

A better understanding of the effectiveness of placed phosphorus fertilisation with struvite for silage maize: A pot experiment

Laura M.E. Ferron^{a,*}, Gerwin F. Koopmans^b, Henry Rommelse^b, Jan Willem Van Groenigen^a, Inge C. Regelink^c

^a Soil Biology Group, Wageningen University & Research, P.O. BOX 47, 6700, AA, Wageningen, the Netherlands

^b Soil Chemistry and Chemical Soil Quality Group, Wageningen University & Research, P.O. BOX 47, 6700, AA, Wageningen, the Netherlands

^c Wageningen Environmental Research, Wageningen University & Research, P.O. BOX 47, 6700, AA, Wageningen, the Netherlands

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ABSTRACT

Struvite is emerging as a circular, slow-release phosphorus (P) fertiliser. However, its effectiveness for crops and its impact on soil P tests still remain largely unclear. We analysed the effectiveness of struvite as a row fertiliser in a pot experiment with maize in a sandy soil and a loamy soil, both exhibiting a low agronomic soil P status. Maize was seeded in both soils, amended with either granular struvite or water-soluble di-ammonium phosphate (DAP) as row fertilisers. A positive control received an excess of sodium phosphate, mixed homogeneously through both soils; a negative control did not receive additional P. We measured the agronomic maize performance at the end of the vegetative growth stage (V10) and assessed the suitability of two established agronomic soil P tests (*i.e.* P-CaCl₂ and P-AL) to predict plant-available P in the soils amended with struvite. Furthermore, we performed an additional batch experiment to better understand the dissolution dynamics of struvite during a prolonged period of extraction of soil in 0.01 M CaCl₂. Placed struvite application led to a lower performance of maize in comparison to DAP in terms of biomass production as well as P uptake (53 and 71 % lower, respectively). Yet the agronomic performance of struvite in general was higher than the performance of the negative control, confirming the potential of struvite as an emerging P fertiliser. Surprisingly, both soils fertilised with struvite showed a significantly higher soil P test than any other P treatment in our pot experiment. This was an artefact due to the dissolution of residual struvite granules during the soil extraction procedures. These results call for a reconsideration of how to interpret P-CaCl₂ and P-AL as a basis for P fertiliser recommendations for soils receiving struvite as a P fertiliser.

1. Introduction

Phosphorus (P) is one of the main nutrients limiting plant growth globally, both on natural and agricultural lands (Du et al., 2020; Hou et al., 2020). Although soils generally do contain large amounts of P, it is usually largely unavailable to plants as most P is part of primary minerals or bound to metal (hydr)oxides and clay mineral edges or present as organic P (Hesterberg, 2010; Yang et al., 2013). To remediate the resulting P-limited plant growth conditions, P fertilisers are applied to agricultural land. The most commonly used mineral P fertilisers, such as triple super phosphate (TSP), come from phosphate rock, which is a non-renewable, limited resource. In addition to being concentrated in the soil of only a few countries, this source will become depleted somewhere in the coming centuries (Cordell et al., 2009; Koppelaar and Weikard,

2013; Walan et al., 2014). This necessitates a rethinking of the origin of the P fertiliser we apply in agriculture. In particular, recycling P from waste streams seems a promising alternative to phosphate rock-based fertilisers (Koppelaar and Weikard, 2013; van Dijk et al., 2016; Vol- laro et al., 2017).

Perhaps the most promising recycled P fertiliser is struvite (Mg(NH₄) PO₄·(6H₂O)_(s)). This salt can be recovered from a wide range of wastes such as wastewater, urine, and digestate (Ahmed et al., 2018; Kataki and Baruah, 2018). Although mainly considered a P fertiliser, struvite contains equimolar amounts of nitrogen (N) in the form of ammonium. Mineral fertilisers containing both ammonium and phosphate such as diammonium phosphate (DAP) are already being used and are soluble in water (Kongshaug et al., 2014). Struvite, however, has a lower solubility than presently used mineral P fertilisers (Degryse et al., 2017).

* Corresponding author.

E-mail address: laura.ferron@wur.nl (L.M.E. Ferron).

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Depending on the experimental conditions and the crops, the slow dissolution of struvite can lead to various effects on agronomic crop performance (Ahmed et al., 2018; Hertzberger et al., 2020). Maize is a crop commonly grown for its grain but also as silage, for which adequate P uptake in the early growth stage is critical for the yield and yet difficult to achieve when its root system is not yet well developed (Bender and van der Heijden, 2015). For this widely used crop, P fertilisation is thus highly recommendable. Struvite fertilisation trials on maize have led to inconclusive results: some studies report a higher performance of maize when fertilized with struvite as compared to a conventional fertiliser counterpart (Robles-Aguilar et al., 2020; Szymańska et al., 2019), while others report a similar performance (Gell et al., 2011; Muys et al., 2021; Nongqwenga et al., 2017; Uysal et al., 2014). In contrast, other studies report a lower maize performance with struvite as compared to a conventional counterpart (Hertzberger et al., 2021; Liu et al., 2011). Although there are various qualities of recovered struvite, a selection of seven ground struvite samples out of the tested eight resulted in the same performance of maize seedlings in a greenhouse trial (Muys et al., 2021). Thus, the variation in study outcomes with respect to maize performance is likely a result of the different experimental setups and the growing conditions of the plant, especially the struvite dose (which is usually excessive for greenhouse pot experiments with maize), the soil P characteristics, and the length of the experiment (Hertzberger et al., 2020). Furthermore, all these studies focused on broadcast application or homogeneous spreading of struvite while maize usually benefits from row application of fertilisers (Alam et al., 2018). Moreover, the fertilization effect of struvite seems to differ per soil (Nongqwenga et al., 2017).

In addition to measuring the agronomic performance of crops after fertiliser application, the use of agronomic soil P tests to extract P from soil provides a more complete insight into the effectiveness of a P fertiliser to raise plant-available P. In the Netherlands, P-CaCl₂ (calcium chloride) and P-AL (acetic acid and ammonium lactate) are used as a combined indicator to classify the agronomic soil P status of arable land and grassland as a basis for P fertiliser recommendation (Commissie Bemesting Grasland en Voedergewassen, 2023). With the introduction of P-CaCl₂ as an intensity method and P-AL as a quantity method (Nawara et al., 2017), it became possible to bring the role of soil P buffering into the Dutch P fertiliser recommendation system as the ratio of P-AL over P-CaCl₂ provides a more mechanistic insight into the availability of P in soil for plant uptake (Reijneveld et al., 2022; van Rotterdam et al., 2012). For determining P-CaCl₂, the multi-nutrient 0.01 M CaCl₂ extraction method based on a soil-to-solution ratio (SSR) of 1:10 (w:v) can be used, which mimics soil solution conditions (Houba et al., 2000; McDowell and Sharpley, 2001). The size of the P-CaCl₂ pool, which is commonly interpreted as a measure of the so-called readily plant-available P (Houba et al., 1996; Nawara et al., 2017; Reijneveld et al., 2022; van Doorn et al., 2023), is determined by P desorption being a function of the P loading of poorly crystalline Fe- and Al-(hydr)oxides in soil and soil properties like pH and soil organic matter (SOM) (Koopmans et al., 2004a; Weng et al., 2011). Poorly crystalline Fe- and Al-(hydr)oxides ([Fe + Al]-ox) and P reversibly adsorbed to these (hydr)oxides (P-ox) can be extracted from soil with 0.2 M acid ammonium oxalate (Schwertmann, 1964). The P loading of Fe- and Al-(hydr)oxides (α) in soil can be calculated as the molar ratio between P-ox and [Fe + Al]-ox (van der Zee and van Riemsdijk, 1988). With P-AL, soil is extracted with a mixture of acetic acid and ammonium lactate (Egnér et al., 1960). The size of the P-AL pool largely depends on the P loading α as well as the amount of P reversibly adsorbed to poorly crystalline Fe- and Al-(hydr)oxides (Schoumans and Groenendijk, 2000; van Doorn et al., 2023). Although P-CaCl₂ and P-AL are established agronomic soil P tests (Nawara et al., 2017; Reijneveld et al., 2022; van Doorn et al., 2023), their reliability has not been tested yet for soils to which struvite has been applied, while other agronomic soil P tests (Mehlich-3, Bray-1, and Olsen P) have been shown to give unreliable results for struvite-amended soils (Gu et al., 2021).

The objective of this study was twofold: first, we sought to assess the suitability of P-CaCl₂ and P-AL as routinely employed agronomic soil P tests for determining plant-available P in soils amended with struvite, and second, we aimed to further the understanding of the effectiveness of struvite as a placed P fertiliser for silage maize. To this end, we performed a 40-day pot experiment with silage maize (V10), using a sandy soil and a loamy soil with contrasting soil properties (including pH) but a low agronomic soil P status for comparing the performance of maize with placed fertilisation of struvite versus placed DAP fertilisation. Placed P fertilisation is often used as an application technique, including in the Netherlands, where the fertiliser is placed in a concentrated manner in or near the row of maize plants (van Schooten et al., 2019; Nkebiwe et al., 2016). Soil samples taken at the end of the pot experiment were used to determine P-CaCl₂ and P-AL so as to assess how plant-available P was related to aboveground biomass production and P uptake by maize. Furthermore, the 0.2 M acid ammonium oxalate extraction method was used to quantify how P-CaCl₂ in the struvite- and DAP-amended soils was related to the P loading α . Lastly, a batch experiment was done to quantify struvite dissolution during soil extraction with 0.01 M CaCl₂ using a shaking time varying from 1 to 24 h instead of the prescribed 2 h (Houba et al., 2000). Our hypotheses are (1) aboveground biomass will be higher for DAP than for struvite and higher on the sandy soil compared to the loamy soil, (2) there will be preferential root growth towards placed P fertiliser, (3) P-CaCl₂ will be higher for a soil treated with DAP than for a soil treated with struvite as struvite will not dissolve fully as opposed to DAP, (4) the P use efficiency (PUE) will be higher for DAP than for struvite and the PUE of both DAP and struvite will be higher for the sandy soil than for the loamy soil.

2. Materials and methods

2.1. Soils

The pot experiment was conducted with a sandy soil and a loamy soil. The sandy soil, a Plaggic Podzol (IUSS Working Group WRB, 2015), was sampled from an extensively managed grassland (Achterberg, the Netherlands), whereas the loamy soil, a Gleyic Fluvisol (IUSS Working Group WRB, 2015), was taken from a floodplain along the river Meuse (Lottum, the Netherlands). Both soils were collected from the top layer (0–25 cm). For the pot experiment, soils were air-dried and sieved over 5 mm. Prior to chemical analysis, a subsample from both soils was oven-dried (40°C) and passed through a 2 mm-sieve. Soil texture was determined via pipette and sieve after removal of CaCO₃ for the sandy soil (Houba et al., 1997) and by near-infrared spectroscopy for the loamy soil (Eurofins Agro, Wageningen, the Netherlands). The loss-on-ignition method (550°C) (Hoogsteen et al., 2015) was used to determine SOM. An extraction with 0.01 M CaCl₂ was used to measure the pH as well as the amounts of readily available N-NO₃, N-NH₄, and P-PO₄ (Houba et al., 2000). Moreover, P-AL (Egnér et al., 1960) was measured and an extraction with 0.2 M acid ammonium oxalate (Schwertmann, 1964) was used to determine P-ox and [Fe + Al]-ox to calculate the P loading α , i.e. the molar ratio of P bound to Fe and Al (hydr)oxides to the number of Fe and Al (hydr)oxides sites available in the soil (van der Zee and van Riemsdijk, 1988). Analytical details of the aforementioned extraction methods are given in Section 2.4.

2.2. Experimental design

We set up a pot experiment with the two soils and four P fertiliser treatments with five replicates each, following a random block design. In order to simulate row fertilisation in our pot experiment, DAP and struvite granules were not mixed homogeneously through the soil but placed 2 cm below the soil surface at the centre of the pot (Fig. S1). The zone with a diameter of 5.5 cm around this centre was named the “fertiliser zone”, i.e. the zone with DAP or struvite, whereas the bulk soil of these treatments did not receive any P fertiliser. The amount of P

applied with struvite and DAP was the same, i.e. 17 kg P ha⁻¹. This P dosage was calculated as the average P dose recommended for both soils as a row fertiliser for maize, based on the initial P-CaCl₂ and P-AL of both soils (Table 1) (Commissie Bemesting Grasland en Voedergewassen, 2023). DAP and struvite were obtained from a commercial supplier (Triferto Fertilizers, Doetinchem, the Netherlands) and a sewage treatment plant for industrial and domestic waste water (Land van Cuijk, Cuijk, the Netherlands), respectively. The required DAP and struvite dosages were calculated using the claimed P contents of these fertilisers. The actual chemical composition of both fertilisers was measured afterwards. To this end, subsamples of DAP and struvite were digested with a mixture of H₂SO₄, salicylic acid, H₂O₂, and selenium (Novozamsky et al., 1983). The N and P concentrations in the digests were measured with a segmented flow analyser (SFA). Furthermore, a subsample of the struvite was digested with Aqua Regia (Houba et al., 1997) to measure the contents of magnesium (Mg) and other elements using an inductively coupled plasma – optical emission spectrometer (ICP-OES). The results are reported in Table S1. In brief, the struvite and DAP contained 57 and 178 g N kg⁻¹ and 122 and 190 g P kg⁻¹, respectively. Furthermore, struvite contained 102 g Mg kg⁻¹. The measured N, P, and Mg contents in struvite and DAP deviated by a maximum of 5 % from the claimed contents. Both fertilisers were used in a granular form as provided by the suppliers. Details about granular size of both fertilisers are given in Table S1. The remaining two P fertiliser treatments of the pot experiment are controls: a negative control (control[-]) without P fertilisation and a positive control (control[+]) receiving P in solution (NaH₂PO₄·H₂O and Na₂HPO₄·H₂O mixed in a 1:1 M ratio) to homogeneously increase soil P availability in the fertiliser zone and the bulk soil alike. For the control[+] treatment, we raised the agronomic P status of both soils from the category “low” to the category “sufficient” (Minister van Landbouw, Natuur en Voedselkwaliteit, 2005). Since the loamy soil had a lower P-CaCl₂ and P-AL than the sandy soil (Table 1), the former received a higher P dosage (64 kg P ha⁻¹) than the latter (47 kg P ha⁻¹). Pots from all treatments received N, Mg, potassium (K), sulphur (S), copper (Cu), manganese (Mn), and boron (B), applied as solutions and mixed homogeneously through the soil, both in the bulk soil and the fertiliser zone. The amounts of N and Mg added with DAP or struvite were subtracted from the intended N and Mg applications (Table S2). A combination of solutions prepared from NH₄NO₃, Mg(NO₃)₂·6H₂O, KCl, CuSO₄·5H₂O, MnSO₄·H₂O, Na₂SO₄·10H₂O, and H₃BO₃ was used to reach a final fertilisation level of 1.20 g N pot⁻¹ for the control[-], control[+], and DAP treatments and 1.23 g N pot⁻¹ for the struvite treatment, 0.28 g Mg pot⁻¹, 1.33 g K pot⁻¹, 0.20 g S pot⁻¹, 0.06 g Cu pot⁻¹, 0.06 g Mn pot⁻¹, and 4 mg B pot⁻¹ for all treatments. The conversion of the recommended fertilisation doses expressed per hectare into doses at the pot scale was done based on the number of maize plants

Table 1
Properties of the two soils used in this study.

Property	Unit	Sandy soil	Loamy soil
Clay	%	2	17
Silt	%	8	46
Sand	%	90	37
SOM ¹	%	4.1	6.4
pH-CaCl ₂	–	5.5	6.5
N-NO ₃ (CaCl ₂)	mg N kg ⁻¹	9.8	5.0
N-NH ₄ (CaCl ₂)	mg N kg ⁻¹	2.8	0.2
P-CaCl ₂	mg P kg ⁻¹	0.37	0.16
P-AL	mg P kg ⁻¹	116	48
P-ox	mg P kg ⁻¹	301	278
[Fe + Al]-ox	mmol kg ⁻¹	71	102
α	–	0.14	0.09

¹Soil organic matter.

²P-CaCl₂ refers to P-PO₄ as measured by SFA in a 0.01 M CaCl₂ soil extract (Houba et al., 2000).

³P loading of poorly crystalline Fe- and Al-(hydr)oxides (van der Zee and van Riemsdijk, 1988).

in the field, using a typical maize plant density of 100,000 plants ha⁻¹ (van Schooten et al., 2019), while we had one maize plant in each pot at the start of our pot experiment.

Each pot (volume = 4 L and ϕ = 20 cm) was filled with 4.2 kg of sandy soil or 3.8 kg of loamy soil including the combination of solutions with the above-mentioned fertilisers and additional demi-water to reach 60 % of the maximum soil water holding capacity. Four maize seedlings (pregerminated in the dark at 25C for 72 h) of the variety Movanna (Zaadhandel Neutkens, Vessem, the Netherlands) were planted in a circle, 2.7 cm from the centre of each pot. Seven days after planting, the best-performing plant was selected and the remaining three plants were removed to ensure the presence of one viable maize plant in each pot (Figure S1). Each pot was watered every other day with demi-water. Each week, the five blocks were rotated clockwise to minimize possible effects of local differences in plant growth conditions in the greenhouse. Plant height was weekly measured. The greenhouse temperature was kept at 25C and relative air humidity was 65 %. Artificial light was supplied for one hour before and after sunset. On day 40 at which some of the maize plants had almost reached the end of the vegetative growth stage (V10), the experiment was terminated: maize shoots were harvested and roots and soil were sampled, as described hereafter.

2.3. Plant analyses

After harvest, maize shoots were oven-dried (70C), followed by measuring their biomass and grinding of the material. Total N and P in the ground biomass were measured by digesting a subsample according to Novozamsky et al. (1983), followed by measurement of the N and P concentrations with a SFA. The uptake of N and P by maize was calculated by multiplying the harvested dry biomass with the N and P contents. The PUE was calculated as follows: $PUE(\%) = \frac{P_1 - P_0}{P} \times 100$ with F being the amount of P fertiliser added per pot, P₀ the P uptake of the control[-] treatment, and P₁ the P uptake of the fertilised pot. The PUE was used as a measure of the agronomic effectiveness of a fertiliser.

For all treatments, roots were sampled at two locations in the pot: the fertiliser zone and the bulk soil (Figure S1). To do so, we extracted two soil cylinders (ϕ = 5.5 cm and height = 15 cm) at equidistance from the maize stubble. The soil sampled with the cylinders was sieved (2 mm) to separate the roots from the soil. Next, the soil taken with the cylinder from the bulk soil was mixed with the rest of the bulk soil to create one composite bulk soil sample, whereas the soil taken with the cylinder from the fertiliser zone was used as it was. The collected roots were further cleaned by washing over a 0.5 mm-sieve and collected carefully with tweezers. Roots were dried (70C) and their biomass was measured. We calculated the ratio of root biomass in the fertiliser zone and the bulk soil as a measure for the maize root distribution in the potted soil.

2.4. Soil analyses

The soil samples of the first three blocks out of the in total five blocks were oven-dried (40C) and 2 mm-sieved prior to chemical analysis. The soil samples were extracted with 0.01 M CaCl₂ at a SSR of 1:10 (w:v) with horizontal shaking for two hours (160 S min⁻¹) and centrifugation (1800 x g for 10 min) following Houba et al. (2000). One part of the supernatant was used to measure the pH and the other part was 0.45 μ m-filtered (Aqua 30/0.45 CA Whatman) and acidified by adding 25 μ L 5 M HCl for sample conservation before chemical analysis. We determined phosphate (P-PO₄) (hereafter referred as P-CaCl₂), dissolved organic carbon (DOC), N-NO₃, N-NH₄, and total dissolved N (Nts) with an SFA and Al, Fe, K, Mg, and total dissolved P (TDP) with an ICP-OES. The results of the pH and TDP, DOC, N-NH₄, N-NO₃, and Nts measurements are shown in Table S3. Determination of P-AL was done by extracting soil with a mixture of acetic acid and ammonium lactate (pH = 3.75) at a SSR of 1:20 (w:v) with horizontal shaking for four hours (160 S min⁻¹),

centrifugation (1800 x g for 10 min), and 0.45 µm-filtration (Egnér et al., 1960). In the resulting extract, we measured P-PO₄ using the SFA to obtain P-AL. To determine P-ox and [Fe + Al]-ox, soil was extracted with 0.2 M acid ammonium oxalate following Schwertmann (1964), using a SSR of 1:20 (w:v) with horizontal shaking for two hours (160 S min⁻¹) in the dark and centrifugation (3000 x g for 10 min). We then measured P, Al and Fe in the resulting extract with an ICP-OES. Knowing P-ox and [Fe + Al]-ox, the P loading α was calculated (van der Zee and van Riemsdijk, 1988). The results of the 0.2 M acid ammonium oxalate extraction are shown in Table S4. To assess the possibility of struvite dissolving during 0.01 M CaCl₂ extraction after the standard shaking time of 2 h as prescribed by Houba et al. (2000) had ended, we performed an additional batch experiment using a shaking time of one to up to 24 h on soil samples from the fertiliser zone of the pot experiment, as well as the original soils freshly mixed with struvite and DAP. This experiment is described in further detail in Section S3.1.

2.5. Statistics

Statistical analyses were performed with R (version 3.6.0, “Planting of a Tree”) and the *stats*, *agricolae* and *dplyr* packages. The normality and homoscedasticity of the data were checked with the Shapiro and Bartlett tests, respectively. We performed two-way analyses of variances (ANOVA) with interaction (soil, fertilisation treatment, and soil:fertilisation treatment interaction) after checking on whether the residuals followed normality. When the normality assumption was not satisfied, we log-transformed the data. This was the case for the results on plant P uptake, P-ox, and P loading α . In the case of heteroscedasticity, we used the varIdent variance structure (*nlme* package) by treatment to correct it (Zuur et al., 2009). The blocks did not have a significant effect on the results and so were not included in the analyses. When the ANOVA yielded a significant difference among treatments, we proceeded with Tukey’s honestly significant difference (HSD) test, which is indicated below the bars for the fertilisation effect and above the bars for the soil:fertilisation interaction effect. For the root distribution ratio, we excluded the control[-] from the statistical analysis, because the variation of the root distribution ratio for this treatment was higher than for the other treatments but the actual root biomasses in the control[-] were very low. On average per pot, sampled root biomass were 0.021, 0.123, 0.192, and 0.091 g for the control[-], control[+], DAP, and struvite treatments, respectively. Furthermore, we excluded the control[-] for P-CaCl₂ of both soils as it was the only way to meet the assumptions for the ANOVA, while P-CaCl₂ was low for the control[-] of both soils.

3. Results and discussion

3.1. The two contrasting soils both had a low P status

The sandy soil and the loamy soil differed in texture (Table 1). The sandy soil had little clay, while the loamy soil contained more clay. The soils had a different pH, with the sandy soil being acidic, whereas the loamy soil had a near-neutral pH. The loamy soil had a higher SOM content than the sandy soil. Furthermore, the loamy soil had a higher [Fe + Al]-ox content than the sandy soil, whereas the amount of reversibly adsorbed P-ox was slightly lower. Consequently, the P loading α for the loamy soil was lower than for the sandy soil. This is in line with the higher P-CaCl₂ of the sandy soil compared to the loamy soil. Yet, the P loading α is rather low for both soils when compared to a large set of representative Dutch agricultural topsoils (Koopmans et al., 2006). The soil P status of both soils was classified as “poor”, based on P-CaCl₂ and P-AL (Minister van Landbouw, Natuur en Voedselkwaliteit, 2005).

3.2. Struvite led to a lower biomass production and P uptake than DAP

According to our first hypothesis, aboveground biomass would be higher for DAP than for struvite and higher on the sandy soil compared

to the loamy soil. For the control[+], DAP, and struvite treatments, maize reached a significantly higher aboveground biomass production in the loamy soil compared to the sandy soil (Fig. 1A). Overall, the biomass was highest for the control[+] and DAP treatments, followed by the struvite treatment, and then the control[-] treatment irrespective of the soil. For the loamy soil, the differences in biomass between the four P treatments were in line with the general effect of P fertilisation. For the sandy soil, however, this was not the case. For the latter, the biomass of the struvite treatment did not differ significantly from the results of the control[-] and control[+] treatments. Overall, our first hypothesis was confirmed. Moreover, the biomass results correspond closely to those of the maize plant height (Figure S2).

The results of the nutrient uptake by maize (Fig. 2A and B) are reasonably in line with those of the aboveground biomass production. For the control[+] and DAP treatments, N and P uptake were significantly higher for the loamy soil than for the sandy soil, whereas no difference was found between both soils for the struvite treatment. Regardless of the soil, N and P uptake was lowest for the control[-] treatment. For the struvite treatment, N and P uptake was significantly higher than for the control[-] treatment, but significantly lower than for the control[+] and DAP treatments. For both soils, the soil:fertilisation interaction effect on N uptake aligns with the overall effect of P fertilisation, whereas this was not the case for P uptake. For the sandy soil, P uptake of the DAP treatment was significantly higher than for the three other P treatments. However, in the sandy soil, P uptake of the struvite treatment did not differ significantly from the results of the control[-] and control[+] treatments. The control[-] treatment had a significantly lower P uptake than the control[+] treatment. For the loamy soil, P uptake was significantly different for all four P treatments, with the control[+] treatment having the highest P uptake, followed by the DAP treatment, the struvite treatment, and then the control[-] treatment. Compared to the control[-] treatment, the higher N and P uptake of the DAP and struvite treatments of both soils (with the exception of P uptake from the struvite treatment of the sandy soil) is most likely facilitated by proliferation of maize roots in the fertiliser zone (Fig. 1B). This will be further discussed in Section 3.3.

The stoichiometric ratio of N and P in the aboveground biomass has widely been used to investigate the nature of nutrient limitation for the growth of natural vegetation as well as several crops including maize (Bélanger et al., 2012; Koerselman and Meuleman, 1996; Sadras, 2006; Ziadi et al., 2007). Overall, regardless of the soil, P fertilisation helped to alleviate P limitation for both soils, as the N:P ratio was highest for the control[-] treatment, followed by the struvite treatment, and then the control[+] and DAP treatments (Fig. 2C). These results were in line with those of the overall effect of P fertilisation on the aboveground biomass production of maize (Fig. 2A). For the control[-] treatment of both soils, the N:P ratio was far above 16, which is used as a threshold to indicate P limitation for natural vegetation (Koerselman and Meuleman, 1996). This is in accordance with the classification of the agronomic soil P status of both soils as “poor”, based on the initial P-CaCl₂ and P-AL (Table 1). However, the sandy soil of the control[-] treatment had a higher N:P ratio (i.e. 49.3) than the loamy soil (i.e. 30.0), although the sandy soil had both a higher initial P-CaCl₂ and P-AL (Table 1). Furthermore, P application in the control[+] treatment of the loamy soil lowered the N:P ratio to below 14, which would indicate N limitation (Koerselman and Meuleman, 1996). However, this was not the case for the sandy soil, as the N:P ratio of this treatment was significantly higher and well above 16. For the DAP and struvite treatment, no significant differences in N:P ratio were found between the sandy soil and the loamy soil. The N:P ratio was well above 16 for both soils when struvite was applied. In case of DAP addition, the N:P ratio was between 14 and 16. Hence, DAP was more effective in alleviating P limitation than struvite, although the amounts of P applied with both fertilisers were the same (Table S2). This may be attributed to the poor solubility of struvite, while DAP has a higher solubility and quickly dissolves in soil (Talboys et al., 2016).

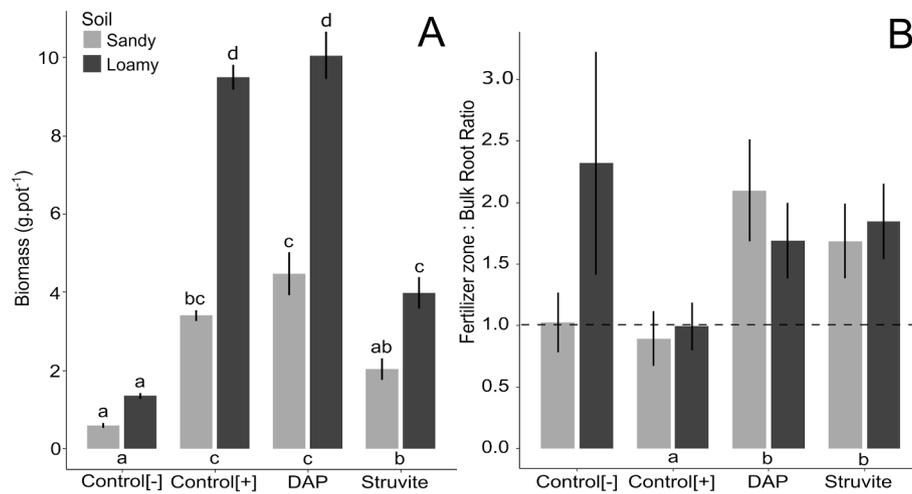


Fig. 1. Aboveground biomass production of maize (A) and root biomass ratio (B) for the four P treatments of the sandy soil and the loamy soil ($n = 5$). The dashed line in Fig. 2B at a root ratio = 1 indicates an equal root biomass in the fertiliser zone and in the bulk soil sample. Because of a very low root biomass of the control[-] treatment for both soils, the latter was excluded from statistical analyses of the root ratio. The error bars represent the standard error of the mean and letters indicate the results of the Tukey test (fertilisation treatment alone below the bars and soil:fertilisation treatment interaction above the bars). The soil:fertilisation treatment interaction was not significant for the root ratio.

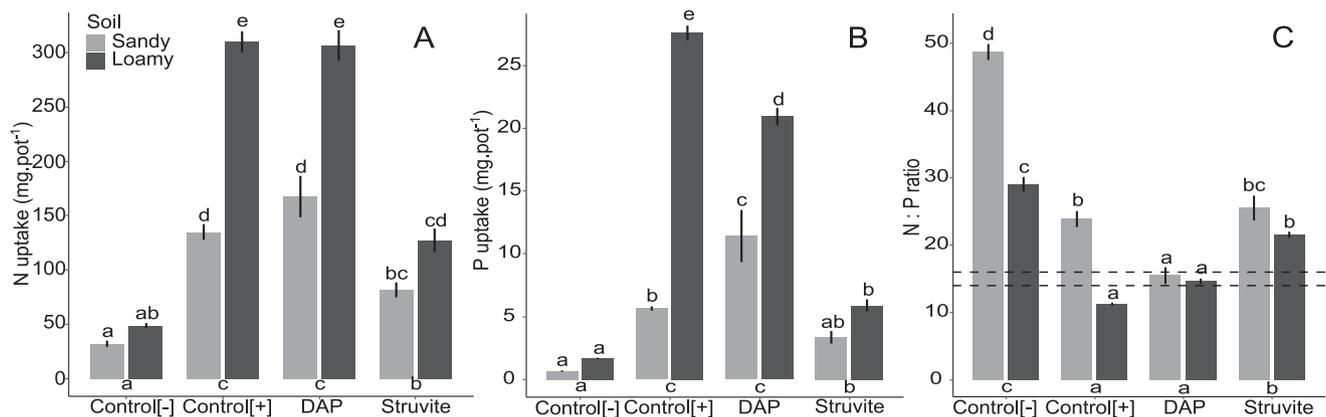


Fig. 2. Nitrogen (N) uptake of maize (A), P uptake (B), and the N:P ratio (C) for the four P treatments of the sandy soil and the loamy soil ($n = 5$). The dashed lines at a N:P ratio = 14 and 16 represent thresholds for N- and P-limited growth of natural vegetation, respectively (Koerselman and Meuleman, 1996). The error bars represent the standard error of the mean and letters indicate the results of the Tukey test (fertilisation treatment alone below the bars and soil:fertilisation treatment interaction above the bars).

3.3. Maize roots preferentially grew towards placed struvite and DAP

As stated in our second hypothesis, there will be preferential root growth of maize towards placed P fertiliser. For both soils, DAP was equally effective in increasing the aboveground biomass production of maize as the P fertiliser applied in the control[+] treatment (Fig. 1A). Likewise, struvite had the same effectiveness as the control[+] treatment for the sandy soil. This is likely the effect of placed DAP and struvite application in the fertiliser zone (Figure S1). In soils with a low agronomic P status such as used here (Table 1), P placement in a small volume of soil leads to a locally larger increase in plant-available P than the same amount of P mixed homogeneously through a larger soil volume, thereby enhancing both P uptake and crop yield (Grant et al., 2001; Lu and Miller, 1993). According to a meta-analysis of Nkebiwe et al. (2016), P fertiliser placement led to a 14.3% higher crop yield than broadcast P application for equal amounts of P applied. Remarkably, the amounts of P applied with DAP and struvite to the fertiliser zone of both soils (i.e. 17 kg P ha⁻¹) in our pot experiment were even ~ 3 and ~ 4 times lower than the amounts of P mixed through the entire volume of potted soil for the control[+] treatment of the sandy soil (i.e. 47 kg P ha⁻¹) and the loamy soil (i.e. 64 kg P ha⁻¹), respectively. Plants can

respond to P limitation as in the control[-] treatment of the sandy soil and the loamy soil (Fig. 2C) by various adaptations including root proliferation in regions of the soil with an elevated level of plant-available P (Grant et al., 2001). For this reason, we sampled roots in equal volumes of soil in the fertiliser zone and in the bulk soil of the four P treatments of both soils (Figure S1). The ratio of the root biomass in the fertiliser zone to the root biomass in the bulk soil was close to one for the control[-] treatment of the sandy soil (Fig. 1B). Hence, no root proliferation took place in the fertiliser zone, which is understandable as no P was applied at all in this treatment. For the control[-] treatment of the loamy soil, however, the ratio was unexpectedly above one. Since the root biomass in the control[-] treatment of both soils was very low (see Section 2.5), these ratios were sensitive to variation as any root fragment collected in either the fertiliser zone or the bulk soil may have had a large impact on the resulting ratios. For this reason, the ratios of the control[-] treatments of both soils were not included in further statistical analyses. The ratio for the control[+] treatment of both soils did not significantly differ from one, meaning there was no preferential root growth in one zone or the other. This was to be expected since the P fertiliser was mixed through the entire volume of potted soil. For the DAP and struvite treatments of both soils, the ratios were higher than the ratios of the

control[+] treatment and significantly higher than one. Hence, root proliferation took place in the fertiliser zone, probably enabling the maize plant to directly tap P from the locally elevated pool of plant-available P resulting from placed DAP and struvite application. Hence, these results are in line with our second hypothesis.

3.4. Struvite remnants in soil at the end of the pot experiment led to elevated P-CaCl₂ and P-AL

With our third hypothesis, P-CaCl₂ is hypothesized to be higher for a soil treated with DAP than for a soil treated with struvite as struvite will not dissolve fully as opposed to DAP. In Fig. 3, the results of P-CaCl₂ and P-AL measured in the bulk and fertiliser zone samples are presented. For both soils, the bulk soil samples of all P treatments, except those from the control[+] treatment, had statistically the same P-CaCl₂ and the same P-AL. The P-CaCl₂ and P-AL in the bulk soil samples of the control[+] treatment were higher as P was mixed through the entire volume of potted soil. For the fertiliser zone of the control[+] treatment, P application caused P-CaCl₂ to increase to $1.77 \pm 0.06 \text{ mg kg}^{-1}$ for the sandy soil and to $7.90 \pm 1.40 \text{ mg kg}^{-1}$ for the loamy soil. Although the loamy soil of the control[+] treatment had a higher P-ox than the sandy soil (Table S4), its P loading α (i.e. 0.14) was lower than the α of the sandy soil (i.e. 0.19) resulting from the higher [Fe + Al]-ox content of the loamy soil (Table 1). Hence, P application in the control[+] treatment was more effective in raising P-CaCl₂ for the loamy soil than for the sandy soil, despite the lower P loading α of the loamy soil. For the treatments with DAP and struvite, the same effect was observed.

The difference in the effectiveness of P application between both soils to raise P-CaCl₂ could be the result of a different P buffering capacity (Ehlert et al., 2003). This is illustrated by plotting the P loading α against P-CaCl₂ in Fig. 4, revealing the P adsorption isotherm of both soils (Koopmans et al., 2004b). Since the adsorption isotherm of the loamy soil has a lower slope at any given P-CaCl₂, this soil has a lower P buffering capacity than the sandy soil (Ehlert et al., 2003). This may be due to the higher pH and SOM content of the loamy soil (Table 1). Adsorption of P to Fe- and Al-(hydr)oxides decreases when the pH increases (Antelo et al., 2010; Weng et al., 2012). Furthermore, P may compete with organic matter for binding to poorly crystalline Fe- and Al-(hydr)oxides as both P and organic matter bind to metal-(hydr)oxides,

which lowers P adsorption (Antelo et al., 2007; Hiemstra et al., 2010; Regelink et al., 2015; Weng et al., 2012).

There is one aspect which complicates the interpretation of the adsorption isotherms in Fig. 4. Remnants of struvite granules were clearly visible when the fertiliser zone samples were taken from both soils at the end of the pot experiment. There were no discernible DAP granules. This explains why the levels of TDP, N-NH₄, and Mg in the 0.01 M CaCl₂ soil extracts of the fertiliser zone samples from the struvite treatment of both soils were significantly higher than those of the other P treatments (Table S3). In an additional batch experiment, we extracted one fertiliser zone soil sample from each P treatment with 0.01 M CaCl₂, using a shaking time varying from one to 24 h as opposed to the standard shaking time of 2 h (Houba et al., 2000), which is commonly interpreted as the time required to reach equilibrium for the desorption of multiple nutrients including N-NH₄ and P-PO₄ (Koopmans et al., 2004a, 2004b; van Erp et al., 1998). The results of this batch experiment are discussed in detail in Section S3.2 of the Supplementary Information. In brief, clear indications for the dissolution of residual struvite were found, as significant quantities of N-NH₄ and P-PO₄ were released from the struvite-amended sandy soil and loamy soil in 0.01 M CaCl₂ when the shaking time increased from one to 24 h (Figure S3). For the DAP-amended loamy soil, some residual DAP dissolution was observed, whereas this was hardly the case for the sandy soil. The results are consistent with the well-known difference in dissolution behaviour between both P fertilisers as struvite has a lower solubility than DAP, leading to slow struvite dissolution with time (Talboys et al., 2016). Following the approach of Yang et al. (2023), we used the increase in the amount of N-NH₄ extracted by 0.01 M CaCl₂ from a shaking time of 2 to 24 h as a proxy to quantify residual struvite and DAP dissolution. For the sandy soil, 14 % of the initially added amount of struvite dissolved during the batch experiment, whereas this was 12 % for the loamy soil. This is likely an underestimation for both soils, because residual struvite dissolution still seemed to continue at a shaking time of 24 h in the batch experiment. Since N-NH₄ mainly binds to clay mineral particles (Wang et al., 2021; Wang and Alva, 2000), struvite dissolution is further underestimated for the loamy soil. Because of the lower P buffering capacity of the loamy soil, the increase in P-PO₄ in 0.01 M CaCl₂ from a shaking time of 1 to 24 h was especially prominent for this soil (i.e. 50.2 mg kg^{-1}), as indicated by the arrow in Fig. 4. The same applies to the increase in P-PO₄

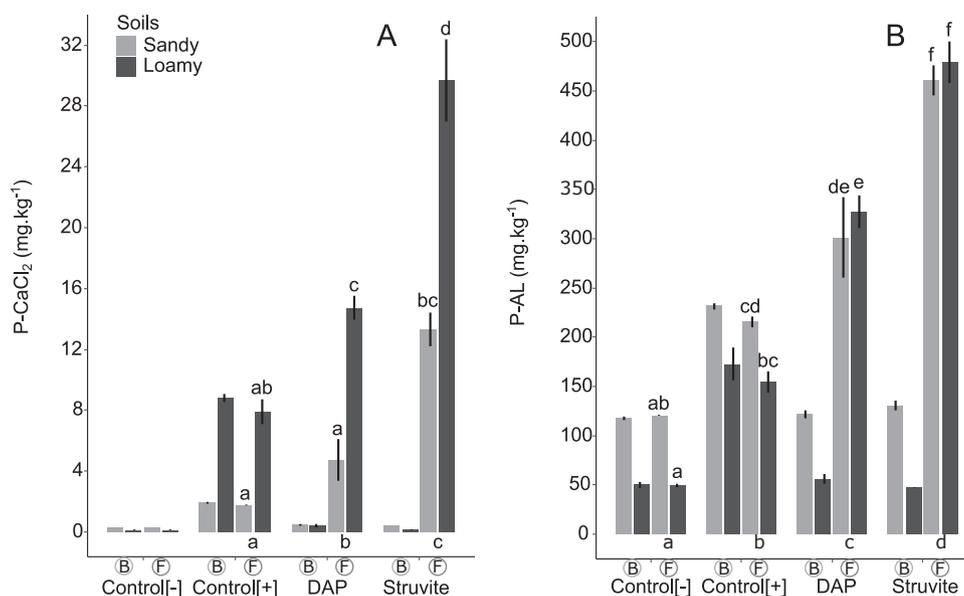


Fig. 3. The amounts of P-CaCl₂ (A) and P-AL (B) extracted from the soil samples taken from the bulk soil (circled B) and the samples from fertiliser zone (circled F) of the four P treatments of the sandy soil and the loamy soil (n = 3). The error bars represent the standard error of the mean and letters indicate the results of the Tukey test (fertilisation treatment alone below the bars and soil:fertilisation treatment interaction above the bars). Bulk soil samples violated normality assumption so ANOVA was not performed for this part of the data. Furthermore, the results of the bulk soil and the fertiliser zone soil were not statistically compared.

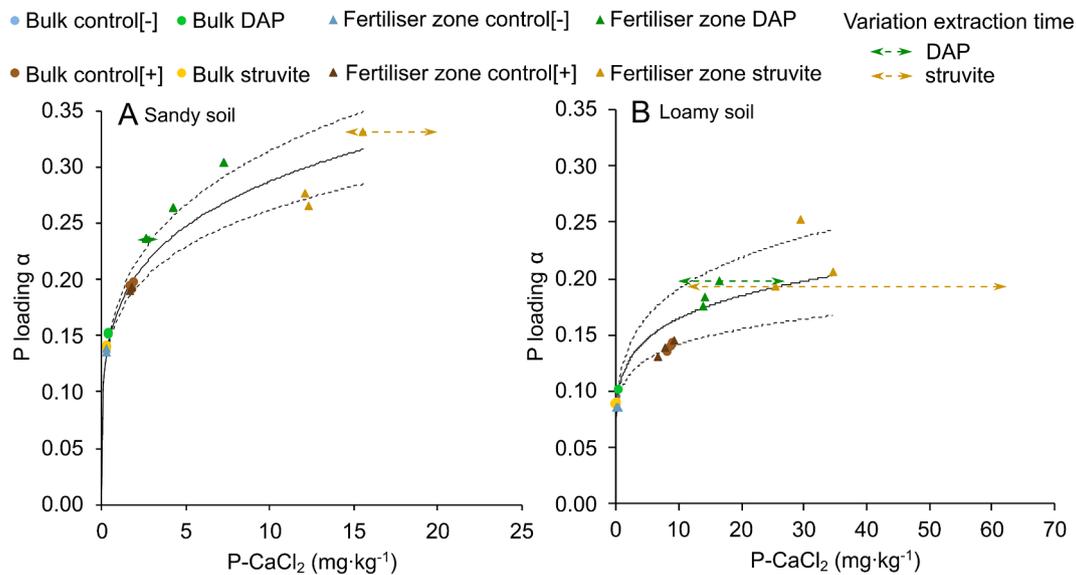


Fig. 4. The P loading α calculated as the molar ratio between P-ox and $[Al + Fe]_{ox}$ plotted against P-CaCl₂ extracted from the bulk and fertiliser zone samples from the four P treatments of the sandy soil (A) and the loamy soil (B) ($n = 3$). The P-CaCl₂ is based on the prescribed shaking time of 2 h (Houba et al., 2000). To guide the eye, a Freundlich equation has been fitted to the results of both soils, with $K_F = 0.17$ and $n = 0.22$ for the sandy soil and $K_F = 0.11$ and $n = 0.17$ for the loamy soil. The dotted lines on both sides of the Freundlich equation represent the 95 %-confidence interval. The arrows with dotted lines for one of the three fertiliser zone soil samples from the DAP and struvite treatments of both soils represent the results of the additional batch experiment in which the fertiliser zone samples were extracted with 0.01 M CaCl₂ using a shaking time varying from 1 to 24 h. Since the effect of shaking time on the extraction of P-PO₄ from the DAP-amended sandy soil by 0.01 M CaCl₂ in the batch experiment was limited, the arrow with the dotted line cannot be seen in the figure.

extracted with 0.01 M CaCl₂ from the DAP-amended loamy soil (i.e. 16.1 mg kg⁻¹). For the struvite-amended sandy soil, the increase in P-PO₄ was much less (i.e. 5.3 mg kg⁻¹), as this soil has a higher P buffering capacity.

The adsorption isotherms in Fig. 4 were based on P-CaCl₂, determined according to the standard protocol of Houba et al. (2000) including the prescribed shaking time of 2 h. However, dissolution of remnants of struvite in both soils and DAP in the loamy soil may have led to an accumulation of P-PO₄ in 0.01 M CaCl₂ already within 2 h of shaking, as revealed by the results of our additional batch experiment. As such, P-CaCl₂ does not reflect the actual equilibrium for P-PO₄ desorption from these soils. To this, the possible uncertainty in P-CaCl₂ must be added, because any variation in the standard shaking time of 2 h or the time taken to centrifuge and filter the 0.01 M CaCl₂ soil suspensions after shaking will have had an impact on the extracted amount of P-PO₄, as is clearly demonstrated by the arrows for the struvite-amended soils and the DAP-amended loamy soil in Fig. 4.

The presence of remnants of struvite in both soils and DAP in the loamy soil may have played a role as well when determining P-AL in the fertiliser zone samples (Fig. 3). Application of P in the control[+] treatment led to a higher P-AL for the sandy soil and the loamy soil, as P-AL increases with P loading α and P-ox (Table S4) (Schoumans and Groenendijk, 2000; van Doorn et al., 2023). Likewise, P-AL increased even further in the fertiliser zone samples of the DAP and struvite treatments of both soils, which can, in part, again be explained by the increase in P loading α and P-ox (Table S4). Since the pH of the extraction solution of P-AL is low (i.e. pH = 3.75) (Egnér et al., 1960), residual struvite and DAP will probably have dissolved in the mixture of acetic acid and ammonium lactate, leading to an overestimation of P-AL. The same is likely true for the 0.2 M acid ammonium oxalate extraction method, due to the low pH of the extraction solution (i.e. pH = 3.0) (Schwertmann, 1964). This is supported by the recovery of P by P-ox from P added with struvite (i.e. 104 %) and DAP (i.e. 82 %) to the fertiliser zone of the sandy soil and the loamy soil (see Section S2; Table S5). The lower P recovery from DAP can, in part, be explained by P uptake by maize (Table S5), which was significantly higher for the DAP-amended soils than for the struvite-amended soils (Fig. 2B). Hence, P-ox

and the resulting P loading α may have been overestimated for the struvite-amended soils and the DAP-amended loamy soil, similar to P-AL.

These results show the shortcoming of traditional soil P extraction methods like P-CaCl₂ and P-AL for soils treated with poorly soluble mineral P fertilisers, as they are unable to separate P desorbed from soil itself from P dissolved during the extraction of soil from remnants of mineral P fertiliser. This leads us to reject our third hypothesis.

3.5. P-CaCl₂ and P-AL have less diagnostic value for struvite-amended soils in our pot experiment

For the fertiliser zone samples, both P-CaCl₂ and P-AL are highest for the struvite treatment of the sandy soil and the loamy soil (Fig. 3). However, this is not at all reflected in the aboveground biomass production and P uptake by maize, which are highest for the control[+] and DAP treatments of both soils (Fig. 1A and Fig. 2B). Indeed, when establishing the relationship between biomass and P uptake on the one hand and P-CaCl₂ measured in the fertiliser zone soil samples from the four P treatments on the other, the data points of the struvite treatment of both soils and the data points of the DAP treatment of the loamy soil deviate strongly from the empirical relationship fitted to the results of the control[-] and control[+] treatments of both soils (Fig. 5). Similar results were found for P-AL (Figure S4). Hence, these soil P tests have less diagnostic value when classifying the agronomic soil P status for soil samples taken from zones of the soil where struvite has been placed as in our pot experiment. This can be explained by the dissolution of remnants of struvite in both soils and DAP in the loamy soil during the extraction of soil when determining P-CaCl₂ and P-AL, as discussed in Section 3.4. Dissolution of struvite has been shown as well for other established agronomic soil P tests like Mehlich-3, Bray-1, and Olsen P (Gu et al., 2021). Although the maize plants grown in the DAP and struvite treatments proliferate their roots in the direction of the fertiliser zone for both soils (Fig. 1B), they do form some roots in the bulk soil of these P treatments. As such, the biomass and P uptake by maize does not depend only upon the level of plant-available P in the fertiliser zone soil but depend on the plant-available P level in the bulk soil as well. However,

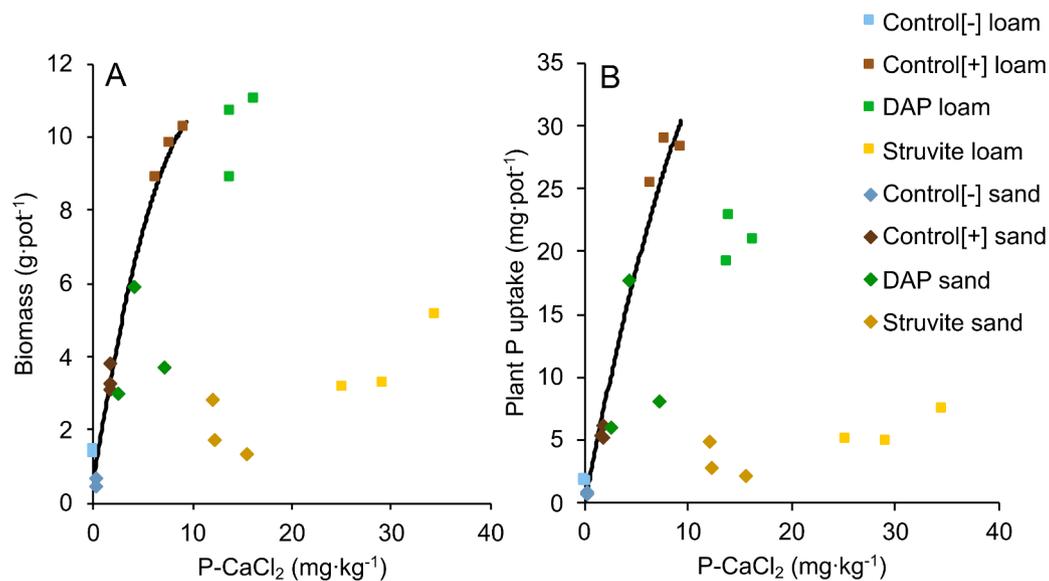


Fig. 5. The aboveground biomass of maize (A) and P uptake (B) plotted against the P-CaCl₂ measured in the fertiliser zone samples, taken from the four P treatments of the sandy soil and the loamy soil ($n = 3$). The P-CaCl₂ is based on the prescribed shaking time of 2 h (Houba et al., 2000). To guide the eye, a quadratic relationship was fitted to the results of the control[-] and control[+] treatments of the sandy soil and the loamy soil.

the levels of P-CaCl₂ and P-AL in the bulk soil of the DAP and struvite treatments were much lower than those in the fertiliser zone soil (Fig. 3). Consequently, the use of P-CaCl₂ and P-AL measured in soil samples taken from zones of the soil where P fertiliser has been placed would lead to an overestimation of the level of plant-available P for crops.

3.6. The phosphorus use efficiency of struvite was lower than that of DAP

According to our fourth hypothesis, the PUE will be higher for DAP than for struvite and the PUE of both DAP and struvite will be higher for the sandy soil than for the loamy soil. The PUE values are presented in Table 2. For the loamy soil, the PUE of the DAP treatment is significantly higher than for the control[+] treatment. Although the PUE in the sandy soil did not show significant differences between P treatments, the same pattern as in the loamy soil was observed. This demonstrates the beneficial effect for crops when placing P fertiliser in a limited volume of soil rather than mixing it through the entire volume of potted soil, even though less P was applied with DAP and struvite than with the control [+] treatments of both soils (Table S2) (see Section 3.3). For the loamy soil, the PUE of the DAP treatment is significantly higher than the PUE of the struvite treatment. The same was observed for the sandy soil. This is due to the higher solubility of DAP compared to struvite (Section 3.4). The latter is supported by the results of our additional batch experiment (see Section S3.2; Figure S5). Furthermore, the PUE of the DAP treatment is higher for the loamy soil than for the sandy soil, because P uptake by maize was significantly higher for the loamy soil (Fig. 2B). For the struvite treatment, the PUE of the sandy soil and the loamy soil are

Table 2

Phosphorus use efficiency (%) for the three P treatments of the sandy soil and the loamy soil. The standard error of the mean is indicated in brackets ($n = 3$). Results of the Tukey tests are indicated in letters for the loamy soil. The one-way ANOVA was not significant for the sandy soil. For the control[+] treatment, P was mixed through the entire volume of potted soil, while DAP and struvite were placed in the fertiliser zone (Fig. S2). The P doses for the control[+] treatment were higher than the P dose used for the DAP and struvite treatments (Table S2).

P treatment	Sandy soil	Loamy soil
Control[+]	1.0 (0.1)	4.0 (0.2) a
DAP	5.6 (2.1)	11.0 (0.6) b
Struvite	1.4 (0.5)	2.3 (0.5) a

reasonably similar, because P uptake was the same for both soils (Fig. 2B). Since maize growth was more P-limited on the sandy soil (Fig. 2C) and because the sandy soil had a lower pH (Table 1), struvite was hypothesized to have a higher agronomic effectiveness for the sandy soil than for the loamy soil. This would have been in line with the higher solubility of struvite at lower pH (Bhuiyan et al., 2007; Talboys et al., 2016), which is an important soil property in determining the agronomic effectiveness of this P fertiliser (Degryse et al., 2017; Hilt et al., 2016). Although a larger part of the added struvite may have dissolved in the sandy soil compared to the loamy soil, as suggested by the results of the additional batch experiment (see Section S3.3), the P released from struvite dissolution was probably bound to a greater extent in the sandy soil leading to a lower soil P availability. However, the latter remains speculative because remnants of struvite granules complicated the measurement of P-CaCl₂ and P-AL in both the sandy soil and the loamy soil (Fig. 4). We can only partially accept our fourth hypothesis: DAP did lead to a higher PUE than struvite but the PUE of both fertilisers were higher for the loamy soil than for the sandy soil.

A limitation of the setup of our pot experiment when it comes to the incomplete dissolution of struvite is the relatively short duration of the maize growing period of 40 days, which is shorter than the growing period of maize under field conditions. Yet, at the end of the 40 day-period used in our pot experiment, the maize plants of the control[+] and DAP-treatment almost reached the end of the vegetative growing period (10 leaves). The temperature effect of the greenhouse will have impacted the growth of maize just as temperature may have affected struvite dissolution (Bhuiyan et al., 2007). In any case, maize plants may have benefited from optimal growth conditions in the greenhouse such as additional lighting to supplement natural light and soil watering conditions. Therefore, our results need to be confirmed under field conditions and for a wider variety of soils.

4. Conclusions

Although placed DAP and struvite fertilisation in general were effective in increasing the aboveground biomass production and P uptake by silage maize as compared to the treatment in which P was withheld in our 40-day pot experiment, struvite had a lower agronomic effectiveness than DAP. This was due to the higher solubility of DAP. Nearly all DAP had dissolved during the pot experiment in the acidic

sandy soil, while some residual DAP was likely present in the loamy soil with a near-neutral pH. For struvite, however, remnants of the fertiliser were visually detected at the end of the pot experiment. Nevertheless, a larger part of the added struvite had probably dissolved in the sandy soil than in the loamy soil. Yet the biomass was higher for the loamy soil, although there was no difference in P uptake between both soils. This was explained by the lower P buffering capacity of the loamy soil. Consequently, P released from the dissolution of struvite was more effective in raising readily plant-available P in the loamy soil than in the sandy soil. Furthermore, for these two soils P-CaCl₂ and P-AL over-estimated biomass and P uptake by maize when the soil contained remnants of struvite, due to struvite dissolution during the extraction procedures. These results necessitate a critical rethinking of how to interpret P-CaCl₂ and P-AL as a basis for P fertiliser recommendations for soils receiving struvite as a P fertiliser.

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CRedit authorship contribution statement

Laura M.E. Ferron: Methodology, Formal analysis, Data curation, Visualization, Writing – original draft, Writing – review & editing. **Gerwin F. Koopmans:** Resources, Supervision, Methodology, Investigation, Conceptualization, Formal analysis, Writing – original draft, Writing – review & editing. **Henry Rommelse:** Conceptualization, Methodology, Investigation, Data curation. **Jan Willem van Groenigen:** Funding acquisition, Supervision, Writing – review & editing. **Inge C. Regelink:** Resources, Supervision, Conceptualization, Methodology, Investigation, Formal analysis, Writing – review & editing.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Jan Willem van Groenigen reports financial support was provided by Horizon 2020 European Innovation Council Fast Track to Innovation. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.geoderma.2024.116939>.

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