



Closing global P cycles: The effect of dewatered fish sludge and manure solids as P fertiliser

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ABSTRACT

The aim of this study was to contribute to closing global phosphorus (P) cycles by investigating and explaining the effect of fish sludge (feed residues and faeces of farmed fish) and manure solids as P fertiliser. Phosphorus quality in 14 filtered and/or dried, composted, separated or pyrolysed products based on fish sludge or cattle or swine manure was studied by sequential chemical fractionation and in two two-year growth trials, a pot experiment with barley (*Hordeum vulgare*) and a field experiment with spring wheat (*Triticum aestivum*). In fish sludge, P was mainly solubilised in the HCl fraction ($66 \pm 10\%$), commonly being associated with slowly soluble calcium phosphates, and mean relative agronomic efficiency (RAE) of fish sludge products during the first year of the pot experiment was only $47 \pm 24\%$. Low immediate P availability was not compensated for during the second year. Thus efforts are needed to optimise the P effects if fish sludge is to be transformed from a waste into a valuable fertiliser. In manure solids, P was mainly soluble in H_2O and 0.5 M NaHCO_3 ($72 \pm 14\%$), commonly being associated with plant-available P, and mean RAE during the first year of the pot experiment was $77 \pm 19\%$. Biochars based on fish sludge or manure had low concentrations of soluble P and low P fertilisation effects, confirming that treatment processes other than pyrolysis should be chosen for P-rich waste resources to allow efficient P recycling. The field experiment supported the results of the pot experiment, but provided little additional information.

1. Introduction

Closing nutrient loops by recycling of organic waste sources as fertiliser has received increased attention. One poorly utilised organic resource is waste from aquaculture production. Global aquaculture production is thriving, driven by the increasing global demand for farmed fish, and accordingly the amount of organic waste from aquaculture production is increasing. Norway has become the world's largest producer of farmed salmonids (salmon, trout etc.) (FAO, 2020), by utilising its production advantages deriving from the long coastline and location by the Gulf Stream. Due to increased production, the composition of salmon feed has changed considerably over recent decades, with an increasing proportion of marine ingredients being replaced by plant-based ingredients (Aas et al., 2019). In Norwegian production, salmon feed now contains on average 70% plant-based ingredients, including soya mainly imported from Brazil (Laksefakta, 2018). Agriculture and also aquaculture are thus indirectly highly dependent on the input of phosphorus (P) fertiliser, which is often derived from mineable rock phosphate, a non-renewable and geographically limited resource.

In Norway, salmon farms are placed strategically to dilute feed residues and faeces (i.e. fish sludge) in the sea, thus deliberately wasting P with land-based origin. Aquaculture can only expand sustainably if fish sludge is recycled in biological cycles, e.g. as fertiliser in agriculture.

In 2019, 14,000 tonne (t) P were lost to the sea with Norwegian fish sludge (Broch and Ellingsen, 2020). In comparison, Norwegian farmers apply 12,000 t P annually in the form of animal manure (Hamilton et al., 2016) and 9000 t P in the form of mineral fertiliser (Norwegian Food Safety Authority, 2021). In contrast to other large fish farming nations, Norway imposes rather strict requirements where land-based fish farms must collect fish sludge before discharging wastewater, to avoid pollution of ecologically vulnerable coastal areas. In 2019, 102 t P were collected with fish sludge, mainly from freshwater-based hatcheries (Broch and Ellingsen, 2020). Increasing amounts are expected to be collected with the ongoing trend of moving post-smolt production and salmon farms on land and with (semi-)closed production systems in the sea, both in Norway and internationally. Instead of disposal to municipal wastewater system, filtered or sedimented fish sludge may be utilised as a co-substrate in centralised biogas or composting plants, or de-watered

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and dried before utilisation as fertiliser (Rosten et al., 2013). To date, treatment technologies for fish sludge have mainly been designed with the aim of lowering the treatment costs and reducing odour, while production of high-quality fertiliser products has not been a treatment aim. Lack of information on the fertilisation effects of fish sludge is one of the main bottlenecks to closing blue-green nutrient cycles in practice.

Another challenge to efficient utilisation of fish sludge as fertiliser in agriculture is its accumulation in soils along the Norwegian coastline, which already receive excess P through animal manure. As in many industrialised countries, increasing specialisation of Norwegian agricultural production over time has resulted in livestock-intensive areas with high P soil status and no or low P fertiliser requirements (Bechmann, 2014). Therefore, resource-efficient utilisation of P in fish sludge and manure requires transportation to crop production areas in need of P fertilisation, but the bulky nature of untreated fish sludge and manure makes transportation over long distances energy- and cost-intensive. Low-technology treatments such as mechanical separation, filtration or thermal treatment (e.g. pyrolysis) can be applied to fish sludge or animal manure to reduce the water content (Rosten et al., 2013; Jensen, 2013).

To date, few studies have examined the effect of fish sludge as fertiliser. Based on a pot experiment with barley (*Hordeum vulgare*) and two field experiments with spring cereals, Brod et al. (2017) concluded that dried, freshwater-based fish sludge can have relative agronomic efficiency (RAE) of 50–80% (compared with mineral fertiliser) when applied as nitrogen (N) fertiliser. Teuber et al. (2005) and Celis et al. (2008) also observed N fertiliser effects of salmon sludge when applied at increasing rates to ryegrass (*Lolium multiflorum* L.) in Chile. Yogeve et al. (2020) found significant P fertilisation effects on a lettuce (*Lactuca sativa*) crop of digested sludge of African catfish (*Clarias gariepinus*) raised in a recirculating aquaculture system (RAS). Brod et al. (2015) found good P fertilisation effects of dried salmon sludge on ryegrass, but only in the acidic soil pH range. Systematic investigations of the P fertilisation effects of fish sludge have not been conducted previously.

More studies have been performed on the P fertilisation effect of products based on animal manure. In general, mechanical separation seems to result in manure solids with similar P plant availability as the original manure substrate (e.g. Gasser et al., 2012; Christel et al., 2016). In contrast, pyrolysis has been shown to decrease P plant availability in biochar based on manure, with the P quality decrease being greater with higher process temperature (e.g. Bruun et al., 2017). However, due to large variations in composition depending on e.g. type of livestock, feeding, bedding and treatment technology, more data on the relationship between P compounds in manure solids and P plant availability are required to allow its efficient use as a substitute for mineral fertiliser.

The aim of this study was to investigate the P fertiliser quality of fish sludge and manure solids subjected to various treatment methods. The fertiliser quality was assessed by (1) comparing the P fertilisation effects of organic products based on fish sludge or animal manure with those of mineral P fertiliser, and (2) assessing the P quality in organic products based on fish sludge or animal manure based on chemical P characterisation.

Definition: In this paper, the term *fish sludge* refers to the mixture of feed residues and faeces of farmed smolt, and is not to be confused with e.g. fish processing waste.

2. Materials and methods

2.1. Organic products

In total, 14 organic products were included in the experiments; six products based on fish sludge and eight products based on animal manure (Table 1). Origin and treatment of the various products are described in Table 1. All analyses were performed on dried (55 °C) and milled samples. Low drying temperature was used to avoid possible alteration of P quality by higher temperature.

Table 1
Description of the 14 organic products.

No.	Product	Description
1	Fish sludge 1	Sludge from freshwater-based flow-through smolt hatchery at Flatanger Settefisk (2018) or Sisomar (2019). Treated in drum filter before gravimetric sedimentation, flocculation, addition of chemical polymer and dewatering by screw press, and then thermal drying (~50 °C). Technology developed by Sterner AS.
2	Fish sludge 2	Sludge from freshwater-based recycling aquaculture system (RAS) hatchery for smolt at MOWI, Steinsvik. Treatment in drum filter before addition of chemical polymer and dewatering by belt filter and centrifuge, and thermal drying (~70 °C) for >2 h. Technology developed by Scanship AS.
3	Fish sludge 3	Sludge from freshwater-based smolt hatchery Sævareid Fiskeanlegg (both flow-through and RAS system). Treatment in drum filter before dewatering by belt filter and drying by thermal mechanical dryer (~100 °C). Technology developed by Fjell Technology Group AS.
4	Fish sludge 4	Sludge from freshwater-based RAS smolt hatchery Lerøy Midt, Belsvik. Treatment in drum filter before addition of chemical polymer and dewatering by belt filter.
5	Fish sludge 5	Filtered fish sludge from four freshwater-based hatcheries using RAS-systems (two owned by MOWI and each one owned by Lerøy Midt and SalMar), composted together with treated wood waste in a composting reactor for ~14 days. Composting temperature varied between 45 and 65 °C. Wood particles >1 mm were removed by sieving. Technology developed by Global Green Energy AS (GGE), Norway.
6	Cattle manure 1a	Solid fraction after separation of slurry by screw press. For more information, see Cattle manure 1b.
7	Cattle manure 1b	Granules produced from wet fraction after Cattle manure 1a, followed by anaerobic digestion, nitrification, filtration and air-drying. Technology developed by Knut Vasdal, Skien, Norway.
8	Cattle manure 2	Slurry sampled on a dairy farm in the Netherlands and anaerobically digested before separation by a screw press (1000 µm). Here, the solid fraction was used.
9	Swine manure 1	Co-treated swine manure from 55 farms in the region Achterhoek, Netherlands. The manure was separated by a screw press (500 µm). Here, the solid fraction was used.
10	Swine manure 2	Swine manure co-digested with cattle manure, whey and other wastes from the food and feed industry (Achterhoek, Netherlands). The digestate was separated by a decanter centrifuge. Here, the solid fraction was used.
11	Horse manure	Horse manure composted with wood chips, sampled on a horse farm in Toten, Norway.
12	Digestate-food waste/manure	Digestate based on anaerobic treatment of swine and cattle manure (~25% on dry matter basis) and source-separated household, catering and food industry waste. Sampled at GREVE Biogas, Tønsberg, Norway.
13	Biochar-swine manure	Solid fraction of Dutch swine manure, dried before pyrolysis at 650–750 °C for 10–20 min (Gollenbeek et al., 2018). The product is marketed in the Netherlands as 'EcoChar' by Mavitec.
14	Biochar-fish sludge	From flow-through smolt hatchery Nordlaks Smolt, Innhavet. Treatment by drum filter, followed by dewatering by belt filter with vacuum suction and steam-drying (~170 °C) and pyrolysis at 600–650 °C for 20–30 min. Technology developed by AquaGreen.

Dry matter (DM) content was determined after drying at 105 °C. The pH_{H2O} value was determined in deionised H₂O in a solid:solution ratio of 1:10 or in liquid samples, by conductometry. Total nitrogen (N) was determined by the modified Kjeldahl method (EN 13654-1, 2001). Ammonium (NH₄-N) was determined by the Kjeldahl method (steam distillation without preceding digestion) and nitrate (NO₃-N) was determined after extraction with 0.0125 M CaCl₂ in a solid:solution ratio

of 1:10 or in the liquid samples. Phosphorus (P) and all other nutrients and heavy metals were determined by ICP-MS after digestion in aqua regia ($\text{HNO}_3 + \text{HCl}$). Carbon (C) concentration was calculated based on loss of ignition determined by incineration at 550 °C.

To determine the fractions of inorganic and organic P, samples were digested in 6 M H_2SO_4 with or without prior incineration at 550 °C (Møberg and Petersen, 1982). Ortho-P in the extracts was analysed by the molybdenum blue method according to Murphy and Riley (1962) to give total and inorganic P concentrations. Organic P was calculated as the difference between total and inorganic P.

Phosphorus fractions of different solubility were analysed in duplicate using the sequential fractionation scheme developed by Hedley et al. (1982) and modified by Sharpley and Moyer (2000) for application to organic products. Samples of 1 g were extracted in 200 mL deionised H_2O for 1 h and then extracted in sequence in 200 mL 0.5 M NaHCO_3 (labile P), 0.1 M NaOH (P adsorbed to Fe-/Al-(hydr)oxides or Fe-/Al-phosphates) and 1 M HCl (stable Ca-phosphates), each for 16 h. After centrifugation at 1160 relative centrifugal force for 20 min, total P in the extracts was analysed by ICP-OES. Residual P in the samples was analysed by ICP-OES after digestion of the dried residual sample in concentrated HNO_3 in an ultraclave. Phosphorus recovery was calculated based on the sum of all fractions compared with total P concentration determined after digestion in aqua regia.

2.2. Pot experiment

To study the immediate and residual plant availability of P in the organic products, a pot experiment was conducted using 3 L pots and two soil types; a limed mixture of nutrient-deficient sand (3 kg) and sphagnum peat (0.3 kg; 20 vol-%), and a silty clay loam (USDA). The sandy-sphagnum peat model soil was chosen based on the results of previous experiments with the same growing medium, where plants clearly responded to P fertilisation (Brod et al., 2015; Øgaard and Brod, 2016). The loam was chosen to represent a typical natural agricultural soil and based on its low content of plant-available P (P-AL), which indicated P demand. The loam, classified as a Stagnosol in the World Reference Base for Soil Resources (NIBIO, 2021a), was collected from the area where the field experiment was located (see 2.3). Selected chemical properties of the soils are shown in Table 2.

Fertilisation rates were calculated based on total P content and a dose of 45 mg P, equivalent to 30 kg P ha^{-1} (assuming 20 cm topsoil depth), was applied to each pot. However, due to an artefact in P analysis, the product Cattle manure 1a was applied at too high a rate (75 mg P pot^{-1} , equivalent to 45 kg P ha^{-1}). During data treatment, the fertilisation effect of Cattle manure 1a was adjusted to allow for comparison with the other treatments, based on the assumption that plants respond linearly to P application up to and beyond application of 45 mg P pot^{-1} . The organic products were compared with a treatment providing no P fertilisation (NoP) and mineral control treatments that received $\text{Ca}(\text{H}_2\text{PO}_4)_2$ at a rate of 22.5 mg P pot^{-1} (1/2 MinP) and 45 mg P pot^{-1} (MinP). All other nutrients were applied as solution in amounts regarded as sufficient: N equivalent to 210 kg ha^{-1} as $\text{Ca}(\text{NO}_3)_2$, K equivalent to 210 kg ha^{-1} as K_2SO_4 and Mg equivalent to 20 kg ha^{-1} as

MgSO_4 , in addition to Fe, Mo, Mn, Cu, B and Zn. For products with a high $\text{NH}_4\text{-N}$ content, mineral N application was reduced to equate to the expected N effect of the product; i.e. mineral N application to pots with Digestate-food waste/manure was reduced from 210 to 73 kg N ha^{-1} based on the assumption that 85% of mineral N in the product would be available to plants (NIBIO, 2021b). There were three replicates per treatment. The set-up of the experimental design can be found in Table S1 (Supplementary material).

In October 2018, 15 barley seeds (*Hordeum vulgare* var. Heder) were sown per pot and thinned out to 12 plants after germination. The pots were randomly placed on tables in a greenhouse. Heating was provided if the temperature dropped below 18 °C during the day and 12 °C at night. Artificial light (white light from halogen lamps) was used to provide a photoperiod of 16 h day^{-1} . The plants were watered by weighing to 60%, and later 70%, of water-holding capacity three times a week. Approximately six weeks after set-up of the experiment, when the first awns were visible (development stage Zadoks 50; Zadoks et al., 1974), aboveground biomass was harvested by cutting the plants with scissors at 2 cm above the soil surface. Plant material was dried at 60 °C, DM production per pot was recorded and the plant material was milled. Nitrogen concentration was determined using a CN analyser and P concentration by ICP-MS after digestion with concentrated HNO_3 in a microwave. Phosphorus uptake (mg pot^{-1}) was computed by multiplying DM production by plant tissue P concentration. After harvest, the 54 pots with the model soil were stored at 4 °C.

In October 2019, approximately 10 months after harvest, soil samples were taken from each pot. The soil was dried at 40 °C and sieved, and $\text{pH}_{\text{H}_2\text{O}}$ was analysed in deionised H_2O in a solid:solution ratio of 1:2.5 (v/v). P-AL was analysed by ICP-OES, after extraction of 1 g air-dried soil in 20 mL AL-solution with 0.1 M ammonium lactate and 0.4 M acetic acid adjusted to pH 3.75 according to Egnér et al. (1960). To study residual P effects, the stored pots were prepared by removing stubble and the remaining plant roots were divided and mixed into the soil before all plant nutrients except P were again applied to the soil as solution. Residual P effects were compared with three control treatments: NoP (2018: 0 kg MinP ha^{-1} ; 2019: 0 kg MinP ha^{-1}); residual effect of MinP (2018: 30 kg MinP ha^{-1} ; 2019: 0 kg MinP ha^{-1}); MinP (2018: 15 kg MinP ha^{-1} ; 2019: 30 kg MinP ha^{-1}). Again, 15 barley seeds were sown per pot, thinned out to 12 plants and aboveground biomass was harvested after about six weeks. The plant material was analysed for N and P concentration as described. Cattle manure 1a was not included in data treatment for P uptake or P-AL in the soil, due to initially too high P fertilisation.

2.3. Field experiment

A 2-year field experiment was conducted at Øsaker (59.32°N, 11.04°E) in south-eastern Norway to study the P effect of five organic products selected among those included in the pot experiment. These were: Fish sludge 1, Cattle manure 1a and b, Digestate-food waste/manure and Biochar-fish sludge. Normal annual precipitation at Øsaker is 978 mm, half of which occurs during the period May–September, and normal mean monthly air temperature in the growing season is 11–17 °C

Table 2
Chemical properties of the soils used in pot experiments.

Soil type	Organic matter %	pH	Total P mg kg^{-1}	P-AL ¹ mg kg^{-1}	$\text{CaCl}_2\text{-P}^2$ mg kg^{-1}	Water-holding capacity g kg^{-1}	Volume kg L^{-1}
Model soil	0.3	–	219	30	0.47	238	–
Silty clay loam	2.6 ³	6.1	–	54 (48–66)	1.92	522	1.1

¹ Extraction of 1 g air-dried soil in 20 mL AL-solution with 0.1 M ammonium lactate and 0.4 M acetic acid adjusted to pH 3.75 according to Egnér et al. (1960), analysed by ICP-OES. In fertilisation planning, a P-AL value of 0–50 is characterised as low, 50–70 as intermediate/optimum, 70–100 as moderately high, 100–140 as high, and >140 as very high.

² Extraction of 1 g air-dried soil in 20 mL 0.0025 M CaCl_2 ; colorimetric analysis of ortho-P by molybdenum blue method according to Murphy and Riley (1962).

³ Estimated based on ignition at 550 °C and clay content according to Riley (1996).

(where normal is defined as average for the period 1991–2020). The soil at the experimental site is a silty clay loam Stagnosol (NIBIO, 2021a) the same that was used in the pot experiment (Table 2).

Cereal was grown in the year before the experiment and the stubble was left standing during the winter, after which the soil was harrowed twice in spring. The experiment was established on May 18th, 2018, and repeated treatments were applied on May 8th, 2019. The experiment had a randomised block design with three replicates. Each plot measured 3.0 m × 8.0 m and plants were harvested on a 1.5 m × 6.5 m sub-area within each plot. The set-up of the experimental design can be found in Fig. S1 (Supplementary material). Organic products were applied based on total P content, equivalent to 30 kg P ha⁻¹. The effect of the organic products was compared with a treatment providing no P fertilisation (NoP) and mineral fertiliser control treatments (Yara Opti-P 0–20–0 at a rate of 15 kg P ha⁻¹ (1/2 MinP) and 30 kg P ha⁻¹ (MinP)). In 2018, Cattle manure 1a was applied at too high a rate due to the previously described artefact in P analysis that affected the pot experiment. Mineral N fertiliser (Yara Opti-NK 22–0–12) was applied at a rate of 120 kg N ha⁻¹ in both 2018 and 2019. For all organic products, the amount of readily plant-available N was estimated as described in Table S2 (Supplementary material) and application of mineral N fertiliser was reduced accordingly. The soil was again harrowed within two hours after fertiliser application, and spring wheat (*Triticum aestivum* L. var. Mirakel) was sown. To study P availability throughout the growing season, aboveground biomass was sampled from each plot by cutting four 0.5-m rows, and P concentration was analysed as described previously. In 2018, plants were sampled at development stage Zadoks 39, and in 2019 at development stages Zadoks 37 and 45 (Zadoks et al., 1974). Since the growing season of 2018 was extraordinarily warm and dry, the field was harvested early, on August 7th. In 2019, the field was harvested on September 16th. Yield and grain DM were recorded, and grain samples were taken from each plot for analysis of P concentration as described previously. Phosphorus uptake in grain was computed by multiplying P concentration by DM production, and converted to a per hectare basis. In 2019, due to a sampling error, one replicate was missing from two treatments (NoP; Cattle manure 1a). Straw residues were left on the plots.

Soil samples were taken at 20 cm depth in each plot before fertiliser application in spring 2018 and after harvest in both 2018 and 2019, and analysed for P-AL as described previously. In addition, easily releasable P in the soil samples was extracted with 0.0025 M CaCl₂ for 1.5 h (1:20 w/v) and measured by the molybdenum blue method according to Murphy and Riley (1962).

2.4. Data analysis

Relative agronomic efficiency (RAE) was calculated for the organic products based on P uptake during the first year of the pot experiment:

$$RAE = 100 \times \frac{X_1}{P \text{ applied}}$$

$$X_1 = \frac{(Y_1 - b)}{a}$$

where: P applied = Amount of P applied with organic product (mg P pot⁻¹); Y₁ = P uptake in aboveground biomass as effect of organic product; X₁ = Amount of MinP (mg P pot⁻¹) resulting in equally high P uptake in biomass as application of organic product at 45 mg P pot⁻¹; a and b = Slope and intercept obtained from linear regression with Y = P uptake (mg P pot⁻¹) after MinP application and X = Application rate of MinP (0, 22.5 and 45 P pot⁻¹).

Apparent P use efficiency (PUE %) during the pot experiment was calculated as:

$$PUE = \frac{P \text{ uptake}(P+) - P \text{ uptake}(NoP)}{P \text{ applied}} \times 100$$

where: P uptake (P+) = P uptake in aboveground biomass of plants that received mineral or organic P fertiliser treatment (mg P pot⁻¹); P uptake (NoP) = P uptake in aboveground biomass of plants that received no P fertiliser treatment (mg P pot⁻¹; average of n = 3); P applied = For 2018: Total P amount applied with fertiliser treatment. For 2019: Total P amount applied with fertiliser treatment in 2018 minus P amount removed with aboveground biomass in 2018.

For the field experiment, P balance was calculated as:

$$P \text{ balance} = P \text{ applied} - P \text{ removed}$$

where: P applied = P amount applied with mineral fertiliser or organic products in 2018 and 2019; P removed = P removed with yield in 2018 and 2019.

Analysis of variance (ANOVA) was performed to identify significant differences between organic products in the distribution of P in sequentially extracted fractions. ANOVA was also applied to study the effect of fertiliser treatments on P uptake, N/P ratio in biomass and RAE in the pot and field experiment, and pH_{H2O} and P-AL in the soil. Analysed data were checked for normal distribution (normal quantile plots) and homogeneity of variance (residual vs fitted plots). To perform multiple comparisons, Tukey's honestly significant difference (HSD) multiple comparison test was used (α = 0.05).

T-tests were conducted to compare products based on fish sludge (products 1–5) with products based on animal manure (products 6–12) for chemical characteristics and effect in the pot experiment. For chemical characteristics, products sampled in both 2018 and 2019 were included, giving n = 6 for fish sludge and n = 10 for animal manure products. Results are expressed as mean ± standard deviation.

3. Results

3.1. Chemical characteristics of organic products

The organic products contained 6.6–65.9 g P kg⁻¹ DM (Table 3). The total P concentration was on average higher in products based on fish sludge (31.5 ± 10.6 g P kg⁻¹ DM) than in products based on animal manure (13.3 ± 7.5 g P kg⁻¹ DM). Both biochars contained considerably higher P concentrations than products that had not undergone thermal treatment. In all organic products, P was mainly present as inorganic P, with the inorganic P fraction varying between 72 and 98% of total P.

Sequential P fractionation revealed that in products based on animal manure, P was mainly soluble in H₂O and 0.5 M NaHCO₃ (72 ± 14%) (Fig. 1). These first two steps of the sequential extraction scheme are commonly associated with soluble and plant-available P. In products based on fish sludge, only 17 ± 2% of P was soluble in H₂O and 0.5 M NaHCO₃ and the sum of these fractions was significantly smaller than in products based on animal manure. In products based on fish sludge, most of the P was found in the HCl-soluble fraction (66 ± 10%), significantly more than in products based on animal manure (11 ± 7%). Phosphorus in both biochars was also mainly found in the HCl-soluble fraction (75 ± 2% in Biochar-fish sludge; 66 ± 8% in Biochar-swine manure). The HCl-soluble fraction is commonly associated with stable calcium phosphates. The sum of all fractions in the sequential fractionation comprised 81–127 (105 ± 15)% of total P in the organic products (P recovery) (Table 3).

The N/P ratio was low relative to plant requirements in all organic products except Digestate-food waste/manure (N/P = 8.3 in 2018 and 14.3 in 2019). For all other products, the N/P ratio varied between 0.2 and 4.0, and there was no significant difference in terms of N/P ratio between products based on fish sludge or animal manure. The K concentration, in contrast, was significantly higher in products based on animal manure (30 ± 15 g K kg⁻¹ DM) than in products based on fish sludge (1.6 ± 1.3 g K kg⁻¹ DM).

In their present form, only five (Products 1, 2, 3, 7 and 14) of the 14 products included in the study fulfilled the new European Union (EU) requirements on solid or liquid organic fertiliser products (European

Table 3

Selected chemical characteristics of the organic products used in the pot experiment (2018) and the field experiment (2018 and 2019). DM = dry matter.

No.	Organic product	Year	DM	pH	TOC ¹	Total P	Inorg. P	Total N	NH ₄ -N	C/N	N/P	K S Ca Mg			
			%		% of DM	g kg ⁻¹ DM	% of total P	g kg ⁻¹ DM	% of total N			g kg ⁻¹ DM			
1	Fish sludge 1	2018	96	5.6	50	21.5	75	68.2	1.2	6.7	3.2	0.7	1.8	46	2.0
		2019	92	–	47	17	–	35	2.9	10.9	2.1	0.5	2.1	33	–
2	Fish sludge 2	2018	83	5.3	46	36.9	86	68.6	4.9	6.7	1.9	1.1	0.1	84	4.1
3	Fish sludge 3	2018	92	5.3	39	45.8	81	34.2	2.6	11.4	0.7	1.1	0.0	77	3.8
4	Fish sludge 4	2018	7	5.2	51	31.5	89	74.8	16.2	6.8	2.4	2.7	5.5	95	4.1
5	Fish sludge 5	2018	68	6.0	30	36.2	98	32.5	6.0	9.4	0.9	3.7	5.3	73	5.4
6	Cattle manure 1a	2018	22	8.0	48	10.0 (6.0 ²)	72	23.9	8.4	18	4.0	29	4.0	16	5.1
		2019	24	9.0	48	7.0	–	20	1.7	24	2.9	–	5.3	16	–
7	Cattle manure 1b	2018	84	8.0	44	14.0	73	52.5	1.1	7.2	3.7	39	6.6	32	8.2
		2019	89	9.2	36	15	–	39	1.6	9.3	2.6	–	8.5	39	–
8	Cattle manure 2	2018	24	8.4	42	10.9	97	23.3	74.4	18.1	2.1	24	5.0	20	9.7
9	Swine manure 1	2018	24	8.5	42	25.5	95	26.9	62.5	15.6	1.1	15	6.0	30	16
10	Swine manure 2	2018	23	7.9	40	27.2	96	30.1	58.8	13.3	1.1	16	11	29	14
11	Horse manure	2018	18	7.1	40	6.6	77	26.8	3.6	14.9	4.1	18	3.5	26	6.6
12	Digestate-food waste/ manure	2018	5	7.5	41	10.0	84	83.0	67.0	5.1	8.3	50	8.2	22	6.0
		2019	5	7.9	43	6.5	–	93.5	62.6	4.6	14.3	52	7.6	26	4.3
13	Biochar-swine manure	2018	90	9.4	32	65.9	98	15.4	3.1	21.1	0.2	41	7.9	113	33
14	Biochar-fish sludge	2018/ 19	100	8.8	33	61.1	93	56.4	0.4	7.4	0.9	15	1.8	113	12

¹ Total organic carbon estimated based on loss of ignition (550 °C) divided by a factor of 1.72.

² Initial analysis based on which fertilisation rates in 2018 were calculated.

Union, 2019/1009). Products 4, 6, 8, 9, 10, 11 and 12 contained too low concentrations of N, P and K by mass to qualify as organic liquid or solid EU fertiliser products. The remaining two products exceeded EU minimum requirements regarding heavy metal contents, Product 5 (Fish sludge 5, to which wood waste was added during treatment) due to elevated Cd, Zn and Cu concentrations and Product 13 (Biochar-swine manure) due to elevated Zn and Cu concentrations. The organic products studied were not analysed for hexavalent chromium (Cr VI), inorganic arsenic (As), biuret (C₂H₅N₃O₂), or pathogens, analyses which will also be required by the new EU fertiliser regulation, but all other specified contaminants were analysed and are presented in Table 4.

3.2. Pot experiment

Barley plants clearly responded to P application on the model soil, as indicated by a linear increase in P uptake in aboveground biomass (y) as function of increasing MinP application rate (x) ($y = 0.39x + 5.1$; $R^2 = 0.97$). For the loam soil, however, there was no response of barley to P application, despite the soil P-AL level indicating P demand (54 mg kg⁻¹ soil; Table 2). Loam pots without P application (NoP) had equally high P uptake in aboveground biomass as pots that had received the optimum P application (MinP) ($y = 0.01x + 28.6$; $R^2 = 0.01$). In the following, we therefore only present results relating to the model soil.

There were large differences in immediate plant availability of P,

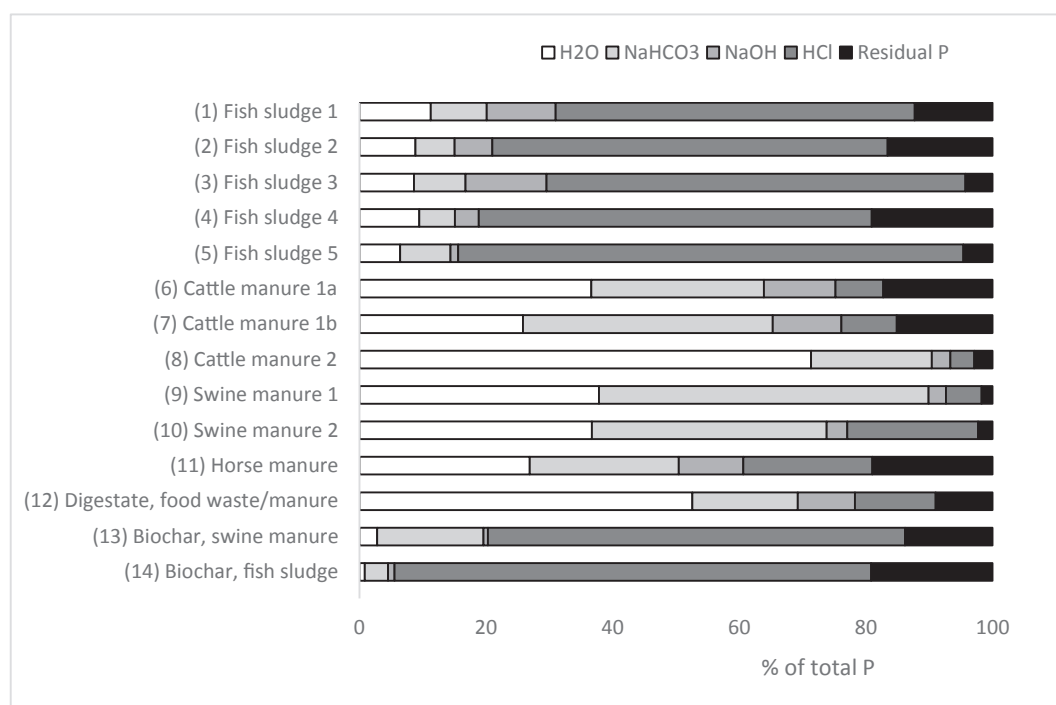


Fig. 1. Distribution of total phosphorus (P) in the organic products into different P fractions according to the sequential fractionation scheme, originally described by Hedley et al. (1982).

Table 4

Heavy metal concentrations (mg kg^{-1} DM) in organic products used in the pot experiment (2018) and the field experiment (2018 and 2019). (X) indicates that the heavy metal concentration exceeds the threshold for use in agriculture (European Union, 2019/1009).

No.	Organic product	Year	Cd	Zn	Pb	Hg	Ni	Cu	Cr
1	Fish sludge 1	2018	0.99	390	1.9	0.011	<1.5	12	9.6
		2019	0.40	390	<0.5	0.018	1.7	13	3.7
2	Fish sludge 2	2018	0.98	560	2.4	0.14	4.1	26	16
3	Fish sludge 3	2018	0.47	540	2.3	0.11	24	24	6.7
4	Fish sludge 4	2018	0.84	470	2.8	0.098	2.8	22	11
5	Fish sludge 5	2018	2.4 (X)	1300 (X)	34	0.26	9.6	2500 (X)	61
6	Cattle manure 1a	2018	<0.1	98	2.5	0.04	2	16	5.3
		2019	<0.1	110	1.1	0.15	3.9	21	2.7
7	Cattle manure 1b	2018	0.68	260	3	0.033	4.5	58	6.6
		2019	0.47	350	3.8	0.021	12	73	6.3
8	Cattle manure 2	2018	0.15	140	3.4	<0.01	4	37	7
9	Swine manure 1	2018	0.18	450	2	0.017	3.2	170	6.3
10	Swine manure 2	2018	0.2	460	2.6	0.032	20	160	8.5
11	Horse manure	2018	0.68	240	3.1	<0.01	5.5	35	5
12	Digestate-food waste/manure	2018	0.17	310	3.7	<0.07	9.1	84	9.5
		2019	–	320	–	–	–	93	–
13	Biochar-swine manure	2018	0.55	2000 (X)	7.6	<0.01	21	530 (X)	38
14	Biochar-fish sludge	2018/19	0.75	670	3	0.074	<1.5	34	10

measured as P uptake in aboveground biomass, as an effect of the various fertiliser treatments (Fig. 2a), but only differences $>7.1 \text{ mg P pot}^{-1}$ were statistically significant. Several products based on animal manure from swine and cattle resulted in almost equally high P uptake as MinP and, on average, products based on animal manure resulted in higher P uptake ($18.5 \pm 0.8 \text{ mg P pot}^{-1}$) than products based on fish

sludge ($13.4 \pm 0.9 \text{ mg P pot}^{-1}$). The RAE of the organic products was on average also higher for products based on animal manure ($77 \pm 19\%$) than for products based on fish sludge ($47 \pm 24\%$).

Low P availability in products based on fish sludge during the first year after application was not compensated for during the second year (Fig. 2b). On average, only $2.9 \pm 2.5\%$ of the residual P in organic

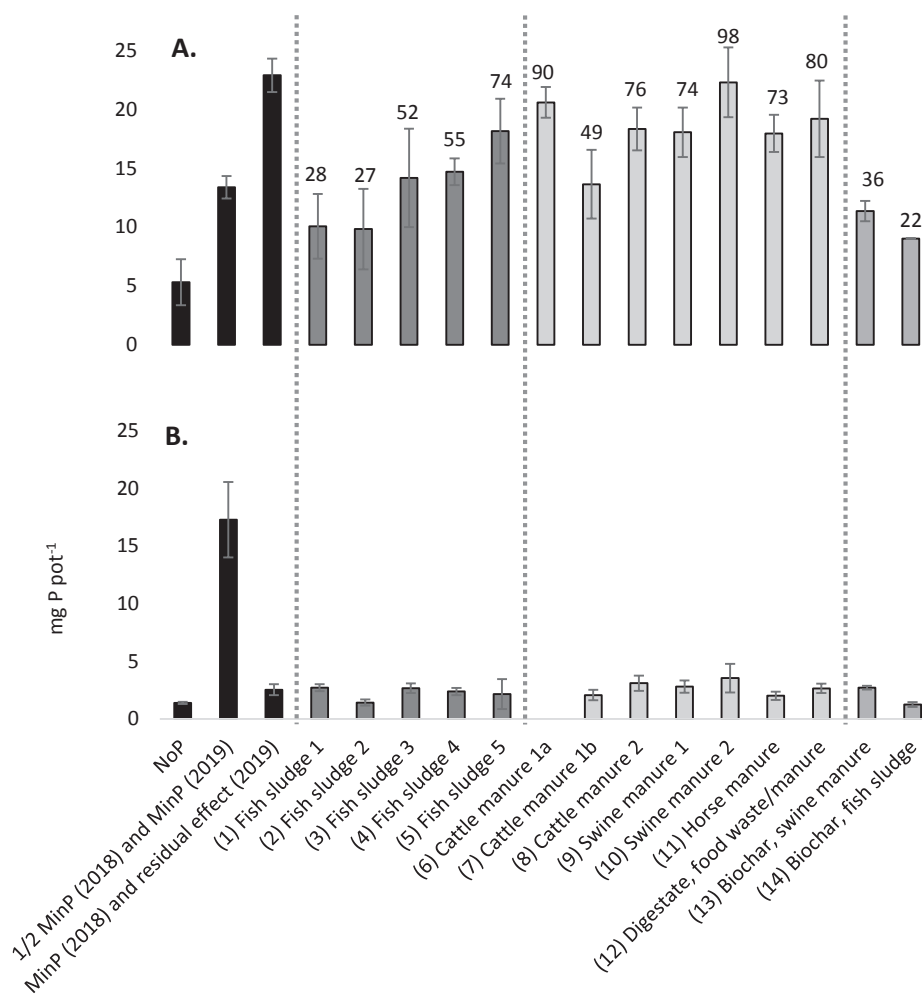


Fig. 2. Phosphorus (P) uptake in barley (mg P pot^{-1}) during the pot experiment as an effect of the different fertiliser treatments. A) First year effect (2018) and B) residual effect (2019). Error bars represent the standard deviation within each treatment. In (A), numbers indicate relative agronomic efficiency (RAE, %) calculated for the organic products.

products based on fish sludge was taken up in aboveground biomass during the second year after application. Plants utilised on average only $5.7 \pm 3.9\%$ of the residual P in organic products based on animal manure, but the utilisation rate was significantly higher than for products based on fish sludge.

Neither of the two biochars (Biochar-swine manure and Biochar-fish sludge) resulted in significantly higher P uptake than the NoP control treatment and the residual P effect of the biochars was low or not measurable (Fig. 2a, 2b).

Analyses of soil samples taken in 2019 before the second part of the pot experiment (residual P effect) indicated that there was no significant effect of fertiliser treatments on the pH of the model soil. The $\text{pH}_{\text{H}_2\text{O}}$ varied between 6.0 and 6.7 (results not shown). The P-AL level in the soil was low for all fertiliser treatments. The lowest levels were found for the NoP treatment and pots receiving Digestate-food waste/manure ($11 \text{ mg P-AL kg}^{-1}$ soil), significantly lower than in pots receiving Fish sludge 5 ($17 \text{ mg P-AL kg}^{-1}$ soil). Apart from that, no significant effects of the fertiliser treatments on P-AL in the soil were detected (results not shown).

3.3. Field experiment

In 2018, the field experiment resulted in very low yields due to the unusually warm and dry weather in that year and no irrigation. In 2018, average grain yield was $0.9 \pm 0.2 \text{ t ha}^{-1}$ and average P uptake $2.9 \pm 0.6 \text{ kg ha}^{-1}$, with no significant differences between the fertiliser treatments in terms of effect on grain yield or P uptake. In 2019, grain yields were higher, on average $4.3 \pm 0.2 \text{ t ha}^{-1}$. The average P uptake in grain was $18 \pm 1.6 \text{ kg ha}^{-1}$, but again there were no significant differences between the fertiliser treatments.

There were no clear differences between the fertiliser treatments in their ability to release P early during the growing season in 2018 or 2019. Only Fish sludge 1 at Zadoks 37 in 2019 gave significantly higher P concentration in aboveground biomass ($5.5 \text{ g P kg}^{-1} \text{ DM}$) than the unfertilised NoP treatment and Biochar-fish sludge ($4.5 \text{ g P kg}^{-1} \text{ DM}$), but this difference had levelled out by Zadoks 45 (average P concentration for all treatments $3.6 \pm 0.4 \text{ g P kg}^{-1} \text{ DM}$).

In autumn 2019, after two years of P application, P-AL was significantly higher in soil samples from the MinP plots ($71 \text{ mg P-AL kg}^{-1}$ soil) than in soil samples from the NoP plots ($44 \text{ mg P-AL kg}^{-1}$ soil). None of the organic products resulted in a significant increase in P-AL in soil compared with the NoP treatment. In 2018, when the field experiment was established, there were no systematic differences in soil P-AL values between the plots (results not shown).

Fig. 3a shows the relationship between P balance (kg P ha^{-1}) and change in P-AL (converted to kg P ha^{-1}) as an effect of the different fertiliser treatments during two consecutive seasons (spring 2018 to

autumn 2019). Fertiliser treatments with data points below the 1:1 line resulted in a smaller increase in P-AL than indicated by the P surplus. This means that part of the P surplus was bound so strongly to the soil that it was not released in the P-AL analysis. This was the case for all the organic products. Fig. 3b shows the relationship between P balance (kg P ha^{-1}) and change in easily releasable $\text{CaCl}_2\text{-P}$ (converted to kg P ha^{-1}) as an effect of the different fertiliser treatments during two consecutive seasons (spring 2018 to autumn 2019). As can be seen, P in the organic products was less soluble in soil than MinP.

4. Discussion

4.1. P quality in fish sludge

The results showed that if fish sludge is to be transformed from a waste into a valuable P fertiliser product, great effort will have to be devoted to optimising the P fertilisation effects of the sludge. The immediate plant availability of P in products based on fish sludge was significantly lower than in products based on animal manure (Fig. 2a), and low immediate P effects were not compensated for by higher residual P effects (Fig. 2b).

Sub-optimal fertilisation effects of products based on fish sludge during the pot experiment were in agreement with the results of the sequential P fractionation. Most of the P in products based on fish sludge was not solubilised until extraction with HCl (Fig. 1), which is commonly associated with the presence of slowly soluble calcium phosphates, and there was a negative relationship between the HCl-soluble P fraction and RAE in the first year of the pot experiment, when all 14 organic products were included (Fig. 4b). The results presented here are in good agreement with those in an earlier study in which X-ray powder diffraction analysis revealed that a dried, freshwater-based fish sludge sample contained P in the form of chlor-fluor apatite, findings confirmed by solid-state ^{31}P MAS-NMR (Brod et al., 2015).

The content and quality of P in fish sludge will be affected by the two substrates it contains, i.e. feed residues and fish faeces. Fish sludge from salmon hatcheries contains on average 50% feed residues, but the variation is high (Aas et al., 2016). It is unknown whether increasing fish size (i.e. post-smolt and salmon) affects the ratio of feed residues in fish sludge and the quality of P contained therein. In the products included in this study, 75–89% of total P in fish sludge was in the form of inorganic P except in Fish sludge 5, to which wood waste was added during treatment and in which almost all P was inorganic (98%). The ratio of organic P in fish sludge can be expected to increase with increasing ratio of plant-based ingredients (e.g. soya) in the feed residues, but this remains to be explored.

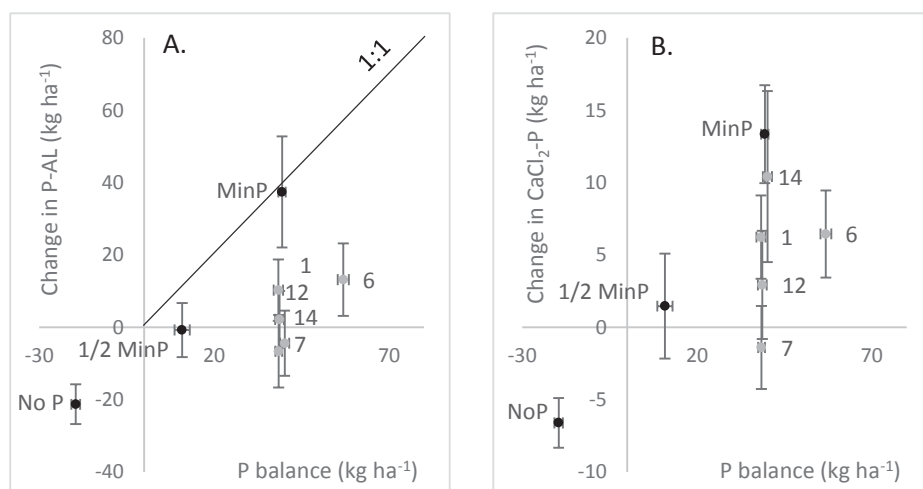


Fig. 3. Relationship between phosphorus (P) balance (kg P ha^{-1} ; $n = 3$ and $n = 2$ for NoP and Cattle manure 1a) and change in A) P-AL and B) $\text{CaCl}_2\text{-P}$ (kg P ha^{-1} ; $n = 3$) as an effect of the different fertiliser treatments during two consecutive seasons at Øsaker (spring 2018 to autumn 2019). 1 = Fish sludge 1; 6 = Cattle manure 1a; 7 = Cattle manure 1b; 12 = Digestate-food waste/manure; 14 = Biochar-fish sludge. Product 6 was unintentionally applied at a higher rate than the other organic products.

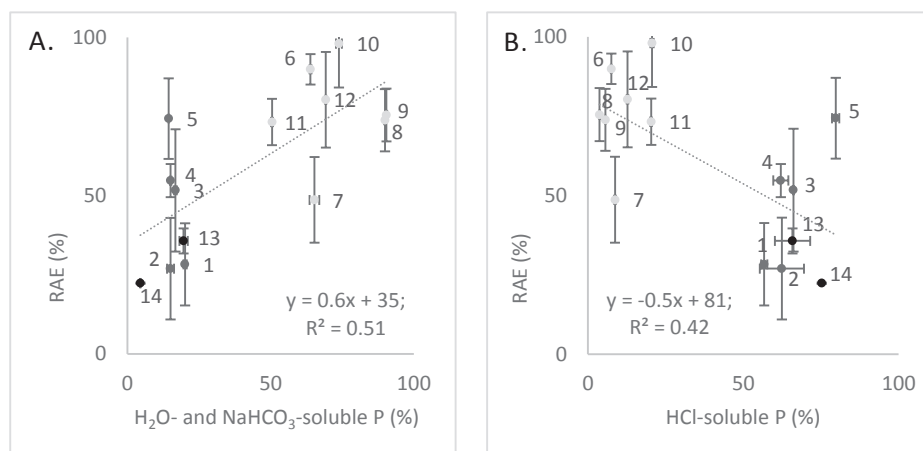


Fig. 4. Relationship between A) H₂O- and 0.5 M NaHCO₃-soluble phosphorus (P) (n = 2) and B) HCl-soluble P (n = 2) and relative agronomic efficiency (RAE, %) of organic products in the pot experiment (n = 3). 1 = Fish sludge 1; 2 = Fish sludge 2; 3 = Fish sludge 3; 4 = Fish sludge 4; 5 = Fish sludge 5; 6 = Cattle manure 1a; 7 = Cattle manure 1b; 8 = Cattle manure 2; 9 = Swine manure 1; 10 = Swine manure 2; 11 = Horse manure; 12 = Digestate-food waste/manure; 13 = Biochar-swine manure; 14 = Biochar-fish sludge.

4.2. P quality in manure solids

The results confirmed that mechanical separation of cattle or swine manure, with or without anaerobic digestion before separation, can facilitate resource-efficient distribution of P in manure, without negatively affecting the good P fertiliser effect of the untreated manure.

In the first year of the pot experiment, all but one of the manure solid products tested resulted in equally high P uptake in aboveground biomass as MinP, which is in agreement with the results of the sequential P fractionation. The majority of total P in manure solids was soluble in H₂O and 0.5 M NaHCO₃ (Fig. 1). There was therefore a positive relationship between the sum of H₂O-soluble and 0.5 M NaHCO₃-soluble P and RAE in the first year of the pot experiment, when all 14 organic products were included (Fig. 4a).

These results confirm findings in previous studies on the P solubility of various cattle or swine manure solids in soil incubations and/or growth experiments, all reporting similar P availability to the respective mineral P fertiliser reference (e.g. Kuligowski et al., 2010; Gasser et al., 2012; Christel et al., 2016). An exception was Cattle manure 1b, a granular, dry product, which showed quite low RAE (49%) in the first year of the pot experiment, despite having a similar fraction of easily soluble P as the other manure products. This may be explained by the granular form retarding solubilisation in soil.

4.3. P quality in biochar

Our results are in agreement with previous findings showing that pyrolysis of manure reduces P availability in the biochar compared with the feedstock (reviewed in Kratz et al., 2019). In the pot experiment, neither of the two biochars significantly increased P uptake in aboveground biomass compared with the unfertilised NoP treatment (Fig. 2a). The sequential P fractionation indicated that this was due to pyrolysis transforming the P in fish sludge or manure into complex P minerals, e.g. stable calcium phosphates (Fig. 1). Low plant availability of P in the biochars was not compensated for by higher residual P effects during the second year of the pot experiment (Fig. 2b), even though biochar is often described as a slow-release P fertiliser (Glaser and Lehr, 2019).

Both biochars used in our study were produced at around 650 °C. Previous studies indicate that P-solubility in the biochars could have been higher if fish sludge and manure had been pyrolysed at lower process temperatures. Huang et al. (2018) observed crystallisation of Ca phosphate minerals at pyrolysis temperatures > 450 °C when different manures were used as feedstock, while Bruun et al. (2017) found significant formation of hydroxyapatites when manure-dominant biogas digestate was pyrolysed at temperatures > 600 °C. However, Bruun et al. (2017) also clearly demonstrated decreased immediate P availability even in biochar produced at 300 °C compared with the feedstock.

Biochar application to agricultural land has been promoted as an

effective way of storing stable C in soils and reducing climate gas emissions to the atmosphere, while at the same time improving soil quality (Lehmann et al., 2006). Based on the results obtained here, we concluded that P-rich substrates in general are not suited for production of biochar, and that fish sludge and manure should be processed in other ways that contribute to effectively closing global P cycles.

4.4. Practical implications

The solubility of calcium phosphates in organic fertilisers is dependent on soil pH, with lower pH enhancing solubilisation of calcium phosphates and higher pH retarding solubilisation (Brod et al., 2015). Our growth experiments were performed under slightly acid conditions and the fish sludge contained slowly soluble calcium phosphates, so soil pH can be expected to have influenced the P fertilisation effects.

In all products tested except the liquid Digestate-food waste/manure, the N concentration relative to P was too low to meet the requirements of barley or wheat (NIBIO, 2021b). During dewatering, ammonium (NH₄⁺) and/or nitrate (NO₃⁻) will follow the liquid phase and NH₄⁺ may volatilise to the atmosphere as NH₃, especially at high pH (e.g. manure) or under high temperature conditions (e.g. drying of fish sludge). All products based on fish sludge also lacked K in relation to N and crop demands, since K⁺ mainly occurs in soluble form and therefore will also accompany the liquid phase during mechanical dewatering. Poorly balanced products will have to be optimised to allow for efficient nutrient recycling. Teuber et al. (2005), too, suggested that sea salmon sludge can be applied as fertiliser to land, but should be complemented with additional N and K.

In practice, nutrient imbalances can be counteracted by combining organic materials with mineral fertiliser components, either as separate additions or by enriching the organic products with mineral nutrient components. Combining the organic products tested in this study with mineral nutrient sources would result in organo-mineral fertiliser products complying with EU fertiliser regulations (European Union, 2019/1009), and would also solve the issue of too low nutrient concentrations for seven of the products to classify as organic fertiliser products in the European Union.

5. Conclusions

This study sought to contribute to closing global P cycles by determining the P fertiliser effects of dewatered fish sludge (feed residues and faeces of farmed fish) and manure solids. Growth experiments and chemical P characterisations indicated that the P fertilisation effects of fish sludge must be optimised if fish sludge is to be transformed from a waste into a valuable P fertiliser product. The results confirmed that mechanical separation of cattle and swine manure can facilitate resource-efficient distribution of P from livestock-intensive areas to arable areas where P input is needed. Sequential chemical fractionation

supported and explained the differences seen in P fertilisation effects. Imbalanced nutrient ratios in fish sludge and manure solids should be counteracted by combined application with other sources of nitrogen and potassium, if necessary, to allow for efficient nutrient recycling. The results indicated that pyrolysis should not be the preferred treatment method for P-rich fish sludge or manure, due to the formation of complex and slowly soluble P compounds.

CRedit authorship contribution statement

Eva Brod: Conceptualization, Methodology, Formal analysis, Investigation, Data curation, Writing – original draft, Visualization, Funding acquisition. **Anne Falk Øgaard:** Conceptualization, Methodology, Investigation, Writing – review & editing, Project administration, Funding acquisition.

Declaration of Competing Interest

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

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.wasman.2021.08.041>.

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