bench. After about 10 days, the number of plants in each pot was cut back to one.

Pot Management

Three times a week, the VWC in the top 6 cm of soil in three randomly selected pots was measured using a soil moisture meter to assess the volume of water needed to be added to return the soil in the pot to the target VWC range. Furthermore, additional N in the form of urea (46% N) was applied to all of the corn P-fertilizer treatments (excluding the No P/-N control) on 5 June 2019, 40 days after planting, as the recommended mid-season N application at a field-equivalent rate of 266 kg N/ha for corn (Table 1; Espinoza and Ross, 2008).

Pot Deconstruction

The experiment was terminated on the 79th day, 15 July 2019 (Fig. 2). Each corn pot was manually deconstructivity sampled, separating the plant into belowground, cob-plus-husk, and stem-plus-leaves portions for dry matter and elemental tissue P concentration determinations. The separated plant portions from each pot were oven-dried at 66.6 °C for five days and then weighed for dry matter determinations. Subsamples were then taken from each plant tissue portion and were mechanically ground to 2 mm to determine tissue P concentrations by acid digestion (USEPA, 1996) followed by inductively coupled plasma optical emission spectrometry (ICAP-OES) (Soltanpour et al., 1996).

Statistical Analyses

Based on a completely randomized experimental design, a one-factor analysis of variance (ANOVA) was conducted with SAS 9.4 (SAS Institute, Inc., Cary, N.C.) using the PROC GLIMMIX procedure to evaluate the effect of the fertilizer treatment on plant response. Treatment means were separated by a least significant difference test at the alpha level of 0.05. Significance was judged at $P < 0.05$.

Results and Discussion

Plant Properties

All measured corn plant properties differed ($P < 0.05$) among P-fertilizer treatments (Table 2). Corn stem-plus-leaves dry matter was numerically largest from TSP, which did not differ from that in the DAP, ECST, RP, MAP, and No P/+N control treatments. Stem-plus-leaves dry matter from the No P/-N control treatment was numerically smallest among all treatments. Stem-plus-leaves dry matter from the two struvite treatments (ECST and CG) did not differ from each other, and both were similar to that from the RP, MAP, and No P/+N control treatments. The mean stem-plus-leaves dry matter from TSP and DAP, which did not differ, was 1.3 times larger than that from the No P/-N control treatment.

Corn cob-plus-husk dry matter was numerically largest from CG, which was similar to that in the ECST treatment, and both did not differ from that in the TSP, MAP, RP, DAP, and No P/+N control treatments (Table 2). Cob-plus-husk dry matter from the No P/-N control treatment was numerically smallest among all treatments. The cob-

Table 1. Summary of the fertilizer grade and nitrogen (N), phosphorus (P), and magnesium (Mg) concentrations of each fertilizer-nutrient source used in the greenhouse pot experiment (i.e., ECST, CG, TSP, MAP, DAP, RP, and urea).

		Nutrient Concentration (%)		
Fertilizer	Fertilizer Grade	N	P	Mg
ECST†	9-52-0	9.3	22.8	5.7
CG	6-27-0	5.7	11.7	8.3
TSP	0-41-0	0.0	18.2	0.6
MAP	11-48-0	11.0	20.9	1.5
DAP	18-42-0	18.1	18.3	0.7
RP	0-17-0	0.0	7.6	0.3
Urea	46-0-0	46.0	0.0	0.0

† Electrochemically precipitated struvite (ECST), Crystal Green (CG), triple superphosphate (TSP), monoammonium phosphate (MAP), diammonium phosphate (DAP), and rock phosphate (RP).

Fig. 2. The first repetition of corn fertilizer treatments at 2 (top), 6 (middle), and 11 (bottom) weeks after planting. Treatment order (left to right): 1) unamended control without added P or N (No P/-N), 2) unamended control that did not have added P, but had added N (No P/+N), 3) triple superphosphate (TSP), 4) monoammonium phosphate (MAP), 5) diammonium phosphate (DAP), 6) rock phosphate (RP), 7) Crystal Green (CG), and 8) electrochemically precipitated struvite (ECST).

plus-husk dry matter from the ECST, CG, TSP, MAP, DAP, RP, and the No P/+N control treatments, which did not differ, was 3.9 times larger than that from the No P/-N control treatment.

Belowground corn dry matter was numerically largest from ECST, which did not differ from that in the RP treatment (Table 2). Both ECST and RP were applied as powder forms, thus having larger surface areas to react with the soil and water compared to fertilizers in pellet forms, and root-excreted organic acids could have helped solubilize RP, which in turn, could have increased concentrations of plant-available P to increase dry matter production in the various plant parts (Oburger et al., 2011). Belowground corn dry matter was numerically smallest from the No P/-N control, which did not differ from that in the MAP, TSP, DAP, CG, and No P/+N control treatments. Belowground corn dry matter from both struvite treatments (ECST and CG) differed from each other, and CG did not differ from TSP, MAP, DAP, and both control treatments (No P/+N and No P/-N). Additionally, belowground corn dry matter from the ECST treatment was 2.0 times greater than that from CG, and ECST was also 1.9 times greater than that from the CG, TSP, DAP, and both control (No P/+N and No P/-N) treatments, which did not differ.

Tissue Properties

Corn tissue P concentrations (Table 3) differed ($P < 0.05$) among P-fertilizer treatments. Corn belowground tissue P concentration was numerically largest from CG and DAP, which did not differ from that in the TSP and MAP treatments. Belowground tissue P concentration was numerically smallest from the No P/-N control treatment, which was similar to that in the ECST, RP, and the No P/+N control treatments. Furthermore, corn belowground tissue P concentrations from CG was 1.4 times larger than that from the ECST treatment. Slower dissolution of the CG pellet material may have kept the P in the active root zone, whereas more rapid dissolution of the crystalline ECST material may have allowed P to move away from the active root zone in the pot and become somewhat less available to active roots. The mean belowground tissue P concentration from CG, TSP, MAP, and DAP, which did not differ, was 1.4 times greater than that from ECST, RP, and both control (No P/+N and No P/-N) treatments, which did not differ.

Corn cob-plus-husk tissue P concentration (Table 3) was numerically largest from ECST, which did not differ from that in the MAP, DAP, and the No P/-N control treatments. Cob-plus-husk tissue P concentration was numerically smallest from the No P/+N control, which was similar to the CG, TSP, and RP treatments. Cob-plus-husk tissue P concentrations from ECST was 1.2 times larger than that from CG. The ECST-P was derived from a synthetic rather than an actual wastewater, as was the CG-P. It is possible that the CG-P had additional associated compounds or complexes that rendered the P somewhat less mobile once in the plant than the relatively cleaner ECST-P. Cob-plus-husk tissue P concentration from CG did not differ from that in the TSP, MAP, DAP, RP, and the No P/+N and No P/-N control treatments. The cob-plus-husk tissue P concentration from ECST was 1.4 times larger than that from the No P/+N control treatment.

Table 2. Summary of the effects of fertilizer amendment on corn belowground, cob-plus-husk, and stem-plus-leaves tissue dry matter.

	Corn Tissue P Elemental Concentrations		
Treatment	Belowground Dry Matter	Cob-plus-Husk Dry Matter	Stem-Plus-Leaves Dry Matter
	----------	g	----------
ECST[†]	28.7 a[‡]	11.1 a	35.8 ab
CG	14.1 c	13.4 a	33.8 b
TSP	15.4 c	13.2 a	37.7 a
MAP	16.6 bc	12.4 a	35.5 ab
DAP	15.1 c	11.6 a	36.7 a
RP	24.9 ab	12.3 a	35.8 ab
No P/+N	15.3 c	11.9 a	35.4 ab
No P/-N	13.9 c	3.14 b	27.7 c
P-value	**0.03**[§]	**< 0.01**	**< 0.01**

[†] Electrochemically precipitated struvite (ECST), Crystal Green (CG), triple superphosphate (TSP), monoammonium phosphate (MAP), diammonium phosphate (DAP), rock phosphate (RP), unamended control that did not have added P, but had added N (No P/+N), and unamended control without added P or N (No P/-N).

[‡] Means in a column with different letters are different at $P < 0.05$.

[§] Bolded values are significant at $P < 0.05$.

Corn stem-plus-leaves tissue P concentration (Table 3) was numerically largest from ECST, which differed from all other treatments. Stem-plus-leaves tissue P concentration was numerically smallest from the No P/+N control, which differed from all other treatments. Stem-plus-leaves tissue P concentration from both struvite treatments (ECST and CG) differed from one another, and the stem-plus-leaves tissue P concentration from CG was similar to that from the TSP, MAP, DAP, and the No P/-N control treatments. The mean stem-plus-leaves tissue P concentration from ECST was 1.2 times greater than that from the CG treatment and also 1.8 times greater than that from the No P/+N control treatment. The stem-plus-leaves tissue P concentration mean from CG, TSP, MAP, DAP, and the No P/-N control, which did not differ, was 1.5 times larger than that from the No P/+N control treatment.

Although yield was not measured in this study due to terminating the study before the corn plants reached full maturity, cob-plus-husk and stem-plus-leaves tissue P concentrations (Table 3) from ECST were greater than that from CG and TSP, which suggests that corn yields would have been at least similar, and perhaps greater, from ECST than yields from CG and TSP. Additionally, the larger cob-plus-husk and stem-plus-leaves tissue P concentrations for ECST than from CG or TSP, coupled with the lower belowground tissue P concentration from ECST than from CG or TSP, suggests that the P from ECST was more mobile in the plant than the P from CG or TSP. The relatively greater purity of the ECST material than that of the CG or TSP material may have contributed to mobility differences, as well as could have led to slightly different forms of P that were taken up by the plant roots from the various fertilizer-P sources.

Conclusions

There were differences in the degree of plant response depending on the fertilizer-P source. Both struvite treatments had at least similar, and in some cases even greater, plant responses in corn to several other commonly used fertilizer-P sources. These results provide not only more useful information on how wastewater-recycled nutrients such as struvite, in crystalline (ECST) or pelletized (CG) form, perform as compared to other commercially available P fertilizers, but also further reasons why more research should be conducted on not only the implementation of struvite as a fertilizer-P source but also struvite's potential impact on sustainable food production and the preservation of water resources. However, more research is still required in order to verify the large potential benefits of not only using struvite as a recycled-P fertilizer, but P recovery from wastewater as an alternative approach to improve wastewater quality and provide a sustainable source of fertilizer-P for further agricultural production.

Acknowledgments

This study was funded by the University of Arkansas Honors College in the form of an Honors College research grant. Thank you to Dr. Trent Roberts for providing the corn seeds used in this study.

Table 3. Summary of the effects of fertilizer amendment on corn belowground, cob-plus-husk, and stem-plus-leaves tissue P elemental concentrations.

	Corn Tissue P Elemental Concentrations		
Treatment	Belowground Tissue P	Cob-plus-Husk Tissue P	Stem-Plus-Leaves Tissue P
	----------------	g/kg	----------------
ECST[†]	0.9 b[‡]	2.7 a	1.6 a
CG	1.3 a	2.3 bcd	1.4 b
TSP	1.2 a	2.3 bcd	1.3 bc
MAP	1.1 a	2.5 ab	1.3 b
DAP	1.3 a	2.5 ab	1.4 b
RP	0.8 b	2.1 cd	1.2 c
No P/+N	0.9 b	2.0 d	0.9 d
No P/-N	0.8 b	2.4 abc	1.4 b
P-value	**< 0.01**[§]	**0.01**	**< 0.01**

[†] Electrochemically precipitated struvite (ECST), Crystal Green (CG), triple superphosphate (TSP), monoammonium phosphate (MAP), diammonium phosphate (DAP), rock phosphate (RP), unamended control that did not have added P, but had added N (No P/+N), and unamended control without added P or N (No P/-N).

[‡] Means in a column with different letters are different at $P < 0.05$.

[§] Bolded values are significant at $P < 0.05$.

Literature Cited

Ashley, K., D. Cordell, and D. Mavinic. 2011. A brief history of phosphorus: from the philosopher's stone to nutrient recovery and reuse. Chemosphere 84:737–746.

Cordell, D., J. Drangert, and S. White. 2009. The story of phosphorus: global food security and food for thought. NATO ASI Ser., Ser. I. 19:292–305.

Espinoza, L., and J. Ross. 2008. Fertilization and liming. In: L. Espinoza, J. Ross, editors, Arkansas Corn Production Handbook. MP437. Univ. of Arkansas., Div. of Agric., Coop. Ext. Serv., Little Rock. p. 23–27.

Filippelli, G.M. 2011. Phosphate rock formation and marine phosphorus geochemistry: the deep time perspective. Chemosphere 84:759–766.

Hallas, J., C. Mackowiak, A. Wilkie, and W. Harris. 2019. Struvite phosphorus recovery from aerobically digested municipal wastewater. Sustainability 11:1–12.

Jarvie, H.P., A.N. Sharpley, D. Flaten, P.J. Kleinman, A. Jenkins, and T. Simmons. 2015. The pivotal role of phosphorus in a resilient water–energy–food security nexus. J. Environ. Qual. 44:1049–1062.

Liu, Y., S. Kumar, J. Kwag, and C. Ra. 2012. Magnesium ammonium phosphate formation, recovery and its application as valuable resources: a review. J. Appl. Chem. Biotechnol. 88:181–189.

Massey, M.S., J.G. Davis, J.A. Ippolito, and R.E. Sheffield. 2009. Effectiveness of recovered magnesium phosphates as fertilizers in neutral and slightly alkaline soils. Agron. J. 102:323–329.

Oburger, E., D.L. Jones, and W.W. Wenzel. 2011. Phosphorus saturation and pH differentially regulate the efficiency of organic acid anion-mediated P solubilization mechanisms in soil. Plant Soil 341:363–382.

Rahman, M.M., M.A. Salleh, U. Rashid, A. Ahsan, M.M. Hossain, and C.S. Ra. 2014. Production of slow release crystal fertilizer from wastewaters through struvite crystallization–a review. Arabian J. Chem. 7:139–155.

Soil Survey Staff. 2017. Captina series. Web Soil Survey. USDA-NRCS. Accessed 7 February 2020. Available at: https://soilseries.sc.egov.usda.gov/OSD_Docs/C/CAPTINA.html

Soltanpour, P.N., G.W. Johnson, S.M. Workman, J.B. Jones Jr., and R.O. Miller. 1996. Inductively coupled plasma emission spectrometry and inductively coupled plasma-mass spectroscopy. *In*: J. M. Bigham et al., editors, Methods of Soil Analysis: Part 3 Chemical Methods. SSSA Book Ser. 5. Madison, Wisconsin. p. 91–140.

Talboys, P.J., J. Heppell, T. Roose, J.R. Healey, D.L. Jones, and P.J. Withers. 2015. Struvite: a slow-release fertilizer for sustainable phosphorus management? Plant Soil 401:109–123.

USEPA. 1996. United States Environmental Protection Agency. Method 3050B: acid digestion of sediments, sludges, and soils. Revision 2. Environmental Sampling and Analytical Methods Program. USEPA. Accessed 7 February 2020. Available at: https://www.epa.gov/esam/epa-method-3050b-acid-digestion-sediments-sludges-and-soils