

**EVALUATING STRUVITE AS A PHOSPHORUS FERTILIZER: DISSOLUTION
DYNAMICS, SOIL TESTING CHALLENGES, AND PLANT BIOAVAILABILITY IN
CONTRASTING SOILS**

By

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ABSTRACT

Sweekriti Subedi, M.Sc., The University of Manitoba, April 2026. Evaluating struvite as a phosphorus fertilizer: dissolution dynamics, soil testing challenges, and plant bioavailability in contrasting soils. Advisor: Dr. Joanne Thiessen Martens

Recovering phosphorus (P) from wastewater as struvite offers a sustainable, slow-release alternative to finite phosphate rock fertilizers. However, struvite's low water solubility challenges standard soil testing protocols, potentially decoupling soil test P (STP) from actual plant availability. This thesis investigated the dissolution dynamics, laboratory extractability, and plant bioavailability of struvite compared to soluble monoammonium phosphate (MAP) in two contrasting soils: an alkaline clay and an acidic sand. Through a 140-day soil incubation study and a two-phase plant bioassay utilizing buckwheat (*Fagopyrum esculentum* Moench) and Italian ryegrass (*Lolium multiflorum* Lam.), this research assessed how residual struvite granules, sample preparation methods, and plant-induced mobilization affect Olsen-P concentrations and subsequent crop P uptake.

The incubation study revealed that standard preparation methods, particularly soil grinding, artificially inflated early Olsen-P concentrations when residual struvite granules were retained in the sample. Physical fragmentation accelerated the dissolution of unreacted fertilizer by the chemical extractant that would otherwise remain stable. The subsequent bioassay demonstrated that while struvite efficacy was highly soil-dependent, matching MAP performance in the acidic sand but not significantly differing from unfertilized controls in the alkaline clay, the elevated Olsen-P readings in the struvite treatments were largely analytical artifacts rather than a reflection of true bioavailable P. When residual granules were removed prior to planting, ryegrass P uptake declined to control levels across both soils. Consequently, initial Olsen-P exhibited a weak correlation with actual plant P uptake ($r^2 = 0.24$). Collectively, these findings emphasize that

standard chemical extractions do not adequately capture the complex, time-dependent dissolution kinetics inherent to the granular form of recovered struvite. Furthermore, both the physical grinding of samples, which artificially accelerates dissolution and the direct extraction of P from residual granules can lead to significant overestimations of available P unless methodological adjustments are implemented.

FOREWORD

This thesis was prepared in manuscript format in compliance with the guidelines of the Department of Soil Science, University of Manitoba. This thesis is organized into four chapters. Chapter 1 serves as a general introduction and literature review, providing essential background on phosphorus (P) management in crop production and broader P cycling. It elaborates on the potential fertilizer value of recovered struvite, specifically addressing its inherently low water solubility, complex plant-soil interactions, and the resulting implications for standard soil testing methodologies. Chapters 2 and 3 are independent manuscripts. Chapter 2 (Effects of Residual Struvite Granules and Sample Preparation on Soil Test Phosphorus Across Contrasting Soils in an Incubation Study) investigates how laboratory grinding creates analytical artifacts that obscure struvite's actual dissolution dynamics in Olsen-P tests. Chapter 3 (Plant Phosphorus Uptake and Olsen-P Dynamics in Contrasting Soils Amended with Struvite) builds on this by using a growth chamber bioassay to compare these soil test results against actual plant P uptake across an alkaline clay and an acidic sand. Finally, Chapter 4 provides an overall synthesis providing a summary of the key experimental findings, discusses the broader agronomic and analytical implications for interpreting soil test results in systems utilizing struvite, and offers recommendations for future study.

Contribution of Authors

The contributions of the authors for Chapters 2 and 3 are as follows:

- Sweekriti Subedi: Conducted experiments, collected and analyzed data, interpreted results, wrote the original manuscript draft
- Joanne Thiessen Martens: Conceptualized the experiments, provided primary administration and supervision of the project, secured project funding, and reviewed and edited manuscript drafts.
- Francis Zvomuya: Helped in data analysis, and reviewed manuscript drafts
- Inoka Amarakoon: Helped in data analysis, and reviewed manuscript drafts

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1. INTRODUCTION

Phosphorus (P) is an essential macronutrient for global food production, yet modern agriculture relies almost entirely on the extraction of mined phosphate rock. This finite, non-renewable resource is becoming increasingly scarce and expensive, posing a long-term risk to global food security. Furthermore, global P reserves are geopolitically concentrated, leaving import-dependent agricultural regions vulnerable to supply chain disruptions and price shocks (Cordell et al., 2009; Brownlie et al., 2023). Compounding these supply risks, the inefficient use of applied P leads to agricultural runoff, combined with uncaptured P in municipal and industrial waste streams, frequently entering surface waters. This nutrient loading is the primary driver of severe eutrophication, leading to toxic algal blooms and the degradation of critical freshwater ecosystems, such as Lake Winnipeg in Manitoba (Schindler et al., 2012).

To simultaneously address resource depletion and environmental pollution, agricultural systems must transition toward a circular P economy. The recovery of P from wastewater and industrial effluents in the form of struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) represents a highly promising strategy (Kataki et al., 2016; Kooij et al., 2020). Struvite is a recycled, slow-release fertilizer (Rahman et al., 2014) distinguished by its characteristically low water solubility (Kratz et al., 2019). Its unique dissolution kinetics provide distinct environmental and agronomic benefits, reducing the immediate risk of P runoff and seedling toxicity while offering a sustained nutrient supply over the growing season or subsequent crop rotations (Kataki et al., 2016; Talboys et al., 2016).

Despite these benefits, integrating struvite into conventional farming systems presents significant methodological and agronomic challenges. Standard diagnostic tools, particularly rapid soil test P

(STP) methods like Olsen-P, were historically designed and calibrated for highly soluble conventional fertilizers (Schoenau & O'Halloran, 2008; Olsen et al., 1954). Because struvite physically persists in the soil as residual granules, standard chemical extractants can artificially dissolve the fertilizer during laboratory analysis. This extraction artifact severely inflates test results, decoupling soil test values from actual plant availability and leading to inaccurate fertilizer recommendations (Gu et al., 2021). Accurately managing struvite requires a modernized understanding of how it interacts with specific soil properties, fertilizer particle size, root exudates, and laboratory sample preparation.

The remainder of this chapter is organized to systematically review these dynamics. Section 1.1 outlines the role of P in agriculture and the environment, followed by a review of P cycling in Section 1.2. Sections 1.3 and 1.4 detail the general chemical properties of struvite and its characteristically low solubility. Section 1.5 examines struvite's behaviour within the soil, while Section 1.6 evaluates its agronomic value and the critical influence of particle size. Section 1.7 reviews how plant interactions, such as root exudation and crop species selection, drive agronomic P uptake. Section 1.8 details the methodological implications and limitations of current soil testing protocols. Finally, Section 1.9 presents the overall objectives and hypotheses of this research.

1.1. Phosphorus in Agriculture and the Environment

Phosphorus is a primary growth-limiting nutrient in many soils; only 10-25% of applied P is directly available to crops in the year of application, with the rest retained by minerals or organic matter, prompting routine over-application of fertilizers (Yang et al., 2025). Phosphorus deficiency significantly harms plant health, leading to stunted growth, smaller roots, and delayed maturity. It also makes it difficult for plants to absorb other essential nutrients, such as nitrogen, which

ultimately limits crop yields. This scarcity is a major issue in semi-arid to humid regions such as the Canadian Prairies and areas with calcareous soils where adequate fertilization and improved efficiency are essential to prevent soil degradation and maintain production (Heuer et al., 2017).

Recent literature emphasizes the urgent need to improve P use efficiency (PUE) in agriculture because natural phosphate rock reserves are finite and facing geopolitical supply risks (Nesme & Withers, 2016). To solve this resource scarcity, researchers are focusing on ‘closing the loop’ by recycling P from waste streams. Key technologies currently being developed include recovering P from sewage sludge and aquaculture waste, as well as using struvite precipitation to extract nutrients from wastewater (Kooij et al., 2020). Beyond the challenges of resource scarcity, the environmental impact of current P management is a significant concern. Agricultural P runoff is a primary driver of water pollution, causing harmful algal blooms and oxygen-depleted ‘dead zones’ in water systems ranging from local streams to coasts (Wurtsbaugh et al., 2019). Even minor increases in P levels above safety thresholds can severely degrade freshwater quality, a problem primarily driven by inorganic fertilizers, which account for 47% of the global P load in large lakes and are rising most rapidly in developing countries (Fink et al., 2018).

1.2. Phosphorus Cycling

Unlike carbon or nitrogen, the P cycle is unique because it lacks a significant gaseous phase. Consequently, P movement and transformations occur exclusively in solid or liquid phases. According to Weil & Brady (2016), the primary natural source of P is rock phosphate entering terrestrial systems via tectonic uplift and subsequent weathering. These authors further note that over time P is then added to the soil through the slow dissolution of rock phosphate, the breakdown of organic matter (OM), and from the settling of dust suspended in the atmosphere. A vast majority

of soil P exists in forms that plants cannot absorb, despite high total abundance in nature. This unavailability frequently makes P a limiting factor for plant productivity in managed and natural landscapes (Shen et al., 2011).

Limited natural P availability necessitates the use of fertilizers to maximize crop production. Mined rock phosphate is the primary source for this, accounting for roughly 90% of all P fertilizers (Cordell et al., 2009). While early models predicted that global rock phosphate production would peak around 2030 (Cordell et al., 2009), subsequent re-evaluations of reserves particularly in North Africa have extended the projected timeline for depletion to several centuries (Geological Survey, 2024). However, despite this abundance in physical reserves, the supply chain remains highly vulnerable. The concentration of over 70% of the world's remaining high-quality reserves in a few geopolitical regions creates significant risks of price volatility and supply disruptions, as evidenced by recent market shocks (Brownlie et al., 2023).

Global agricultural systems depend heavily on phosphate fertilizers to improve yields. To satisfy the food demands of a rapidly expanding population, farmers must continuously add P fertilizers to their fields. Despite heavy application, in calcareous soils, P availability is severely restricted by calcium precipitation, often resulting in a PUE of only 10-25% (Etesami, 2019). These reactions create complexes with low solubility, a broader process known as P retention that encompasses both precipitation and sorption (Ibrahim et al., 2022). This retention temporarily reduces P availability to plant roots, creating a critical need to enhance plant-available P using sustainable technologies. According to Liu et al. (2021), long term over-application results in the accumulation of P in intensively managed cropland which increases the risk of losses through soil erosion and runoff. Consequently, agricultural fertilizers and manure are major non-point sources of pollution,

directly linking farming practices to eutrophication and hypoxia in aquatic ecosystems (Liu et al., 2021).

Recognizing this critical disconnect between finite phosphate reserves and the growing environmental burden of nutrient pollution has renewed interest in closing the P loop. A circular economy framework prioritizes the efficient use of P already within the system. Instead of relying on new extraction, this strategy focuses on recovering and redistributing anthropogenic waste streams to agricultural lands where they are required (Nicksy et al., 2022). A closed-loop P economy is essential for long-term sustainability, requiring two main strategies. First, we must improve PUE through crop breeding and better farming systems that utilize ‘legacy’ soil P, the substantial P reserves that have accumulated in agricultural soils due to decades of fertilizer and manure applications exceeding crop removal (Sharpley et al., 2013). Second, we must prioritize the recycling of nutrients. There is broad consensus that recovering P from waste streams is critical for managing the global cycle (Hall et al., 2020). One effective method for P recovery is precipitating struvite from wastewater.

1.3. General Properties of Struvite

Struvite is a white crystalline mineral composed of equal molar concentrations of magnesium, ammonium, and phosphate (Yilmazel & Demirer, 2011). Struvite contains significantly lower concentrations of impurities and toxic metals, such as Cd, Ni, Zn, and Cr, compared to triple superphosphate (TSP) (Hall et al., 2020). Due to this high purity and its ability to meet strict regulatory standards, struvite recovered from human waste has recently been approved for use in organic farming in Europe (Kokulan et al., 2024) and Canada (Canadian General Standards Board, 2026).

Commercially, Ostara Nutrient Recovery Technologies, Inc. (Vancouver, Canada) uses their WASSTRIP (waste-activated, sludge-stripping technique to remove internal P) processes to chemically precipitate struvite from anaerobic digestors using their patented Pearl technology (Liu et al., 2013). Crystal Green Pearl® (5-28-0 with 10% Mg) is a commercial-grade, high-purity granular fertilizer manufactured by Ostara Nutrient Recovery Technologies (Ostara Nutrient Recovery Technologies Inc.). However, a practical drawback to wide-scale commercialization is that current precipitation technologies still require high operational costs and energy consumption (Ghosh et al., 2019).

Researchers have evaluated the fertilizer value of struvite recovered from various waste streams, including sewage sludge (Mehta et al., 2018), dairy manure (Hilt et al., 2016), and swine manure (Katanda et al., 2019). While chemically pure struvite inherently has a consistent theoretical P content, the actual P concentration and purity of the recovered product can fluctuate. Although the chemical composition of the initial waste stream poses challenges, the final purity is dictated primarily by the efficiency of the specific pre-treatments and crystallization processes (Kataki et al., 2016).

1.4. Low Solubility of Struvite

Struvite is considered a slow-release fertilizer (Rahman et al., 2014) due to its limited water solubility. According to Kratz et al. (2019) only 6.6% of the P in struvite is water-soluble. This stands in contrast to common P sources like monoammonium phosphate (MAP) and TSP, which dissolve rapidly in water. The water solubility of Crystal Green® is approximately 4% (Ostara Nutrient Recovery Technologies Inc.). However, low water solubility does not mean the nutrient

is entirely unavailable; for instance, its dissolution in citric acid is comparable to standard ammonium phosphate fertilizers (Rech et al., 2018).

Beyond its physical form, the dissolution behaviour of struvite is heavily governed by the physicochemical properties of the soil, primarily pH and P-sorption capacity. Struvite crystals are characterized by a low specific gravity of 1.7 and solubility behaviour that is highly pH dependent (Degryse et al., 2017; Everaert et al., 2018). Struvite becomes significantly more soluble under acidic conditions; specifically, solubility decreases gradually as pH rises from 7 to 8.5 (Bhuiyan et al., 2007). Degryse et al. (2017) quantified this dependency, finding that in acidic soils, granules dissolved at a rate of 0.43 mg struvite day⁻¹, but in alkaline conditions, this rate declined sharply to just 0.03 mg day⁻¹. This delayed initial solubility in high-pH conditions can restrict early-season P diffusion, which sometimes gives highly soluble fertilizers an early-season growth advantage over struvite, as observed in canola (*Brassica napus* L.) grown in alkaline soil (Ackerman et al., 2013).

Although this delayed release dynamic requires careful management for early-stage crop development, struvite's low solubility provides a distinct environmental advantage. By releasing P slowly, struvite helps minimize the risk of eutrophication (Kratz et al., 2019). For example, using a rainfall simulation experiment, Everaert et al. (2018) found that when simulated rainfall occurred in the days immediately following fertilizer application, 42% of the P applied as MAP was lost to surface runoff. In contrast, the slow-release struvite exhibited runoff losses of less than 3% under the same timeframe. Furthermore, Thiessen Martens et al. (2023) demonstrated that after 20 days of soil incubation, Olsen-P concentrations near struvite granules remained significantly lower (30-122 mg kg⁻¹) than those near MAP granules (435-1063 mg kg⁻¹). This restricted initial P diffusion

limits the pool of vulnerable, highly soluble P in the soil right after application, thereby substantially reducing the risk of water pollution.

1.5. Struvite Behaviour in Soil

How struvite releases P in soil is a complex process governed by several interacting factors, primarily soil pH, texture, and the concentration of elements like calcium and magnesium. Interestingly, while environmental factors often drive nutrient cycling, the dissolution of struvite appears largely unaffected by temperature. In a controlled soil incubation, Thiessen Martens et al. (2023) demonstrated that the behaviour of P from both struvite and MAP fertilizers remained consistent across cold (4°C) and warm (22°C) conditions. This indicates that intrinsic soil properties and the physical characteristics of the fertilizer are the primary drivers of P dissolution, rather than seasonal temperature fluctuations.

Beyond soil properties, the physical form of the fertilizer itself is a primary driver of release rates. Because struvite dissolution occurs primarily at the particle surface, the higher specific surface area of smaller particles fundamentally accelerates the rate of P release in both soil and solution (Degryse et al., 2017). Thermodynamic and kinetic modeling confirms this relationship, demonstrating that total dissolution resistance increases with particle size; smaller particles exhibit significantly lower resistance to both surface reactions and diffusion (Chen et al., 2023). In a soil incubation study and accompanying pot experiment, Degryse et al. (2017) observed that finely ground struvite mixed uniformly through the soil dissolved quickly, achieving an agronomic effectiveness similar to MAP. In contrast, granular struvite dissolved much more slowly (0.03-0.43 mg d⁻¹) and was notably less effective, particularly in alkaline environments where solubility is heavily restricted (Everaert et al., 2018).

When applied in granular form, struvite's P diffusion in soil is much slower and occurs over a much smaller spatial area compared to highly soluble sources like MAP (Thiessen Martens et al., 2023; Degryse et al., 2017). This restricted diffusion is further influenced by soil texture and mineralogy. Generally, soils with higher clay and silt content retain P more strongly, reducing immediate availability compared to sandier soils (Ruiz-Navarro et al., 2023). In the calcareous soils of Manitoba, for example, P retention is driven by exchangeable calcium, magnesium, and clay content (Ige et al., 2005). While these factors bind P, a high retention capacity can also act as a "P sink." By rapidly removing dissolved phosphate from the soil solution and maintaining a concentration gradient, these soils can encourage the further solubilization of struvite granules (Degryse et al., 2017).

Evaluating the true agronomic effectiveness of these dissolution dynamics can be challenging due to the baseline P status of the test soils. In a meta-analysis comparing struvite to soluble fertilizers, Hertzberger et al. (2020) found that testing fertilizers in soils with inherently high available P creates a positive confounding effect. If the soil already has abundant P, the crop can compensate for struvite's slow initial release by drawing directly from existing soil reserves. This masks the fertilizer's delayed availability, making struvite falsely appear just as effective as highly soluble P sources during early crop development.

1.6. Value as a Fertilizer, Including Consideration of Granule/Particle size

Although struvite has low water solubility, its P is readily labile under specific conditions. Using long-duration resin and bicarbonate extractions, Meyer et al. (2018) demonstrated in a controlled environment that over 95% of total P could be recovered from finely ground struvite. This high

potential availability suggests that struvite can be as effective as conventional fertilizers, provided its dissolution is not physically restricted by its form.

The physical form, specifically the trade-off between powder and granules, is a primary controller of the P release profile in struvite. In a study assessing nitrous oxide emissions due to struvite application, Yang et al. (2023) found that powdered struvite resulted in higher plant P uptake and yields compared to granular forms, performing similarly to conventional soluble fertilizers. This increased efficacy is attributed to the higher specific surface area of the powder, which reduces dissolution resistance. Similarly, utilizing a six-month incubation study across four varying soil textures, Anderson et al. (2020) quantified the profound impact of physical form of struvite on dissolution. After just two weeks, finely ground struvite yielded a 76.2 mg kg^{-1} average increase in initial water-soluble P, compared to only 14.0 mg kg^{-1} from intact struvite pellets. However, over the full six-month period, the available P from the ground treatment gradually decreased, whereas the intact pellets provided a sustained, steadily increasing P release.

Meta-analyses and multi-study syntheses confirm that struvite dissolution is inversely proportional to particle size (Hertzberger et al., 2020; Degryse et al., 2017). Because of this rapid dissolution, finely ground struvite often matches the efficacy of water-soluble P fertilizers during early vegetative growth. However, researchers must be cautious when interpreting these results; for instance, the pot study by Meyer et al. (2018) likely increased struvite's dissolution by finely grinding it to less than 120 micrometers, which increased its surface area. As noted by Degryse et al. (2017), such extensive grinding artificially increases surface area to a point that likely overestimates the dissolution and performance of the granular products typically used by farmers in the field. Consequently, there is a critical need to better understand how granular struvite

interacts with standard assays and to determine if current environmental and agronomic guidelines are applicable.

1.7. Plant Interactions and Agronomic Uptake with Struvite

The use of struvite as a potential alternative P source has been studied across several agronomic crops, including corn (*Zea mays* L.), soybean (*Glycine max* (L.) Merr.), wheat (*Triticum aestivum* L.), canola, and ryegrass (*Lolium* spp.), to analyze how its unique dissolution properties affect nutrient uptake. Overall, most studies confirm that struvite can perform comparably to commercial highly soluble P sources over a full growing season. In a greenhouse study using a P-deficient silt loam, Barak and Stafford (2006) reported that corn fertilized with struvite achieved similar dry matter production to corn treated with diammonium phosphate (DAP). Furthermore, the struvite-fertilized corn demonstrated a highly efficient aboveground P uptake, reaching 177% relative to the DAP treatment. Similar comparable efficacy has been observed in greenhouse trials using ryegrass; the application of struvite resulted in plant yield and P uptake levels comparable to those achieved with conventional MAP, positioning it as a viable, sustainable alternative (García-Díaz et al., 2024; Deinert et al., 2023). As Meyer et al. (2018) demonstrated using isotopic tracers in a greenhouse pot experiment, because plant uptake correlates closely with resin-extractable P rather than initial water solubility, it is the gradual dissolution over time, not immediate solubility that serves as the true driver of plant P uptake.

Despite this overall efficacy, the slow dissolution of struvite presents a complex dynamic, offering distinct early-season agronomic challenges. Because it does not dissolve rapidly, struvite can fail to supply sufficient P during the critical early growth stages of the crop. For example, in a controlled growth chamber study, Ackerman et al. (2013) reported a 28% decrease in early canola

biomass with struvite compared to MAP, attributing this reduced efficacy to the soil's high pH (7.7) substantially inhibiting the fertilizer's initial dissolution. Similarly, Talboys et al. (2016) demonstrated that when struvite granules were placed with the seed, they did not supply P as rapidly as DAP during the early vegetative stages of spring wheat. However, by harvest time, struvite matched DAP in terms of P uptake, yield, and fertilizer recovery, even though only 26% of the mass of applied struvite granules had dissolved.

However, this restricted early-season solubility also provides specific agronomic advantages. Because struvite has minimal salt toxicity, it can be placed directly with the seed without causing seedling injury. This safe, localized placement actively encourages root proliferation directly around the granules (Talboys et al., 2016; Katanda et al., 2019). Independently, the initially low P supply provided by struvite can be highly advantageous for mycorrhizal-dependent crops. By utilizing a tomato genotype model in P-deficient soil, Di Tomassi et al. (2021) demonstrated that wild-type mycorrhizal plants fertilized with struvite produced 24% greater shoot biomass than those fertilized with highly soluble MAP. This suggests the slow-release nature of struvite actively synergizes with early arbuscular mycorrhizal colonization to enhance long-term nutrient scavenging.

As demonstrated by Talboys et al. (2016), crops ultimately often overcome the early-season lag in P availability as the growing season progresses. This recovery is not merely a function of time, but of the plant's active role in altering the soil chemistry. Plants actively influence P dynamics through root exudates and continuous uptake patterns, essentially acting as a biological "sink" that maintains a concentration gradient to continuously drive the dissolution of the slow-release granules (Richardson et al., 2009). This sustained availability provides notable residual benefits in multi-year crop rotations. Katanda et al. (2016) evaluated sequential cropping in a greenhouse and

found that while struvite produced similar dry matter yield to MAP in the first crop phase for both canola and wheat, it produced significantly greater yields in the second and third crop phases, indicating superior long-term P availability.

To maximize the utilization of slow-release soil P, selecting crops with inherent genetic capabilities for P-mining is a viable strategy. Research from controlled growth chamber experiments indicates that buckwheat (*Fagopyrum esculentum* Moench) can efficiently utilize Ca-bound P fractions, such as rock phosphate, due to localized rhizosphere acidification. By testing both sand-culture and highly calcareous soils, Zhu et al. (2002) demonstrated that this acidification mechanism allowed buckwheat to take up nearly 10-fold more P from rock phosphate than spring wheat. Furthermore, in a 30-day pot experiment using sandy soil, Mäkelä et al. (2020) confirmed that buckwheat develops robust root systems that exhibit highly efficient P uptake even when supplied with alternative, non-synthetic P sources. This scavenging ability is driven by marked rhizosphere acidification (Raniero & Santner, 2023). Talboys et al. (2016) found that struvite solubility is greatly enhanced in the presence of these organic acid anions; consequently, buckwheat was significantly more effective at mobilizing and utilizing P from struvite granules than spring wheat. Because these root-induced changes actively mobilize struvite P, growing strong organic-acid exuders can effectively "prime" the soil, increasing P availability for a subsequent crop. Therefore, incorporating such crops into rotation systems presents a highly practical approach for maximizing the agronomic efficiency of struvite.

However, the efficacy of buckwheat as a net P-mobilizer for subsequent crops is highly conditional and heavily dependent on biomass management. While buckwheat efficiently scavenges P, short-term evaluations, such as a 21-day growth study by Possinger et al. (2013), indicate that this enhanced uptake does not necessarily translate to increased P availability in the bulk soil. Instead,

the plant can act as a significant P sink, rapidly accumulating the mobilized P into its own tissue and leaving the soil depleted if the biomass is physically removed. Furthermore, even if the biomass is retained as green manure, its subsequent mineralization can be hindered by tissue quality. As demonstrated by Hansen et al. (2022) using radioisotope tracing, the high carbon-to-phosphorus (C:P) ratio of mature buckwheat residue can trigger rapid microbial P immobilization, which can temporarily restrict inorganic P availability for a subsequent crop. Similarly, Arcand et al. (2011) demonstrated that buckwheat green manures often produce only a marginal increase in soil P bioavailability, a response observed strictly when very high rates of rock phosphate were applied. Despite these management challenges, sequentially planting a strong organic-acid exuder like buckwheat can actively mobilize struvite-P, theoretically enhancing biological P availability for a subsequent responsive crop like Italian ryegrass (*Lolium multiflorum* Lam.) (Zhu et al., 2002; Talboys et al., 2016; Meyer et al., 2018; Raniro & Santner, 2023).

1.8. Implications for Soil Testing and Methodology for Struvite

Soil test P methods were originally designed for soils treated with highly water-soluble P fertilizers like MAP. Upon application to alkaline or calcareous soils, the soluble P from these conventional fertilizers dissolves rapidly and subsequently precipitates as metastable calcium phosphates, primarily dicalcium phosphate dihydrate (DCPD) and anhydrous dicalcium phosphate (DCP), before slowly transitioning into less soluble forms like octocalcium phosphate (Beaton et al., 1963). The Olsen-P method is specifically designed to extract these secondary reaction products by utilizing a sodium bicarbonate solution to precipitate soil calcium, thereby driving the dissolution of labile P from compounds like DCPD and OCP (Schoenau & O'Halloran, 2008; Olsen et al., 1954).

The primary purpose of STP methods is to estimate the pool of plant-available P and calibrate those measurements to predicted crop responses (Mallarino & Blackmer, 1992; McKenzie et al., 2003). These calibrations form the basis for accurate fertilizer recommendations, allowing agronomists to determine the exact rate of fertilizer required to optimize yield without risking environmental losses. However, because these standard interpretations and critical thresholds were developed using highly water-soluble P fertilizers, they are often inadequate for making accurate recommendations in soils containing residual, slow-release sources like struvite (Gu et al., 2021).

Struvite is predominantly processed and applied in agricultural settings as a granular fertilizer. Commercially available struvite fertilizers (such as Crystal Green Pearl®) are manufactured as granules to ensure compatibility with existing agricultural handling, storage, and application equipment (Everaert et al., 2017). Struvite's low water solubility and granular application mean a significant portion of each granule may remain undissolved after a growing season, providing residual P for subsequent crops. This persistence is central to its slow-release behaviour but problematic for soil testing (Gu et al., 2021).

When standard extraction methods are applied to soils containing residual struvite granules, they sometimes underestimate the total P that will become available to the plant over the growing season, particularly if the soil samples are not ground prior to extraction. Across a three-year field trial evaluating a corn and soybean rotation, Thompson et al. (2013) evaluated broadcast applications of struvite versus TSP. They found a critical disconnect: while plant P uptake and yield from struvite were equal to or occasionally higher than TSP, the post-harvest soil tests (Bray-1, Mehlich-3, and Olsen) mostly showed no difference between the sources, and in some instances, reported lower residual P for struvite. While standard chemical extractions do not recover 100% of applied P even for highly soluble sources due to natural soil retention processes, the recovery

rate for soluble fertilizers is notably higher. For example, during a 20-day laboratory incubation using two contrasting low-P soils, Thiessen Martens et al. (2023) demonstrated that standard testing recovered roughly 40% of the added P from soluble MAP, compared to only 1-5% recovery from struvite in the same soil environment, noting that undissolved struvite remnants were carefully removed prior to extraction. This discrepancy highlights the fundamental limitation of using standard rapid extractions to evaluate slow-release fertilizers.

Conversely, the physical presence of residual struvite in soil samples can generate a contrasting methodological issue: an overestimation of immediate P availability. If residual granules are collected and subjected to the chemical extraction phase, the extractants themselves can artificially dissolve the intact fertilizer. Gu et al. (2021) demonstrated that common STP methods (Mehlich-3, Bray-1, Olsen, Resin, and Haney) partially dissolve intact struvite granules during the extraction process itself. This artificial dissolution inflated measured P by 20 to 3900% across various soil textures and pH levels. Furthermore, by testing these methods in an inert quartz sand medium, they proved that the chemical extractants alone independently dissolved 10 to 59% of the added struvite, artificially inflating availability regardless of soil chemistry. To prevent the overestimation of Olsen-P caused by the dissolution of residual fertilizer, Thiessen Martens et al. (2023) found it necessary to manually remove struvite granule remnants prior to extraction during their incubation work. Similarly, in a pot experiment evaluating banded fertilizers for maize grown in low-P sandy and loamy soils, Ferron et al. (2024) compared granular struvite against water-soluble DAP. Despite struvite resulting in lower crop P uptake than DAP, P concentrations in extractions with calcium chloride and ammonium lactate were artificially high. Again, this overestimation occurred because residual struvite granules dissolved during the chemical extraction process, inflating the test results and failing to reflect the lower actual bioavailability observed in the plants.

Collectively, these field and pot experiments demonstrate a critical decoupling between STP values and actual plant P uptake for struvite-amended soils.

This dynamic generates an analytical paradox, creating two opposing risks: the false inflation of immediate P availability if granules dissolve during extraction (Gu et al., 2021; Degryse et al., 2017), and conversely, the undervaluation of long-term supply if the test ignores slow-release dynamics (Kratz et al., 2019). Consequently, resolving these conflicting interpretations requires the selection of extraction methods and frameworks specifically calibrated to struvite's dissolution kinetics, soil pH, and the soil's P-fixing capacity (Degryse et al., 2017). Therefore, different guidelines are needed for interpreting STP values in soils treated with low water-solubility P fertilizer like struvite.

1.9. Objectives and Hypotheses

The overall objectives of this research are to investigate how struvite dissolves in soil, both in the presence and absence of plants, and to assess how this dissolution process influences plant-available P and the outcomes of soil tests in soils amended with struvite.

Chapter 2

Objective: The specific objective of this chapter was to assess how sample preparation, the inclusion or exclusion of residual struvite granules, and soil type impact STP concentrations in soil samples taken at various intervals over a 140-day incubation following struvite application.

Hypotheses: To address this objective, the following hypotheses were formulated and tested:

1. Including residual struvite granules in soil samples will result in higher Olsen-P concentrations compared to their removal.
2. Preparing samples by grinding will increase extractable P concentrations relative to unground samples.
3. Soil types will not affect the extractable P concentrations resulting from these sample preparation methods.

Chapter 3

Objective: The specific objective of this chapter was to examine the relationships between plant P uptake and soil Olsen-P concentrations in two contrasting soil types previously amended with struvite, both with and without the presence of plants.

Hypotheses: To address this objective, the following hypotheses were formulated and tested:

1. In soils treated with struvite, growing plants immediately after struvite application to the soil will increase P availability to a subsequent crop, compared to unplanted soils.
2. In soils treated with struvite, growing plants immediately after struvite application will lead to stronger correlations between plant P uptake and Olsen-P concentrations, compared to soils without plants.
3. In soils treated with struvite, plant P uptake will be better correlated with Olsen-P when residual struvite granules are removed from the soil, compared to when residual granules are retained.
4. The relationship between Olsen-P and plant P uptake will not be affected by soil type.

1.10. Thesis Structure

This thesis follows the formatting and organizational guidelines of the Department of Soil Science at the University of Manitoba. The thesis is outlined in four chapters, beginning with Chapter 1, which serves as a general introduction and comprehensive literature review. This is followed by two primary research chapters (Chapter 2 and Chapter 3), which are written in manuscript format. Finally, Chapter 4 provides an overall synthesis of the study, integrating the findings, and discussing their limitations and future research.

Chapter 2: Effects of residual struvite granules and sample preparation on soil test phosphorus across contrasting soils in an incubation study.

Chapter 3: Plant phosphorus uptake and Olsen-P dynamics in contrasting soils amended with struvite.

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2. EFFECTS OF RESIDUAL STRUVITE GRANULES AND SAMPLE PREPARATION ON SOIL TEST PHOSPHORUS ACROSS CONTRASTING SOILS IN AN INCUBATION STUDY

2.1. Abstract

Phosphorus (P) is an essential crop nutrient derived from non-renewable phosphate rock, and while depletion estimates vary, our continued reliance on it poses severe long-term risks to global food security. Struvite ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$) recovered from municipal wastewater offers a promising alternative due to low solubility, low contamination, and slow-release properties. However, these slow-release and low-solubility characteristics also pose challenges for standard soil testing protocols, as residual undissolved granules either artificially elevate extractable P concentrations or remain undetected, depending on sample preparation methods and solubility of the fertilizer compound in the extractant. This study evaluated the effect of residual struvite granules and soil grinding on Olsen-P concentrations over time in two contrasting soils: an alkaline clay and an acidic sand. An incubation experiment (A) was conducted with two soil types (20 g soil), four fertilizer treatments (i.e., MAP, Struvite with residual granules retained for extraction, Struvite with residual granules removed before extraction, and Control; P application rate 215 mg kg^{-1}), and two sample preparation methods (grinding or no grinding) over 140 days. A parallel experiment (B) used the same treatments but with 1 g of soil to allow a commercial lab to use a standard Olsen P extraction without grinding and subsampling, and with a reduced sampling schedule over 140 days. Experiment A showed that when residual struvite granules were retained in the soil sample for extraction, grinding produced very high early Olsen-P concentrations (182 mg kg^{-1}), whereas unground samples showed much lower values (54.5 mg kg^{-1}) because intact granules did not fully interact with the extractant. Under grinding, the Retained and Removed

treatments remained significantly different until Day 84; without grinding, they differed only on Day 1, indicating that physical fragmentation strongly drives early extractable P. Experiment B showed a similar pattern. Olsen-P in MAP treatments decreased steadily over time in both experiments, with no effect of grinding in Exp A. In Experiment B, soil type had a greater influence on Olsen-P concentrations in the Struvite-Retained treatments than on MAP treatments. These experiment-specific differences highlight how sample preparation and extraction conditions shape the interpretation of P availability from slow-release struvite fertilizers in soil tests.

2.2. Introduction

Phosphorus is an essential macronutrient for crop production; however, its primary source is mined phosphate rock, which is a finite and non-renewable resource. While early estimates warned that rock phosphate (PR) could be exhausted within 50 to 100 years (Cordell et al., 2009), recent assessments confirm that global reserves are sufficient. Instead, the primary threat to long-term food security arises from the extreme concentration of these reserves in just a few countries, which heavily exposes import-dependent regions to geopolitical instability and price shocks (Brownlie et al., 2023). Global fertilizer production for agriculture requires the annual extraction of approximately 19 million metric tonnes of elemental P from finite phosphate rock reserves (U.S. Geological Survey, 2024). Per capita demand for P has increased by 38% in the last 50 years (Cordell et al., 2022). However, because of inefficiencies within the food production and consumption cycle, 80% of the extracted P is lost from the cycle before reaching the consumer, while only 20% ultimately reaches the food consumed by the global population (Chen & Graedel, 2016). Much of this consumed P is subsequently excreted; when this human waste is not recycled back into food production, it often ends up in water bodies, causing water quality issues and

eutrophication. This problem is worsened by runoff from agricultural fields and animal production facilities (Childers et al., 2011).

Struvite ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$) is a P-rich mineral that forms through crystallization and precipitation from municipal wastewater and sewage sludge. Struvite stands out among recycled P products as a promising alternative for agricultural use because it contains very few contaminants and is economical compared to other P recovery methods (Schneider et al., 2019). Struvite recovery through precipitation or crystallization effectively excludes heavy metals, producing a product that meets regulatory standards (Kataki et al., 2016). Struvite is often recommended as a suitable slow-release fertilizer due to its low water solubility. Research has shown that struvite performs comparably to conventional, highly soluble P fertilizers such as monoammonium phosphate (MAP) and triple superphosphate (TSP) in terms of final yield, P uptake, and fertilizer recovery efficiency at harvest (Talboys et al., 2016). Recycling P from waste streams as fertilizer can close regional and global P cycles, reducing environmental losses and dependency on mined P fertilizers (Schröder et al., 2011). Struvite, as a recycled P fertilizer, not only contributes to the circular economy by recovering P from waste streams but can also reduce environmental impacts. Struvite, used instead of traditional P fertilizers, is expected to reduce P runoff from agricultural fields because it dissolves slowly in water. For instance, (Wilson et al., 2025) observed that P loss to snowmelt in the Northern Great Plains was consistently lower than predicted following struvite application. This should help farmers and policymakers meet P reduction goals for surface water bodies (Everaert et al., 2018). Struvite recovered from human waste (sewage) has recently been approved for use in organic farming in Europe (Kokulan et al., 2024) and Canada subject to strict composition and safety criteria (Canadian General Standards Board, 2026). One practical

drawback of struvite recovery is that current technologies are associated with high operational costs and energy consumption (Ghosh et al., 2019).

Struvite dissolution is heavily influenced by the particle surface area; thus, the rate of P release in both soil and solution is accelerated by the higher specific surface area of smaller particles (Degryse et al., 2017). Incubation experiments across various soils demonstrated a distinct trade-off based on physical form: finely ground pellets yielded significantly higher initial water-soluble P, whereas unground pellets provided a sustained release over six months (Anderson et al., 2020). These results confirm that particle size is a critical factor in controlling the P release profile.

Soil physicochemical properties play a critical role in determining struvite's reactivity and bioavailability. Degryse et al. (2017) identify soil pH as a primary governor of dissolution, while also noting that dissolution rates are enhanced in soils with high P sorption capacity. In alkaline soil, the dissolution of finely ground struvite has been shown to be more rapid than that of granular struvite (Degryse et al. 2017; Everaert et al. 2017). Soil P retention capacity, which is driven by exchangeable calcium, magnesium, and clay content in the calcareous soils of Manitoba (Ige et al., 2007), plays a critical role in controlling struvite dissolution rates. High retention capacity effectively acts as a P sink, rapidly removing dissolved phosphate from the soil solution and maintaining a concentration gradient that encourages further release. This sink mechanism is known to enhance the solubilization of struvite (Degryse et al., 2017), mirroring the dynamics observed with conventional fertilizers like MAP in similar soil conditions (Duminda et al., 2017).

The rationale for this study arises from the need to deepen our understanding of struvite dissolution in soils. The physical presence of fertilizer granules in soil samples can alter soil P measurements, as the chemical extraction process may dissolve residual granules that were not actually plant-

available in the soil solution (Degryse et al., 2017). Gu et al. (2021) systematically showed that common soil test P (STP) methods (Mehlich-3, Bray-1, Olsen, Resin, Haney) partially dissolve intact struvite granules during extraction, inflating measured P by 20-3900% depending on method, soil pH, and clay content. Previous research found that STP concentrations were higher than expected when remnants of struvite granules were included in the soil samples (Degryse et al., 2017). Previous soil incubation and pot experiments revealed that finely ground soil samples, which included undissolved struvite granules, exhibited higher Olsen-P than soils amended with the soluble fertilizer MAP (Thiessen Martens, unpublished data). This suggests that soil tests, particularly when finely ground samples are used, may overestimate P availability by extracting P directly from undissolved struvite granules rather than from the soil solution. This occurs because the standard STP extraction solutions promote the dissolution of residual struvite granules during the extraction phase, thereby overestimating the pool of P available to plants (Gu et al., 2021). The impact of sample preparation, such as grinding, on the accuracy of these tests is another key factor, as grinding may artificially increase P availability during extraction by breaking down struvite granules. However, a critical challenge remains in accurately assessing P availability; previous studies suggest that standard soil tests may induce an extraction artifact by shifting chemical equilibria, thereby dissolving residual struvite granules during the analysis rather than measuring the P actually released into the soil solution (Degryse et al., 2017). Consequently, resolving these conflicting interpretations requires the selection of extraction methods and frameworks specifically calibrated to struvite's dissolution kinetics, soil pH, and the soil's P-fixing capacity (Degryse et al., 2017).

The objective of this study was to assess how sample preparation, the inclusion or exclusion of residual struvite granules, and soil type impact STP concentration in soil samples taken at various

intervals over a 140-day incubation following struvite application. We hypothesized that including residual struvite granules in soil samples would result in higher Olsen-P concentrations compared to their removal, as the Olsen extractant may extract P directly from the undissolved granules rather than only from the soil matrix. Furthermore, it was hypothesized that preparing samples by grinding would further increase extractable P concentrations relative to unground samples, as the physical reduction of particle size through grinding increases the specific surface area of the fertilizer, thereby accelerating the dissolution of struvite during the extraction process. To test these hypotheses, two soil incubation experiments, in which residual struvite granules were either retained in or removed from soil before extraction, were conducted using different scales of observation and analytical workflows. Experiment A utilized a 20 g soil mass to evaluate the effects of sample preparation (grinding vs. no grinding) and was conducted at the University of Manitoba to allow for a modified extraction approach. In contrast, Experiment B utilized 1 g soil with smaller struvite granules and no grinding treatments, with analysis performed by a commercial soil laboratory to provide a standard industrial comparison. Both experiments maintained a consistent P application rate of 215 mg P kg⁻¹ soil to ensure results were comparable across different experimental scales.

2.3. Materials and Methods

2.3.1. Experiment and Treatment Design

2.3.1.1. Experiment A

The study followed a randomized complete block design with three replications incorporating a factorial combination of two soil types, four fertilizer treatments, two grinding options, and 10 sampling dates over a 140-day incubation, requiring a total of 480 vials of soil. Detailed

descriptions of the soil characteristics and fertilizer treatments are provided below. Two soil processing treatments were applied prior to extraction: a "no grinding" and a "grinding" treatment. All treatment combinations were subjected to repeated measurements during an incubation period of 140 days, with sampling occurring on days 0, 1, 4, 7, 14, 28, 56, 84, 112, and 140.

Each of three shelves in the incubator served as a single replicate (block). Within each block, a full factorial combination of two soil types, four fertilizer treatments, and two grinding levels was randomly assigned. To maintain the integrity of each experimental unit, the vials associated with all 10 sampling dates for a specific soil-treatment combination were grouped together in the incubator as a single unit within their assigned block. This arrangement ensured that any micro-environmental variation within the incubator was captured by the blocking factor, while ensuring consistent conditions for each temporal sequence of a treatment.

2.3.1.2. Experiment B

This study followed a randomized complete block design with three replications incorporating a factorial combination of two types of soil, four fertilizer treatments, and five sampling dates over a 140-day incubation, requiring a total of 120 vials of soil. The soil and fertilizer treatments remained the same as in Experiment A. However, there was no sample grinding treatment, and a subset of sampling dates (Day 0, 7, 14, 56, and 140) was used. This experiment used the same approach to blocking and grouping of vials for sampling dates as Experiment A.

2.3.2. Treatment Factors

2.3.2.1. Soil Types

Two soil types were tested: one soil was alkaline clay (Libau location, 50°14'30.1"N 96°43'39.5"W; Dencross series, Gleyed Rego Black Chernozem); the other was acidic sand (Woodmore location, 49°07'15.2"N 96°54'12.0"W; likely Lenswood or Glenhope series, Gleyed Rego Black Chernozem). Soils were collected in autumn 2024 from the upper 15-cm layer of annual crop fields near Libau (clay) and Woodmore (sandy) in Manitoba. Field-moist soils were hand-sieved to pass a 10-mm screen to remove crop residue and stones. The clay soil was air dried for at least 12 h at 30 °C, whereas the sandy soil was not dried as the moisture content was already low. Both soils were stored for two months and then ground to pass through a 2-mm sieve prior to experiment set-up. To determine the moisture content of the air-dried soil, samples (~200 g) were oven-dried at 105 °C until a constant weight was reached. The water-holding capacity for each soil type was established by packing air-dry soil into transparent columns and incrementally adding water to the surface. The container capacity was defined as the maximum amount of water required to fully wet the soil column without resulting in drainage. Based on these parameters, the target moisture contents for the clay and sandy soils were set at 0.40 g g⁻¹ and 0.16 g g⁻¹, respectively.

2.3.2.2. Fertilizer Treatments

The fertilizer treatments were designed to quantify the release of P directly from struvite granules to the Olsen extractant by comparing samples with residual struvite granules retained to samples with residual struvite granules removed from soils incubated with struvite. Thus, the fertilizer treatments included: (1) Struvite with granules retained during Olsen-P extraction (Struvite Retained), (2) Struvite with residual granules removed from soil prior to Olsen-P extraction

(representing the P released from struvite to the surrounding soil) (Struvite Removed), (3) MAP with granules retained (MAP), and (4) an unfertilized control in which no fertilizer was added to the soil to serve as a baseline to compare the effects of the fertilizer treatments. All fertilizers were applied to the soils at the beginning of the incubation (Day 0). In the Struvite Retained treatment, visible fertilizer granules were left in the soil sample throughout the process. Conversely, in the Struvite Removed treatment, any visible granules were carefully removed from the soil at the sampling date before air drying the samples. The two fertilizers tested were the commercial struvite fertilizer Crystal Green Pearl® (N-P₂O₅-K₂O analysis 5-28-0 plus 10% Mg; Ostara Nutrient Recovery Technologies Inc., Vancouver, BC) and commercial MAP (N-P₂O₅-K₂O analysis 11-52-0, CropKing Inc., Lodi, OH). To ensure a relevant comparison, MAP was used as our baseline fertilizer due to its similar N:P ratio to struvite and because it is widely used in Manitoba.

2.3.3. Experiment Set-up

2.3.3.1. Experiment A

In this experiment, the fertilizer was added to the soil and thoroughly mixed before being packed into vials measuring 70 mm in height and 25 mm in internal diameter. Ten granules of struvite (1.5 mm) or two granules of MAP, totaling 4 mg P, were added to 20 g of soil in individual vials, achieving a P addition of approximately 215 mg P kg⁻¹ of soil. While this rate is high compared to field recommendations, it was chosen to represent the high P concentration found in the immediate reaction zone of a fertilizer band.

The vials were incubated at a temperature of 20°C, with deionized water added on Day 0 after fertilizer addition and as necessary throughout the incubation period to maintain the desired moisture level. The Day 0 vials were sampled immediately after fertilizer and water addition to

establish the initial treated concentration without incubating, while the remaining vials were organized within the incubator shelves.

2.3.3.2. Experiment B

A parallel experiment to Experiment A was conducted with the following distinctions: Use of two struvite granules (0.9 mm), a MAP solution, and 1 gram of soil per incubation unit, to achieve the same P addition rate as Exp A. To ensure precise and uniform delivery of P to each experimental unit, MAP was applied as a dissolved solution rather than in solid form. This method was necessitated by the extremely small mass of MAP required per vial (0.00094 g), which fell below the reliable weighing threshold of a standard analytical balance, even after the fertilizer was finely ground. A stock solution was prepared for each soil type by dissolving MAP in distilled water to achieve specific concentrations based on the soil's target moisture requirements. For the clay soil, a solution with a concentration of 0.0036 g ml⁻¹ was prepared, while a concentration of 0.0057 g ml⁻¹ was used for the sandy soil. An aliquot of the respective solution (0.26 ml for clay soil and 0.16 ml for sandy soil) was pipetted into each vial. This liquid approach ensured that the precise dose of MAP was delivered uniformly throughout the soil matrix while maintaining the target moisture content for each soil type.

The Day 0 vials were sampled immediately to establish the initial treated concentration without incubating, while the remaining vials were organized within the incubator compartments.

2.3.4. Post-Incubation Sample Processing

At each designated sampling date, a destructive sampling approach was employed, whereby the specific vials assigned to that date were removed from the incubator. For Struvite Removed

treatments, any remaining struvite granules were manually and meticulously recovered from the moist soil immediately upon sampling. Following the removal of these granules, the entire soil content of each vial was air-dried at 32° C. Once a constant air-dry weight was achieved, samples assigned to the grinding treatment were finely ground using a Christy & Norris Lab Mill (Christy Turner Ltd., Ipswich, UK) to pulverize soil aggregates and fertilizer granules. All the samples were stored for 20 weeks and analyzed for Olsen-P concentrations.

2.3.5. Sample Analysis

2.3.5.1. Experiment A

For unground samples, the entire soil content of each experimental subunit (20 g) was analyzed for Olsen-P without subsampling to ensure a representative ratio of fertilizer granules to soil. For ground samples, a 10 g subsample was analyzed due to soil mass reduction during the grinding procedure. The extraction followed the standard protocol described by Olsen et al. (1954), modified to accommodate the larger sample size. While the standard procedure typically utilizes 1 g of soil and 20 mL of extractant, this study maintained the required 1:20 soil-to-extractant ratio by scaling the volume of the extractant (0.5 M NaHCO₃, pH 8.5) according to soil sample mass: 400 mL for each 20 g unground sample, and 200 ml for each 10 g ground sample. The soil-extractant suspension for each sample was placed in a 1000 ml transparent plastic bottle, agitated on a reciprocal shaker for 30 minutes, and subsequently filtered through Whatman No. 42 filter paper. The concentration of P in the resulting filtrate was determined colorimetrically using the ascorbic acid-molybdate blue method of Murphy and Riley (1962). Following colour development, the absorbance of the samples was measured at 882 nm using a spectrophotometer. Final Olsen-P

concentrations were calculated by comparing absorbance readings against a standard calibration curve of known P concentrations.

2.3.5.2. Experiment B

Like Experiment A, the entire units were analyzed for Olsen-P without subsampling. The Olsen extraction and P determination were performed by a commercial soils lab. Olsen P was determined by extracting 1 g of soil with 20 ml of extractant in 50 ml Erlenmeyer flasks without grinding. The suspension was shaken for 30 minutes on a reciprocal shaker and filtered through Whatman No. 42 filter paper. Phosphorus concentration in the extracts was quantified using the molybdate blue method on a FIAlyzer-1000 series flow injection analyzer (FIALab, Seattle, WA).

2.3.6. Statistical Analyses

Experiments A and B were analyzed separately using the PROC GLIMMIX procedure in SAS® OnDemand for Academics (version 9.4, SAS Institute Inc., Cary, NC). For both experiments, a mixed-model ANOVA, with block as a random factor, was employed to evaluate the main and interactive effects of fertilizer treatment, soil type, and sampling date on Olsen-P concentrations; for Experiment A, the effect of soil grinding was also included as an additional fixed factor. All factors were modeled as fixed effects with sampling date modeled as a repeated measurement using the spatial power covariance structure to account for the unequal spacing between dates and the Kenward–Roger denominator degrees of freedom method. Prior to analysis, the assumptions of ANOVA were rigorously confirmed. The Shapiro-Wilk test was used to verify the normal distribution of residuals, with a W-statistic > 0.9. To meet the requirement for homogeneity of variance, residual plots were visually inspected and covariance among treatment groups was tested. For both experiments, data were analyzed using a lognormal distribution because the data

violated Gaussian assumptions. Results are reported as back-transformed LSMeans and standard errors to facilitate a meaningful interpretation of the P concentration values. When significant main effects or interactions were detected, least-squares means (LSMeans) were compared using Tukey's HSD test at a significance level of $\alpha = 0.05$. To explain the interactive effects including sampling date, the data were sliced by sampling date to compare treatment means within each specific time point with significant differences represented by the letters in the figures. For these interactions, the data were also sliced by the combined treatment factors other than date to determine if values changed significantly across dates (letters not shown in the figures).

2.4. Results

2.4.1. Soil Properties

The two soils used in this study differed in characteristics relevant to P availability, struvite dissolution, and P retention. Both soils were initially low in labile P pools but differed in other properties that could be expected to affect struvite dissolution and P adsorption and/or precipitation processes in soils (Table 2.1). In particular, the clay soil had higher pH and higher concentrations of organic matter, clay, Ca, Mg, and carbonate than the sandy soil. These properties suggest a strong potential for P retention through Ca-P precipitation and adsorption to clay surfaces. The lower pH in our sandy soil creates conditions that generally favour faster dissolution of struvite granules.

Table 2.1 Baseline chemical properties of experimental soils used in the incubation study.

Soil Property	Libau (Clay)	Woodmore (Sandy)
Olsen-P, mg kg ⁻¹	5	7
pH (in water)	8.1	6
Nitrate-N, kg ha ⁻¹	51.5	2.2
Exchangeable K, mg kg ⁻¹	334	62
Sulphate-S, kg ha ⁻¹	13.5	11.2

Exchangeable Ca, mg kg ⁻¹	6955	929
Exchangeable Mg, mg kg ⁻¹	1497	157
Calcium carbonate, CCE % ^a	11.7	0
CEC, meq 100g ⁻¹ (summation of cations) ^b	48.2	7.9
Organic matter, %	6.3	1.6
Texture Class	Clay	Sandy Loam / Loamy sand

^a CCE: Calcium Carbonate Equivalent

^b CEC: Cation Exchange Capacity

2.4.2. General Patterns in Olsen-P Across Experiments

Across both experiments, Olsen-P concentration in the unfertilized control remained close to initial background levels throughout the incubation period, confirming that changes in P availability were driven by fertilizer additions rather than incubation conditions alone. Fertilizer application increased Olsen-P substantially, but the magnitude and persistence of this increase differed depending on several factors, including fertilizer type (struvite vs. MAP), whether struvite granules were retained or removed, the use of grinding during sample preparation in Experiment A, soil type (clay vs. sandy), and the duration of incubation.

2.4.3. Experiment A (In-House Analysis)

Experiment A examined the combined effects of fertilizer treatment, soil type, grinding, and repeated measurements over time on Olsen-P concentrations. A highly significant Fertilizer × Grinding × Date interaction ($p < 0.0001$; Table 2.2) indicated that the effect of sample preparation depended strongly on both fertilizer type and incubation duration. Similarly, a significant Soil × Fertilizer × Date interaction ($p = 0.0002$) demonstrated that the P release patterns of the different fertilizers varied over time depending on the soil type.

Table 2.2 Analysis of variance (p-values) for the effects of soil type, fertilizer type, grinding, incubation time, and their interactions on soil Olsen-P concentration during Experiment A.

Factor	Olsen-P
	mg kg ⁻¹
Soil	
Libau	41.91 (0.84)
Woodmore	48.05 (0.96)
Fertilizer	
Control	12.20 (0.35)
MAP	115.50 (0.28)
Str Retained	92.17 (0.89)
Str Removed	31.25 (2.62)
Grinding	
Yes	48.09 (0.96)
No	41.87 (0.84)
ANOVA	p-values
Soil (S)	0.08
Fertilizer (F)	<0.0001
Grinding (G)	<0.0001
Date (D)	0.0004
S × F	<0.0001
S × G	<0.0001
S × D	<0.0001
F × G	<0.0001
F × D	<0.0001
G × D	<0.0001
S × F × G	<0.0001
S × F × D	0.0002 ^a
F × G × D	<0.0001 ^a
S × F × G × D	0.30

^a The highest-order significant interactions are plotted in Figure 2.1(Fertilizer × Grinding × Date) and Figure 2.2 (Soil × Fertilizer × Grinding).

2.4.3.1. Interactive effects of fertilizer treatment, sample preparation, and incubation duration

The analysis of variance revealed a significant three-way interaction, indicating that P dynamics were driven by complex relationships between the fertilizer type, physical disturbance (grinding), and time. In the MAP and Control treatments, sample preparation had no significant effect on

Olsen-P concentrations (Figure 2.1A, 2.1B). Values remained similar within each sampling date regardless of grinding, confirming that highly soluble fertilizers like MAP dissolve rapidly upon soil contact, leaving no residual granules to be affected by mechanical disturbance. Consequently, MAP exhibited a consistent temporal pattern: high initial extractable P followed by a 30–40% decline across the incubation, consistent with rapid dissolution and subsequent P fixation. Struvite Retained (ground) showed very high early Olsen-P concentrations but declined sharply after Days 28, likely due to the adsorption or precipitation of solubilized P into less extractable pools. In contrast, the Struvite Removed treatment showed a gradual increase in Olsen-P during the mid to late incubation period (after day 56) reflecting slow and progressive dissolution of residual granules releasing P into the soil. The unfertilized control remained stable at low concentrations throughout the 140-day period.

In contrast to MAP, grinding had a pronounced effect on measured Olsen-P concentration in the Struvite Retained treatment (Figure 2.1D), due to residual granules remaining in the soil at sampling. Across the early incubation period (0-56 days), grinding consistently produced significantly higher Olsen-P concentrations in the Struvite Retained treatment compared to no-grinding, often more than double the concentration observed in the no-grinding treatment (Figure 2.1D). The high Olsen-P concentrations in the ground Struvite Retained treatment reflect the physical breakup of struvite granules during grinding, which increases particle surface area and allows previously inaccessible P to dissolve into the Olsen extractant. In contrast, when samples were not ground, intact granules provided less exposed surface area for dissolution into the extractant, resulting in substantially lower Olsen-P concentration (generally $\leq 100 \text{ mg kg}^{-1}$). The influence of grinding diminished after Day 56, as the granules progressively dissolved or weathered and the difference between ground and unground samples narrowed. An isolated spike

in Olsen P was observed on Day 0 for the ground samples in the Struvite Removed treatment. Because the struvite granules were extracted manually prior to the experiment, it is highly probable that trace granules were inadvertently left behind in this specific sample. The subsequent grinding process would have pulverized these residual granules, vastly increasing their surface area and resulting in the temporary artifact of elevated extractable P. The rapid convergence of the ground and unground treatments by Day 1 supports this as an isolated incident rather than a sustained treatment effect.

The effect of granule inclusion is also evident in Figure 2.1, where the Struvite Retained and Struvite Removed treatments show distinct early incubation patterns. When samples were ground, the Struvite Retained treatment produced significantly higher Olsen-P than the Struvite Removed treatment from Day 0 through Day 112 (Fig 2.1 C and D). This sustained difference reflects the contribution of crushed granule fragments to the extracted P pool in the Struvite Retained treatment, as these particles were small enough to be dissolved directly during the Olsen extraction. Without grinding, however, the contrast between Struvite Retained and Struvite Removed was less pronounced but still distinct. The two treatments differed significantly until day 14, after which their values converged rapidly. In the absence of grinding, intact struvite granules exposed minimal surface area to the extractant and dissolved only slowly over time, limiting their contribution to measured P.

Even with minimal surface area, the Olsen extractant superficially dissolved intact granules during the testing procedure. Because little dissolution of struvite granules had occurred by Day 0, we can estimate this direct extraction artifact by looking at the initial difference between the unground treatments. Based on the difference between the unground Struvite Retained and Struvite Removed samples at Day 0, the extraction process itself caused an artificial release of approximately 56 mg

P kg⁻¹ directly from the intact granules. By Day 140, the difference between unground Struvite Retained and Struvite Removed treatments was only 8 mg P kg⁻¹, demonstrating that as the incubation progressed, this artifactual difference steadily decreased. Because the granules remained in the vials for longer periods, they underwent gradual dissolution. As the physical struvite granules dissolved and shrank over time, there was progressively less mass of intact struvite granules remaining to be artificially dissolved during the Olsen extraction, driving the convergence of the two treatments in the later days.

These results demonstrate that including undissolved granules in the extraction inflates Olsen-P when the granules are mechanically fragmented but may underestimate available P when granules remain intact and dissolve gradually. Collectively, these findings indicate that grinding can introduce a methodological bias into the determination of plant-available P in soils amended with struvite. Together, the trends shown in Figure 2.1 demonstrate the strong influence of physical granule condition, sample preparation, and fertilizer chemistry on measured Olsen-P dynamics over time.

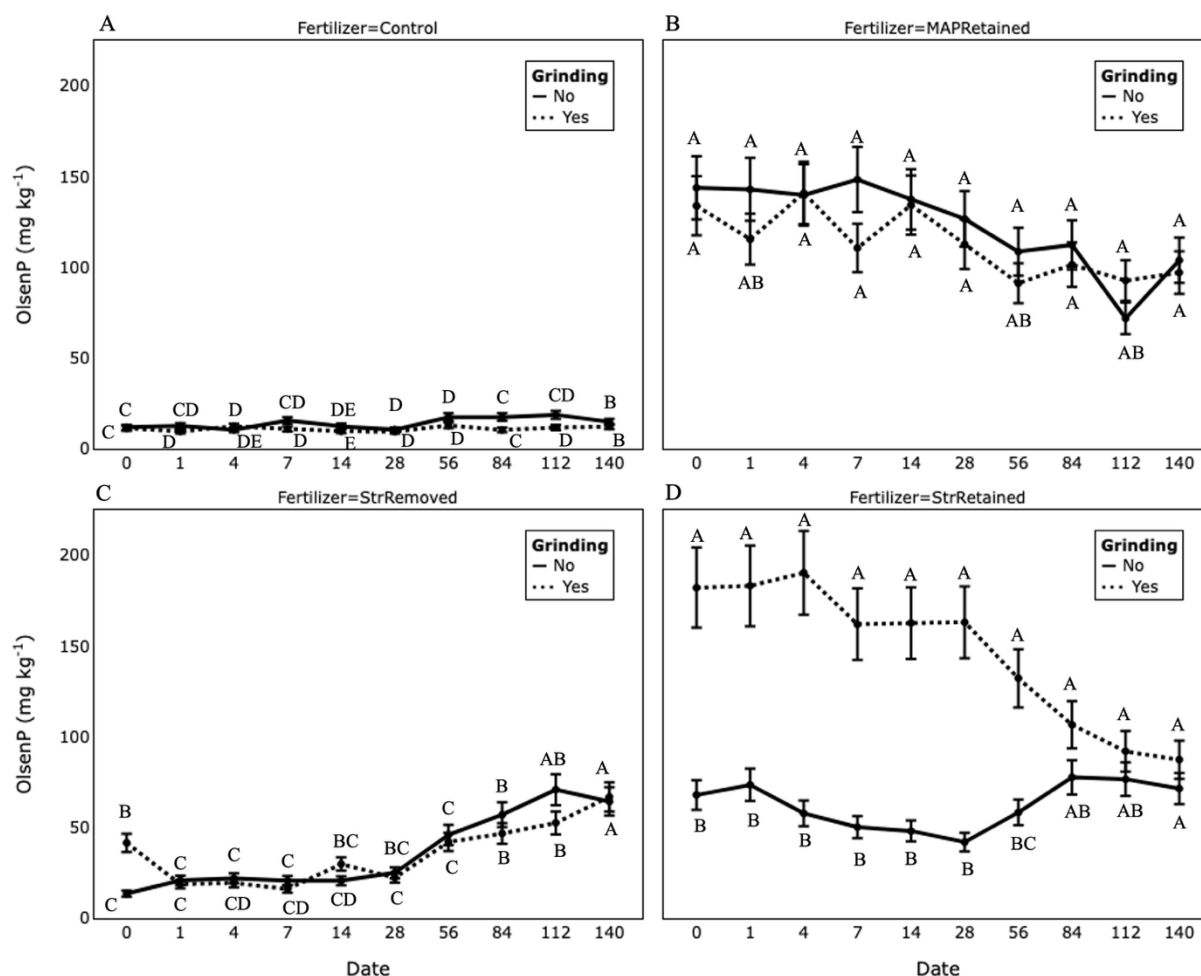


Figure 2.1 Temporal trends in Olsen-P concentration for four fertilizer treatments (Control (A), MAP Retained (B), StrRemoved=Struvite Removed (C), and StrRetained=Struvite Retained (D)) under two grinding conditions (Grinding = No and Grinding = Yes). Data points represent back-transformed treatment means at each incubation day, and error bars show back-transformed standard errors of the mean. Different letters indicate significant differences among grinding and fertilizer treatments within each sampling date according to Tukey's HSD test ($p < 0.05$). Unequally spaced dates are presented with equal spacing to allow clearer visualization of closely spaced early dates.

2.4.3.2. Interactive effects of soil type, fertilizer treatments, and incubation duration

The effect of fertilizer treatments on Olsen-P dynamics over time was also dependent on soil type.

During the early incubation period, MAP and Struvite Retained treatments produced comparably

high Olsen-P concentrations in both soils when averaged across grinding treatments, clearly separating them from the Struvite Removed and Control treatments (Figure 2.2). In the Woodmore soil, the Struvite Removed treatment maintained significantly higher Olsen-P concentrations than the unfertilized Control across all sampling dates, indicating an immediate and persistent release of P into the soil matrix prior to granule removal. In the Libau soil, the Struvite Removed treatment remained statistically similar to the Control during the earliest sampling dates (e.g., Days 0 and 7, Figure 2.2A and C) before diverging and becoming significantly greater later in the incubation. These patterns demonstrate that even when residual granules were removed prior to extraction, a measurable amount of P had already dissolved into the soil. Olsen-P in the Control stayed low throughout, confirming minimal background P supply. When evaluating temporal dynamics for the MAP treatment, in the clay soil (Libau), Olsen-P concentrations remained statistically stable over the 140-day incubation period (mean comparisons not shown). However, in the sandy soil (Woodmore), an initial period of statistical stability was followed by significant differences from preceding dates on Days 56 and 112, contrasting with the steady baseline seen in the clay (mean comparisons not shown).

The Struvite Retained treatments displayed distinct soil-dependent patterns. A crucial divergence occurred after Day 56 in Struvite Retained (Fig 2.2 D): while P availability in the sandy (Woodmore) soil stabilized and remained constant through Day 140, the clay soil continued to exhibit a significant decline. In the clay soil, the Struvite Retained treatment started at a similar initial concentration as in sandy soil but declined more steeply over time, indicating less stable P availability compared to the sandy soil. This contrast highlights the distinct capacity of the acidic sandy soil to maintain soluble P levels, whereas the alkaline conditions in the clay soil drove continuous P precipitation. In the sandy soil (Woodmore), the Olsen-P concentrations in the

Struvite Retained treatment were not significantly different from the MAP treatment (Figure 2.2B and D). Conversely, the statistical relationship between these two treatments in the clay soil (Libau) was highly fluctuating; the MAP and Struvite Retained treatments yielded similar Olsen-P levels during the earliest (Days 0–4) and later (Days 56–112) stages of incubation, but diverged significantly during the intermediate (Days 7–28) and final (Day 140) sampling dates.

In the sandy (Woodmore) soil, Olsen P concentrations showed a more pronounced and sustained increase in the struvite treatments (Figure 2.2C and D) compared to the clay (Libau) soil. While the MAP treatment in Woodmore soil peaked immediately at Day 0, the Libau soil exhibited a slightly delayed peak at Day 4 (Figure 2.2B), suggesting a slower initial dissolution or immediate early fixation in the clay soil. Struvite Removed again showed slow-release behaviour, with small increases at later times and little early availability (Figure 2.2C). This indicates that as P diffused from the granule sites, it remained extractable in the sandy soil but was increasingly fixed in the calcareous clay soil. These differences highlight the strong influence of soil chemistry on fertilizer dissolution patterns: the sandy soil maintained stable P concentrations over time, whereas the alkaline clay soil promoted rapid precipitation and loss of P availability.

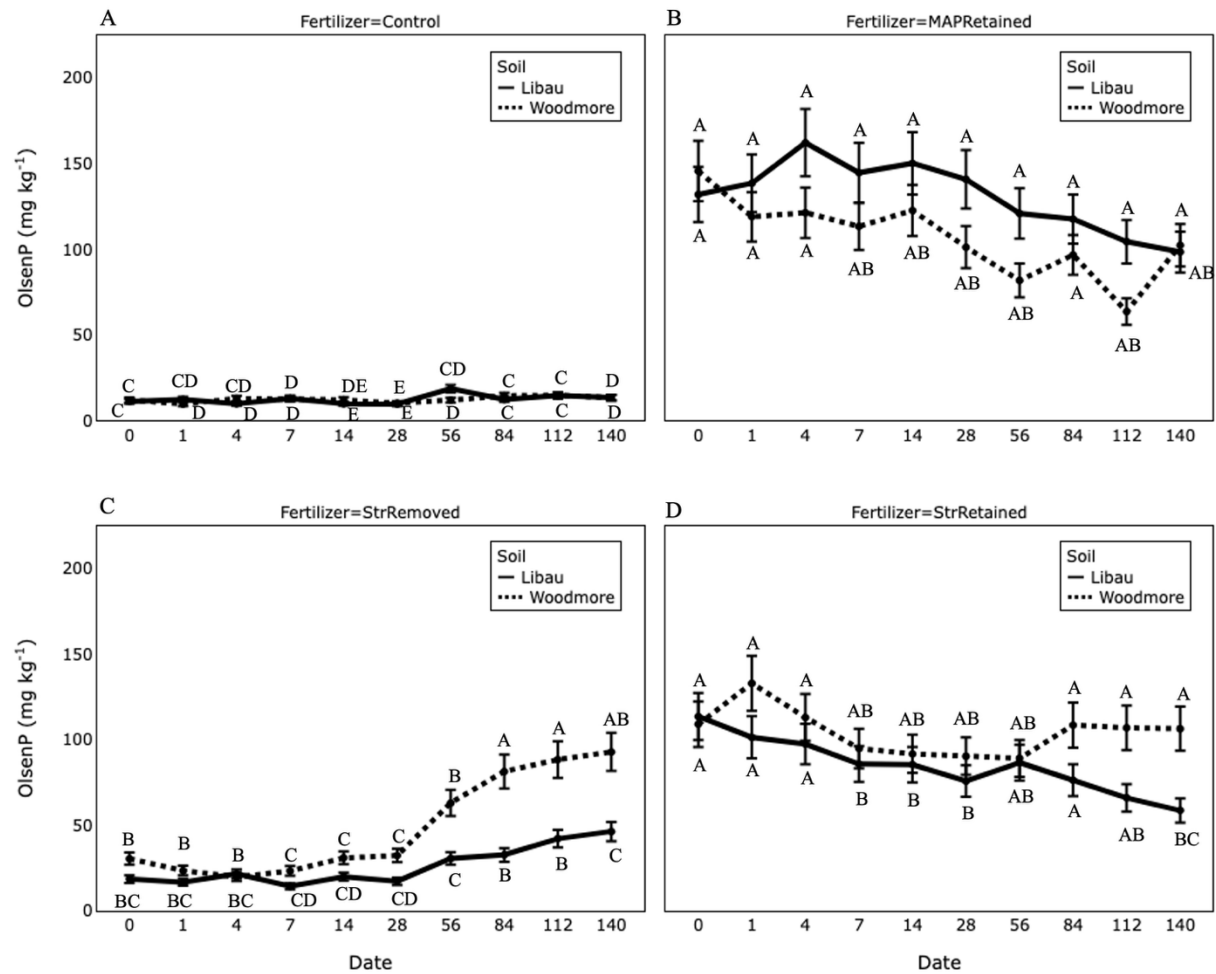


Figure 2.2 Temporal trends in Olsen-P concentration for four fertilizer treatments (Control (A), MAP Retained (B), StrRemoved=Struvite Removed (C), and StrRetained=Struvite Retained (D)) in two soil types (Libau (clay) and Woodmore (sandy)). Data points represent treatment means at each incubation sampling date, and error bars show standard errors of the mean. Different letters indicate significant differences among soil types and grinding treatments within each sampling date according to Tukey's HSD test (p < 0.05). Unequally spaced dates are presented with equal spacing to allow clearer visualization of closely spaced early dates.

2.4.4. Interactive Effects of Soil Type, Fertilizer Treatments, and Incubation Duration in Experiment B (Commercial Lab Analysis)

Experiment B evaluated fertilizer performance across two contrasting soils without grinding. A significant Soil $\times$ Fertilizer $\times$ Date interaction ($p < 0.0001$; Table 2.3) indicated that the relative effect of MAP, Struvite Retained, and Struvite Removed on Olsen-P concentrations changed over time and differed between soils.

Table 2.3 ANOVA results showing p-values for main effects and interactions affecting Olsen-P in Experiment B.

Factor	Olsen-P mg kg ⁻¹
Soil	
Libau	37.28 (1.21)
Woodmore	52.63 (1.70)
Fertilizer	
Control	9.04 (0.34)
MAP	117.36 (4.47)
Struvite Retained	146.58 (5.66)
Struvite Removed	24.77 (0.94)
Date	
0	41.34 (1.68)
7	42.75 (1.70)
14	47.83 (1.90)
56	42.44 (1.69)
140	47.57 (1.89)
ANOVA	p-values
Soil (S)	<.0001
Fertilizer (F)	<.0001
Date (D)	0.0009
S $\times$ F	<.0001
S $\times$ D	<.0001
F $\times$ D	<.0001
S $\times$ F $\times$ D	<.0001 ^a

^aThe highest -order interaction that is significant is plotted in Figure 2.3.

Across both soils in Experiment B, the Struvite Retained treatment consistently produced higher early Olsen-P concentrations than the Struvite Removed treatment, reflecting the contribution of

undissolved granules to extractable P during the initial incubation period (Figure 2.3C, D). However, despite this early separation, the Olsen-P concentrations in the two struvite treatments gradually converged within each soil type as dissolution progressed and granule effects diminished, becoming statistically similar by Day 56 in the alkaline clay (Libau) and Day 140 in the acidic sand (Woodmore).

The two soils exhibited distinctly different temporal responses. In the alkaline clay Libau soil, Olsen-P for Struvite Retained declined sharply, with concentrations approaching but remaining significantly above baseline levels by Day 56 (Figure 2.3D), indicating rapid adsorption or precipitation under high-pH, Ca-rich conditions. In contrast, the acidic, sandy (Woodmore) soil showed no statistically significant changes in Olsen-P for the struvite-retained treatment across the incubation period, and values remained comparatively stable. Although convergence occurred in both soils, the concentration at which treatments converged was much greater in the sandy soil than the clay soil (Figure 2.3C, D). In contrast, MAP treatment values were consistent across both soils (Figure 2.3B), indicating that soil properties exert a much stronger influence on struvite dissolution and P availability than on soluble MAP.

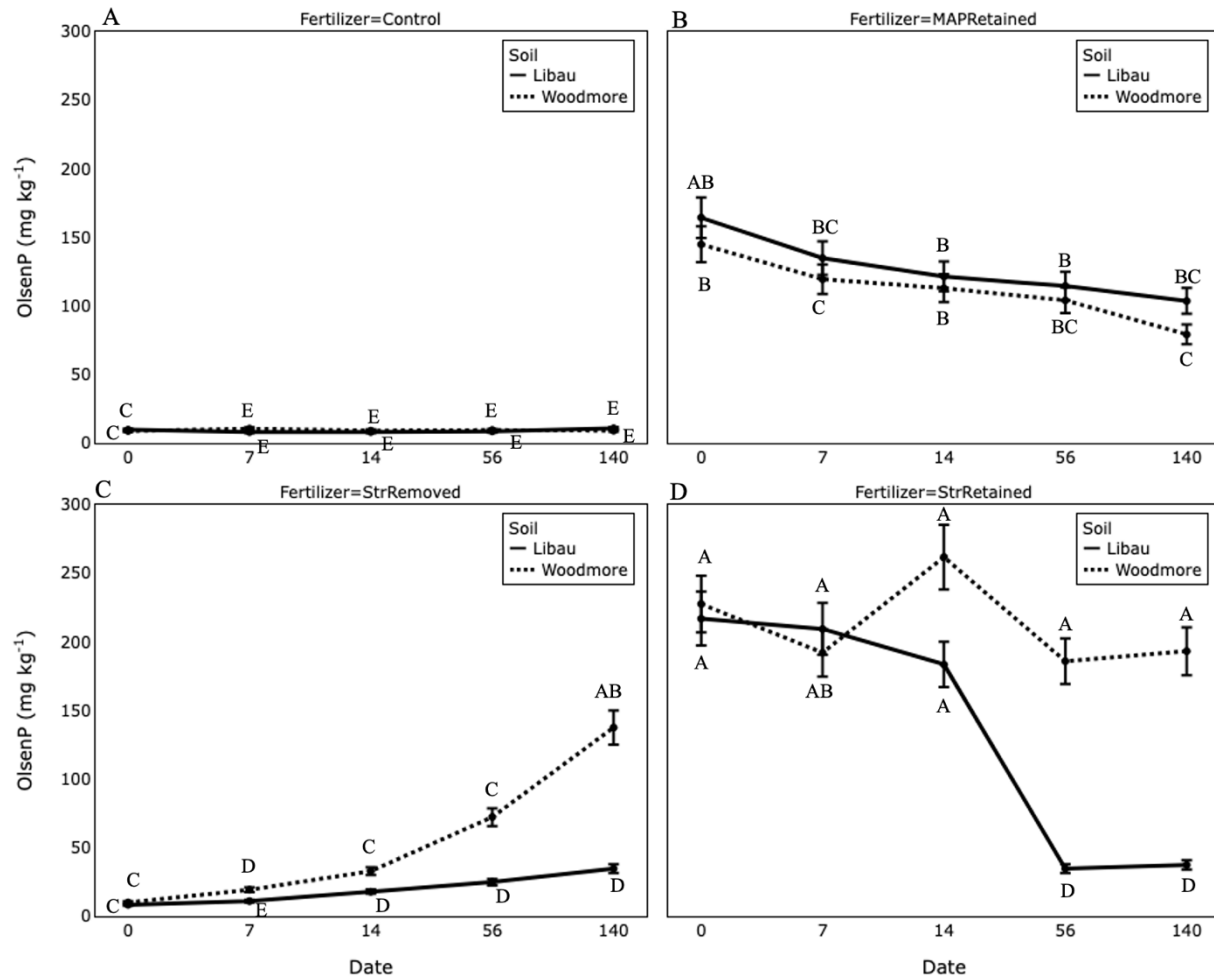


Figure 2.3 Temporal trends in Olsen-P concentration for four fertilizer treatments (Control (A), MAP Retained (B), StrRemoved=Struvite Removed (C), and StrRetained=Struvite Retained (D)) in two soil types (Libau (clay) and Woodmore (sandy)) over 140 days of incubation without plants. Data points represent treatment means at each incubation sampling date, and error bars show standard errors of the mean. Different letters indicate significant differences among soil types and fertilizer treatments within each sampling date according to Tukey's HSD test ($p < 0.05$). Unequally spaced dates are presented with equal spacing to allow clearer visualization of closely spaced early dates.

2.5. Discussion

2.5.1. Distinctive patterns in Olsen-P among fertilizer types

The inherent chemical solubility and physical form of the applied fertilizers played a decisive role in early P release dynamics. The immediate peak in Olsen-P concentrations observed in MAP-

amended soils is consistent with the high water-solubility of MAP, which creates a zone of high P concentration immediately surrounding the granule. As described by Degryse et al. (2017), this rapid dissolution drives P diffusion far into the surrounding soil matrix but also subjects the soluble P to rapid adsorption and precipitation reactions. This explains the sharp initial peak followed by a steady decline in MAP treatments, a pattern well-documented in incubations of soluble P fertilizers (Griffin et al., 2003; Kashem et al., 2004). Upon application, MAP granules rapidly dissolve, flooding the soil solution with orthophosphate (H_2PO_4^-). This creates a localized zone of supersaturation that drives rapid adsorption onto clay surfaces or precipitation with soil cations (calcium in alkaline soils; iron/aluminum in acidic soils), a process collectively known as P fixation (Hettiarachchi et al., 2006).

The Struvite Removed treatment provided the clearest evidence of natural struvite dissolution dynamics, revealing a gradual release of P into the soil over time. The acidic sandy (Woodmore) soil demonstrated considerable release of P from struvite granules to Olsen-extractable forms in the later incubation dates, whereas Olsen-P concentration in the Struvite Removed treatment in the alkaline clay (Libau) soil was still relatively low, even at 140 days of incubation (Figure 2.2C). This delayed release pattern aligns with the kinetic model proposed by Talboys et al. (2016), who demonstrated that while struvite is thermodynamically more soluble in acidic conditions compared to alkaline soil conditions, the release rate under acidic conditions is often still limited by the surface area of the granule and local saturation of the soil solution. Consequently, the Struvite Removed treatment effectively measured only the P that had dissolved and successfully diffused away from the granule into the surrounding soil matrix. This diffusion-limited release aligns with findings by Nascimento et al. (2018) and Thiessen Martens et al. (2023), both of whom

demonstrated that granular struvite exhibits highly restricted dissolution and minimal P diffusion compared to highly soluble sources like MAP.

Extracting soil with the struvite granule still present severely overestimates the true quantity of soil-soluble P, artificially inflating the labile pool through the direct extraction of the undissolved fertilizer (Nascimento et al., 2018; Gu et al., 2021). This methodological artifact perfectly explains why our Struvite Removed treatment yielded significantly lower Olsen-P concentrations than the Struvite Retained treatment during the first 28 days of incubation in experiment A, and until Day 56 in both soil types in experiment B. After this point, the concentrations in the Removed and Retained treatments converged, primarily because the residual struvite granule mass in the soil had significantly declined by the later sampling dates. The temporal trend of high initial labile P followed by a decrease suggests the gradual retention of applied P into less accessible fractions in soil across both fertilizer treatments in which granules were retained in the sample (MAP and Struvite Retained).

2.5.2. Mechanisms Behind P Release: Ground vs. Intact Struvite

The physical form of struvite also strongly affected the P release dynamics in the Olsen extraction, though these differences diminished over time. When soil samples were ground to a powder prior to extraction, the physical breakdown of the granules caused the struvite to behave nearly identically to the soluble MAP source in the early incubation stages (Figure 2.1). In contrast, the unground struvite samples maintained significantly lower Olsen P concentration than MAP until Day 56, reflecting the inherent slow-release nature of the intact struvite granules. This confirms that STP tests for soils amended with struvite are highly sensitive to sample preparation, as grinding artificially accelerates dissolution by increasing the specific surface area, consistent with

the dissolution kinetics described by Talboys et al. (2016). These authors demonstrated that the low surface area contact of intact struvite fertilizer granules with the soil results in slow initial P solubility and a delayed release of P during early growth stages. There was no statistical difference between the ground and unground Struvite Retained treatments from day 84 onward, in our incubation experiment, indicating that after 84 days, the physical form of the fertilizer (intact granule vs. ground powder) no longer limited P extractability in the soil test. This suggests that the granules had sufficiently dissolved or diffused into the bulk soil, reaching a state of chemical equilibrium where the rate of P supply was governed by soil-solution interactions rather than the initial surface area of the fertilizer.

The pronounced divergence in Olsen-P concentrations between ground and unground samples in the Struvite Retained treatment (Figure 2.1D) identifies a critical limitation in standard soil testing protocols when applied to slow-release fertilizers. This finding confirms our hypothesis that sample preparation via grinding would artificially increase extractable P concentrations relative to unground samples. The significant spike in Olsen-P observed in the ground Struvite Retained samples, often double that of the unground samples, is attributed to the pulverization of residual granules. This aligns with findings by Degryse et al. (2017) who demonstrated this physical phenomenon by directly comparing the dissolution of intact granular struvite versus ground struvite (<0.15 mm) homogeneously mixed into soil. In their incubations, intact granules dissolved slowly, restricted by the rate-limiting diffusion of P from the particle surface. In contrast, grinding the struvite exponentially increased the soil-fertilizer contact area, resulting in rapid dissolution comparable to highly soluble MAP. By pulverizing our soil samples prior to extraction, standard laboratory protocols inadvertently replicated Degryse's "ground and mixed" treatment, destroying

the physical barrier of the granule and allowing the alkaline Olsen extractant to rapidly dissolve P that would otherwise remain protected under field conditions.

The absence of a grinding effect in the MAP treatment (Figure 2.1B) and the Struvite Removed treatment (Figure 2.1C) confirms that the spike in the Struvite Retained treatment was not due to adsorbed or precipitated P, but rather the direct dissolution of the struvite fertilizer during analysis. This supports the conclusions of Gu et al. (2020), who observed that high P concentrations in struvite-amended soils are often misinterpreted as high availability when they actually reflect the dissolution of residual granules during extraction steps. The fact that the ground and unground measurements became similar after Day 56 in struvite removed treatment (Figure 2.1C) indicates that the grinding error is most severe in the early weeks, when the fertilizer granules are still mostly intact.

2.5.3. Effects of Residual Struvite Granule Inclusion on Olsen-P

The comparison between Struvite Retained (granules present) and Struvite Removed (granules removed) explains the dissolution kinetics of the fertilizer and the limitations of standard testing. Confirming our hypothesis, the inclusion of residual struvite granules in soil samples consistently resulted in significantly higher Olsen-P concentrations compared to treatments where granules were removed. This discrepancy was evident across both Experiment A and B, supporting the premise that the alkaline Olsen extractant solubilizes P directly from undissolved fertilizer granules, rather than measuring only the P extracted from the soil matrix.

In the ground Struvite Retained treatment, which represents the typical soil laboratory process, Olsen-P concentrations remained elevated during the initial dissolution phase (Figures 2.1 and 2.2), tracking closely with soluble MAP. This trend suggests that the standard method risks

overestimating immediate soil P availability, as unreacted struvite granules were actively dissolving during the extraction procedure itself. Degryse et al. (2017) described this phenomenon as an extraction artifact, noting that standard extractions (like Olsen P) shift the chemical equilibrium, causing undissolved struvite to dissolve during the test itself rather than reflecting the P released into the soil solution. As noted by Gu et al. (2020), the alkaline bicarbonate extractant acts as a chemical driver, solubilizing residual struvite particles via surface reactions that would otherwise remain solid in the soil. Conversely, the Struvite Removed treatment risks underestimating the total P reserves potentially available to the system. By physically removing the granule prior to analysis, this method captures only the P that had successfully diffused into the surrounding soil matrix, thereby excluding the undissolved nutrient reservoir remaining within the granule. The low initial values in the Struvite Removed treatment confirm that struvite behaves as a slow-release source, relying on diffusion gradients to gradually enrich the soil P pool (Degryse et al., 2017).

The eventual statistical convergence of the Struvite Retained and Struvite Removed treatments (Figures 2.1, 2.2, and 2.3) marks a critical inflection point. This similarity suggests that after this convergence, the physical granules have largely dissolved, eliminating the discrepancy between the two methods. By delaying sampling until the granules have dissolved, agronomic planning can avoid the method-dependent biases of pre-convergence testing and rely on a consistent assessment of soil P status.

2.5.4. Olsen-P Dynamics in Two Contrasting Soil Types

The contrasting Olsen-P dynamics between the two soils highlight the profound influence of soil texture and pH on P mobility and retention. In the alkaline, clayey Libau soil, the restricted

diffusion and continuous decline of Olsen-P in experiment A and B align perfectly with the mechanisms described by Nascimento et al. (2018). Using a concentric-ring incubation on a chemically similar alkaline clay soil (Barnes; pH ~8.0), they found that over 70% of applied struvite fertilizer-P remained immediately adjacent to the granule. As in our Libau soil, this severe restriction in P mobility is driven by the high clay content, which provides abundant sorption sites, coupled with high extractable Ca that rapidly precipitates dissolved P. Consequently, as the initial dissolution of the fertilizer progresses, these aggressive adsorption and precipitation mechanisms dominate, driving the steep, ongoing decline in extractable P observed in our Struvite Retained treatment. Consistent with the rapid initial peaks observed in our Struvite Retained treatments in sandy acidic soil in both Experiments A and B, De Lucca Agrelo et al. (2025) found that struvite generated an immediate increase in Olsen-P comparable to water-soluble fertilizers like DAP and SSP within the first week, driven by its high citrate solubility in acidic conditions. The results of our study show that while struvite dissolved in both soils, soil-specific retention processes dominated the longer-term Olsen-P outcomes, causing convergence in Olsen-P concentrations between the Struvite Retained and Struvite Removed treatments, but at different absolute levels in the two soils, depending on soil chemistry. Statistical analysis (path analysis) from Ige et al. (2007) demonstrated that, for Manitoba's neutral to calcareous soils, the factors with the greatest direct influence on P retention are the Mehlich-3 extractable concentrations of calcium and magnesium, alongside oxalate-extractable aluminum. Soil texture (sand or clay content) does not exert a significant direct chemical control; rather, its correlation with retention is an indirect effect driven by its association with the soil's Ca content, particularly the Ca-bearing minerals that dominate P retention.

The divergence in temporal trends due to soil type and fertilizer treatment between Experiments A and B is best explained by the difference in granule size (1.5 mm in Exp A vs. 0.9 mm in Exp B). This aligns with literature which confirms that struvite dissolution and subsequent P availability are heavily dependent on particle size and specific surface area (Degryse et al., 2017; Hertzberger et al., 2021). Specifically, Hertzberger et al. (2021) demonstrated that soil extractable P concentrations were significantly higher for 1.5 mm granules compared to 3.0 mm granules, a size-dependent relationship further supported by Degryse et al. (2017), who found maximum dissolution when struvite was reduced to a powder (< 0.15 mm). The smaller (0.9 mm) granules used in Experiment B (Figure 2.3) possessed a much higher specific surface area than those used in Experiment A, facilitating more rapid soil-granule contact and faster initial dissolution. Degryse et al. (2017) explain that when struvite has maximum contact with the soil particles, the surrounding soil acts as a massive sink. It immediately adsorbs the released P and buffers the pH. This prevents the solution around the fertilizer particles from becoming saturated, allowing the dissolution reaction to proceed rapidly and without hindrance. In contrast, the larger 1.5 mm granules in Experiment A (Figure 2.1C) dissolved much more slowly. Degryse et al. (2017) also explain that when struvite's contact area with the soil is drastically reduced, the granules begin to dissolve and the thin layer of soil water immediately touching the granule surface becomes saturated with dissolved P and Mg. Thus, further dissolution is restricted until dissolved ions physically diffuse away from the granule interface into the bulk soil, making diffusion the rate-limiting factor.

2.5.5. Agronomic Implications and Recommendations for Soil Testing

The methodological artifacts identified in our incubation, specifically the overestimation of Olsen-P due to granule inclusion and sample grinding, pose a significant risk for making accurate

agronomic recommendations to farmers. As Gu et al. (2020) and Anderson et al. (2020) warn, relying on standard ground soil samples in testing procedures can lead us to falsely believe struvite releases P too quickly, masking its actual benefit as a slow-release source. However, the eventual statistical convergence of the Struvite Retained and Struvite Removed treatments, as well as the ground and unground treatments, indicates that the influence of sample preparation diminishes as the struvite granules naturally dissolve during incubation. Given the uncertainty regarding which method more accurately reflects plant-available P during the dissolution phase, this convergence offers a practical agronomic solution: conducting soil tests post-convergence appears to be the most reliable option. Future field studies should investigate whether establishing delayed sampling windows (e.g., advising against soil testing within the first 60 days of struvite application) will solve this problem of overestimating and underestimating STP values. While the methodological bias of grinding disappeared by later days in our controlled laboratory study, this dissolution timeline will likely vary under actual field conditions. Dynamic environmental factors such as fluctuating soil moisture, temperature extremes, and the active release of organic anions by crop roots could significantly alter the physical breakdown rate of intact granules. Therefore, future research must prioritize field-based trials to evaluate the persistence of this extraction artifact across diverse, site-specific climates.

However, chemical extractions alone cannot definitively resolve whether the overestimation of the Struvite Retained method or the underestimation of the Struvite Removed method better reflects actual agronomic value. To determine the true accuracy of these contrasting soil test approaches, they must be evaluated against a biological indicator. Conducting plant bioassay and field calibration tests, where true P uptake is measured in struvite-amended soils, would provide the definitive baseline needed to evaluate these laboratory methods. By correlating actual plant

performance with the Olsen-P values generated from both preparation methodologies, it becomes possible to identify which approach serves as the most reliable predictor of plant-available P.

2.6. Conclusion

This study demonstrated that both sample preparation and soil chemistry strongly influence Olsen-P measurements in struvite-amended soils. Grinding substantially inflated extractable P in Experiment A by fragmenting struvite granules, whereas intact granules in unground samples contributed little to no measured P in the early dates of incubation, highlighting the risk of overestimating plant-available P when soil samples containing residual struvite are finely processed and underestimating plant-available P when residual struvite granules are removed. Experiment B indicated that struvite produced higher early Olsen-P than MAP when granules were present, but its apparent availability declined rapidly in alkaline clay soil and remained relatively stable in acidic sandy soil. Collectively, these findings indicate that standard Olsen-P measurements can be biased by the presence of residual granules, obscuring the actual dissolution dynamics of the fertilizer. Consequently, both soil properties and laboratory handling must be carefully considered when interpreting soil test results for recycled, slow-release fertilizers like struvite.

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3. PLANT PHOSPHORUS UPTAKE AND OLSEN-P DYNAMICS IN CONTRASTING SOILS AMENDED WITH STRUVITE

3.1. Abstract

The recovery of phosphorus (P) from waste streams as struvite offers a sustainable, slow-release alternative to finite phosphate rock fertilizers. However, struvite's low water solubility challenges standard soil testing protocols. Analytical extractants may artificially dissolve residual struvite granules during laboratory analysis, potentially decoupling soil test P (STP) from actual plant availability. This study evaluated the relationship between plant P uptake and Olsen-P concentrations in struvite-amended soils, assessing the impact of plant-induced mobilization and the physical presence of residual struvite fertilizer granules. A two-phase growth chamber experiment was conducted using two contrasting soils: an alkaline clay (Libau) and an acidic sand (Woodmore). Phase 1 evaluated the initial presence or absence of a buckwheat (*Fagopyrum esculentum* Moench) crop across four fertilizer treatments (MAP, Struvite with residual granules retained, Struvite with residual granules removed, and an unfertilized Control). A Phase 2 bioassay subsequently measured cumulative P uptake by Italian ryegrass over 60 days. The experiment showed that prior buckwheat growth depleted rather than mobilized available P, significantly reducing Olsen-P and subsequent ryegrass P uptake. While MAP consistently maximized ryegrass P uptake across both soils, Struvite-Retained efficacy was highly soil-dependent, matching MAP performance in the acidic sand but failing to exceed the Control in the alkaline clay. Crucially, when residual struvite granules were removed prior to Phase 2, ryegrass P uptake plummeted to Control levels in both soils. This confirms that minimal struvite dissolution occurred during Phase

1, and that elevated Olsen-P readings observed in the acidic sand were laboratory artifacts caused by the chemical extractant dissolving the largely intact granules. Consequently, the overall relationship between initial Olsen-P and subsequent ryegrass P uptake was weak ($r^2 = 0.24$) and highly dependent on fertilizer type. These findings emphasize that standard chemical extractions fundamentally fail to capture the complex, time-dependent dissolution kinetics of recovered struvite, making the standard test a poor predictor of actual plant performance without methodological adjustments.

3.2. Introduction

Phosphorus (P) deficiency is a major limitation to crop production on a large share of the world's arable land, especially where farmers have poor access to P fertilizers (Cordell et al., 2009). Modern agriculture relies almost entirely on P fertilizers manufactured from phosphate rock (PR), a non-renewable resource (Cordell et al., 2009). Although estimates of when PR will be depleted vary from several decades to a few centuries, there is broad agreement that continued dependence on mined PR poses long-term risks for food security and exposes import-dependent regions to geopolitical and price shocks (Brownlie et al., 2023). Consequently, interest is shifting toward closing the P cycle. The goal is to recover P from waste to address both scarcity and pollution. Precipitation of struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) from municipal, industrial, and agricultural effluents is emerging as a promising strategy to recycle P into a slow-release fertilizer. This recycled product (struvite) can substitute for traditional inputs and support more circular, sustainable nutrient management in agriculture (Kataki et al., 2016).

Struvite granules have low water solubility and minimal salt toxicity, allowing them to be placed directly with the seed to encourage root proliferation around the granules (Talboys et al., 2016).

This placement results in a slower, more sustained P uptake pattern compared to highly soluble inorganic P fertilizers, which release P rapidly via diffusion but can cause seedling toxicity if placed too close (Katanda et al., 2019). This low solubility can delay P release for plant uptake in some cases but doesn't necessarily restrict yield. In a pot study on spring wheat (*Triticum aestivum* L.), plants fertilized with struvite rather than diammonium phosphate (DAP) achieved similar final grain yields, even though P uptake in the struvite-treated plants was lower in early growth stages compared to those receiving DAP (Talboys et al., 2016). Degryse et al. (2017) found that the dissolution rate of granular struvite is highly dependent on soil pH. In acidic soils, the granules dissolved at a rate of 0.43 mg struvite day⁻¹, but in alkaline conditions, this rate declined sharply to just 0.03 mg day⁻¹. This severely restricted how quickly P could diffuse through the alkaline soil, resulting in significantly lower early-season P uptake by the crop. This inconsistency is often attributed to the interaction between soil pH and crop-specific root mechanisms, such as organic anion exudation, which facilitate struvite dissolution (Hertzberger et al., 2020). For example, Ackerman et al. (2013) found that canola grown in alkaline soil (pH 7.7) yielded less biomass per unit of P when fertilized with struvite compared to monoammonium phosphate (MAP). They attributed this reduced biomass to struvite's delayed initial solubility in high-pH conditions, which gives highly soluble fertilizers an early-season growth advantage. This delayed release dynamic of struvite underscores the necessity of evaluating its impact on early-stage crop growth.

Although struvite is characterized by low water solubility, its dissolution in citric acid is comparable to standard ammonium phosphate fertilizers (Rech et al., 2018). While the in vitro experiment by Talboys et al. (2016) showed that organic anions significantly increased struvite dissolution, their accompanying greenhouse trials demonstrated this effect agronomically. They observed that buckwheat, a crop recognized for high root exudation of organic anions, mobilized

struvite P much more effectively than spring wheat. Buckwheat is consistently characterized as a highly P-efficient species, particularly on calcareous or low-P soils. Buckwheat can take up P efficiently from Ca-bound sources such as PR and CaHPO_4 , with P uptake up to ~10-fold higher than wheat in sand culture and clearly superior performance on a highly calcareous soil (Zhu et al., 2002). It can access both inorganic and some organic P forms, supported by high root surface phosphatase activity and depletion of organic P in the rhizosphere (Mäkelä et al., 2020). In a calcareous and a decalcified chernozem, buckwheat promoted marked rhizosphere acidification and maintained high acid phosphatase activity, especially in the calcareous soil where these root-induced changes correlated with removal of organic P (Raniero & Santner, 2023). Under P limitation, buckwheat developed a large root surface area, high root surface area:shoot ratio, and rapid root proliferation, which confer a high specific P absorption rate even when solution P is low (Mäkelä et al., 2020). As a cover or green manure crop, buckwheat is widely considered effective at mobilizing labile P pools and contributes to P cycling in low-input systems. Its high capacity to utilize Ca-bound and residual P suggests value in rotations or intercropping on calcareous, P-fixing soils to improve overall P use efficiency (Raniero & Santner, 2023). However, its efficacy can be highly conditional. For instance, Arcand et al. (2011) demonstrated that buckwheat green manure residues produced only a marginal increase in soil P bioavailability, a response observed strictly when very high rates of rock phosphate were applied. Although plant root activity can undoubtedly contribute to P mobilization from sparingly soluble fertilizers, it is not always a strict requirement. Plant-free incubation and soil-column studies demonstrate that struvite can substantially increase soil test P (STP) (such as water-soluble and Mehlich-3 P) even in the absence of roots, indicating that soil pH, P-sorption capacity, and extractant chemistry alone can drive appreciable struvite dissolution (De Lucca Agrelo et al., 2025; Gu et al., 2021).

Soil properties strongly regulate how struvite dissolves and releases plant-available P, especially soil pH and P sorption capacity. In dissolution experiments, Talboys et al. (2016) observed single 2.4 mm struvite granules over 42 days in solution across a pH range of 5.5–8.0. Their dissolution curves revealed that initial rates dropped sharply as pH increased, with the maximum P release occurring at lower pH levels. This trend was corroborated in soil environments by Degryse et al. (2016), who compared granule mass loss in acidic versus alkaline soils. They reported significantly higher release rates in acidic soil (0.43 mg P d^{-1}) compared to alkaline soil ($\sim 0.03\text{--}0.05 \text{ mg P d}^{-1}$). In a 2020 meta-analysis, Hertzberger et al. (2020) synthesized data from numerous greenhouse and field studies to compare the efficacy of struvite against soluble P fertilizers like MAP and triple superphosphate (TSP). By correlating relative yield and P uptake with soil acidity, they identified a clear pH dependency. Struvite outperformed soluble fertilizers resulting in higher biomass and P uptake primarily in soils with a pH below 6. However, as soil pH increased, the agronomic performance of struvite declined below that of soluble fertilizers.

Soil test P, particularly the Olsen-P method (Olsen et al., 1954), is one of the main agronomic tools used to assess how much plant-available P a soil can supply and to decide whether, and how much, P fertilizer should be applied. In practice, soils are classified into P status classes (low, moderate, high) based on Olsen-P, and recommended P rates are increased as Olsen-P falls below a crop- and soil-specific critical value (Pedersen et al., 2023). The necessity of soil-specific targets is highlighted by controlled pot experiments, which demonstrate that the critical Olsen-P value required to achieve 90% of maximum ryegrass yield can vary drastically from 7.3 to 194 mg kg^{-1} depending entirely on the soil properties (Tandy et al., 2021). Research by Johnston et al. (2014) on sandy clay loam soils has confirmed a strong positive correlation between STP and plant acquisition, with P uptake increasing more than threefold from 23.3 to 75.2 kg ha^{-1} as initial Olsen-

P levels rose from 4 to 33 mg kg⁻¹. However, soil pH and clay content strongly influenced both the critical Olsen-P and how fertilizer P translated into plant-available P. Increasing clay content (and associated P buffer capacity) reduced the Olsen-P threshold because such soils can maintain a steady solution P concentration at lower test values (Recena et al., 2022). Reviews of P chemistry confirm that maximum P availability to plants typically occurs near neutral pH (≈ 6.5 -7); at lower pH, P is fixed by Fe/Al oxides, and at higher pH as calcium phosphates (Penn & Camberato, 2019). These processes affect both measured Olsen-P and the amount of fertilizer P required to raise Olsen-P to a given target.

A fundamental limitation in understanding effects on STP arises when dealing with low-solubility fertilizer sources. For instance, PR is typically only 5% to 33% as effective as freshly applied superphosphate, primarily due to its severely restricted dissolution in many soils (Bolland & Gilkes, 1990). This limited dissolution drastically reduces the pool of P available for immediate plant uptake. This discrepancy complicates soil testing. Because the Olsen-P method was designed to quantify the readily soluble P provided by fertilizers like superphosphate or MAP, it cannot account for the slow, soil-dependent dissolution of struvite that occurs over a growing season (Kratz et al., 2019). As a result, standard soil tests underestimate the total P that will gradually become available to the crop, yielding misleadingly low values that require separate calibration curves to accurately predict plant response (Bolland & Gilkes, 1990). This analytical challenge is particularly relevant to alternative slow-release fertilizers, such as struvite, where dissolution kinetics may similarly decouple soil test values from actual plant P uptake (Meyer et al., 2018; Kratz et al., 2019). In research using a ³³P tracer to measure P uptake in ryegrass grown in calcareous soil treated with recycled fertilizers like struvite, Meyer et al. (2018) found that while struvite has very low initial water solubility (about 4%), it was 75% as effective as fully soluble

reference fertilizers. Because plant uptake correlated closely with resin- and NaHCO_3 -extractable P, the study demonstrated that gradual dissolution over time rather than immediate water solubility was the true driver of plant P uptake.

The rationale for this study arises from the need to deepen our understanding of struvite dissolution dynamics and to validate diagnostic tools for its effective agronomic use. While struvite is a promising recycled P source, its behaviour in soil, particularly regarding the role of plant roots in mobilizing P from struvite, remains underexplored. Plants can actively influence P dynamics through root exudates and P uptake patterns, potentially accelerating the dissolution of slow-release granules (Richardson et al., 2009). However, this interaction introduces an analytical challenge: the physical presence of undissolved fertilizer granules in soil samples can artificially inflate P measurements, decoupling soil test values from actual plant availability. Conversely, standard soil tests could misleadingly underestimate actual plant uptake if the extractant fails to capture gradual dissolution kinetics. This limitation is frequently observed with low-water-solubility fertilizers, such as the recycled struvite evaluated by Meyer et al. (2018). Consequently, the relationship between plant P uptake and soil Olsen-P must be critically examined to determine how standard protocols require modification for struvite-amended soils. Modifying protocols, such as physically removing residual partially dissolved granules prior to soil extraction, prevents the chemical extractant from artificially inflating the STP, as shown in Chapter 2, and may provide a more realistic measure of P that plants can immediately access compared to analyzing soil samples containing residual struvite granules. However, this practice introduces its own analytical bias: it risks decoupling the STP from actual plant availability by entirely omitting the slow-release P reservoir in residual granules that the crop would continuously access throughout the growing season. To address this methodological gap, this study compares Olsen-P extractions performed

both with and without removing of struvite granules. This comparative approach aims to determine which protocol more accurately reflects actual plant P uptake, or whether standard chemical extractions are fundamentally inadequate for calibrating struvite-amended soils. Validating these diagnostic relationships is essential for optimizing application rates, thereby improving long-term P use efficiency. This research is particularly crucial for regions like Manitoba, where precise P management is required to mitigate nutrient loading into sensitive water bodies such as Lake Winnipeg, supporting both sustainable agriculture and environmental conservation.

The objective of this study was to examine the relationships between plant P uptake and soil Olsen-P concentrations in soils previously amended with struvite, both with and without the presence of plants, to investigate the impact of plant P mobilization on the availability of P in contrasting soil types treated with struvite. We hypothesized that (1) in soils treated with struvite, growing plants immediately after struvite application to soil will increase P availability to a following crop, compared to unplanted soils (due to mobilization of struvite P by plants); (2) in soils treated with struvite, growing plants immediately after struvite application to soil will lead to a stronger relationship between plant P uptake and Olsen-P concentration compared to soils without plants (due to mobilization of struvite P by plants); (3) in soils treated with struvite, plant P uptake will have a stronger relationship with Olsen-P when residual struvite granules are removed from soil, compared to when residual granules are retained (due to artificial inflation of Olsen-P concentrations from dissolution of residual struvite granules); and (4) the relationship between Olsen-P and plant P uptake will not be affected by soil type. The research was conducted in two phases within a controlled environment growth chamber. Phase 1 (Treatment phase), involved applying various treatments, including soils, fertilizers, and plants. In Phase 2 (Assessment phase), plant P uptake was used to evaluate the effects of the treatments from Phase 1.

3.3. Materials and Methods

3.3.1. Experiment and Treatment Design

The experiment followed a randomized complete block design with four replications. Each replication functioned as a single block, containing one complete set of the treatment combinations to control for variations in light and temperature across the growth chamber benches. The factors were soil type (Libau and Woodmore), four fertilizer treatments (described below), and Phase 1 buckwheat plant history (presence vs. absence). The study employed a 2×4×2 factorial arrangement resulting in 16 total treatment combinations. The experimental unit was the pot, each containing one buckwheat plant for Phase 1 and 10 Italian ryegrass plants for Phase 2. At the end of Phase 1, soil samples were analyzed for Olsen-P concentrations to test the effects of soil type, fertilizer, and plant presence. At this time, P uptake was also measured for the buckwheat plants to assess soil and fertilizer effects. Subsequently, at the end of Phase 2, the experiment evaluated the cumulative impact of all treatment combinations on the Italian ryegrass, specifically measuring aboveground plant biomass, tissue P concentration, and P uptake.

3.3.2. Treatment Factors

3.3.2.1. Soils

Two soil types classified as Gleyed Rego Black Chernozems were collected in autumn 2024 from the top 15-cm layer of annual crop fields in Manitoba. The first was an alkaline clay from Libau (50°14'30.1"N 96°43'39.5"W; Dencross series), and the second was an acidic sand from Woodmore (49°07'15.2"N 96°54'12.0"W; likely Lenswood or Glenhope series). Field-moist samples were passed through a 10-mm sieve to remove crop residue and stones. The clay Libau soil was subsequently air-dried for at least 12 hours at 30 °C, while the sandy Woodmore soil

remained undried due to its low initial moisture content. Both soils were stored for five months and ground to pass through a 2-mm sieve prior to the experiment. Both soils had a history of organically managed forage and annual crop production with occasional fallow periods. To determine initial moisture content, air-dried samples (~50 g) were oven-dried at 105 °C to a constant weight. Water-holding capacity was assessed by packing air-dried soil into transparent columns and adding water incrementally; container capacity was defined as the water volume required to fully wet the soil without drainage. Based on these measurements, target gravimetric moisture contents were established at 0.40 g g⁻¹ for the clay Libau soil and 0.21 g g⁻¹ for the sandy Woodmore soil, corresponding to 60% and 47% water-filled pore space, respectively.

3.3.2.2. Fertilizers

The experiment evaluated two commercial struvite fertilizers: Crystal Green Pearl® (N-P₂O₅-K₂O analysis 5-28-0 plus 10% Mg; Ostara Nutrient Recovery Technologies Inc., Vancouver, BC) and commercial MAP (N-P₂O₅-K₂O analysis 11-52-0, CropKing, Inc. Lodi, OH). MAP was selected as the soluble fertilizer for comparison due to its widespread agronomic use in Manitoba and its N:P ratio, which is comparable to that of struvite. The fertilizer treatments in Phase 1 included struvite applied at planting (retained in soil for Phase 2), struvite applied at planting with granules removed prior to Phase 2, and MAP applied at planting as a positive control. An unfertilized treatment (no P applied) was included as a negative control. Both fertilizers were applied in granular form. Struvite granules were ~3 mm in diameter and granular MAP was sieved to isolate granules around 2 mm in diameter, to limit the effect of granule size on P distribution through the soil.

3.3.3. Experimental Set-up, Sample Collection and Analysis

3.3.3.1. Phase 1

Each pot (12 cm long x 12 cm wide x 16 cm high) was set up with air-dried soil equivalent to 2 kg of oven-dry soil and was designed without drainage holes to prevent nutrient loss. Soil moisture was maintained at the previously determined target capacities (section 2.2.1.), with watering occurring approximately three times per week based on the weight of the pots. Fertilizer and seeds were placed in a central row to simulate a crop row, following recommended practices for both fertilizers. For struvite, the fertilizer was placed with the seed, while for MAP, it was placed 1 cm below the seed to avoid seedling toxicity from its high solubility. The P application rate was 20 mg kg⁻¹, requiring approximately 30 granules of 3-mm struvite or 7 granules of MAP.

Buckwheat was grown in half of the pots for a period of four weeks. The target plant population was one plant per pot, achieved by pre-germinating seeds and placing four into each pot, then thinning to one plant after establishment. Both planted and unplanted pots were maintained under typical growing season day/night lighting and temperatures. The pots were placed in a growth chamber room that was maintained between 20 and 22 °C, with 16 h d⁻¹ of artificial light throughout the experiment. To ensure P was the sole limiting variable, all pots (including controls) received a P-free basal nutrient solution. Nutrients were applied at the following rates per kg of soil: 20 mg S, 4 mg Zn, 1 mg B, 1 mg Cu, and 0.4 mg Mo. Nitrogen was balanced to a total rate of 100 mg N kg⁻¹ across all treatments, accounting for the N already supplied by the struvite and MAP fertilizers (Zvomuya et al., 2006). After four weeks of buckwheat growth, plant shoots were cut, dried, weighed, and ground for analysis to determine P uptake by the plants. All harvested plant material was oven-dried at 60 °C for at least 48 hours (until constant weight) and

subsequently ground using a ball mill (Spex 8000 Mixer/Mill; Spex Industries, Metuchen, NJ). To determine total P concentration, 0.5 g of ground tissue was digested in nitric acid (HNO₃) using a digester and analyzed via Inductively Coupled Plasma–Optical Emission Spectroscopy (ICP–OES).

3.3.3.2. Phase 2

At the end of the first phase, after the buckwheat was harvested, the soil from each pot was well mixed and a soil sample of 67 g was taken from each pot. For treatments where struvite was retained, the 67 g soil sample was prepared containing a single struvite granule retrieved from the soil of the corresponding pot, ensuring a representative soil-to-fertilizer ratio. All the soil samples were analyzed for Olsen-P by a commercial lab, where they were ground and subsampled prior to analysis. Soil Olsen-P was determined by extracting 1 g of soil with 20 mL of extractant in 50 mL Erlenmeyer flasks. The suspensions were shaken for 30 minutes on a reciprocal shaker, then filtered through Whatman No. 42 filter paper. Phosphorus concentrations in the filtrate were quantified using the molybdate blue method on a flow injection analyzer (FIAlyzer-1000 series, FIALab, Seattle, WA).

A 120-day bioassay using Italian ryegrass was conducted on the remaining soil to quantify biological P uptake. Each pot was seeded and thinned to a target population of 10 plants, maintained under the same temperature and lighting conditions as in Phase 1. Italian ryegrass was harvested by cutting plant shoots 1–2 cm above the soil surface at four 30-day intervals. Ryegrass samples were processed and analyzed for tissue P concentration using the same approach as for buckwheat, as described in section 3.3.3.1.

3.3.3.3. Calculations

Plant uptake of fertilizer P by the buckwheat crop and Italian ryegrass for each harvest date (PU, mg P pot⁻¹) was calculated as:

$$PU = DMY \times P \text{ conc}$$

where DMY is the aboveground biomass dry matter yield (g pot⁻¹) and P conc is the respective tissue P concentration (mg g⁻¹).

Cumulative P uptake for Italian ryegrass was calculated as the sum of P removal from the first two harvests. Although a third harvest and fourth harvest were conducted, data from these intervals were excluded from the cumulative analysis due to non-uniform plant mortality and stunting across replicates, which rendered the third and fourth harvest dataset inconsistent. Therefore, cumulative uptake analysis was restricted to the first two harvests to maintain a balanced design with four replicates per treatment (n=4) and to ensure statistical reliability.

The biomass-weighted mean of plant tissue P concentration across the first two harvest dates was calculated by dividing the total cumulative P uptake from the first two harvests by the total cumulative aboveground biomass dry matter yield from those same harvests.

3.3.4. Statistical Analyses

Analysis of variance (ANOVA) was conducted using PROC GLIMMIX of SAS® software (version 9.4, SAS OnDemand for Academics; SAS Institute, Cary, NC).

Phase 1 Analyses: Total P uptake, shoot biomass, and tissue P concentration of buckwheat for Phase 1 were analyzed using two-way ANOVA with soil type and fertilizer treatment as fixed

factors, while block was included as a random factor. The model tested the main effects of soil and fertilizer, as well as their interaction.

Phase 2 Analyses: Three-way ANOVA was utilized to analyze shoot biomass, plant tissue P concentration, and cumulative P uptake in ryegrass. The fixed factors included soil type, fertilizer treatment, and the Phase 1 cropping history (i.e., with plants vs. without plants), while random factor included block. Olsen-P concentrations measured at the end of Phase 1 were also analyzed using a three-way ANOVA with the same fixed and random factors. Residual normality was confirmed using the Shapiro-Wilk test (statistic $W > 0.9$). Homogeneity of variance was assessed through visual inspection of residual plots and by testing for covariance heterogeneity among treatment groups. When the ANOVA indicated significant effects, differences between treatment means were compared using Tukey's HSD test at a significance level of $\alpha = 0.05$. For Olsen-P, data were analyzed using a lognormal distribution because the data violated assumption of normality. Results were back-transformed to the original scale for a meaningful interpretation.

Regression analysis was performed using the MIXED procedure in SAS to determine whether Olsen-P at the beginning of Phase 2 serves as a good indicator of the plant P uptake in Phase 2. Separate regression models were run for each categorical factor of interest in interaction with Olsen-P as the continuous predictor variable. In each model, the categorical factor of interest and Olsen-P were specified as fixed effects. Block and the remaining two categorical factors (from: Plant, Fertilizer, and Soil) were treated as random effects. In accordance with our study's objectives, the initial models evaluated all relevant two-way interactions with Olsen-P (Olsen-P $\times$ Plant, Olsen-P $\times$ Fertilizer, and Olsen-P $\times$ Soil) to test the predictions outlined in Hypothesis 3 regarding equal regression slopes across individual experimental factors. When the interaction term was significant, specific pairwise comparisons of the regression slopes for individual

treatments were performed to identify which treatments had significantly different relationships with Olsen-P. Statistical significance for all tests was evaluated at an alpha of 0.05.

3.4. Results

3.4.1. Buckwheat Biomass, P concentration, and P uptake (Phase 1)

In Phase 1, the effects of soil type and fertilizer treatment on buckwheat biomass, P concentration, and P uptake were examined (Table 3.1). ANOVA revealed no significant interaction effects between soil type and fertilizer treatment for buckwheat biomass, P uptake, or tissue P concentration. For the buckwheat crop, the only statistically significant main effect ($p < 0.05$) observed was the effect of soil type on tissue P concentration ($p = 0.001$). The P concentration was greater in the acidic Woodmore soil than the alkaline Libau soil. Since the plant biomass was numerically lower in the acidic Woodmore soil than the alkaline Libau soil, the difference in P concentration did not translate to a difference in P uptake between the two soil types.

For P uptake, there was a marginal main effect of fertilizer treatment ($p = 0.06$). The biomass and P concentration means followed the same ranking for fertilizer treatments (though not significantly different), combining to produce a stronger trend in P uptake. Mean P uptake was lowest in the unfertilized control ($0.72 \text{ mg plant}^{-1}$) and highest in the highly soluble MAP treatment ($1.19 \text{ mg plant}^{-1}$), while the two struvite treatments yielded intermediate values (0.88 and $0.80 \text{ mg plant}^{-1}$). The lack of a significant difference between the struvite treatments and the control is likely due to struvite's slow-release nature; the limited dissolution during the short Phase 1 growth period was insufficient to supply P at rates significantly higher than the control. Furthermore, the similar P uptake between the two struvite treatments is to be expected, as the physical removal of granules was performed after buckwheat harvest.

Table 3.1 Effects of soil type and fertilizer type on the biomass, tissue P concentration, and P uptake for buckwheat.

Factor	Biomass	P concentration	P uptake
	g pot ⁻¹	mg g ⁻¹	mg pot ⁻¹
Soil			
Libau	5.42 (0.51) ^a	1.60 (0.10) b ^b	0.87 (0.13)
Woodmore	4.43 (0.51)	2.16 (0.10) a	0.93 (0.13)
Fertilizer			
Control	4.44 (0.68)	1.67 (0.15)	0.72 (0.17)
MAP	5.55 (0.68)	2.15 (0.15)	1.19 (0.17)
Struvite Retained	5.09 (0.68)	1.96 (0.15)	0.88 (0.17)
Struvite Removed	4.62 (0.51)	1.76 (0.15)	0.80 (0.17)
ANOVA		p-value	
Soil (S)	0.14	0.001	0.60
Fertilizer (F)	0.62	0.15	0.06
S × F	0.90	0.46	0.98

^a Data are expressed as least-squares means; standard errors of the mean (SEM) are provided in parentheses.

^b For significant ANOVA results, means within a column sharing the same letter do not differ significantly according to Tukey's test ($\alpha=0.05$).

3.4.2. Olsen-P Concentrations

A significant main effect of plant presence was observed for Olsen-P concentrations ($p = 0.01$, Table 3.2). Contrary to the initial hypothesis that buckwheat would mobilize and thereby increase available soil P, the presence of Phase 1 buckwheat resulted in a net decrease in Olsen-P compared to unplanted soils. This indicates that the rapid accumulation and removal of P by the buckwheat biomass during the growth phase significantly exceeded any localized P mobilization caused by root exudates, resulting in an overall depletion of the easily extractable P pool.

Table 3.2 Effects of soil type, fertilizer type, and plant presence on the Olsen-P concentrations and on the biomass, tissue P concentration, and cumulative P uptake for ryegrass in Phase 2.

Factor	Olsen-P	Biomass	P concentration	P uptake
	mg kg ⁻¹	g pot ⁻¹	mg g ⁻¹	mg pot ⁻¹
Soil				
Libau	9.84 (0.50)	9.47 (0.21) ^a	0.88 (0.02)	8.53 (0.33)

Woodmore	16.64 (0.85)	5.07 (0.21)	1.45 (0.04)	7.97 (0.33)
Fertilizer				
Control	8.61 (0.62)	5.18 (0.3)	0.95 (0.04)	4.80 (0.46)
MAP	21.41 (1.55)	9.47 (0.3)	1.38 (0.06)	11.79 (0.46)
Str Retained	12.41 (0.9)	7.93 (0.3)	1.35 (0.04)	10.09 (0.46)
Str Removed	11.78 (0.85)	6.51 (0.3)	0.99 (0.03)	6.31 (0.46)
Plant Presence				
Yes	11.64 (0.59) b	6.01 (0.21)	1.13 (0.03)	6.54 (0.33)
No	14.06 (0.72) a	8.54 (0.21)	1.20 (0.03)	9.96 (0.33)
ANOVA		<i>p-value</i>		
Soil (S)	<0.0001	<0.0001	<0.0001	0.24
Fertilizer(F)	<0.0001	<0.0001	<0.0001	<0.0001
Plant Presence(P)	0.01	<0.0001	0.08	<0.0001
S × F	0.007 ^b	0.0004	<0.0001	<0.0001 ^b
S × P	0.34	0.001	0.28	0.0499
F × P	0.59	0.71	0.04	0.10
S × F × P	0.62	0.29	0.07	0.50

^a Data are expressed as least-squares means; SEM are provided in parentheses.

^b The highest-order interactions that are significant for Olsen-P are plotted in Figure 3.1, while those significant for P uptake are plotted in Figures 3.2 and 3.3.

Furthermore, a significant interaction between soil type and fertilizer treatment was observed for Olsen-P concentrations ($p = 0.007$; Figure 3.1), indicating that the availability of P depended strongly on the specific combination of soil type and fertilizer source. MAP application significantly increased Olsen-P relative to the control in both Libau and Woodmore soils. However, the efficacy of struvite in raising Olsen-P was distinct between the two soils. In the Woodmore soil, both Struvite Retained and Struvite Removed treatments increased Olsen-P to levels statistically equivalent to MAP. In contrast, in the Libau soil, struvite application resulted in Olsen-P concentrations that were statistically indistinguishable from the control.

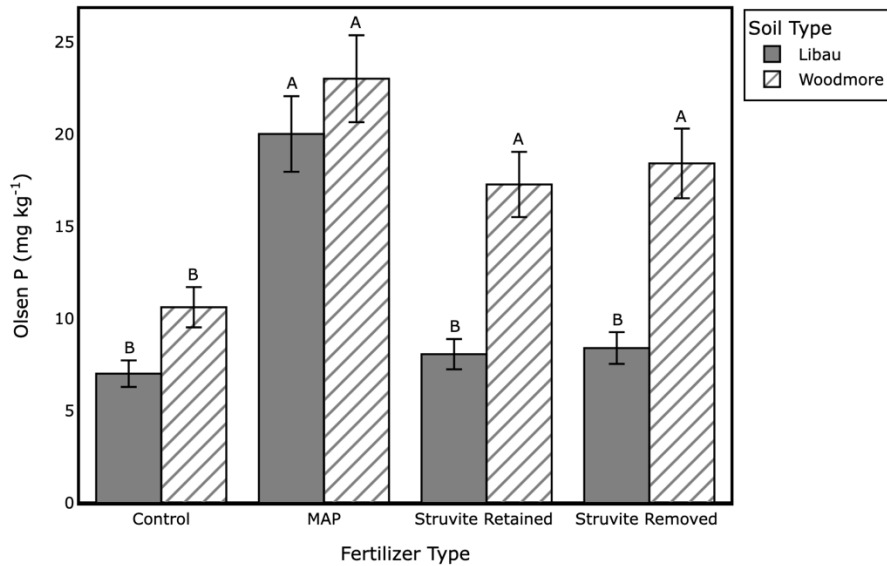


Figure 3.1 Interactive effects of fertilizer treatment (Control, MAP, Struvite Retained, and Struvite Removed) and soil type (Libau and Woodmore) on Olsen-P concentrations at the end of Phase 1. Bar heights represent the least-squares means and error bars indicate standard errors of the mean. Different letters indicate significant differences among treatment combinations according to Tukey's HSD test ($p < 0.05$).

3.4.3. Ryegrass Biomass, P Concentration, and Cumulative P Uptake (Phase 2)

Phase 2 examined the combined effects of fertilizer treatment, soil type, and plant presence on biomass, tissue P concentration, and cumulative P uptake for ryegrass. A significant Soil $\times$ Fertilizer interaction was observed for P uptake ($p < 0.0001$), indicating that the availability and acquisition of P depended strongly on the specific combination of soil type and fertilizer source. Additionally, a significant Soil $\times$ Plant interaction ($p = 0.04$) for P uptake suggested that the influence of Phase 1 cropping history on subsequent ryegrass uptake varied by soil type. Biomass followed a similar pattern, driven by significant Soil $\times$ Fertilizer ($p = 0.0004$) and Soil $\times$ Plant ($p = 0.001$) interactions, alongside a highly significant main effect of soil type ($p < 0.0001$). In contrast, tissue P concentration exhibited a slightly different response; while it shared the strong

Soil $\times$ Fertilizer interaction ($p < 0.0001$), it uniquely displayed a significant Fertilizer $\times$ Plant interaction ($p = 0.04$) rather than a Soil $\times$ Plant effect.

Prior growth of buckwheat significantly reduced subsequent ryegrass P uptake (Figure 3.2). While the overall ANOVA indicated a significant interaction between soil type and plant presence ($p=0.0499$), conservative post-hoc testing (Tukey's HSD) grouped the treatments by main effects. As indicated by the shared letters across soil types, the overwhelming driver of P depletion was the presence of Phase 1 plants rather than the specific soil texture. However, the magnitude of the difference between Phase 1 unplanted and planted treatments was numerically greater in the Libau soil than in the Woodmore soil (Figure 3.2), contributing to the significant interaction. Contrary to our hypothesis that buckwheat would mobilize P, both the Libau and Woodmore soils experienced a statistically equivalent inability to resupply P to the ryegrass following buckwheat growth.

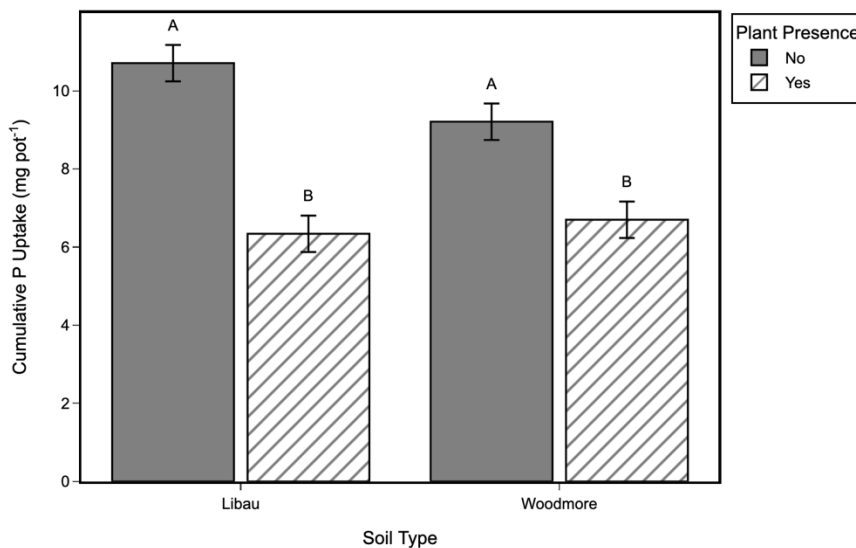


Figure 3.2 Interactive effects of soil type (Woodmore and Libau) and Phase 1 plant presence (No, Yes) on cumulative P uptake by ryegrass. Bar heights represent the least-squares means and error bars represent standard error of the mean. Different letters indicate significant differences among treatment combinations according to Tukey's HSD test ($p < 0.05$).

The interaction between soil type and fertilizer treatment showed that the MAP treatment consistently produced the highest ryegrass P uptake, though the magnitude of this fertilizer response was soil-dependent (Figure 3.3). However, the efficacy of the Struvite Retained treatment varied by soil. In the acidic Woodmore soil, Struvite Retained resulted in P uptake statistically equivalent to MAP and significantly higher than the Libau soil. Conversely, in the alkaline Libau soil, ryegrass P uptake in the Struvite Retained treatment was significantly lower than MAP and did not differ from the Control. When struvite granules were removed (Struvite Removed), P uptake decreased significantly in the Woodmore soil compared to the Retained treatment, dropping to levels statistically comparable to the Control. In the Libau soil, the Struvite Removed treatment was also statistically identical to the Control. These results suggest that struvite granules had not dissolved extensively during Phase 1; consequently, removing the granules removed the primary source of P, leaving soil P levels similar to the unfertilized control. Furthermore, within the control treatments, the Libau soil supported greater P uptake than the Woodmore soil (Figure 3.3), despite the Libau clay starting with less easily extractable P (Olsen-P of 5 mg kg⁻¹) compared to the Woodmore sand (Olsen-P of 7 mg kg⁻¹). This demonstrates the higher P buffering capacity of the clay soil, which acts as a steady reservoir to keep supplying P to the plants, whereas the sand rapidly exhausts its available P.

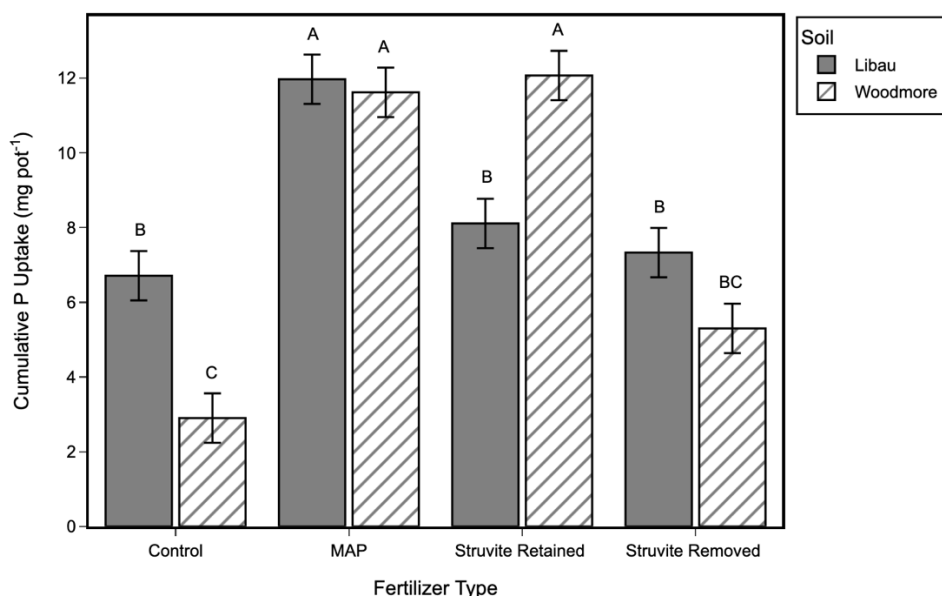


Figure 3.3 Interactive effects of fertilizer type (Control, MAP, Struvite Retained, Struvite Removed) and soil type (Libau and Woodmore) on cumulative P uptake by ryegrass. Bar heights represent the least-squares means and error bars represent standard error of the mean. Different letters indicate significant differences among treatment combinations according to Tukey's HSD test ($p < 0.05$).

3.4.4. Relationship Between Olsen-P and Ryegrass P Uptake

To evaluate the relationship between initial soil Olsen-P (beginning of Phase 2) and cumulative P uptake by ryegrass, a set of mixed model regression was conducted testing multiple categorical factors, including soil type, Phase 1 plant presence, and fertilizer treatment. The interactions for Olsen-P $\times$ Soil (p -value = 0.08) and Olsen-P $\times$ Plant Presence (p -value = 0.92) were not statistically significant, indicating that the relationship between initial Olsen-P and subsequent plant P uptake did not differ across the contrasting Woodmore and Libau soils or due to prior buckwheat growth. However, this relationship was significantly influenced by the fertilizer treatment, as indicated by a significant Olsen-P $\times$ Fertilizer interaction ($p=0.003$; Figure 3.4).

Where the interaction between initial soil Olsen-P and the categorical variable was significant, the relationship between Olsen-P and plant P uptake was analyzed separately for each fertilizer treatment to account for the unequal slopes. This subsequent analysis revealed that the Struvite Retained treatment exhibited a distinct positive relationship, yielding a slope (0.40) that was significantly greater than all other treatments (indicated by the letter A). In contrast, the regression slopes for the Control (-0.28), MAP (0.078), and Struvite Removed (-0.0078) treatments were relatively flat and statistically indistinguishable from one another, all sharing the letter B. The overall regression model, inclusive of initial soil Olsen-P, fertilizer treatment, and their interaction, accounted for 24% of the total variance observed in cumulative P uptake by ryegrass ($r^2 = 0.24$).

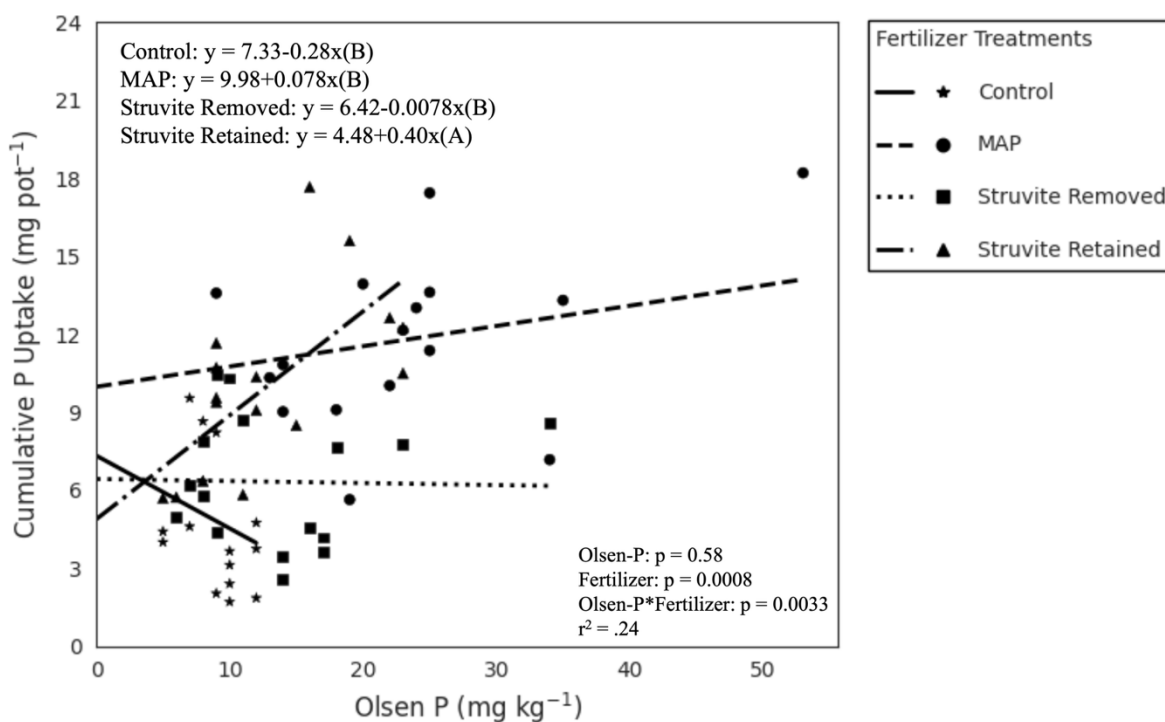


Figure 3.4 Regression relationships between Olsen-P and ryegrass cumulative P uptake in each of the four fertilizer treatments. Data points represent individual observations. Slopes of regression equations for Cumulative P uptake for ryegrass followed by the same upper-case letter are not significantly different based on pair-wise comparisons.

3.5. Discussion

3.5.1. Phosphorus Uptake Dynamics in Buckwheat

3.5.1.1. Effects of Soil

Total cumulative P uptake by buckwheat did not significantly differ between the Libau clay and Woodmore sand treatments. While buckwheat grown in the Woodmore soil exhibited a highly significant increase in tissue P concentration ($p = 0.001$; Table 3.1), this was balanced out because the plants produced less total biomass. This inverse trend: higher P concentration but smaller biomass points to a classic 'concentration effect' within the Woodmore soil. Because the Woodmore soil is highly sandy with a lower water-holding capacity, it is likely that poor early establishment and mild water stress suppressed overall biomass accumulation. Consequently, the P absorbed by these plants was concentrated within a smaller volume of tissue, meaning the higher P concentration was functionally negated by the lower biomass and did not translate to a superior soil P supply compared to the Libau clay.

Despite this concentration effect, analyzing the tissue P concentration remains critical. As emphasized by Arcand et al. (2011), when utilizing a cover crop for nutrient cycling, the primary metric of success is not total dry matter, but the concentration of P accumulated within the tissue. From this perspective, while the acidic Woodmore sand was superior to the alkaline Libau clay at enabling buckwheat to accumulate P, it ultimately fell short of its agronomic goal. Even in the most favourable Woodmore treatments, the maximum buckwheat P concentration failed to reach the critical 2.9 mg g^{-1} threshold as mentioned by Arcand et al. (2011) required for a cover crop to successfully release P to a subsequent crop.

3.5.1.2. Effects of Fertilizer

In contrast to the soil's effect, where different physiological factors neutralized one another, fertilizer treatment exhibited a marginal effect on total P uptake ($p=0.06$; Table 3.1). This result was driven by a synergistic amplification: while the individual effects of fertilizer on early biomass and tissue P concentration were not statistically significant, both parameters followed the same numerical ranking across treatments. These parallel increases amplified the overall effect on P uptake, indicating a biological response to the differing P supplies. The observed pattern, where mean P uptake was numerically greatest in the highly soluble MAP treatment, followed by the Struvite Retained treatment, the Struvite Removed treatment, and finally the unfertilized control, aligns with the well-documented dissolution kinetics of these fertilizers. MAP provides an immediate flush of orthophosphate into the soil solution, readily available for plant uptake. In contrast, struvite is a slow-release compound whose dissolution is heavily dependent on time, soil moisture, and soil acidity (Jiménez et al., 2021; Talboys et al., 2016).

The absence of a significant fertilizer effect on Phase 1 buckwheat biomass (Table 3.1) strongly aligns with established literature regarding this species' early developmental physiology and aggressive P acquisition strategies. Despite the immediate P supply provided by MAP, this rapid availability did not translate into significantly increased structural growth compared to the unfertilized control. During the initial weeks of growth, crops like buckwheat frequently rely heavily on endogenous P reserves stored within the seed (Grant et al., 2000). As demonstrated by Nadeem et al. (2011) in maize, seed-derived phytate acts as the main P source during early germination, rapidly hydrolyzing to sustain developing tissues before exogenous root uptake becomes the dominant P source. Furthermore, buckwheat possesses well-documented ability to acidify the rhizosphere and dissolve mineral-bound P (Zhu et al., 2002), which effectively

mitigates early P deficiency without relying on exogenous fertilizer. This explains why biomass, tissue P concentration, and total P uptake in the unfertilized control were not significantly different from the highly soluble MAP treatment. These findings mirror field studies where unfertilized buckwheat maintained equal biomass to fertilized plots (Possinger et al., 2013), and support observations that early P applications to buckwheat frequently succeed in elevating tissue P concentrations without translating into increased dry matter production (Arcand et al., 2011).

3.5.2. Reevaluating the Agronomic Benefit of Buckwheat Cover Cropping

Traditionally, buckwheat has been heavily promoted as a rotational tool to improve soil fertility. For example, Mäkelä et al. (2020) broadly recommend its inclusion in crop rotations specifically to activate P sources in calcareous soils. Teboh & Franzen, (2011) similarly concluded that buckwheat leaves higher available P pools, suggesting potential solubilization of P to subsequent crops. However, the severe P-starvation observed in our subsequent ryegrass crop demonstrates that such generalized recommendations must be qualified by crop residue management practices. Our results show that while buckwheat may chemically activate P, its aggressive sink capacity means it hoards that P within its biomass even when highly soluble fertilizers like MAP are present. Therefore, if buckwheat is incorporated into a rotation but the biomass is physically removed (such as for forage or grain export), it functions as a severe net extractor rather than a P provider.

The observation that prior buckwheat growth depleted available P and reduced subsequent ryegrass uptake aligns with findings by Possinger et al. (2013). In the Possinger et al. (2013) study, where they grow buckwheat for a short 21-day growth period in a sandy loam soil, the authors noted that enhanced P uptake by buckwheat did not directly correlate with increased P availability in the bulk soil for a subsequent crop in the short term. Instead of acting as a net mobilizer that

leaves a surplus of available P, the buckwheat in our study acted as a strong P sink, rapidly accumulating the mobilized P into its own harvested biomass and leaving the soil depleted for the subsequent crop.

Furthermore, while the physical removal of the buckwheat biomass in our study clearly transitioned the cover crop into a net P sink, literature suggests that even the retention of its residue may fail to alleviate short-term P deficiency. As demonstrated by Hansen et al. (2022) the mineralization of cover crop residues is highly dependent on specific quality traits, particularly the Carbon-to-Phosphorus (C:P) ratio, which must generally remain below 100 to facilitate net P release. In their radioisotope study, even when buckwheat residues were standardized to deliver the same total P load as mineral fertilizers and allowed to decompose for five weeks prior to planting, the incorporation of this residue resulted in significantly less soil P uptake by the subsequent ryegrass crop than an unfertilized control. While the C:P ratio of the buckwheat biomass was not directly evaluated in our study, its mineralization potential can be assessed through its tissue P concentration. Arcand et al. (2010) established that crop residues generally require a critical P concentration of at least 2.9 mg g^{-1} to achieve net P mineralization. The maximum P concentration observed in the buckwheat tissue during our experiment was only 2.16 mg g^{-1} . Because this value falls well below the critical threshold, it is highly probable that retaining and incorporating our buckwheat residue would have similarly induced microbial P immobilization rather than acting as a viable P source. Therefore, whether the biomass is physically exported as an agronomic sink (Possinger et al., 2013) or retained as a high-carbon residue (Hansen et al., 2022), buckwheat cover cropping alone is fundamentally insufficient to meet the immediate P demands of a fast-growing subsequent crop.

3.5.3. Phosphorus Uptake Dynamics in Ryegrass

3.5.3.1. Effects of Soil

The significant interaction between soil type and fertilizer treatment on cumulative ryegrass P uptake ($p=0.04$; Table 3.2, Figure 3.3) highlights the differing P-retention capacities of the two soils. Despite the Woodmore sand possessing a higher initial concentration of easily extractable P (Olsen-P of 7 mg kg⁻¹), it was the Libau clay (Olsen-P of 5 mg kg⁻¹) that supported greater cumulative P uptake in the absence of fertilizer (Control treatment), when averaged across planted and unplanted treatments. This demonstrates the robust P-buffering capacity of the clay soil (Shirvani et al., 2005). While the sand represents a small, easily accessible pool that is rapidly exhausted by plant uptake, the clay functions as a substantial, steady reservoir, continuously releasing P to the soil solution to meet plant demand over an extended period (Holford, 1997).

Although the initial buckwheat crop successfully mined available P from both environments, the subsequent penalty on ryegrass was disproportionately severe in the Libau clay, as shown by the significant interaction between soil and plant presence. Specifically, prior planting with buckwheat reduced subsequent ryegrass P uptake by approximately 40% in the Libau soil, compared to only a 27% reduction in the Woodmore soil (Figure 3.2), even though buckwheat P uptake was similar between the two soils. This pronounced depletion effect suggests that once the readily available P pool was reduced by buckwheat, the high-fixation nature typical of alkaline clay soils tightly bound a large proportion of the remaining residual P (Holford, 1997; Shirvani et al., 2005). Furthermore, the exceptionally high initial magnesium concentration in the Libau clay (1497 mg kg⁻¹) theoretically suppresses further dissolution of the fertilizer via the common ion effect; a soil solution already heavily saturated with Mg²⁺ shifts the dissolution equilibrium of the magnesium-

based struvite ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$) strongly toward the solid phase (Pinatha et al., 2017). In contrast, the Woodmore sandy soil, possessing an inherently lower fixation capacity, allowed a comparatively greater proportion of its remaining P to stay accessible to the second crop. This observation aligns with the established soil dynamics described by Holford (1997), who noted that sandy soils possess an inherently lower P-fixation capacity, thereby keeping residual P more biologically available rather than locking it into insoluble mineral complexes.

3.5.3.2. Effects of Fertilizer

The contrasting performance of the Struvite Retained treatment across the two soil types (Figure 3.3) provides compelling evidence for the pH-dependent dissolution of struvite. In the acidic Woodmore sand, the Struvite Retained treatment matched the cumulative efficacy of the highly soluble MAP treatment. This indicates that the low soil pH successfully drove the continuous dissolution of the struvite mineral structure over the extended timeframe, releasing plant-available orthophosphate. This mechanism is directly supported by past research on dissolution rate of granular struvite across different pH levels which demonstrates that struvite granules dissolve more than 10 times faster in acidic soils (up to 0.43 mg day^{-1}) compared to alkaline soils ($\sim 0.03 \text{ mg day}^{-1}$) (Degryse et al., 2017). Conversely, the alkaline environment of the Libau clay severely inhibited this dissolution process, rendering the retained struvite agronomically ineffective and statistically indistinguishable from the unfertilized control in terms of P supply to ryegrass. This stark divergence confirms that the utility of struvite as a slow-release fertilizer is fundamentally constrained by the receiving soil's pH (Degryse et al., 2017; Thiessen Martens et al., 2023).

The significant agronomic benefit observed during the 60-day Phase 2 in the Struvite Retained treatment, limited to the acidic Woodmore soil, illustrates the subsequent availability of the

fertilizer. This demonstrates that under favourable pH conditions, retained struvite continues to supply P beyond the initial application period. Talboys et al. (2016) noted that during a single crop cycle, only 26% of the total mass of the applied struvite granules dissolved. This partial dissolution paradigm explains the trajectory of our experiment: the struvite granules remained largely intact and unavailable during the rapid Phase 1 buckwheat cycle but continued to slowly dissolve during Phase 2. By retaining these partially dissolved granules in the soil for Phase 2, the subsequent ryegrass was able to capitalize on this delayed P release, resulting in significantly higher biomass and P uptake compared to treatments where the residual granules were physically removed. Deinert et al. (2023) demonstrated that in Irish grasslands characterized by neutral to acidic loamy soils, struvite functions as a highly effective, direct substitute for soluble mineral fertilizers in short-term perennial ryegrass (*Lolium perenne* L.) production, simultaneously increasing plant dry weights and stimulating acid phosphatase activity. Similarly, Rech et al. (2018) evaluated multiple struvite formulations and found that despite their inherently low water solubility, struvite applied to slightly acidic to neutral soils achieved up to 80% of the P recovery observed with highly soluble TSP within just 38 days.

The inclusion of the Struvite Removed treatment offers vital physical validation of struvite's slow-release dynamics. In the Woodmore soil, physically removing the granules following Phase 1 caused subsequent ryegrass P uptake to plummet to values similar to the control. This confirms that the granules had only partially dissolved during the initial buckwheat growth phase. The fact that the retained granules sustained P uptake equal to MAP in Phase 2 in this soil type proves that struvite functions as an ongoing, sustained P source, provided the soil chemical environment is conducive to continuous, gradual dissolution (Johnston & Richards, 2003). In the Libau soil, the removal of the granules had no statistical impact compared to the retained or control treatments,

further confirming that virtually no dissolution occurred in the alkaline conditions in either Phase 1 or Phase 2.

3.5.4. Reconciling Olsen-P Dynamics with Plant Uptake: Biological Depletion and Methodological Limitations

The significant reduction in Olsen-P concentrations following the Phase 1 growth period ($p=0.01$; Table 3.2) demonstrates that biological uptake acted as a substantial sink, overwhelming any localized chemical mobilization. While buckwheat is well-documented to exude organic acids that solubilize fixed soil P (Tebboh & Franzen, 2011), the P mobilized by these root exudates was likely utilized immediately by the plant for its own growth. Consequently, natural soil buffering could not replenish the labile Olsen-P to compensate for the P removed from the soil in the harvested buckwheat biomass. Interestingly, the drop in soil Olsen-P (approximately 2.5 mg kg^{-1}) was much larger than the amount of P found in the harvested plants (approximately 1 mg pot^{-1} , equivalent to 0.5 mg kg^{-1} soil). Even after accounting for P stored in the roots, the soil lost more available P than the plants took up. This suggests that soil Olsen-P does not always directly translate to plant P content. While we don't have a perfect explanation for this gap, it indicates that the soil was unable to keep its P concentration balanced once the plants started growing. This disproportionate drop shows that plant demand can deplete the soil's measured available pool much faster than the soil can naturally replenish it.

The interaction between soil type and fertilizer source on Olsen-P ($p=0.007$; Figure 3.1) further highlights the complex dissolution dynamics of struvite. In the Woodmore sand, struvite application increased Phase 1 Olsen-P to levels equivalent to MAP, yet this was not reflected in the corresponding buckwheat P uptake. One explanation for this disconnect is a spatiotemporal

mismatch between plant demand and fertilizer dissolution. Buckwheat exhibits rapid early-stage growth, establishing its peak P demand within the first few weeks. Because struvite dissolution is gradual and time-dependent (Talboys et al., 2016), a significant portion of the granules may have dissolved late in Phase 1 releasing P into the soil after the critical window for buckwheat uptake had closed. Therefore, the labile P was present in the soil at the time of the destructive harvest and chemical extraction, but not when the plant actively required it.

Alternatively, this discrepancy may highlight a recognized methodological limitation of using conventional soil extractants on sparingly soluble fertilizers, a dynamic explicitly supported by the findings in Chapter 2. The Olsen-P method utilizes a sodium bicarbonate extractant (Olsen et al., 1954). As demonstrated in our incubation experiments (Chapter 2), simply retaining struvite granules within the soil sample during extraction significantly increases the measured Olsen-P concentrations. Furthermore, the standard laboratory preparation step of grinding the soil samples exacerbates this effect, artificially inflating the Olsen-P concentrations in the retained treatments. It is highly plausible that the undissolved residual struvite granules remaining in the Woodmore soil were inadvertently dissolved during the laboratory extraction process itself. If the chemical extractant solubilized the struvite in the sample tube, it would artificially inflate the available P reading, falsely indicating that the P was available to the plant during the greenhouse phase. This discrepancy suggests a decoupling between standard soil test values and actual agronomic effectiveness, a limitation frequently observed with low-water-solubility fertilizers like struvite (Meyer et al., 2018) and demonstrated in Chapter 2. Recent field studies on Manitoba soils confirm that Olsen-P consistently overpredicts the actual environmental solubility of struvite (Wilson et al., 2025), and incubation studies have shown that sample preparation methods like grinding can artificially spike extractable P readings, supported by our results in Chapter 2.

3.5.5. Predicting P uptake: The Utility of the Olsen-P test

The overall weak relationship between Phase 2 soil Olsen-P and cumulative P uptake by ryegrass ($r^2 = 0.24$), alongside the statistically indistinguishable and flat regression slopes for the Control, MAP, and Struvite Removed treatments, highlights the limitations of the Olsen-P extraction method in low-P soils. This statistical disconnect is heavily supported by field-scale agronomic trials. In a comprehensive study spanning 154 locations, McKenzie et al. (2003) found that various soil P tests accounted for a maximum of only 7% to 27% of the variance in crop fertilizer response. In another example, Kulhánek et al. (2007) evaluated the efficacy of multiple soil P tests across six distinct field sites in the Czech Republic. In their study, barley was grown in sandy loam soils (pH 5.8 to 7.1) under varying climatic conditions and subjected to a gradual increase of P alongside organic and mineral fertilizers at rates up to 52 kg P ha⁻¹ yr⁻¹. Despite the application of fertilizer, their regression analysis revealed no statistical dependence between plant P uptake and the Olsen extraction method ($r^2 = 0.25$), with the method explaining at most 25% of the variance (Kulhánek et al., 2007). This directly mirrors our findings and validates that Olsen-P is an inherently weak predictor of plant-available P in acidic-to-neutral sandy soils, just as it is in highly alkaline clays. Furthermore, our finding that the Olsen-P $\times$ Soil interaction was not significant aligns with their observation that segregating analyses by distinct soil types or incorporating covariates like pH and clay yields only negligible improvements in predictive accuracy. In contrast, Recena et al. (2022) found that environmental drivers, specifically soil pH and clay content, have been shown to explain up to 49% of the variance in Olsen-P thresholds by directly controlling a soil's P buffer and sorption capacity. The physicochemical complexity represented in their study is particularly pronounced in alkaline soils similar to our Libau clay. As demonstrated by Bouray et al. (2024), accurately predicting Olsen-P variability in calcareous environments requires accounting for a highly

heterogeneous web of soil chemistry, including carbonates, cation exchange capacity, and micronutrient interactions. Another reason that Olsen-P fails to accurately predict plant uptake is because this simple chemical extraction ignores the complex biological mechanisms, such as root phosphatase activity, that govern P acquisition when readily available inorganic P is scarce (Recena et al., 2015).

The broad failure of the initial Olsen-P test to predict uptake in our MAP treatment can likely be attributed to the rapid dissolution dynamics typical of highly soluble fertilizers (McKenzie et al., 2003). In these instances, a transient flush of P is often quickly subjected to soil fixation mechanisms before the plant can fully utilize it. However, a distinct and statistically significant positive relationship between Olsen-P and ryegrass P uptake was observed exclusively in the Struvite Retained treatment (slope = 0.40; Figure 3.4). This divergence from the MAP treatment suggests that the Struvite Retained treatment may have avoided the rapid dissolution dynamics often associated with highly soluble fertilizers. Instead, the gradual dissolution of retained struvite likely provided a more consistent supply of P to the soil solution. This mechanism, combined with the varying response across soil types, likely revealed a correlation that was otherwise obscured by rapid P fixation. Specifically, the contrast between the low P uptake in the Libau soil and the significantly higher uptake in the Woodmore soil provided the broad data range necessary for this significant relationship to emerge.

These findings underscore a critical complexity in modern agronomic management: standard chemical soil tests, calibrated for highly soluble fertilizers, may be inadequate for predicting the efficacy of sparingly soluble, recovered nutrients. Notably, however, when struvite granules were retained in the soil sample, Olsen-P did serve as a reliable predictive tool, suggesting that sampling methodology may play an equally important role as the test itself. When the physical granules

were extracted prior to Phase 2 (Struvite Removed), the predictive slope collapsed to levels statistically identical to the Control (-0.0078), suggesting that the ongoing dissolution of the struvite granule, rather than the initial snapshot of dissolved P, was the primary driver of crop nutrition in Phase 2. This has practical implications for soil testing methodology: whether struvite granules are retained or removed during soil sample processing may fundamentally alter the predictive value of Olsen-P in struvite-amended soils. Retaining granules appears to better capture the plant-available P supplied by slowly dissolving struvite, whereas removing them may cause Olsen-P to substantially underestimate crop-available P. Consequently, standardizing how granular struvite is handled during soil sampling and extraction may be an important consideration for improving the accuracy of fertilizer recommendations in soils amended with this recovered nutrient (Gu et al., 2021; Thiessen Martens et al., 2023).

3.6. Conclusion

Building directly upon the chemical constraints of standard extractions, this study demonstrates that Olsen-P poorly predicts actual P availability from slow-release sources like struvite, creating significant risks for agronomic management compared to conventional fertilizers like MAP. While prior cropping with buckwheat is frequently cited as a tool for P mobilization, this experiment revealed it acted as a P sink, depleting available reserves and substantially reducing subsequent ryegrass P uptake. Furthermore, the Phase 2 plant uptake by ryegrass confirmed the pH-dependent dissolution dynamics observed previously: retained struvite matched the efficacy of highly soluble MAP in the acidic sandy soil but was severely inhibited in the alkaline clay, rendering it agronomically ineffective in the high-pH environment. Crucially, the physical removal of residual granules prior to the bioassay caused ryegrass P uptake to decline to unfertilized control levels. This confirms that the elevated Olsen-P readings in samples with retained residual struvite granules

are largely laboratory artifacts caused by the chemical extractant, rather than true reflections of early-season plant availability. Ultimately, these findings reinforce that standard chemical extractions inherently fail to capture the complex, time-dependent dissolution of recovered nutrients like struvite, necessitating targeted methodological adjustments to accurately predict plant performance.

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4. SYNTHESIS

This chapter synthesizes the core findings of the thesis, drawing together the chemical analysis results of the soil incubation study and the biological results of the pot experiment. By evaluating struvite against highly soluble monoammonium phosphate (MAP) across contrasting soil types, this research exposes the limitations of standard soil testing for struvite. The following sections summarize the study's key findings and accomplishments, address its limitations, and offer practical recommendations for testing and managing slow-release fertilizers.

4.1. Key Findings

A controlled soil incubation study (Ch. 2) demonstrated that standard sample preparation methods, specifically physical grinding, artificially inflated Olsen-P concentrations in struvite-amended soils. While unground struvite maintained a distinct release profile, the grinding process increased its solubility to the point where it produced Olsen-P levels comparable to the highly soluble MAP. This suggests that conventional grinding may lead to an overestimation of immediate Phosphorus (P) availability for granular, slow-release fertilizers like struvite. A subsequent bioassay pot experiment (Ch. 3) showed that prior crop (buckwheat) growth depleted rather than mobilized P, and crucially, that removing residual struvite granules after a four-week period dropped subsequent plant uptake to be similar to the control. The bioassay also demonstrated that a standard soil test P extraction was a poor predictor of actual plant performance ($r^2 = 0.24$).

When synthesizing the chemical incubation data (Ch. 2) with the biological bioassay results (Ch. 3), a stark contradiction emerges between laboratory-derived soil test phosphorus (STP) and actual agronomic performance of soils amended with struvite. The incubation study demonstrated that

under certain standard testing conditions (grinding the soil samples before extraction and retaining the struvite granules in soil samples), struvite can yield very high Olsen-P concentrations. However, the subsequent growth chamber pot experiment (Ch. 3) proved that these elevated chemical readings in the soil incubation do not translate to biological availability. The overall relationship between initial Olsen-P and subsequent plant P uptake was fundamentally weak ($r^2=0.24$), indicating that standard soil extractions are poor predictors of immediate agronomic value. Interestingly, the steeper slope observed in the Struvite Retained treatment compared to MAP suggests that this predictive relationship is even weaker for conventional soluble fertilizers than for slow-release sources.

While the ultimate outcomes of the two experiments diverge, their underlying mechanisms are highly complementary, both pointing to the physical integrity of the struvite granule as the source of the soil testing disconnect. The incubation experiment established that physical fragmentation (soil grinding) or forced chemical extraction artificially inflates early P readings by breaking down intact granules. The pot experiment confirmed this artifact biologically. When residual granules were removed prior to the ryegrass bioassay, plant P uptake plummeted to baseline control levels. This proves that minimal natural dissolution occurred during the preceding buckwheat phase, and that the high STP readings were largely a product of laboratory interference rather than natural soil solution dynamics.

Both experiments align in demonstrating that struvite's behaviour is far more dependent on inherent soil properties than highly soluble conventional fertilizers like MAP. In the incubation study (Ch. 2), soil type exerted a significantly greater influence on Olsen-P concentrations for struvite than for MAP, with the acidic sand (Woodmore) yielding statistically higher

concentrations than the alkaline clay (Libau). This chemical variance was perfectly mirrored in the biological pot experiment (Ch. 3), where struvite's efficacy matched MAP in the acidic sand (Woodmore) but failed to provide any agronomic benefit over the unfertilized control in the alkaline clay (Libau).

Finally, synthesizing the two phases of the pot experiment reveals that short-term biological interventions are insufficient to overcome struvite's low solubility. The introduction of a preceding crop (buckwheat) acted solely as a P-depleting factor, failing to biologically mobilize P for the subsequent ryegrass crop. This underscores that the slow-release nature of intact struvite granules persists despite active root systems.

4.2. Project Accomplishments

To the best of our knowledge, this is the first study to empirically isolate and test the physical removal versus the retention of residual struvite granules prior to STP extraction. While previous literature has widely hypothesized that retaining intact granules might overestimate STP, this phenomenon had not been rigorously quantified in a controlled experimental design. By directly comparing the Retained and Removed methodologies, this project successfully transitioned a theoretical warning into validated, quantifiable data, providing the first direct evidence of how profoundly physical fragmentation dictates standard soil test outcomes for slow-release fertilizers.

Beyond the specific mechanical and agronomic findings, a major accomplishment of this project is its contribution to global agricultural sustainability. By rigorously evaluating struvite recovered from municipal wastewater, this research successfully validates a viable pathway to reduce agricultural reliance on finite, non-renewable mined phosphate rock. The data confirms that under

the right soil conditions, recycled P can serve as a highly effective slow-release nutrient. This supports a necessary paradigm shift toward a circular agricultural economy as advocated by many researchers where municipal waste streams are repurposed to meet global food security demands while simultaneously minimizing the environmental risks of P runoff and loss. Furthermore, this project successfully decoupled the concept of extractable STP from actual phytoavailable (plant-ready) P when using struvite. By identifying the specific laboratory artifacts that inflate soil test results, this research provides the foundational knowledge required to develop more accurate diagnostic tools. Establishing a more accurate baseline of plant-available P is a critical accomplishment, as it is the mandatory first step toward preventing the systemic over-fertilization and resource waste that currently hinders P management.

4.3. Limitation and Analytical Gaps

While this study successfully identified the mechanical artifacts caused by including and excluding struvite granules in soil samples, a distinct analytical gap remains regarding how to accurately handle samples shortly after application. The soil incubation data (Ch. 2) clearly demonstrate that retaining granules artificially inflates early Olsen-P values. Conversely, manually removing the granules drastically reduces the Olsen-P value, which may inadvertently underestimate the P that would naturally dissolve into the immediate soil environment over time. This underestimation is supported by the lack of a relationship between Olsen-P and plant P uptake in the struvite-removed treatments in our Chapter 3 results, as well as the lower plant P uptake observed in the Struvite Retained versus Struvite Removed treatments in the Woodmore soil. However, the incubation data (Ch. 2) revealed that both the Retained and Removed treatments statistically converge to similar values between 56 and 84 days. This convergence indicates that the methodological dilemma is

strictly a temporary, early-stage artifact. Until this convergence occurs, chemical extractions alone are insufficient to determine which preparation method more accurately reflects true agronomic value.

An additional practical limitation of this study lies in the physical execution of the Removed treatment methodology. Manually extracting residual, partially dissolved struvite granules from the soil matrix prior to extraction proved to be a highly complex and labour-intensive process. While rigorous protocols were followed to ensure maximum extraction, the fundamental physical reality of soil matrices makes it highly probable that complete, 100% removal was not universally achieved across all samples. Microscopic fragments or highly weathered granules may have been inadvertently retained. Consequently, if trace amounts of residual struvite remained in the Removed samples, they may have marginally inflated the resulting Olsen-P values upon extraction. This highlights a significant methodological constraint: achieving absolute physical separation of a degrading granular fertilizer from surrounding soil aggregates is practically unfeasible, which introduces a slight, unavoidable margin of error when attempting to establish a zero-granule baseline.

While the laboratory and pot studies highlighted the inherent difficulty of manually removing granules from soil matrices, preliminary one-year field trials (Appendix A) conducted alongside this research revealed that this logistical challenge scales exponentially in a true agricultural setting. In a controlled pot, the fertilizer zone is known; in the field, accurately locating the exact fertilizer band to pull a precision soil core becomes increasingly difficult as the crop matures. Once the buckwheat canopy closed, physically identifying the location to sample within or between the rows was highly problematic, a challenge that was even more pronounced in the no-plant

treatments where rows were entirely absent. Furthermore, attempting to visually identify and extract weathered struvite granules from heterogeneous, naturally occurring soil aggregates in field samples proved to be severely impractical. This introduces a major limitation for practical agronomy: precision sampling directly on the fertilizer row is highly susceptible to spatial variability and extreme sampling error, as inadvertently capturing even one intact granule in a field core causes massive, artificial spikes in reported Olsen-P.

4.4. Future Research

While the 140-day incubation study established a statistical convergence of Olsen-P values between the Retained and Removed treatments for ground samples, starting around Day 84, the long-term stability of this trend calls for further investigation. Future laboratory studies should extend incubation periods significantly beyond 140 days to confirm whether this statistical convergence persists indefinitely or if secondary release dynamics occur late in the timeline. Furthermore, physical observations during the sample preparation process confirmed that intact struvite granules remained visibly present in the soil even at the 140-day conclusion of the experiment. The exact timeline required for the complete physical dissolution of these granules in varying soil types remains undefined. To address this, future experimental designs should incorporate a broader spectrum of soil types to explicitly clarify how different soil properties such as pH, texture, and organic matter content influence the degradation dynamics of the granules. Future studies must physically track the granules over a longer period to pinpoint exactly when they completely dissolve in the soil. Understanding this absolute endpoint is vital for mapping the full extent of struvite's slow-release nutrient curve.

To bridge the gap between controlled laboratory findings and practical agronomy, subsequent research must prioritize field-scale experiments across diverse climatic zones and soil types. A critical objective for future studies should be the establishment of specific, delayed sampling windows for soils treated with slow-release recovered nutrients. Because early sampling captures intact granules and heavily skews STP values, field trials must determine the precise duration required such as a 60-day post-application threshold for natural dissolution to occur under specific regional conditions. Pinpointing this timeframe in the field is essential to prevent the systemic overestimation or underestimation of plant-available P. Establishing this timeline in the field requires robust methodologies, such as using buried mesh bags to physically recover and assess granule dissolution over time. Implementing these methods is essential to prevent systemic miscalculations of plant-available P.

Preliminary data from our one-year field study (Appendix A) confirmed that the extraction artifacts identified in the laboratory, specifically the dramatic overestimation of Olsen-P when granules are retained in the sample, do manifest in real-world field conditions before eventually stabilizing later in the season. However, a single growing season is fundamentally insufficient to map the complete dissolution curve and total agronomic value of a slow-release resource. Future research must prioritize multi-year field studies that extend continuous soil and plant tracking well beyond a single crop's lifespan. Investigating the residual P availability of struvite for subsequent crops in a rotation, as well as understanding how climatic conditions impact the physical breakdown of granules remaining after the first harvest, is essential. Only through multi-year, multi-crop tracking can the true, long-term efficiency of recycled P be accurately quantified.

4.5. Recommendations

4.5.1. For Farmers

The superior performance of retained struvite in specific environments demonstrates its efficacy as a slow-release source, particularly in challenging, low-P soils like acidic sands. Farmers operating on these soil types can integrate wastewater-recovered struvite into their nutrient management plans as a sustainable alternative to conventional soluble fertilizers. However, the most critical recommendation for growers is to exercise extreme caution when interpreting standard soil test reports following a struvite application. Because there is a vast difference between total applied P and immediate phytoavailable P, relying on artificially inflated soil tests will lead to severe mismanagement. If a standard lab report overestimates the available P, a farmer might mistakenly withhold necessary supplemental nutrients; conversely, misunderstanding the slow-release curve could lead to unnecessary over-application. Farmers must work closely with agronomists to understand that struvite's value lies in its long-term, slow-release trajectory, and nutrient management plans should be adjusted to account for delayed availability rather than immediate crop uptake. It is particularly important to account for this in alkaline soils, where the delay may be substantially longer and the overall crop response reduced.

4.5.2. For Soil Test Labs

The methodological biases identified in our research present a significant vulnerability for agricultural advisory services. If commercial laboratories subject struvite-amended soils to standard grinding and extraction protocols, they will inadvertently mask the slow-release nature of the fertilizer. As cautioned by Gu et al. (2021) and Anderson et al. (2020), this artificial inflation of early P availability can lead to highly inaccurate agronomic recommendations, prompting

farmers to misunderstand the nutrient release curve of fertilizers applied to their fields. To mitigate this risk, soil testing facilities must either adjust sample preparation protocols to exclude intact granules or attach explicit advisories to soil test reports when slow-release P sources have been recently applied. Furthermore, adopting the delayed sampling windows proposed in future research would offer a practical workaround, ensuring tests are only conducted after the granules have naturally dissolved and the statistical convergence of STP values has been reached.

4.6. References

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APPENDIX

Appendix A: Effects of Residual Struvite on Soil Test Phosphorus in Field Conditions

This was a field study designed to determine the contribution of residual struvite granules to Olsen-P concentrations in struvite-amended soils over time, comparing environments with and without active plant growth. It is hypothesized that these residual granules will steadily increase Olsen-P levels, yielding significantly higher P concentrations in planted soils compared to unplanted controls. This expected difference is driven by the plants' ability to actively mobilize P through root exudates, which naturally enhance the dissolution of the struvite and release more available P directly into the soil solution.

Methods

This research was conducted in the field near Libau, Manitoba (50°14'30.1"N 96°43'39.5"W; Dencross series, Gleyed Rego Black Chernozem) in 2024. The soil at this site has a clay texture, a pH of 8.1-8.2, is low in soil test P (5 mg kg⁻¹), high in nitrate (52 kg ha⁻¹) and potassium (334 mg kg⁻¹), and contains a high level of organic matter (6.3%). The experimental design utilizes a randomized complete block design with three replications, utilizing a split-split-plot arrangement for treatments and sampling locations. The main plot treatments consisting of a control, struvite, and MAP, which are further divided into sub-plots evaluating the presence or absence of plants. To comprehensively assess P dynamics, sub-sub-plot treatments are based on three distinct sampling locations: on-row with fertilizer granules included, on-row with granules removed, and between rows. Sampling on the row with granules included helps determine how both dissolved and residual struvite impact Olsen P concentrations and potentially elevate soil test results. Conversely, sampling on the row with granules removed (which is specific to struvite plots, as it

is unfeasible for MAP and irrelevant for the control) isolates the contribution of dissolved granules; comparing these two on-row methods reveals the specific contribution of residual granules to measured Olsen-P. The between-row samples act as a baseline free from fertilizer or plant influence, and comparing these to the on-row samples provides insight into the broader field-level effects of banded fertilizers. To track these dynamics over time, repeated measurements were taken at weeks 0, 1, 2, 3, 4, 6, and 8 after seeding and fertilizer application, concluding with a final fall sample at week 16.

The overall plot size was 20 m², with dimensions of 10 m in length and 2 m in width. Within each plot, the plant-free and with-plants areas occupied 1 m in width and 2 m in length. The remaining portions of the plots were used for related research conducted by another individual. Seeding was done in 12 rows in each plot, each 10 m long, with 0.15 m row spacing. The plots were planted on June 27, 2024, using a Fabro plot-scale double-disc drill. Struvite and MAP fertilizers were applied directly into the furrows alongside the seeds at the designated rates for each treatment. For the sub-plot treatments, the plant-free area was maintained by manually removing buckwheat plants and any weeds as the plants emerged from the soil and before each soil sample was taken. In this field experiment, buckwheat was seeded at a target density of 150 pl m⁻², with a seeding rate of 256.4 seeds m⁻² or 87 kg ha⁻¹. Struvite (28% P₂O₅) was applied at a rate of 245.52 kg ha⁻¹, requiring 449 g per plot. MAP (52% P₂O₅) was applied at a rate of 132.20 kg ha⁻¹, requiring 242 g per plot. For both fertilizers, the P application rate was 30 kg ha⁻¹.

Soil sampling depth was 5 cm, using a cylindrical probe with approximately 4 cm diameter. Each sample included two sampling points combined into a composite sample. After collecting the soil samples from the field, struvite granules were removed immediately from the ‘fertilizer removed’

samples. Additionally, all samples are carefully inspected to remove any debris. Once cleaned, the soil samples were placed in an air-drying room set at 32°C for 4-5 days, or until thoroughly dried. Samples were then stored at room temperature until further processing. This study used the Olsen-P test to evaluate the availability of P in soil samples. Samples were analyzed by a commercial lab, using standard procedures.

Statistical Analysis

A three-way repeated-measures ANOVA was used to assess the broad effects of Fertilizer Type (Control, MAP, Struvite), Plant Presence (with vs. without), and Time (Week 0–16) on log-transformed Olsen-P concentrations. This model was designed to compare the overall performance and interactions of the different P sources across the entire study period. A separate three-way repeated-measures ANOVA was performed exclusively on the Struvite treatments to investigate spatial P dynamics. This model examined the effects of Sampling Position (on-row with granules vs. on-row with granules removed), Plant Presence, and Time. By isolating the struvite data, this analysis focused on the localized impact of struvite granules and the influence of the rhizosphere on struvite dissolution.

Results

The Olsen-P concentrations measured reflect P availability within the fertilizer band and the shallow soil layer sampled. These results do not represent the overall field P status but provide insights into localized P dynamics near struvite granules and plant roots.

Table A - 1 ANOVA results reflecting the effects of Fertilizer (Control, MAP, Struvite), Plant (with vs. without), and Week (0–16) treatments and their interactions on log-transformed Olsen-P concentrations ($\alpha = 0.05$).

Effect	p-value
Fertilizer	<0.0001
Plant	0.81
Week	<0.0001
Fertilizer*Plant	0.48
Fertilizer*Week	0.0006 ^a
Plant*Week	0.37
Fertilizer*Plant*Week	0.82

^aThe highest-order interaction that is significant is plotted in figure A-1.

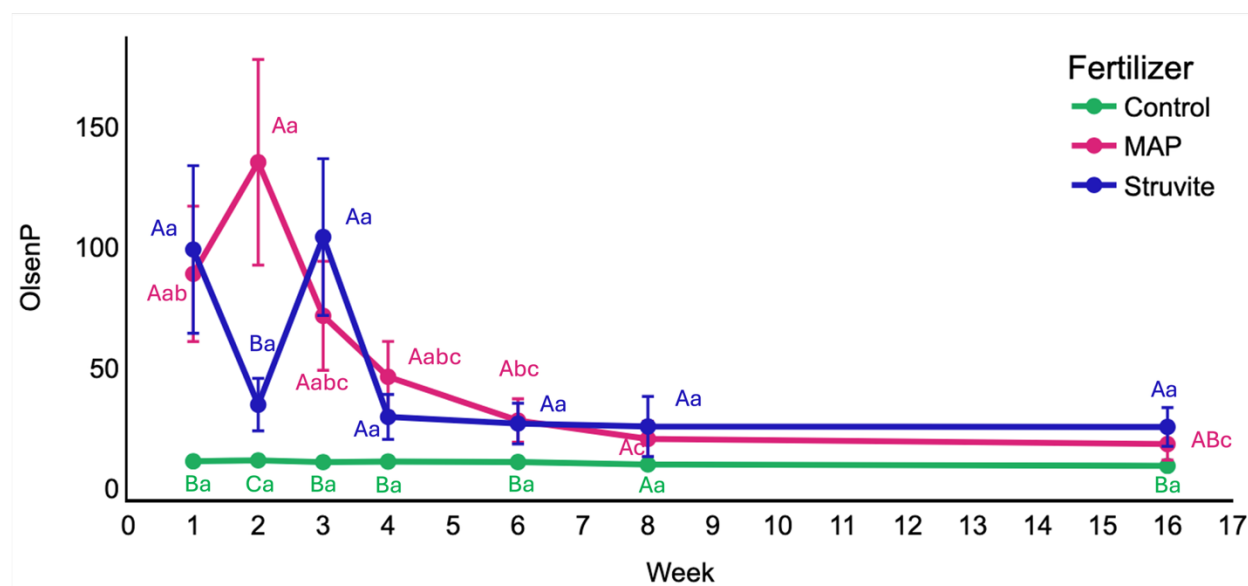


Figure A- 1 Olsen-P concentrations over time in Control, MAP, and Struvite treatments. Values represent soil samples collected from 0–5 cm depth within fertilizer bands. Letters indicate significant differences: capital letters (A, B, C) compare means within each week; lowercase letters (a, b, c) compare means within each fertilizer treatment across weeks ($p < 0.05$).

Table A - 2 ANOVA results for Struvite treatment only, showing effects of Position (on-row with granules, on-row granules removed), Week (0-16), and Plant (with/without) on log-transformed Olsen-P concentrations ($\alpha = 0.05$).

Effect	p-value
Plant	0.86
Position	<0.0001
Week	0.01
Plant*Position	0.10
Plant* Week	0.10
Position*Week	0.001 ^a
Plant*Position*Week	0.83

^aThe highest-order interaction that is significant is plotted in Figure A-2.

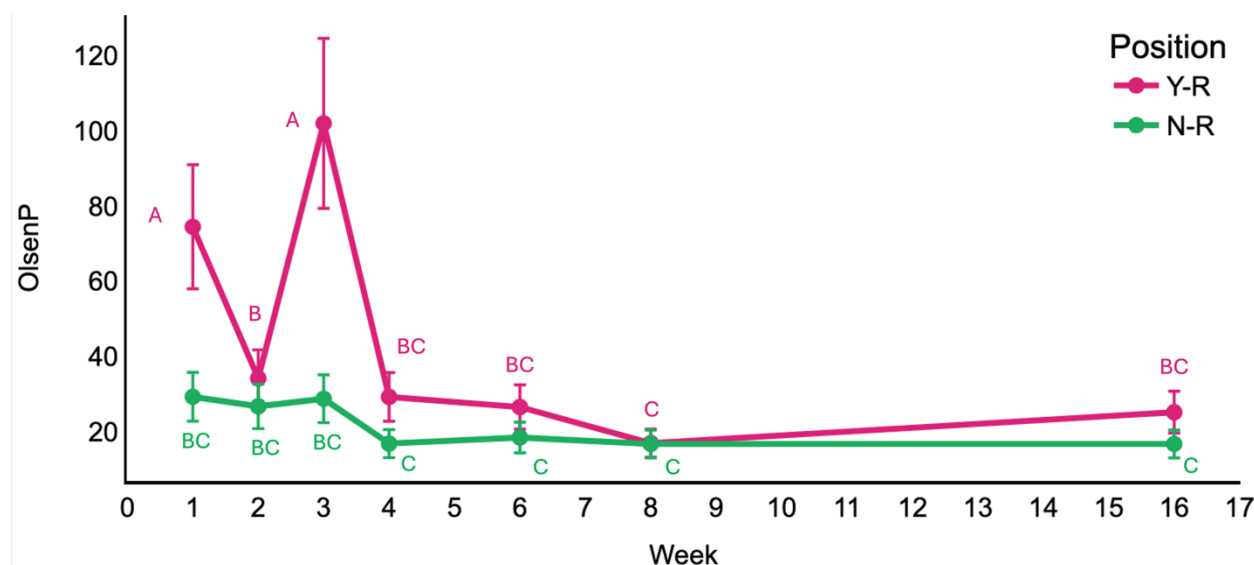


Figure A- 2 Olsen-P concentrations over time at two sampling positions (Y-R: on-row, granules included; N-R: on-row, granules removed) within the Struvite treatment. Letters indicate significant differences: capital letters compare all sampling position and week combinations at $p < 0.05$.

Discussion

The application of both struvite and MAP significantly increased Olsen-P concentrations compared to the control group during the first six weeks of the study, after which the levels became statistically similar to the control. The temporal trends differed between the treatments, with Olsen-P peaking earlier in the MAP plots and increasing more gradually in the struvite plots, further supporting struvite's slow-release characteristics. Early in the study, sampling position also played a key role; soils sampled on-row with fertilizer granules included (Y-R) exhibited higher Olsen-P levels than those with granules removed (N-R), highlighting the initial contribution of residual struvite granules. However, this difference was no longer observed after week 4. Notably, the presence or absence of plants did not significantly influence Olsen-P concentrations in this analysis. Given these findings, future research should focus on monitoring residual struvite granules beyond a single growing season to evaluate their continued contribution to Olsen-P levels in subsequent years.