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Evaluating agronomic soil phosphorus tests for soils amended with struvite

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ABSTRACT

Struvite is an emerging, recycled phosphorus (P) fertilizer of low water solubility (<5%). As a first step towards wide-scale integration of struvite into agricultural systems, distinct interpretation of soil test phosphorus (STP) values for soils amended with struvite may be needed due to the persistence of struvite for months after its application (i.e., residual struvite). However, STP methods were developed for soils amended with highly water-soluble P fertilizers and may not necessarily translate to soils with residual struvite prior to soil testing for P recommendation. We evaluated the potential effects of STP method and edaphic properties (pH, clay content) on STP values for soils with residual struvite. To mimic residual struvite, struvite granules were added to a quartz control and to six soils encompassing a range of pH (4.3, 6.0, 8.1) representative of agricultural soils and with contrasting clay content. The mixtures were then extracted by common STP methods (Mehlich-3, Bray-1, Olsen), Resin, and Haney 3A-2. In the quartz control, dissolution of struvite granules in STP extraction solutions ranged from 59% in Resin to 10% in H3A-2. In soil treatments, apparent dissolution of struvite among STP methods was 19–401% higher for acidic soils with low versus high clay contents. Adsorption experiments confirmed that the disparity in the apparent dissolution of struvite in soils was caused by adsorption of dissolved P on clay minerals. Additionally, for acidic soils with high clay content, scanning electron microscopy coupled with energy dispersive X-ray spectroscopy identified surface deposition of Al and Si on the struvite granule surface during STP extractions, which may have decreased struvite dissolution. Depending on STP method, residual struvite dissolution may overestimate STP concentrations by 20–3900%. Results demonstrate the need to account for the impacts of residual struvite on measured STP values. As a next step, quantifying in situ struvite dissolution rates across soil and cropping conditions is needed to evaluate the extent to which residual struvite may require adjustment of yield-based calibration of STP values.

1. Introduction

Demand for phosphorus (P), a key crop nutrient largely derived from finite resources, is projected to double in the next 20 years, leading to increased interest in the use of P recovered from wastestreams as a fertilizer (Bennett et al., 2001; Cordell et al., 2011). Struvite ($NH_4MgPO_4{\cdot}6H_2O$), a P mineral generated from wastestreams by precipitation, is increasingly seen as a means to reduce point source losses of P while valorizing recovered P as a potential fertilizer (Bennett et al., 2001; Heppell et al., 2016; Trimmer and Guest, 2018). Though it exhibits low water-solubility (generally < 5%), improved solubility in the presence of citrate (27%) and other organic acids secreted by roots raises the possibility of better synchronizing P dissolution with crop needs (i.e., P release from struvite concurrent with crop demand) (Ahmed et al., 2016; Heppell et al., 2016; Everaert et al., 2017). Accordingly, struvite has been proposed to minimize the water-soluble P in bulk soils while maintaining high P phytoavailability in the rhizosphere, thereby optimizing the trade-offs between P availability and loss risk (Margenot et al., 2019). For example, despite the incomplete dissolution of granular struvite (26%, measured by mass difference of remaining granule) as a P fertilizer, struvite can support equivalent crop growth and (apparent) P fertilizer recovery relative to diammonium phosphate (Talboys et al., 2016). However, in contrast to commonly used soluble fertilizers (e.g., ammonium phosphates, superphosphates), the persistence of undissolved struvite at the end of a growing season (i.e., residual) can impact evaluation of soil P availability for the next cropping season.

Phosphorus fertilizer recommendations are commonly based on soil testing to achieve economic optimum returns on P inputs (Menon and

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Chien, 1995). However, soil test P (STP) methods are designed for soils amended with highly water-soluble P fertilizers rather than lowly water-soluble P fertilizers such as struvite or phosphate rock (water solubility < 1%). For soils amended with phosphate rock, Menon and Chien (1995) found soil tests using acidic extractants can overestimate P availability whereas those using alkaline extractants can underestimate P availability due to greater dissolution of residual phosphate rock in the low pH extraction solution. Though values of some STP methods such as Bray-1 and Olsen are generally correlated (Wolf and Baker, 1985), these correlations are significantly influenced by the water solubility of the P source used to fertilize soils because of residual (i.e., non-dissolved) P fertilizer present in the soil at the time of testing (Bolland and Allen, 1987). Thus, distinct interpretations of extraction-based STP values have been established for soils amended with low water solubility phosphate rocks (Bolland and Allen, 1987; Bolland et al., 1989; Menon et al., 1989; Menon and Chien, 1995).

Similar to phosphate rock, residual struvite that remains undissolved in soil after a cropping season (Fig. S1) means that commonly used soil tests (e.g., Mehlich-3, Bray-1) may require adjustment for how absolute values are interpreted. As a first step towards field crop yield-based calibration of struvite application rates, it is necessary to determine the extent to which the deviation of STP values can be influenced by the residual struvite in the tested soils (Margenot et al., 2019). Limited field-scale evaluation of struvite as fertilizer suggests that soil STP values may be more sensitive to struvite than conventionally employed highly water-soluble P fertilizers, depending on the STP method (Thompson, 2013). In Iowa maize systems, struvite-amended soils sampled after harvest – and thus susceptible to residual struvite effects – exhibited lower values of Bray-1 and Olsen, but not Mehlich-3, compared to soils amended with triple superphosphate (TSP) (Thompson, 2013). This likely reflects a differential response of residual struvite to the Mehlich-3 tested soils but not in the Bray-1 test extractions.

Dissolution of residual struvite in the soil sample during the soil test extraction is likely to entail some degree of STP overestimation, depending on the pH and composition of STP extractants (e.g., organic acids, chelating ligands). A linear decrease in dissolution rate (mg d^{-1}) of granular struvite (2.9 mm diameter) with increasing pH across a soil pH gradient of 5.9 to 8.5 (1:1 water) (Degryse et al., 2017) suggests that acidic extractants such as Mehlich-3 (pH 2.5 and buffered) or Bray-1 (pH 2.7 and unbuffered) may solubilize P from residual struvite. Despite its formation in alkaline pH conditions (pH 8.5–9.0), struvite has also unexpectedly shown appreciable dissolution (~60%) by $NaHCO_3$ extraction (0.5 M, pH 8.5) after the Resin extraction (~40% dissolution) in 16 h sequential extractions (Meyer et al., 2018) However, fine grinding (<120 μm) of struvite as applied by Meyer et al. (2018) likely maximized its dissolution by increasing particulate surface area, thus overestimating dissolution compared to granular struvite (Degryse et al., 2017). Given that granular struvite (1 to 3 mm) generated by precipitation (Talboys et al., 2016; Degryse et al., 2017) is well-suited to the practical needs of dry fertilizer application (Hanna and Sawyer, 2001; Liu et al., 2016), dissolution of finely ground struvite may not be relevant in agronomic contexts. Additionally, greater adsorption of anions such as phosphate with increasing clay content (Gu et al., 2016; Wang et al., 2017) suggests that adsorption of phosphate dissolved from residual struvite – or any other residual P fertilizer – on clay minerals during the extraction would likely lead to lower apparent STP values.

Thus, the objective of this laboratory study was to evaluate the potential effect of residual struvite on values of five different STP methods and the impacts of soil properties on the STP values. The widely used STP methods of Mehlich-3, Bray-1 and Olsen were evaluated, as well as the recently developed Haney test (H3A-2) that uses a combination of organic acids (Haney et al., 2010). Finally, due to its universality across soil pH and P source (Tiessen and Moir, 1993) and common use in biogeochemical assessments (Johnson et al., 2003; Gu and Margenot, 2020), the sink-based Resin test was evalauted. We quantified dissolution of residual granular struvite by these five STP methods using a matrix of quartz (control) and six soils that engendered edaphic contrasts of pH and clay. We hypothesized that low pH of STP extractants and soils would cause greater dissolution, and high clay content would decrease the apparent dissolution of struvite. This study serves as a first step to identify potential impacts of residual struvite on STP values, which has direct implications for future yield-based calibration of STP values on soils amended with struvite.

2. Materials and methods

2.1. Struvite properties

Struvite granules of size guide number (SGN) 300 were obtained (9–13 mg $granule^{-1}$) as Crystal Green® with a P content of 12.2% (28% P_2O_5) reported by Ostara Inc. (Chicago, IL) and confirmed by 1 M nitric acid digestion and molybdate colorimetry (Murphy and Riley, 1962) as 12.4 ± 0.2% (n = 3). To control for particle size effects, struvite granules were constrained to 2.0–2.8 mm diameter by dry sieving prior to dissolution experiments.

2.2. Soil test P (STP) methods

Five STP methods for soil available P were selected (Table 1) to represent the majority of STP used in agronomic P management (Hopkins, 2015) as well as soil biogeochemistry. The choice of STP method is often dictated by soil pH due to pH-specific mechanisms of P extraction (Havlin et al., 2013). Mehlich-3 (Mehlich, 1984) and Bray-1 (Bray and Kurtz, 1945) are prescribed for acidic soils (pH < 7) but are not applicable to alkaline soils (pH > 7) because the acidic extraction solution dissolves calcium bound P (Ca-P) forms, and can lead to overestimating crop-available soil P (Elrashidi, 2010). In contrast, Olsen (Olsen et al., 1954) was developed for alkaline soils, for which it is the STP method of choice in the US and internationally (Elrashidi, 2010; Havlin et al., 2013). The recently developed H3A-2 is proposed for use in both acidic and alkaline soils, and is designed to mimic root exuded organic acids (Haney et al., 2010). In contrast to extraction-based tests that are specific to soil pH, Resin test is applicable to all soils as a sink-based method.

Two struvite granules (0.020–0.030 g total mass) were added to 1.0 g quartz or soil and extracted in quadruplicate using each STP method detailed in Table 1. For soils, only the appropriate STP methods based on the soil pH (e.g., alkaline soils extracted with Olsen and H3A-2 only) were conducted. Acid-washed quartz (<63 μm) was used to mimic the physical matrix effect of a soil sample. Soil extracts were filtered through Whatman® Grade 42 filter paper. In contrast to these four extraction-based tests used in agronomy, the sink-based Resin test is conducted over a longer time period (16 h) and with a wider solid: solution ratio (1:40). Anion exchange membrane (1 × 4 cm, VWR International, West Chester, PA) was loaded with bicarbonate as the counterion (Cheesman et al., 2010). Inorganic P on the membrane was desorbed by shaking for 1 h in 20 ml of 0.25 mol L^{-1} H_2SO_4 (Cheesman et al., 2010). Orthophosphate P concentration was quantified as molybdate-reactive P by colorimetry (Murphy and Riley, 1962).

To identify whether the dissolution of struvite by different STP methods is caused by the extractant solution chemistry or by the extraction time and solid: solution ratio, struvite granules were extracted in deionized (DI) water in quadruplicate for varying durations (5 min, 30 min, 16 h) and solid: solution ratios (1:10 or 1: 20) that matched STP methods.

Dissolution of P (%) from struvite was calculated for each STP method to account for variation in the mass of individual struvite granules, using the following equation:

Table 1
Parameters for soil test phosphorus (STP) methods used to evaluate hypothesized impacts of dissolution of residual struvite on STP values.

	Extraction time (min)	Recommended soil pH	Extractant pH	Extractant Composition	Solid: solution	Refs.
Resin	960	any	7.0	Deionized water + anion-exchange membrane loaded with sodium bicarbonate	1:40	Tiessen and Moir (1993)
Mehlich-3	5	<6.5	2.5 (buffered)	0.2 M acetic acid 0.25 M ammonium nitrate 0.015 M ammonium fluoride 0.013 M nitric acid 0.001 M EDTA	1:10	Mehlich (1984)
Bray-1	5	<6.5	2.7	0.025 M hydrochloric acid 0.03 M ammonium fluoride	1:10	Bray and Kurtz (1945)
H3A-2	5	4.5–8.6	4.4	0.010 lithium citrate 0.003 M citric acid 0.003 M malic acid 0.004 M oxalic acid	1:10	Haney et al., 2010
Olsen	30	>6.5	8.5	0.5 M sodium bicarbonate	1:20	Olsen et al. (1954)

$$\text{P Dissolution}(\%) = \frac{\left(Pconcentration_{soil+fertilizer} - Pconcentration_{soil}\right) \times V_{total}}{Total\ P\ amount\ in\ granule} \times 100\%$$

In which, $Pconcentration_{soil+fertilizer}$ and $Pconcentration_{soil}$ represent the P concentrations in the STP extracts of fertilizer-added soil and original soil, respectively. $V_{total}$is the volume of extractants, which is 10 ml for Mehlich-3, Bray-1 and H3A-2, and 20 ml for Olsen, (Havlin et al., 2013), with 40 ml for Resin for extraction and 20 ml solution for eluting sorbed P.

2.3. Soils

Six surface soils under agricultural land use were selected to furnish differences common in pH (4–8) and clay content, while also furnishing a range of background STP values (Table 2). While these soils vary in other properties that may interact with soil test extractants, the three pH values and comparison of relatively low versus high clay are meant to evaluate the extent to which dissolution of the remaining struvite in soil test extractants can be influenced by these two major soil properties. As such, these soils are meant to illustrate variation in dissolution of residual struvite, but are not intended for projecting recommendations based on the limited soil set evaluated.

Soils were collected from the plow layer (0–20 cm depth), air-dried and sieved to < 2 mm prior to analyses. Soil pH was determined in DI water (1:2 m/v). Total C was measured by dry combustion and corrected for carbonate content, which was quantified seperately via gravimetric loss by acidification, to estimate total organic C (TOC) (Ramnarine et al., 2011). Soil pH encompassed low (strongly acidic, 4.3), middle (weakly acidic, 6.0) and high (alkaline, 8.1) values (Table 2). For each of the three soil pH values, two soils were selected to furnish contrasting clay contents (0–55%) in order to test for hypothesized clay effects on STP results (i.e., adsorption of dissolved P). Based on these properties, soils are referred to as $pH_{xx}HC$ or $pH_{xx}LC$, in which xx designates one of three pH values, and HC and LC refer to high clay and low clay, respectively.

2.4. Adsorption experiments

Preliminary data showed that the apparent dissolution of struvite was lower in the high clay than low clay soils for soils with acidic pH. To estimate the potential impacts of P adsorption on clay surfaces during STP extractions, adsorption experiments were therefore conducted for $pH_{4.3}HC$ and $pH_{6.0}HC$ for Mehlich-3, Bray-1 and H3A-2 tests. Certified phosphate-P reference solution (1000 mg P L^{-1}, CAT#5839.1-16, Ricca Chemical Company, Arlington, TX 76012) and 10.0 ml extractant were sequentially added to 1.0 g soil and subjected to the extractions in triplicate. The amount of P reference solution was adjusted to achieve an initial solution P concentration of 0, 4, 8, 16, 32, 64 μg ml^{-1} phosphate. Soil extracts were filtered through Whatman® Grade 42 filter paper, and P concentration was quantified by molybdate colorimetry (Murphy and Riley, 1962). The expected STP concentrations would be the sum of the soil background STP concentrations (i.e., without struvite) and the added phosphate if no adsorption occurred during STP extractions. Linear regression of the measured P concentrations versus the expected concentrations was conducted to test the degree of the deviation of measured P concentrations from the expected P concentrations, i.e., greater slope indicates greater adsorption of P by soil clays. This linear regression equation was then used to calculate the true dissolution of struvite that accounts for the adsorption of P in the high clay and acidic soils during dissolution of residual struvite during soil test extractions.

Table 2
Soil classification, general properties, and soil test phosphorus (STP) concentrations (mg P kg^{-1} soil) in the soils with standard deviations showed in parentheses. N.A. signifies not applicable, as Olsen test is not used for acidic soils and Mehlich-3 and Bray-1 tests are not used for alkaline soils.

Soil	USDA classification	pH (1:2 water)	Clay (%)	OC (%)	Resin	Mehlich-3	Bray-1	H3A-2	Olsen
$pH_{4.3}HC$	Oxisol	4.3	55.0	2.4	10.6 (0.6)	3.4 (0.4)	22.2 (1.4)	7.7 (0.9)	N.A.
$pH_{4.3}LC$	sandy, siliceous, hyperthermic Typic Alaquods	4.3	2.5	2.7	16.5 (0.9)	5.5 (0.5)	7.5 (0.8)	6.2 (0.3)	N.A.
$pH_{6.0}HC$	fine, smectitic, mesic Aquic Argiudoll	6.0	31.7	3.7	99.7 (5.6)	50.8 (5.2)	149.3 (2.1)	113.9 (9.4)	N.A.
$pH_{6.0}LC$	fine-loamy, kaolinitic, thermic Typic Kandiudult	6.0	7.5	0.8	47.3 (1.9)	64.0 (2.3)	197.7 (13.7)	12.4 (3.0)	N.A.
$pH_{8.1}HC$	loamy-skeletal, carbonatic, hyperthermic Lithic Udorthent	8.1	22.5	2.3	24.6 (1.4)	N.A.	N.A.	8.4 (1.2)	11.1 (0.7)
$pH_{8.1}LC$	fine-loamy, mixed, superactive, mesic Typic Natrargid	8.1	0.1	0.2	41.8 (1.1)	N.A.	N.A.	9.4 (2.2)	3.2 (0.3)

$pH_{4.3}HC$: pH 4.3, high clay; $pH_{4.3}LC$: pH 4.3, low clay; $pH_{6.0}HC$: pH 6.0, high clay; $pH_{6.0}LC$: pH 6.0, low clay; $pH_{8.1}HC$: pH 8.1, high clay; $pH_{8.1}LC$: pH 8.1, low clay.

2.5. Surface morphology and chemical composition of pure struvite and residual struvite after STP extraction

To complement laboratory analyses of solution chemistry and soil clay impacts on struvite dissolution, scanning electron microscope (SEM) images were collected and elemental chemical analysis were conducted for pure struvite and residual struvite recovered from Mehlich-3 extraction for $pH_{4.3}HC$ soil using an Inspect JSM-7800F (JEOL, Japan) SEM at the acceleration voltage of 3.0 kV with an energy-dispersive X-ray analysis spectrometer (EDS). Remaining residual struvite granules after extraction were isolated using forceps, and then gently washed using DI water to remove any physically surface attached materials. The residual struvite granules were oven-dried at 65 °C for 48 h to remove water for SEM-EDS analysis. Though heating may alter the crystalline structure of struvite (Farhana, 2015), removal of water is necessary to achieve the requirements of vacuum for the SEM instrument.

2.6. Statistical analyses

To evaluate the differences in apparent dissolution of struvite across STP methods and soils, including the quartz control and the six soils, one–way analysis of variance (ANOVA, Tukey test $\alpha = 0.05$) were conducted separately for each soil test using PROC GLM in SAS v9.4. The normality and homoscedasticity of the residuals were confirmed by using the Kolmogorov-Smirnov test's and Levene's test, respectively.

3. Results

3.1. Background concentrations of soil test phosphorus in the soil materials

Soil test P values varied by three orders of magnitude (3.2–197.7 mg kg^{-1}) across STP methods, and were greatest in $pH_{6.0}HC$ and $pH_{6.0}LC$ soils, followed by $pH_{4.3}HC$ and $pH_{4.3}LC$ soils, and the lowest in $pH_{8.1}HC$ and $pH_{8.1}LC$ soils (Table 2). For a given soil, STP values varied by up to an order of magnitude among STP methods. For example, STP by H3A-2 was 14-fold lower than by Bray-1 test for $pH_{6.0}LC$ soil (12.4–197.7 mg kg^{-1}).

3.2. Struvite dissolution in DI water

Struvite dissolution in DI water (0.6–15.2%) was significantly higher with wider solid: solution ratio of 1: 20 and longer extraction time (16 h) than solid: solution ratio of 1: 10 and shorter extraction time representative of STP extraction parameters (Fig. 1). For the relatively brief extraction times (≤30 min) used in common STP methods compared to Resin (>16 h), struvite dissolution in DI water was similar and unaffected by the solid: solution ratio.

3.3. Apparent dissolution of residual struvite by STP methods

Struvite dissolution among STP methods (control treatments) was distinct for Resin and methods with highly acidic extraction solutions (i.e., Mehlich-3 and Bray-1, Fig. 2). Struvite dissolution decreased in the order of Resin (59.3 ± 7.2%), Mehlich-3 (24.0 ± 2.0%) and Bray-1 (24.0 ± 2.4%) to Olsen (16.0 ± 1.5%) and H3A-2 (10.1 ± 1.2%). Except for Olsen test, the greater dissolution in extraction-based tests with lower pH of extractants (Mehlich-3, Bray-1) than in higher pH H3A-2 supported the hypothesis of extractant pH impacting struvite dissolution.

The influence of soil pH on struvite dissolution mainly depended on clay contents for acidic soils except the Resin test (Fig. 2, Table 3, Table S1). No significant differences in struvite dissolution by Resin test were observed between the control and soils of pH 6.0 and 8.0 (60–71%). Soils with pH 4.3 showed greater apparent dissolution with the Resin test (86–92%) than other soils. Soil pH only influenced

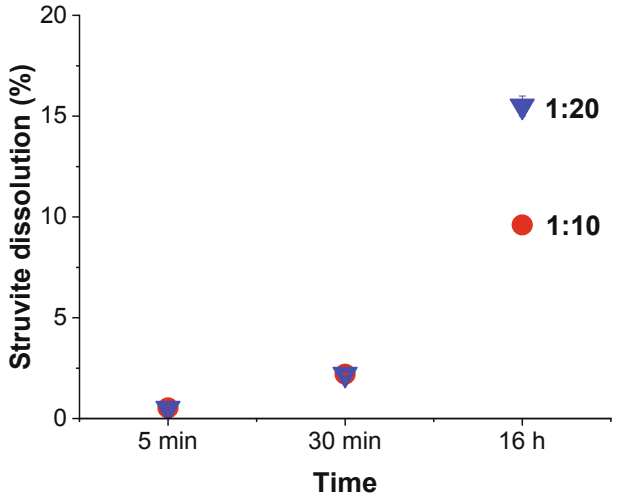

Fig. 1. Struvite dissolution (%) in deionized water in the presence of quartz as a control substrate with time (5 min, 30 min, 16 h) and different solid: solution ratios (1:10 or 1:20). Standard deviations are shown as bars for the mean value, indicated by the circle (1:10 ratio) or inverted triangle (1:20 ratio).

apparent dissolution in Mehlich-3 for soils with low clay contents, with 23% lower dissolution in $pH_{4.3}LC$ than $pH_{6.0}LC$. In contrast, soils with high clay contents yielded greater differences in apparent dissolution than soils with low clay contents. For example, the apparent dissolution of struvite in Bray-1 was much lower for high clay soils than low clay soils. Apparent dissolution in high clay soils was not influenced by soil pH but in low clay soils was 71% higher at pH 4.3 than pH 6.0. Moreover, for Mehlich-3, Bray-1 and H3A-2 at pH 4.3 and pH 6.0, when comparing apparent dissolution at the same pH, low clay soils generally had greater dissolution (19–401%) than high clay soils. However, at pH 8.1, there appeared to be no difference in struvite apparent dissolution between the two contrasting clay contents.

3.4. True dissolution of residual struvite by selected STP methods in high clay soils

Significant adsorption of P during the Mehlich-3, Bray-1, and H3A2 extractions was observed, with the expected P concentrations consistently higher than the measured P concentrations (Fig. 3). The slopes for the $pH_{4.3}HC$ soil with different STP methods were generally greater than for $pH_{6.0}HC$ soil. The cut-off values for all the treatments were overall consistent with the corresponding background STP concentrations. Linear regression to account for adsorption revealed that true dissolution proportions of struvite for both high clay acidic soils and the three STP methods were 22–98% higher than the apparent dissolution (Fig. 4). The extent of soil-specific adsorption was soil-specific and also appeared to be STP method-specific.

3.5. Surface morphology and chemical composition of residual struvite after STP extraction

Scanning electron microscopy revealed qualitative differences in surface morphology and chemical composition between pure struvite and residual struvite following Mehlich-3 extraction in $pH_{4.3}HC$ soil. The pure struvite surface had more obvious pore structures and exhibited greater microgranularity with limited but notable lamellar structure (Fig. 5a-b). After extraction of a struvite granule in soils by Mehlich-3, which mimics residual struvite being present in the soil sample during STP measurement, the surface of the residual struvite appeared smoother with a greater abundance of clastic fine particles. Changes in struvite surface chemical composition before and after Mehlich-3 extraction suggested surface deposition of Al and Si ions

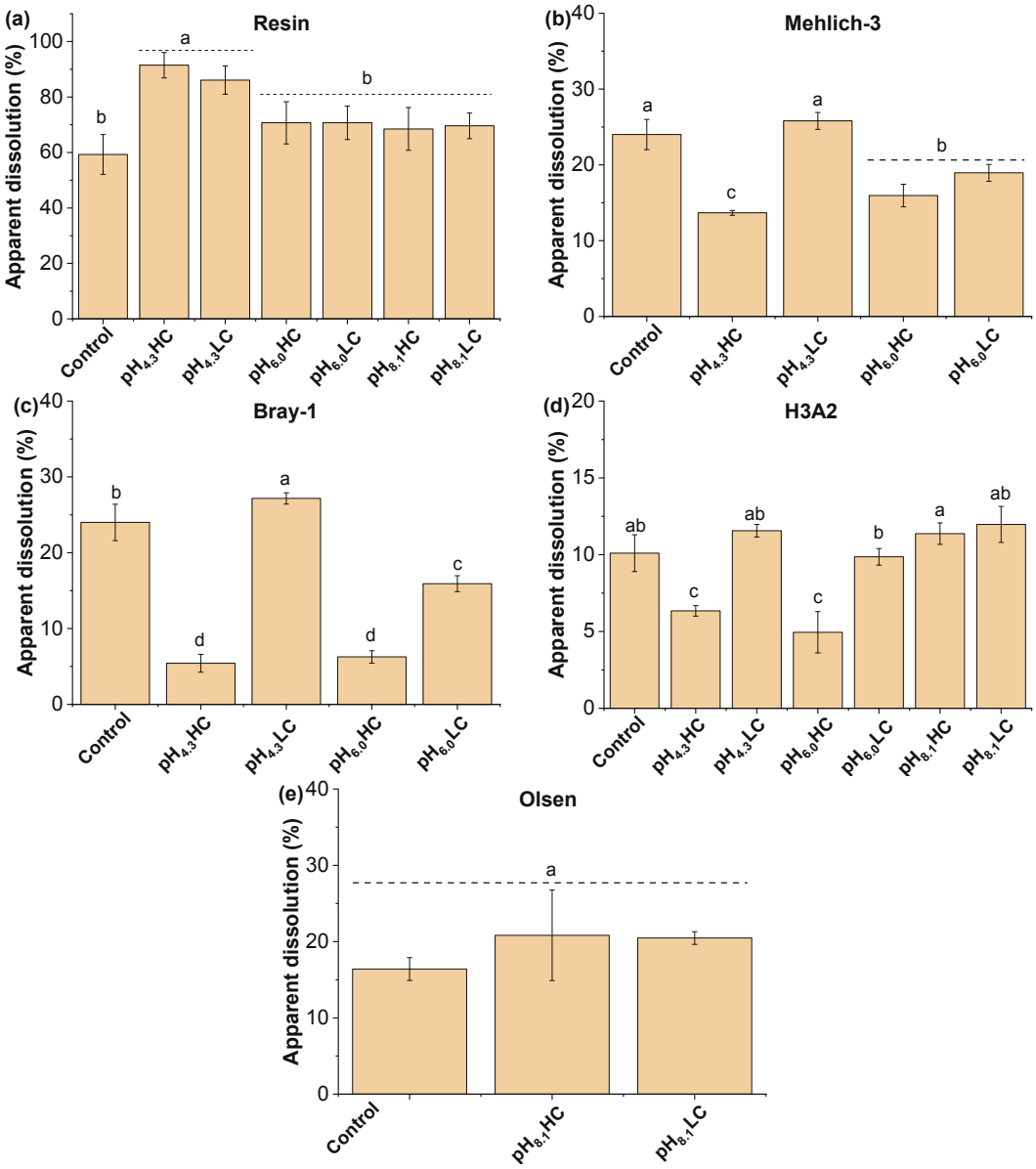

Fig. 2. Apparent dissolution proportion of struvite in substrate of quartz (control) and soils by soil test P (STP) methods. The means separation was conducted by Tukey test ($p < 0.05$). Letters indicate differences among the soils. As Olsen test is not used for acidic soils and Mehlich-3 and Bray-1 tests are not used for alkaline soils, these STP were not performed for acidic and alkaline soils, respectively. Note that the y-axis differs by STP methods. $pH_{4.3}$HC: pH 4.3, high clay; $pH_{4.3}$LC: pH 4.3, low clay; $pH_{6.0}$HC: pH 6.0, high clay; $pH_{6.0}$LC: pH 6.0, low clay; $pH_{8.1}$HC: pH 8.1, high clay; $pH_{8.1}$LC: pH 8.1, low clay.

derived from soils (Fig. 5c). Total P and Mg concentrations were lower relative to pure struvite, and Al and Si totaled 11.1% of mass on the surface evaluated by EDS.

3.6. Residuality and dissolution of MAP compared to struvite

The focus of this study is on struvite, as MAP dissolution is believed to be rapidly and presumably no residual MAP granule will persist. For examle, using a greenhouse study employing the $pH_{6.0}$HC soil, nearly 6-fold more struvite remained after only 35 days (~77%) compared to MAP (~13%) (Table S2). In a field experiment also on the $pH_{6.0}$HC soil, 138 days after struvite application there remained 35–50% of struvite but none of the MAP in struvite-MAP blends (Table S3). However, to show how chemical behavior of highly water-soluble P fertilizers in STP extractants may differ from struvite, a parallel experiment was conducted to quantify MAP dissolution in STP extractants with quartz or soils using the same parameters. The mean proportion of dissolved MAP for treatment of acidic soils was generally lower in HC than LC soils (Table S4), suggesting adsorption of P dissolved from granules. The mean proportion of dissolved MAP was generally greater than that for struvite in all treatments except the resin test. The greater dissolution of struvite than MAP by resin but not by other STP tests suggests resin test induced greater dissolution of struvite. The greater standard deviation (Table S4) for the MAP than the struvite by extraction based STP tests suggests MAP is more sensitive to minor differences in methodological parameters of STP methods such as shaking time.

4. Discussion

4.1. Water-solubility of struvite

Differences among STP methods in extraction time and solid: solution ratio were unlikely to contribute to the observed variation in struvite dissolution in soils because struvite dissolution in water was similar

Table 3
Example of anticipated orthophosphate concentrations (mg P kg^{-1} soil) for soil test P (STP) methods due to residual struvite. An example residual struvite mass of 2 mg was used because this is equivalent to 20% of an intact granule, i.e. assuming 80% dissolution over the growing season before soil sampling in the fall. This 2 mg of struvite (0.024 mg P) was present in 1.0 g soil during the soil test. The dissolution proportion of struvite as reported in Fig. 2 was then used to estimate apparent STP values that would result. Standard deviations are shown in parentheses. N.A. signifies not applicable, as Olsen test is not used for acidic soils and Mehlich-3 and Bray-1 tests are not used for alkaline soils.

	Resin	Mehlich-3	Bray-1	H3A-2	Olsen
$pH_{4.3}HC$	413.2 (20.8)	61.7 (1.4)	24.5 (5.3)	28.6 (1.5)	N.A.
$pH_{4.3}LC$	388.8 (23.0)	116.5 (5.1)	122.6 (3.4)	52.2 (1.9)	N.A.
$pH_{6.0}HC$	319.2 (34.3)	72.1 (6.7)	28.3 (3.7)	22.4 (6.1)	N.A.
$pH_{6.0}LC$	319.2 (27.1)	85.6 (5.0)	71.9 (4.7)	44.5 (2.4)	N.A.
$pH_{8.1}HC$	309.3 (34.8)	N.A.	N.A.	51.3(3.2)	94.0 (6.8)
$pH_{8.1}LC$	314.3 (20.8)	N.A.	N.A.	54.0 (5.3)	92.4 (3.7)

$pH_{4.3}HC$: pH 4.3, high clay; $pH_{4.3}LC$: pH 4.3, low clay; $pH_{6.0}HC$: pH 6.0, high clay;
$pH_{6.0}LC$: pH 6.0, low clay; $pH_{8.1}HC$: pH 8.1, high clay; $pH_{8.1}LC$: pH 8.1, low clay.

across extraction time (5 or 30 min) and solid: solution ratio (1: 10 or 1:20) used in agronomic STP methods (i.e., excluding Resin test) (Table 1). Enhanced dissolution of struvite with wider solid: solution ratio and under longer extraction time such as in the Resin test could help explain discrepancies in reported values of struvite water solubility, in addition to varying particle size. For example, 3% dissolution of granular struvite (based on quantification of dissolved P) was reported in a 2 h extraction at a solid: water ratio of 1:200 (Degryse et al., 2017) and 0.8–1.9% dissolution with a solid: water ratio of 1:50 (Cabeza et al., 2011) though the extraction duration was not specified. On the other hand, including the extraction parameters is critical to indicate the water-solubility of struvite for future studies. The positive effects of longer extraction time (16 h) and wider solid: solution ratio on struvite dissolution means that *in situ* dissolution of struvite over growing season under conditions of high soil moisture may be higher than proposed based on *in vitro* or soil incubation studies (e.g., Talboys et al., 2016).

4.2. Comparisons among various soil P tests

Struvite dissolution by the sink-based Resin test was much greater (>68%) than by the extraction-based agronomic tests (<28%). Struvite dissolution by Resin was also remarkably higher than previous reports of ~ 40% dissolution using < 120 μm struvite for a 16 h extraction without a resin membrane at a solid: water ratio of 1:20 (Meyer et al., 2018), suggesting an interaction among particle size, solid: water ratio and presence of a phosphate sink collectively determine struvite dissolution. As has been found for phosphate rock, the residuality of struvite means that sink-based approaches that do not use acidic extractants may offer a more realistic assessment of crop-available soil P (Bolland, 1993; Menon and Chien, 1995).

The composition and pH of extractant solution of extraction-based STP methods influenced apparent struvite dissolution. Greater apparent struvite dissolution by Mehlich-3 and Bray-1 than by H3A-2 may be attributed to lower extractant pH due to the acidity-driven dissolution (Liu et al., 2016; Talboys et al., 2016; Degryse et al., 2017). Despite containing citric acid and other organic acids known to promote struvite dissolution (Talboys et al., 2016), both apparent and true dissolution of struvite in H3A-2 was much lower than reported

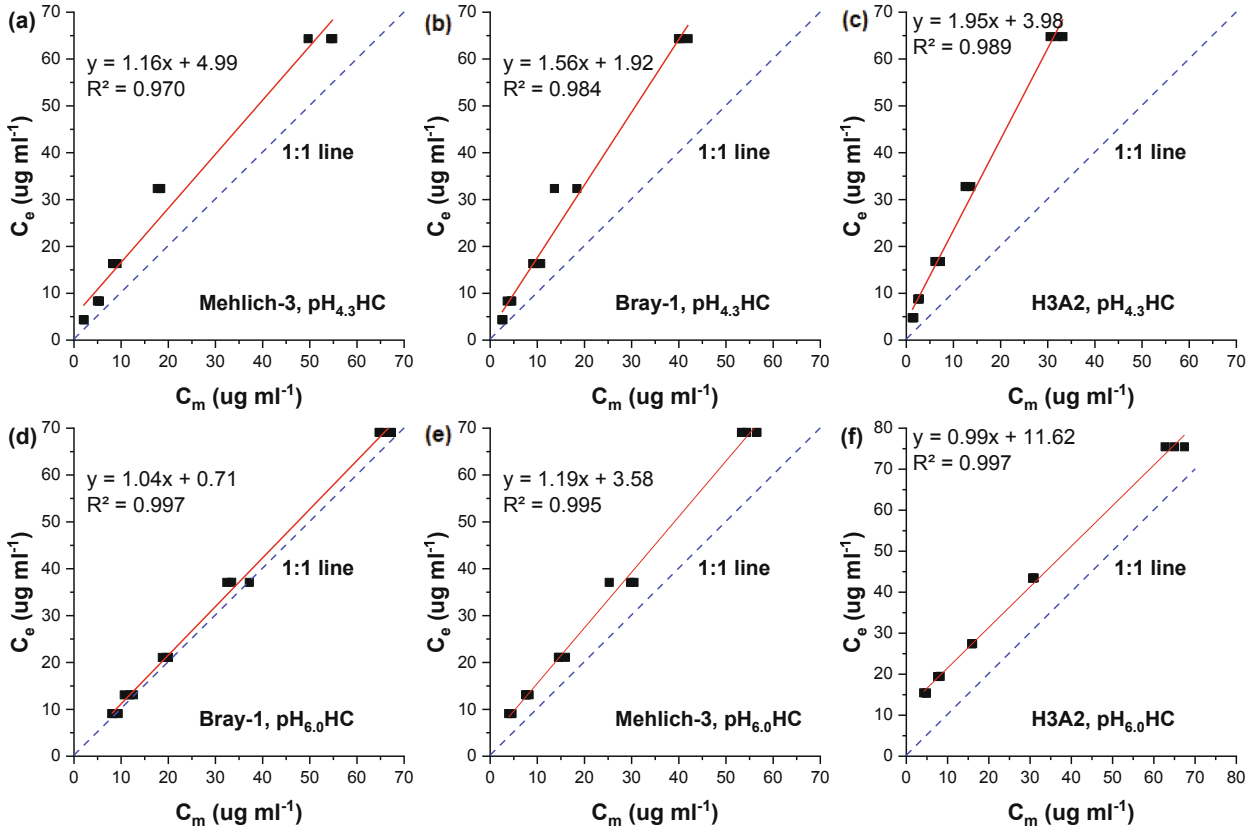

Fig. 3. Linear regressions of the measured orthophosphate concentrations (C_m) and expected concentrations (C_e) as calculated from adsorption experiments. Adsorption experiments were conducted by adding certified phosphate reference solution to achieve an additional P concentrations of 0, 4, 8, 16, 32, 64 μg ml^{-1} phosphate in 1.0 g soils and 10.0 ml extractants of Mehlich-3, Bray-1 and H3A-2, and extracted in triplicate for two high clay acidic soils. The expected orthophosphate concentration in the centrifuge tube was calculated as the sum of the background soil test P in the soil and the added additional P concentrations. $pH_{4.3}HC$: pH 4.3, high clay; $pH_{6.0}HC$: pH 6.0, high clay.

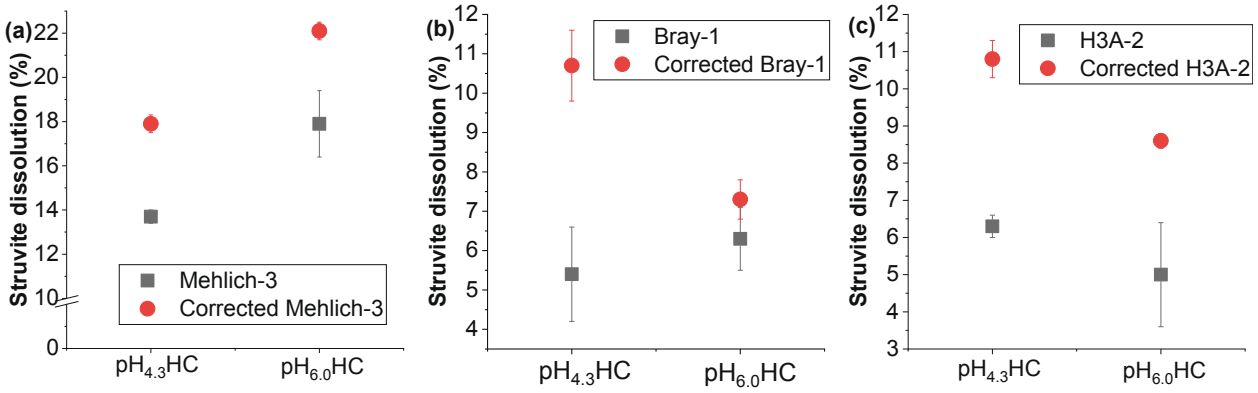

Fig. 4. Struvite dissolution in two high clay acidic soils corrected for soil adsorption of orthophosphate, in soil test P extraction solutions of Mehlich-3, Bray-1 and H3A2 methods. $pH_{4.3}HC$: pH 4.3, high clay; $pH_{6.0}HC$: pH 6.0, high clay.

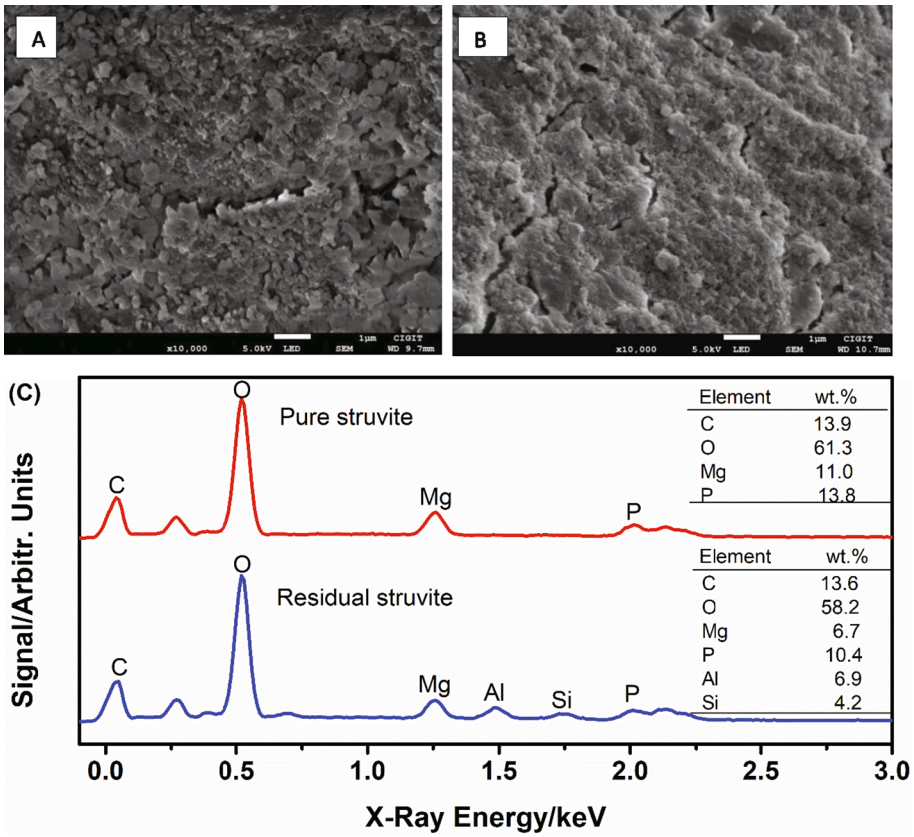

Fig. 5. Scanning electron microscopy (SEM) images and elemental distribution on surfaces of pure struvite (A) and residue struvite (B) after Mehlich-3 extraction of $pH_{4.3}HC$ (pH 4.3, high clay) soil using scanning electron microscope coupled with energy-dispersive X-ray spectroscopy (EDS). Despite the possible transformation of struvite and residual struvite to magnesium phosphate minerals during the oven-drying pretreatment (65 °C), the appearance of Al and Si on the residual struvite is consistent with potential surface deposition of soil-derived Al and Si ions. Note that carbon (C) cannot be reliably determined by EDS due to C contamination inherent to SEM analyses and poor sensitivity of EDS to this element.

previously. For example, up to 94% of struvite was dissolved in 2% citric acid solution over 30 min in Meyer et al. (2018). This is likely due to the lower concentrations of total organic acids (<0.3%) and shorter duration (5 min) of the H3A-2 method.

The lower dissolution of struvite in Olsen (16.4%) compared to 60% dissolution in a similar extraction solution (0.5 M $NaHCO_3$, pH 8.5) reported by Meyer et al. (2018) was likely due to the finer grinding and 28-fold longer duration (0.5 vs 16 h) in their study. Given the four dominant surface species ($\equiv$MgOH, $\equiv MgOH_2^+$, $\equiv$POH and $\equiv PO^-$) involved in acid-base reactions on the struvite surfaces under alkaline conditions (7.5 < pH < 11) (Wei et al., 2018), the $\equiv MgOH_2^+$ on the granular struvite surface likely reacted with HCO_3^- and CO_3^{2-} in the Olsen extractant (pH 8.5), leading to the unexpected greater dissolution than by H3A-2. This is consistent with the moderate crop availability of finely ground struvite in alkaline soils (Meyer et al., 2018).

4.3. Clay dependence of pH impacts

The distinct influences of soil pH on apparent dissolution of struvite are anticipated because its dissolution was favored in low pH extractants. Greater apparent dissolution by Mehlich-3 and Bray-1 tests in $pH_{4.3}LC$ than in $pH_{6.0}LC$ soil is consistent with acidity-driven struvite dissolution (Talboys et al., 2016). However, for agronomic STP methods recommended for acidic soils of Mehlich-3, Bray-1, and H3A-2, we identify a significant influence of clay contents on apparent dissolution of residual struvite given that the differences in the solid:solution ratios and extraction duration cannot explain the marked differences in the apparent dissolution of struvite subjected to a given STP extraction.

Given that the choice of STP method is made on the basis of soil pH, when interpreting STP values the clay contents of acidic soils should be considered due to the underestimation of P availability from residual fertilizers with low water solubility such as – but not limited to – struvite. The degree of P adsorption during STP extractions may be related to soil clay contents because the generally greater slopes of the linear regression equation for the $pH_{4.3}HC$ soil than for the $pH_{6.0}HC$ soils coincided with the greater clay contents of the former (Fig. 3 and Table 2). It is also notable that P adsorption was expected to occur in the high clay acidic soils for Resin test, but was not observed. The absence of P adsorption during the Resin test suggests that P adsorption during extraction-based tests is a weaker factor than the increased dissolution induced by the resin membrane sink effect (i.e., the rapid removal of soluble P from solution by resin favors stronger dissolution of struvite).

4.4. Possible mechanisms for the clay dependence of pH impacts

The lower measured dissolution of struvite in high clay than in low clay acidic soils was likely caused by adsorption of dissolved P onto clay mineral surfaces and inhibited dissolution by surface precipitation. Although soil clay and OC contents are generally positively correlated (Rasmussen et al., 2018), the effects of lower apparent dissolution in the high clay soils than low clay soils can be at least partially ascribed to the influences of clay contents because clay, not OC, dominantly binds soil solution phosphate (Havlin, 2014). Adsorption of P dissolved from struvite onto clay surfaces partially led to the underestimation of apparent dissolution by up to 98% (Fig. 4). Clay minerals may not be completely dissolved by the extractants, which can provide reactive sites for P adsorption. In extracting solutions with low pH (e.g., Mehlich-3, Bray-1), the low solution pH ($\leq$ 2.7) favored P adsorption because P adsorbed on mineral surface is typically inner-sphere complex via ligand exchange process (Essington, 2015) and because low pH favored the protonation of surface hydroxyl group and hence the ligand exchange process (Sparks, 2003; Wang et al., 2017). The solution chemistry of these struvite-extractants systems is complex and P adsorption alone is likely insufficient to explain the observed absence of clay impacts for Mehlich-3 test on slightly acidic (pH 6.0) soils.

Second, struvite dissolution itself was likely lowered by the surface deposition of Al and Si ions. The rate-limiting step of struvite dissolution is the diffusion of constituent ions from the granule surface and/or the desorption of ions from the crystal adsorption layer (Babić-Ivančić et al., 2002). Thus, surface deposited ions could retard struvite dissolution, consistent with previous studies demonstrating the negative impacts of the OC (or PO_4^{3-})-metal ternary complex, and/or surface precipitation of other minerals on minerals of diffusion-controlled dissolution (Fein, 2002; Hinkle et al., 2015).

No nitrogen was detected by EDS, suggesting the possible transformation of struvite to magnesium phosphate minerals during the oven-drying (65 °C) pretreatment, as 60–70% of ammonium N can be lost from struvite heated at 60° ± 0.5 °C via thermal decomposition (Farhana, 2015). However, the appearance of Si and Al on the residual struvite surface can still indicate the surface deposition of soil-derived elements because these are likely to remain on the surface even after drying. Further work is needed to indicate whether the surface deposition process is due to surface precipitation of Al and Si ions and/or physical blocking of pores by Al and Si minerals. In addition, consumption of reactive ions like H^+, F^- and $EDTA^{2-}$ in the extraction solution by Fe and Al minerals may have further attenuated dissolution of struvite in high clay soils. The higher dissolution of struvite in $pH_{4.3}HC$ and $pH_{6.0}HC$ soils by Mehlich-3 than by Bray-1 or H3A-2 was likely caused by the greater concentration of these reactive ions and the buffered pH in Mehlich-3 extraction solution.

4.5. Soil test phosphorus for struvite of agriculture use

Struvite is a key nutrient recovery product from waste streams (Trimmer et al., 2017; Trimmer and Guest, 2018) and offers a means to recirculate P already in the human trophic chain to mitigate reliance on finite phosphate rock reserves (Kok et al., 2018; Weeks and Hettiarachchi, 2019). By enabling P removal from wastewater, generation of struvite enables point sources to decrease effluent P loads (Margenot et al., 2019), and its reuse in agroecosystems to meet crop P needs can then decreasie off-farm P losses to surface waters by virtue of its low water solubility (Everaert et al., 2018; Margenot et al., 2019). In addition, the significantly lower cadmium (Cd) concentrations of struvite than conventional P fertilizers such as phosphate rock and triple superphosphate can avoid the risk of secondary Cd contamination (Gu et al., 2020).

To realize the benefits of struvite for environmental and agricultural goals, its integration into agronomic systems requires consideration of soil testing methods used as a basis for P fertilization practices. To this end, we demonstrate that that residual struvite can dissolve during the soil test extraction step and thus impact STP values depending on STP method and soil properties (overestimation of 20–3900%, Table S1). Specifically, STP values may be overestimated by Mehlich-3 and Bray-1 used for acidic to circumneutral soils due to dissolution of particulate struvite in these strongly acidic extractants (pH $\approx$ 2.5). To assess the potential impact of residual struvite on STP values, considering the weathering of struvite in field soils and reasonably assuming 2 mg residual struvite (20% mass of a 2 mm diameter granule) in 1.0 g soil, the incomplete dissolution of residual struvite would contribute 22–123 mg P kg^{-1} soil to extractant-based STP values. For the sink-based Resin test, residual struvite would result in extreme increases of STP values (+309–413 mg P kg^{-1} soil). This increase in STP value is significantly greater than even excessive STP values (e.g., 100 mg kg^{-1}) and far above the economic optimum of STP values (e.g., 25–35 mg kg^{-1} for Mehlich-3) for crops such as maize, soybean, and wheat (Murrell et al., 2016). DThus, for agronomic STP methods, values would be overestimated, but for some of soils and tests the lower magnitudes of overestimation cannot be easily differentiated

Even assuming the small granules of the residual struvite can be physically removed from a soil sample prior to performing STP measurements, the need to hand-pick residual struvite remains questionable in a scenario of large-scale struvite adoption as a fertilizer and subsequent soil testing (Kok et al., 2018). Residual struvite will continue to supply P to crops in the subsequent cropping season (i.e., the second year after application) and hence ignoring its contribution could led to over-application of P fertilizer. In contrast, including a small residual granule of struvite in the soil sample would cause overestimation of STP concentrations and hence lead to insufficient P application. Consequently, the dissolution rate of struvite in field under various edaphic and cropping conditions should be quantified to gauge the persistence of residual struvite and its impact on STP. Future field-scale studies should therefore build on preliminary work relating STP values in soils amended with struvite to crop P response in order to establish guidelines for interpreting STP values for the use of struvite as a renewable P fertilizer.

5. Conclusion

As the first step toward large-scale implementation of struvite as a P fertilizer, this study assessed whether the residuality of struvite in soils may impact measured STP values. To this end, we evaluated how STP method and key edaphic properties (pH, clay content) influence dissolution of struvite particles remaining in the soil at the time of soil sampling (i.e., residual struvite), and identify consequences for interpreting STP results used to inform P fertilization rates. Struvite dissolution differed markedly by STP methods with up to 5-fold differences among methods. For acidic soils that are tested using Mehlich-3, Bray-1 and H3A-2, apparent dissolution was 19–401% higher for low versus high clay soils due to the adsorption of dissolved P from struvite. The impacts of soil pH on struvite dissolution depended on the clay contents for acidic soils. Depending on STP method, residual struvite dissolution

can overestimate STP concentrations by 20–3900%. Similar to phosphate rock, this study suggests the need to quantify *in situ* (i.e., field and seasonal scales) dissolution rates of struvite across soil and cropping conditions to evaluate persistence of residual struvite, its impact on STP values, and how these may merit STP interpretations specific to struvite-amended soils.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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