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Plant phosphorus availability of pyrolysed pig slurry related to ammonium and nitrate nutrition

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Abstract: Excessive slurry applications in regions with intensive livestock production are overloading soils with phosphates, which can lead to water pollution. Pyrolysis of pig slurry solids creates a fertiliser that is potentially efficient to store and transport, hence creating the opportunity to export it from affected regions. This study aims to quantify the plant availability of phosphorus (P) from the pyrolysed pig slurry in different soils and in combination with the nitrogen application in the form of nitrate (NO_3^-) and ammonium (NH_4^+), respectively. A pot experiment with maize seedlings (*Zea mays* L., cv. Amadeo) was conducted under glasshouse conditions to assess changes in plant-available phosphate from pyrolysed and freeze-dried solids in three contrasting topsoils with pH values of 5.2, 6.7 and 7.4 (in 0.01 mol/L $CaCl_2$). In two separate positive control treatments, P was applied in the form of rock phosphate and $Ca(H_2PO_4)_2$, respectively, instead of processed pig slurry. To eliminate nitrification in the treatment fertilised with NH_4^+, the synthetic nitrification inhibitor 3,4-dimethylpyrazol phosphate (DMPP) was utilised. The plant P availability of the pyrolysed and freeze-dried product exceeded the plant P availability of rock phosphate on all tested soils, but pyrolysis lowered it compared to the freeze-dried treatment. Furthermore, the NH_4^+ nutrition improved plant P availability compared to the NO_3^- nutrition. This indicates that pyrolysis potentially leads to the formation of tri- or octa-calcium phosphates rather than crystalline apatite and that the acidification of the rhizosphere by NH_4^+ nutrition led to the solubilisation of P. Pyrolysis is a promising treatment for making a plant available P fertiliser, however freeze-drying led to an even better result. For the future, both procedures need to be compared economically to achieve optimal utilisation of the scarce resource P.

Keywords: phosphorus recycling; nitrification inhibition; plant phosphorus availability

Phosphorus (P) is an important plant nutrient; its supply in agricultural soils varies widely worldwide. Many soils in South America and Africa have a rather low P supply, whereas agricultural topsoils in Western Europe (e.g., Germany, Spain, Great Britain, Italy) are well supplied (Barberis et al. 1996). This high P supply probably resulted from the heavy use of basic slag phosphate (a by-product of the steel industry) and organic fertilisers derived from livestock farming (e.g., pig slurry). Especially in districts with high livestock density, P supply exceeds crop needs, leading to P leaching into deeper soil layers (Leinweber 1996). Ultimately, the P reaches groundwater, which in turn contaminates it.

In Northwest Germany (e.g., the rural districts of Emsland, Vechta and Oldenburg), this was addressed by prohibiting additional P fertilisation after enrichment with the element was observed (Werner et al. 1988). This raises the question of how to deal with organic fertilisers from livestock farming, since their production cannot be regulated by stricter P fertilisation regulations.

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While the most sensible strategy would be a reduction in livestock numbers, thereby reducing the volume of imported nutrients from feed and produced organic fertiliser, it seems economically inapplicable to farmers. Another option would be the export of organic fertilisers to regions with lower livestock density and higher crop P demand. This strategy is challenged by the low nutrient concentrations in organic fertilisers compared to mineral fertilisers (Mengel and Krikby 2001), which increase the cost of transportation and storage per unit of nutrient. There are various methods to increase the nutrient concentration of organic fertilisers by removing undesired components: slurry can be separated into a liquid and a solid phase by centrifugation. Following this separation, struvite (NH_4MgPO_4) precipitation could be used to extract nitrogen (N), phosphorus (P), and magnesium (Mg) from the liquid phase. The solid phase could undergo various procedures to remove residual moisture and further increase the nutrient concentration. The simplest approach is drying the solid phase. To further enrich the product with P, combustion could be an option, but carbon (C) and N would be eliminated. This concept was already tested with bio-based fertilisers (5.23% P) produced from turkey litter ash by Frick et al. (2025). Another approach for P enrichment is pyrolysis, which conserves C in the product. Regardless of the approach, plant available P in the end product should be the most important parameter for choosing a procedure, since the resulting fertiliser will only be accepted in practical agricultural production if it is comparable to other (conventional) P fertilisers.

Especially high-energy procedures could alter the chemical composition of the product; therefore, the objective of this study is to analyse the plant P availability of the pyrolysed solid phase of pig slurry. To achieve a comprehensive overview, this needs to be done on various soils. N nutrition needs to be taken into account as well, since Vogel et al. (2020) observed an interaction between N nutrition, rhizosphere pH and plant P availability. Only if the product achieves a high plant P availability, pyrolysis can be considered a suitable procedure for producing competitive P fertilisers from pig slurry.

MATERIAL AND METHODS

Fertiliser and soil preparation. To produce a suitable fertiliser for testing, a pig slurry was separated by centrifugation into a liquid and a solid phase. The liquid phase was not further considered since it only contained approximately 20% of the total P.

The solid phase was further processed by letting it air-dry until a solid sample could be collected. The sample was frozen at –20 °C until further processing. One aliquot was freeze-dried to remove all remaining moisture. This product was used as a positive control treatment to check whether pyrolysis changes the P availability.

Another aliquot was used for pyrolysis: the pyrolysis process was executed in a laboratory reactor at 500 °C under a N_2 atmosphere for 10 min. The pyrolysis product, as well as the freeze-dried product, was ground to pass a 1 mm sieve to ensure homogeneity in particle size. Final products achieved P concentrations of 2.46% in the freeze-dried solid phase of a pig slurry (FSPS) and 6.56% in the pyrolysed solid phase of a pig slurry (PPS). P solubility of the pyrolysis product was checked in water (1.4%), 2% citric acid (6.5%), neutral ammonium citrate (5.7%) and 2% formic acid (49.5%) which indicate a mediocre plant P availability.

Three cambisols (0–0.25 m depth) differing in soil pH and CAL-extractable P were collected from arable land located in the Rhine valley close to Bingen, Germany. They were classified into an acidic soil, a neutral soil and an alkaline soil. Their pH value, CAL-extractable P and the crop before sampling are shown in Table 1. All soils were air-dried and then ground with an earth shredder until they passed a 4 mm sieve.

Experimental design and analysis procedures. The experiment was conducted in Mitscherlich I pots in a greenhouse at the experimental station of the Institute of Plant Nutrition in Giessen (50°35'53.30"N, 8°40'1.56"E) during the vegetation period of 2019. To compare the generated products with commercially available products, a rock phosphate (14% total P) and a $Ca(H_2PO_4)_2$ (19.5% total P, applied as triple superphosphate) treatment were added, as well as

Table 1. pH value, CAL-extractable phosphorus (P) and previous crop of the soils used in the pot experiment

Soil name	pH in 0.01 mol/L $CaCl_2$	CAL-extractable P (mg P/kg)	Previous crop
Acidic soil	5.2	69.8	maize
Neutral soil	6.7	62.5	winter wheat
Alkaline soil	7.4	7.3	grassland

a P_0 treatment, which did not receive any P fertilisation. N was either supplied with $Ca(NO_3)_2$ (YaraLiva™ 15.5% N) for the NO_3^- treatment or $(NH_4)_2SO_4$ + 3,4-dimethylpyrazol phosphate (DMPP; 21% N) for the NH_4^+ treatment. The P content of the applied DMPP was disregarded since its concentration in the product is negligible for the plant's nutrition.

Each treatment contained four replicates, resulting in a total of 120 pots for the experiment (5 P treatments × 2 N treatments × 3 soils × 4 replicates = 120).

Since two of the soils were relatively high in CAL-extractable P, all soils were mixed with quartz sand (DORSILIT® Nr. 5F particle size: 1–1.8 mm) in a ratio of 3 kg soil and 3 kg quartz sand. Each soil-sand mixture was fertilised with 75 mg P/kg substrate according to the P treatments and with 300 mg N/kg substrate according to the N treatments. The relatively high N fertilisation was applied in order to consider the N concentration of FSPS (2.96% N) and therefore eliminate N-driven yield effects. Furthermore, the substrates were treated with 250 mg K (K_2SO_4), 30 mg Mg ($MgSO_4$), 4.6 mg B (H_3BO_3), 0.11 mg Mo (NH_4-molybdate), 20 mg Mn ($MnSO_4$), 10 mg Zn ($ZnSO_4$) and 5 mg Cu ($CuSO_4$) per kg. N, P, K and Mg were applied in solid form, and the micronutrients were applied in a watery solution. Fertilisers were carefully mixed with the substrate. After that, the fertilised substrate was filled into pots. The substrate was then moistened with deionised water to 60% of its maximum water holding capacity. After four days of incubation, soil samples were taken from each pot. Each sample from a pot consisted of three homogeneously mixed subsamples. These soil samples were dried at 40 °C in an oven, ground and sieved through a 1 mm sieve.

Ten seeds per pot of maize (*Zea mays* L., cv. Amadeo) were then sown, and the plants were thinned to five plants per pot ten days after germination. Forty days after sowing, the maize plants were harvested. Shoot samples were dried to constant weight at 105 °C, and the shoot dry weight was determined gravimetrically. The samples were ground finally before determining the P concentration. Phosphorus was determined colourimetrically after dry ashing at 550 °C as an NH_4-molybdo-vanado-P-complex at 450 nm (Gericke and Kurmies 1952). The N concentration in the shoot was determined using an elemental analyser (Unicube® trace, Elementar Analysensysteme GmbH, Langenselbold, Germany). In the soil samples, the concentration of exchangeable NO_3^- and NH_4^+ was measured in a 0.01 mol/L $CaCl_2$ extract (soil-to-solution 1:10 according to Houba et al. 1986). The CAL method was used for the extraction of available soil-P (Schüller 1969).

Statistics. The data were analysed using linear models. For each parameter, a linear model was fitted using the lm interface of R (version 4.1.1, R Core Team 2026). Outliers were identified using Cook's distance. If the Cook's distance exceeded 1, the data point was considered an outlier and excluded from further analysis. From the linear model, an ANOVA was conducted, and treatment means were estimated using the "emmeans" package (version 1.10.4, Lenth and Piaskowski 2026). Multiple comparisons were conducted, and *P*-values were adjusted using the false discovery rate (FDR) method (Benjamini and Hochberg 1995) to account for the multiple-testing problem. The results were then compiled into a compact-letter-display (Piepho 2018) using the "multcomp" package (version 1.4-17, Hothorn et al. 2008). Plant parameters for three pots were omitted due to a reduced number of vital plants.

RESULTS AND DISCUSSION

Soil analyses. Since the soil samples were collected 4 days after the incubation period began, no significant changes in soil pH were expected, nor could they be quantified. Therefore, the pH analysis is not shown.

Regarding the N concentration of the soil, we could observe that most of the applied NO_3^- was detected by the analysis (Figure 1). For NH_4^+ the recovery was not as good as for NO_3^-. Especially for the alkaline soil we detected strong NH_4^+ losses. These could be explained by ammonia volatilisation, since this process is increasing with increasing soil pH. The acidic and neutral soils show reduced NH_4^+ losses in comparison to the alkaline soil, which underlines this hypothesis. Another explanation for a lower detection of NH_4^+ is NH_4^+ fixation (Nieder et al. 2011). The nonexchangeable NH_4^+ can not be detected with the applied method, therefore explaining a part of the discrepancy between the applied and the detected NH_4^+. The absence of NO_3^- in the $(NH_4)_2SO_4$ + DMPP treatment (Figure 1) confirmed the efficacy of the nitrification inhibitor, hence confirming the inhibition of nitrification.

The CAL-extractable P concentration was increased by the FSPS, PPS, and $Ca(H_2PO_4)_2$ treatments compared to the P_0 treatment on all tested soils (Figure 2). The highest values were recorded for $Ca(H_2PO_4)_2$,

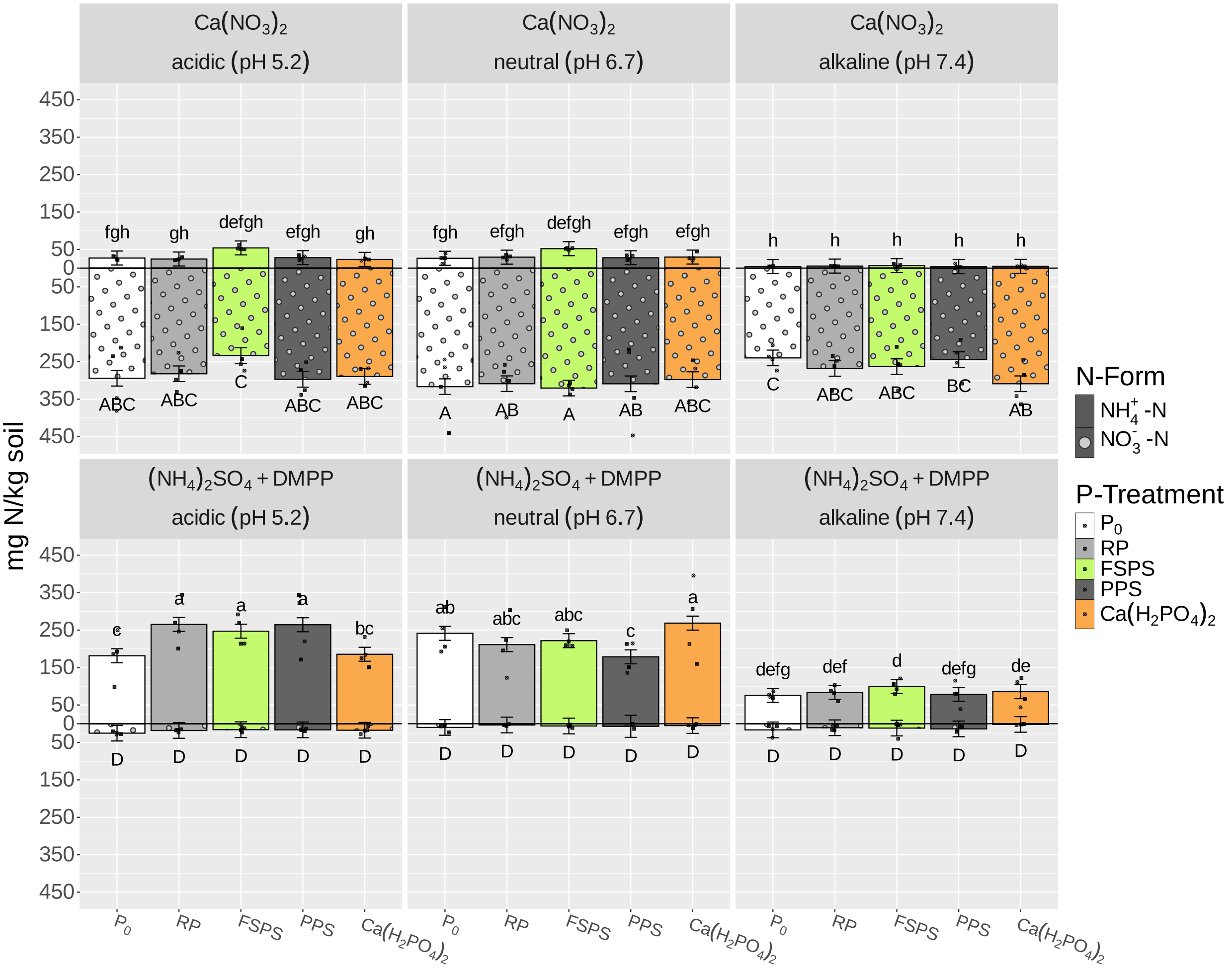

Figure 1. Effect of nitrogen (N) fertilisation (300 mg N/kg soil) in the form of $Ca(NO_3)_2$ or $(NH_4)_2SO_4$ + DMPP on the concentration of NO_3^- (solid columns) and exchangeable NH_4^+ (dotted columns) in soils before sowing of maize, where P was applied (75 mg P/kg soil) in different forms (P_0 – no P fertilisation; RP – rock phosphate; FSPS – freeze-dried solid phase of a pig slurry; PPS – pyrolysed solid phase of a pig slurry; $Ca(H_2PO_4)_2$ – triple superphosphate). Uppercase letters indicate significant differences for NO_3^-; lowercase letters indicate significant differences for NH_4^+. Columns which share at least one letter of the same case cannot be shown to differ significantly ($P \geq 0.05$). *P*-values were adjusted using the false discovery rate (FDR) method. Error bars indicate the modelled standard error (SEM). Raw data points are indicated by black dots

which falls in line with its solubility characteristics. FSPS performed slightly worse in some cases but there is no systemic evidence that soil or N treatment caused this on their own. Concentrations for PPS were lower than FSPS and $Ca(H_2PO_4)_2$, indicating a change in chemical composition of the pig slurry by pyrolysis. Weber et al. (2014) observed the same reduction in CAL-extractable P concentration for pyrolysed meat bone meal. But even though the performance was reduced compared to FSPS, PPS outperformed rock phosphate (in five out of six cases significantly). This finding indicates that very little apatitic -P was formed through the pyrolysis, if any. We assume that tri- or octo-calcium phosphate compounds could have been created. Hernandez-Mora et al. (2024) reported the detection of such compounds in chars processed from manure.

Plant analyses. In Figure 3, we compiled the aboveground biomass yield of the maize plants. P fertilisation led to the expected P treatment-dependent increase in biomass. The data reflect the findings of the earlier-discussed CAL-extractable P results (Figure 2). Within the $Ca(NO_3)_2$ treatment, the yield is strongly driven by the solubility of the used P fertiliser, regardless of the soil. The rock phosphate treatment was outperformed by all other P treatments

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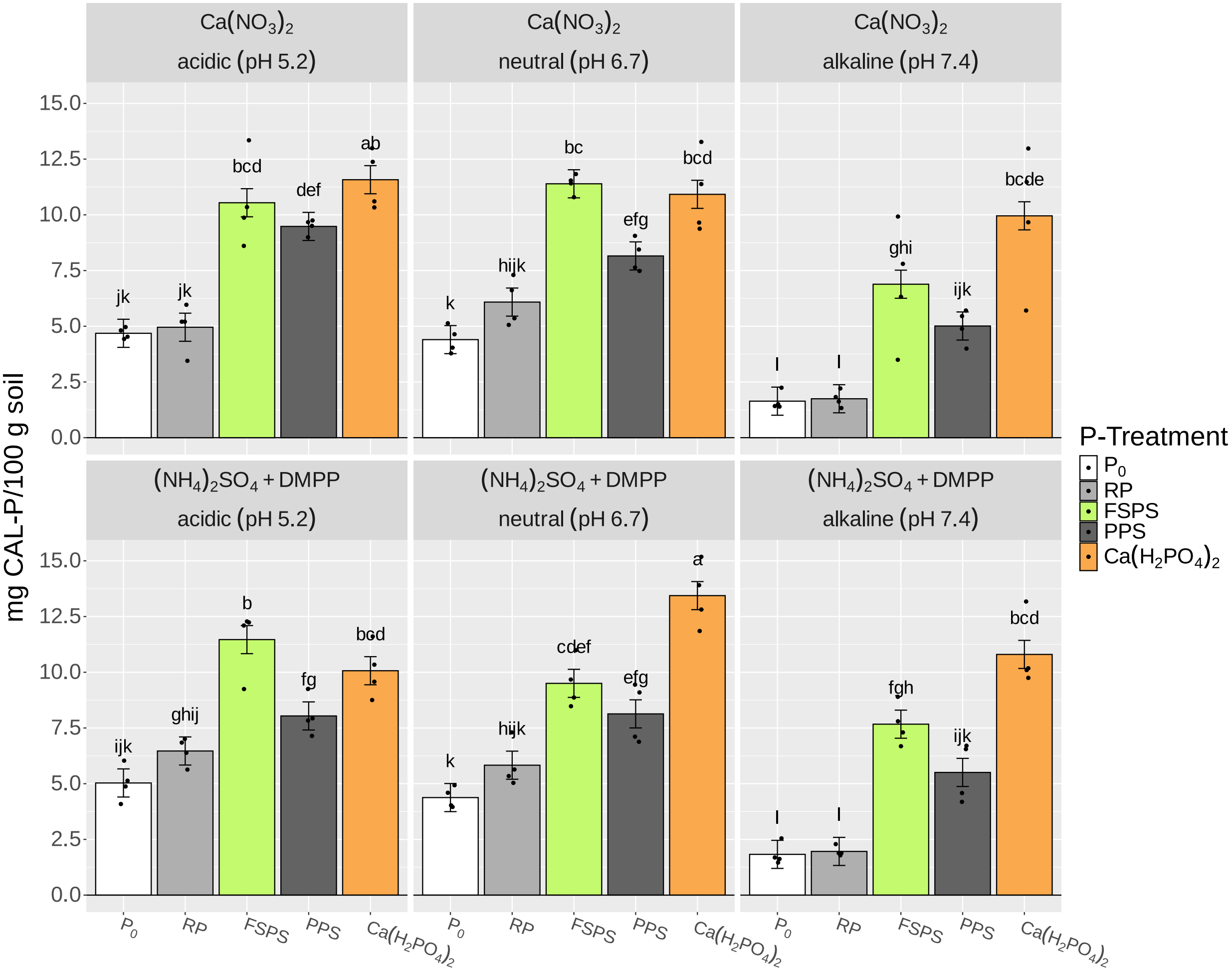

Figure 2. Effect of nitrogen (N) fertilisation (300 mg N/kg soil) in the form of $Ca(NO_3)_2$ or $(NH_4)_2SO_4$ + DMPP on the CAL-extractable P in soils before sowing of maize, where P (75 mg P/kg soil) was applied in different forms (P_0 – no P fertilisation; RP – rock phosphate; FSPS – freeze-dried solid phase of a pig slurry; PPS – pyrolysed solid phase of a pig slurry; $Ca(H_2PO_4)_2$ – triple superphosphate). Lowercase letters indicate significant differences. Columns which share at least one letter cannot be shown to differ significantly ($P \geq 0.05$). *P*-values were adjusted using the FDR method. Error bars indicate the modelled standard error (SEM). Raw data points are indicated by black dots

and did not differ from the unfertilised P_0 treatment. FSPS and $Ca(H_2PO_4)_2$ showed no difference in the yield response, while PPS fell between them and rock phosphate. Arguably, FSPS performed so well due to its residual N concentration, which could increase yields. The findings of the N-analysis refute this argument since all treatments (and their combinations) showed that plant N concentrations exceeded the critical concentration of 3.8% given by Bergmann (1992). Also, the N fertilisation of 300 mg N per kg soil was chosen to ensure that N-driven yield effects were only caused by the N form or its interaction with the P treatment and not by N deficiency. The yield response of the $(NH_4)_2SO_4$ + DMPP treatment illustrates this in an intriguing manner for the neutral and acidic soil: the biomass generally exceeded the $Ca(NO_3)_2$ treatment and on acidic soil even the rock phosphate treatment showed no significant difference to the $Ca(H_2PO_4)_2$ treatment. As discussed earlier, on alkaline soil, this observation disappears since there were high NH_4^+ losses. Hence, the observation on that soil is very similar for $Ca(NO_3)_2$ and $(NH_4)_2SO_4$ + DMPP. For the neutral and acidic soil, there were no significant differences between $Ca(H_2PO_4)_2$ and FSPS or PPS treated plants under the $(NH_4)_2SO_4$ + DMPP treatment.

Figure 4 shows plant P uptake. The $(NH_4)_2SO_4$ + DMPP treatment enhanced the uptake of all treat-

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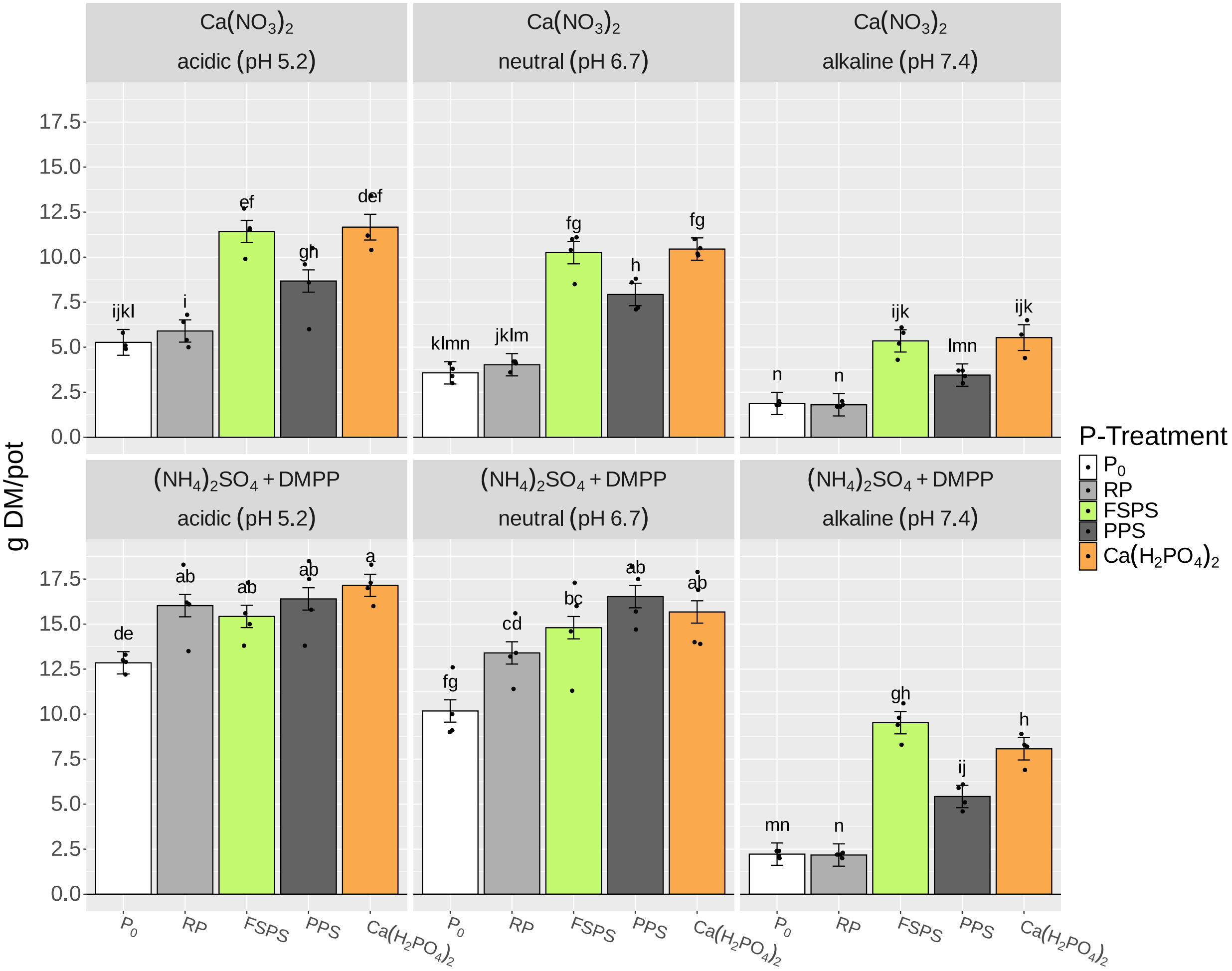

Figure 3. Aboveground biomass yield of maize treated with different phosphorus (P)-fertilisers (75 mg P/kg soil) under nitrogen (N) fertilisation (300 mg N/kg soil) in the form of $Ca(NO_3)_2$ or $(NH_4)_2SO_4$ + DMPP on three different soils, where P was applied in different forms (P_0 – no P fertilisation; RP – rock phosphate; FSPS – freeze-dried solid phase of a pig slurry; PPS – pyrolysed solid phase of a pig slurry; $Ca(H_2PO_4)_2$ – triple superphosphate). Lowercase letters indicate significant differences. Columns which share at least one letter cannot be shown to differ significantly ($P \geq 0.05$). P-values were adjusted using the FDR method. Raw data points are indicated by black dots

ments on the acidic and neutral soils. As observed for the biomass, the rock phosphate treatment failed to outperform the P_0 treatment under NO_3^- supply but drastically improved under NH_4^+ supply. In contrast to this observation, PPS showed an increase in plant P uptake on the acidic and neutral soil compared to the P_0 treatment, regardless of N-form. When comparing those observations to the FSPS treatment, the reduction in plant P availability by pyrolysis becomes even more evident. This falls in line with the observations of Steckenmesser et al. (2017), who showed that the pyrolysis reduced the P availability of sewage sludge. They reported that the thermal conditions (500 °C) could induce whitlockite synthesis, which is a mineral that is comparable to apatite regarding P availability.

Taking everything into account, the strongest influence on plant growth and plant P availability was exerted by the N form. It is known that a NO_3^--based nutrition leads to a physiological alkaline effect, while a NH_4^+-based nutrition leads to an acidic effect (Schubert and Yan 1997). This effect includes the rhizosphere (Römheld 1986) and therefore affects the P acquisition of the plant. This is backed by the findings of Riley and Barber (1971), who reported higher plant P uptake of maize supplied with NH_4^+ compared to NO_3^-, and Mengel (1986), who observed an increased solubility of rock phosphate on low pH

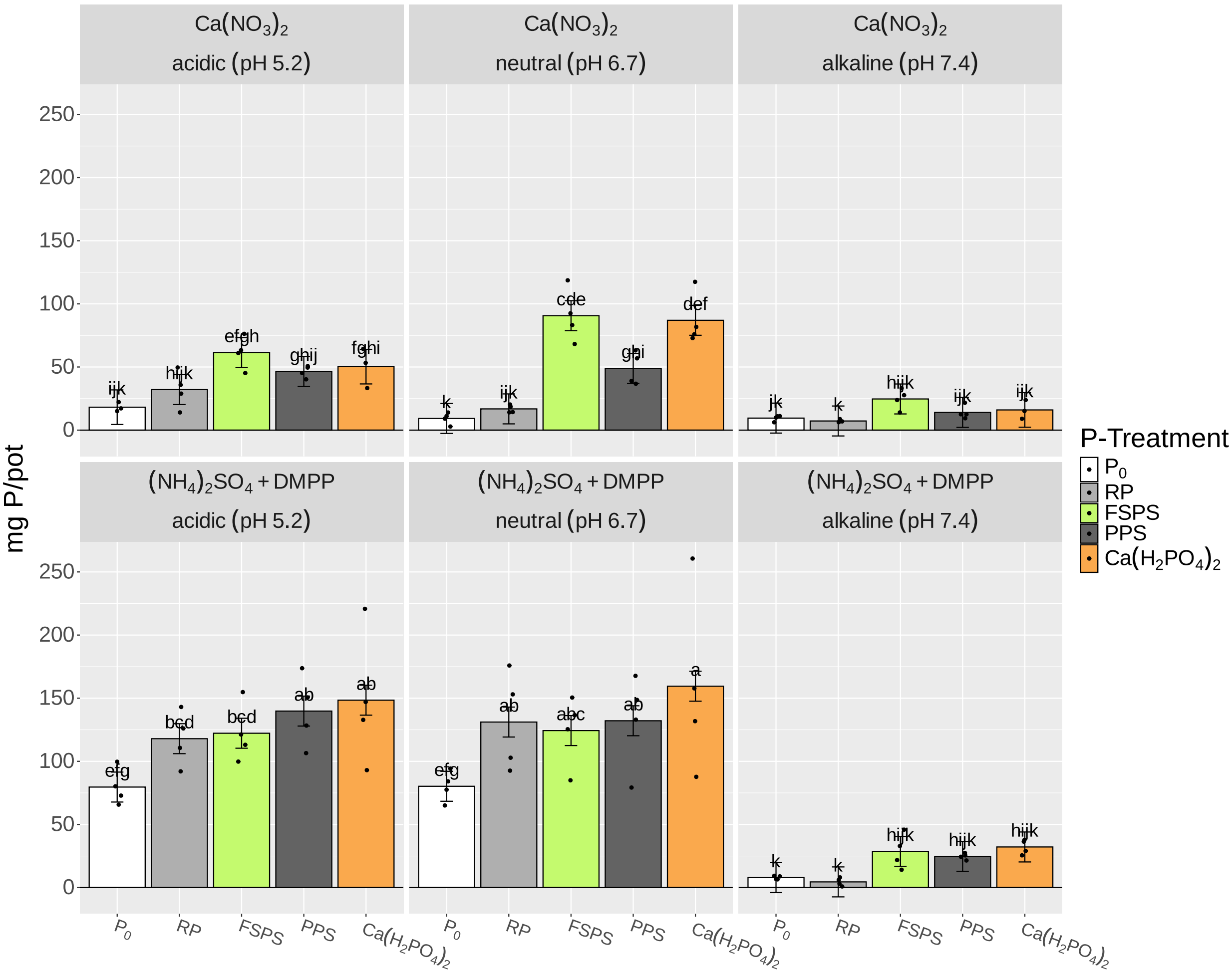

Figure 4. Phosphorus (P) uptake of maize treated with different P-fertilisers (75 mg P/kg soil) under nitrogen (N) fertilisation (300 mg N/kg soil) in the form of $Ca(NO_3)_2$ or $(NH_4)_2SO_4$ + DMPP on three different soils, where P was applied in different forms (P_0 – no P fertilisation; RP – rock phosphate; FSPS – freeze-dried solid phase of a pig slurry; PPS – pyrolysed solid phase of a pig slurry; $Ca(H_2PO_4)_2$ – triple superphosphate). Lowercase letters indicate significant differences. Columns which share at least one letter cannot be shown to differ significantly ($P \geq 0.05$). *P*-values were adjusted using the FDR method. Raw data points are indicated by black dots

soils. We found that the acidification induced by NH_4^+ supply promoted the solubilisation of P from rock phosphate as well as PPS. Rahmatullah et al. (2006) observed this effect on rock phosphate even when a mixed fertiliser (ammonium sulfate nitrate) was applied with an addition of a synthetic nitrification inhibitor (DMPP). Vogel et al. (2020) also reported this for calcium phosphate from pyrolysed sewage sludge. They furthermore showed that DMPP increased NH_4^+ fixation using K-edge microXANES spectroscopy and the Moglivekina method. The fixed NH_4^+ can be released over time and therefore could act as N source for plants at a later stage.

The limits of using NH_4^+ nutrition to enhance P availability from pyrolysed pig slurry became obvious in our experiment as well. This study shows that proton buffer capacity of soils needs to be taken into account when considering combined NH_4^+ and P fertilisation. After losing a considerable amount of the applied N through ammonia volatilisation on the alkaline soil, a high buffer capacity can lead to the effect that the released protons from the NH_4^+ uptake by the plant can be buffered. This reduction of acidification was also observed for N_2-fixing red clover roots on an alkaline soil (Hauter and Mengel 1988). When the proton buffer capacity is low, like on the acidic and neutral soil, the effect on P uptake was astonishing, since acidification was not lessened. However, a NH_4^+-based nutrition is not only a question of pH and buffer capacity. Liu and von Wirén (2017) reported that NH_4^+ nutrition at low concentrations increased plant growth while high NH_4^+ concentrations induce a toxicity, which is the result of a strong change in the cytosolic pH (Kosegarten et al. 1997). For our experiment we could not observe such effects but they need to be taken into account when working with NH_4^+ as a N source.

In conclusion, our objective was to assess whether pyrolysis could be applied to create a plant-available P fertiliser from pig slurry. We showed that the pyrolysis product reached P availability comparable to that of commercially available fertilisers ($Ca(H_2PO_4)_2$ and rock phosphate) under certain conditions, but freeze-drying led to even better results. This needs to be thoroughly assessed by future studies, since the enrichment in P concentration comes at the cost of reduced availability. Also, the energy costs of this procedure need to be evaluated and taken into account when searching for the best procedure. There are technical enhancements to the pyrolysis process that could improve the outcome (e.g., recovering N from exhaust gas, adding compounds to achieve a desired P form), but these also increase process costs. The data indicate that, if economically suitable, pyrolysis would be a procedure to enrich organic fertilisers in P while maintaining a mediocre P availability. This study also highlighted the importance of N nutrition for plant P availability. The use of a stabilised NH_4^+-based N nutrition (e.g. synthetic nitrification inhibitor) can be recommended for this purpose if the soil pH allows it.

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