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Quality of fish sludge as fertiliser to spring cereals: Nitrogen effects and environmental pollutants

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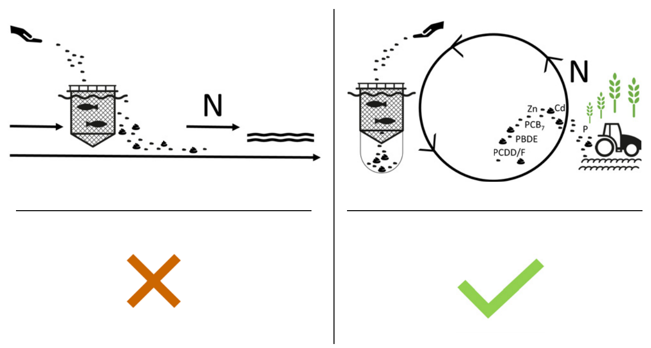
HIGHLIGHTS

- Fertiliser quality of fish sludge (i.e. feed residues and faeces) is largely unknown.
- Nutrient composition in fish sludge is unbalanced relative to crop requirements.
- Six fish sludge products showed variable N effects in a 2-year field experiment.
- Soil incubation can give an indication of N quality in fish sludge products.
- PCB_7, $PBDE_7$ and PCDD/F + DL-PCB in fish sludge are reported for the first time.

GRAPHICAL ABSTRACT



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ABSTRACT

The aim of this study was to contribute to development of organic fertiliser products based on fish sludge (i.e. feed residues and faeces) from farmed smolt. Four dried fish sludge products, one liquid digestate after anaerobic digestion and one dried digestate were collected at Norwegian smolt hatcheries in 2019 and 2020. Their quality as fertilisers was studied by chemical analyses, two 2-year field experiments with spring cereals and soil incubation combined with a first-order kinetics N release model. Cadmium (Cd) and zinc (Zn) concentrations were below European Union maximum limits for organic fertilisers in all products except one (liquid digestate). Relevant organic pollutants (PCB_7, $PBDE_7$, PCDD/F + DL-PCB) were analysed for the first time and detected in all fish sludge products. Nutrient composition was unbalanced, with low nitrogen/phosphorus (N/P) ratio and low potassium (K) content relative to crop requirements. Nitrogen concentration in the dried fish sludge products varied (27–70 g N kg^{-1} dry matter), even when treated by the same technology but sampled at different locations and/or times. In the dried fish sludge products, N was mainly present as recalcitrant organic N, resulting in lower grain yield than with mineral N fertiliser. Digestate showed equally good N fertilisation effect as mineral N fertiliser, but drying reduced N quality. Soil incubation in combination with modelling is a relatively cheap tool that can give a good indication of N quality in fish sludge products with unknown fertilisation effects. Carbon/N ratio in dried fish sludge can also be used as an indicator of N quality.

1. Introduction

Bioeconomy can only succeed in practice if organic waste residues are utilised efficiently in biological cycles. With growing global demand for farmed fish, organic waste from aquaculture production is becoming an increasing challenge, including in Norway, the world's largest producer of farmed Atlantic salmon (*Salmo salar*) (FAO, 2022). In 2019, 66.000 ton

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(t) nitrogen (N) were lost to the sea with farmed fish excrement and feed residues (i.e. fish sludge) from Norwegian aquaculture systems (Broch and Ellingsen, 2020). In comparison, Norwegian farmers apply 106.000 t N annually in the form of mineral fertiliser (Norwegian Food Safety Authority, 2021). Fish sludge is currently mainly collected by land-based systems producing smolt, young salmon being adapted to freshwater before acquisition of seawater tolerance. Land-based systems include both recirculation aquaculture systems (RAS) and flow-through systems cleaning effluent water before discharge. Increasing amounts of fish sludge are expected to be collected with the ongoing trend for moving post-smolt production and salmon farms from open fish farm systems in the sea to land and with (semi-)closed production systems in the sea, both in Norway and internationally. Aquaculture can only expand sustainably if fish sludge is transformed from a waste problem into a valuable resource.

Despite fish sludge containing high concentrations of valuable plant nutrients, including N and phosphorus (P), little attention has been devoted to optimising treatment processes towards efficient reuse, e.g. as high-quality bio-based fertiliser products. To date, treatment technologies for fish sludge have mainly been designed with the aims of lowering costs and reducing odour. Fish sludge de-watering and treatment occurs in several successive steps where the water first passes through a drum filter, followed by de-watering by belt filters or through sedimentation. In some cases, chemical polymers are added to collect the sludge in larger particles before further de-watering by a centrifuge or screw press to around 30 % dry matter (DM). In most cases, fish sludge is heat-dried to around 90 % DM. Alternatively, fish sludge can be utilised as a co-substrate in aerobic digestion, with biogas and anaerobic digestate as final products (Rosten et al., 2013).

The fertiliser effect of products based on fish sludge is still largely unknown, because only a few studies have examined its quality. 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 smolt sludge can have relative agronomic efficiency (RAE) of 50–80 % compared with mineral fertiliser when applied as N fertiliser. Lenz et al. (2021) reported significant growth of lettuce following application of sludge from an aquaponic system rearing tilapia (*Oreochromis niloticus*) as fertiliser to soil. Teuber et al. (2005) and Celis et al. (2008) observed N fertiliser effects of salmon sludge when applied at increasing rates to ryegrass (*Lolium multiflorum* L.) in Chile. Systematic investigations of the N fertilisation effects of fish sludge subjected to various treatment methods have not been conducted previously.

Another challenge to efficiently closing blue-green nutrient cycles is lack of knowledge about the presence and importance of undesirable components, including heavy metals and organic pollutants, in fish sludge. This is also the reason given by the European Union (EU) for why fish sludge is currently not included in the list of organic materials that can be used for production of CE-marked fertiliser products (Annex II in European Union 2019/1009). According to Brod et al. (2017), in Norway use of land-based salmon sludge as fertiliser is limited by elevated zinc (Zn) and/or cadmium (Cd) concentrations. Similar and/or higher Zn and Cd concentrations have been reported for Chilean land-based salmon sludge (Madariaga and Marín, 2017; Salazar and Saldana, 2007). Zinc is added to fish feed as an essential element in salmon nutrition (Silva et al., 2019), while Cd is an undesired and toxic metal accompanying marine feed ingredients, particularly fish meal and fish oil from pelagic fisheries, where global environmental pollutants accumulate. Pollutants such as polychlorinated dibenzo para dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs)/dioxin-like polychlorinated biphenyls (DL-PCBs), non-dioxin like PCBs (PCB_7) and polybrominated diphenyl ether (PBDEs) have received much attention from a food safety perspective, but to our knowledge their occurrence in fish sludge has not been studied previously.

The aim of this study was thus to investigate the quality of fish sludge as N fertiliser to spring cereals. The fertiliser quality of six fish sludge products subjected to various treatment methods was assessed based on (1) the N fertiliser effect, studied in field experiments and in a soil incubation experiment combined with a first-order kinetics N release model and (2) presence of environmental pollutants, including heavy metals and organic pollutants.

Note that in this paper the term *fish sludge* refers to a 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. Fish sludge products

2.1.1. Fish sludge sampling

Six fish sludge products originating from five land-based RAS or flow-through systems for smolt were used in field experiments conducted in 2019 and 2020. These comprised four dried fish sludge products (Fish sludge 1, Fish sludge 2, Fish sludge 3, Fish sludge 4), a liquid digestate after anaerobic digestion (Digestate) and a dried digestate (Dried digestate). In 2020, the dried digestate was a mixture of digestate and undigested fish sludge (Dried digestate + fish sludge). The treatment processes for all fish sludge products are described in detail in Table S1 in Supplementary material (SM), while Fig. S1 in SM indicates the locations where the fish sludge products were collected. All fish sludge products were collected in February/March, in both 2019 and 2020. In addition, four previously collected fish sludge products were analysed but not included in the field experiment. These were: Fish sludge 2-b and 2-c, originating from the same hatchery as Fish sludge 2, and Fish sludge 4-b and 4-c, treated by the same method as Fish sludge 4, but obtained from a different hatchery.

2.1.2. Characterisation

Dry matter content was determined by drying at 105 °C and organic matter content after incineration at 550 °C. The pH was determined in deionised H_2O in a solid:solution ratio of 1:10, and directly in the liquid product Digestate. Total N was determined by the modified Kjeldahl method (EN 13654–1:2001, 2001), and ammonium (NH_4-N) and nitrate (NO_3-N) after extraction in 2 M KCl or directly in the liquid product Digestate by spectroscopy (FIA). Phosphorus and all other nutrients and heavy metals were determined by ICP-OES or ICP-MS after digestion in concentrated nitric acid in a microwave oven (EN ISO 11885:2009, 2009). Chloride (Cl) concentration was measured by ion chromatography. Carbon (C) concentration in the fish sludge products was determined by the Dumas method (EN 13654–2:2001, 2001) using a CN analyser, after drying at 40 °C or freeze-drying (Digestate 2019) and milling.

2.1.3. Organic pollutants

Analyses of organic pollutants were performed at the laboratories of the Institute of Marine Research, Norway, using methods accredited under ISO 17025:2005. The fish sludge samples were analysed for 17 PCDD/F (2,3,7,8-TCDD; 1,2,3,7,8-PeCDD; 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDD; 1,2,3,7,8,9-HxCDD; 1,2,3,4,6,7,8-HpCDD; OCDD; 2,3,7,8-TCDF; 1,2,3,7,8-PeCDF; 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDF; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8,9-HxCDF; 2,3,4,6,7,8-HxCDF; 1,2,3,4,6,7,8-HpCDF; 1,2,3,4,7,8,9-HpCDF, and OCDF) and DL-PCB congeners (non-ortho PCBs 77, 81, 126 and 169, mono-ortho PCBs; 105, 114, 118, 123, 156, 157, 167, and 189), which have been assigned a WHO-TEF 2005 and are included in the current EU maximum limit for food (Van den Berg et al., 2006), seven indicator PCBs (PCB_7; PCB-28, -52, -101, -118, -138, -153, -180) and seven indicator congeners of PBDE ($PBDE_7$; PBDE-28, -47, -99, -100, -153, -154, -183), which the European Food Safety Authority has identified as being of primary interest (EFSA Journal, 2011). PBDE-209 analysis was only available for fish sludge sampled in 2020. All analyses were performed as described in Moxness Reksten et al. (2020). Concentrations below the limit of quantification (LOQ) were not included in the sum of PCDD/F + DL-PCB, PCB_7 and $PBDE_7$ (lower bound (LB) LOQ estimation). In 2019, the sampled amount of Digestate was too small for analysis of organic pollutants.

2.1.4. Feed samples

Feed samples were collected from all hatcheries at the same time as fish sludge samples in both 2019 and 2020. The feed samples were crushed in a mortar by hand before analysis of C and N using a CN analyser. In 2019, the feed samples were analysed for heavy metals as described, while in 2020 the feed samples were analysed for the same organic pollutants as the fish sludge products.

2.2. Field experiments

Two 2-year field experiments were established in south-eastern Norway in 2019 to study the effect of the six fish sludge products as N fertiliser (Table S2 in SM). One experiment was established at Øsaker (59°32′N; 11°04′E), and one at Rakkestad (59°27′N; 11°21′E) (Fig. S1). Field experiments and treatments were placed on exactly the same spots during both years (Fig. S2 in SM), and the results for 2020 therefore show the combined same-year and residual effect of fertiliser treatments applied in 2019. The soils at Øsaker and Rakkestad are both silty clay loams classified as Stagnosols (NIBIO, 2022). Additional information on the soils, including selected chemical properties, is provided in Table S3 in SM.

Normal annual sum of precipitation (mean for 1991–2020) is 960 mm at (Yr Øsaker, 2021) and 920 mm at (Yr Rakkestad, 2021), half of which occurs during the period May–September. Normal mean monthly air temperature in the growing season is 11–17 °C at Øsaker and 10–16 °C at Rakkestad for the single months in the period May–September. In 2019, April was warmer than normal mean at both Øsaker and Rakkestad, while the summer was characterised by relatively abundant rain except in July. In 2020, April and June were warmer than normal at both sites, whereas May was colder and July was exceptionally cold and wet at both sites.

The field experiments had a randomised block design with three replicates (Fig. S2). Each plot measured 3.0 m x 8.0 m (2.0 m x 8.0 m for Digestate in 2020) and plants were harvested on a 1.5 m x 6.5 m sub-area within each plot. Fish sludge products were applied based on total N concentration, equivalent to 120 kg N ha^{-1}, in both 2019 and 2020. The fertilisation rate for Digestate was corrected for estimated ammonia (NH_3-N) losses during application, equivalent to 15 % of NH_4-N (NIBIO, 2021). Nitrogen effects of the fish sludge treatments were compared with an unfertilised control treatment (No N) and with mineral fertiliser (YaraMila Fullgjødsel® 22-3-10 (NPK)) at a rate of 60 kg N ha^{-1} (½ Min N) and 120 kg N ha^{-1} (Min N). All fertiliser products were applied by hand before harrowing within 2 h after fertiliser application. Fertilisation rates and amounts of total N, NH_4-N and total P applied in each fertiliser treatment and year are presented in Table S2, and the set-up of the experimental design can be found in Fig. S2 in SM.

At Øsaker, cereal was grown in the year before the experiment and the stubble was left standing during the winter. The soil was harrowed twice in spring and then the experiment was established on 8 May 2019, and repeat treatments were applied on 22 April 2020. At Rakkestad, grass was grown in the year before the experiment and was ploughed down before harrowing and field establishment on 15 May 2019. The soil was ploughed again in spring 2020 and repeat treatments were applied on 22 April 2020. Spring cereal was sown within two days after fertiliser application at both sites. At Øsaker, spring wheat (*Triticum aestivum* var. Mirakel) was grown in both 2019 and 2020, while at Rakkestad barley (*Hordeum vulgare* var. Brage) was grown in 2019 and oats (*Avena sativa* var. Haga) in 2020. The field at Øsaker was harvested on 16 September 2019 and 17 September 2020, and the field at Rakkestad on 13 September 2019 and 2 September 2020. Straw residues were left on the plots.

Crude grain yield was recorded at harvest by an experimental combine harvester and grain samples were taken from each plot and dried in cold air (< 20 °C). In 2019, due to a sampling error, one replicate was missing from each of three treatments at both sites (No N, Fish sludge 1, Fish sludge 3). At Øsaker in 2019, one additional replicate was missing from the Min N treatment. Grain DM content was analysed by near-infrared (NIR) (InfraTecTM NOVA) and grain yield was calculated at the standardised water content (15 %). Protein content was determined by NIR (InfraTecTM NOVA) and N concentration in grain was estimated based on protein content divided by 6.25. Nitrogen uptake in grain was calculated for each plot by multiplying yield and N concentration in grain and converted to a per-hectare basis. Hectolitre weight was also determined for whole grain samples. Further, in 2020 grain samples were milled and digested in concentrated nitric acid in a microwave oven for analysis of P, Zn and Cd concentration by ICP-MS. Seven individual grain samples (from treatments Min N, Fish sludge 1, Fish sludge 2, Fish sludge 3, Fish sludge 4, Digestate, Dried digestate + fish sludge; all block 2) were collected from the field at Rakkestad for screening of organic pollutants as described for the fish sludge products.

Soil samples were taken at 20 cm depth before fertiliser application in spring 2019 in plots of three randomly selected fertiliser treatments (No N, ½ Min N and Fish sludge 2) at both sites. After harvest in 2020, soil samples were taken in all plots at both sites. Soil samples were air-dried or dried at 40 °C and sieved (<2 mm), and pH was analysed in deionised H_2O in a solid:solution ratio of 1:2.5 (*v*/v). Extractable P (P-AL) was analysed by ICP-OES, after extraction of 1 g dried soil in 20 mL solution with 0.1 M ammonium lactate and 0.4 M acetic acid adjusted to pH 3.75 according to Egnér et al. (1960). Three individual soil samples (fertiliser treatments Min N, Fish sludge 2 and Digestate; all block 2) were selected from the field at Rakkestad for screening of organic pollutants as described for the fish sludge products.

2.3. Incubation experiment and nitrogen release model

Soil-fertiliser incubations were conducted over a full growing season to study N release from samples of the fish sludge products used in the field experiment in 2020. Fish sludge 2-c, sampled some weeks earlier from the same smolt hatchery as Fish sludge 2, was also included in the incubations. An unfertilised control incubation (No N) was included as reference.

Topsoil (0–20 cm) taken from a sandy loam classified as an Endostagnic Cambisol at Apelsvoll (60°42′N, 10°51′E) (NIBIO, 2022) was used for the incubations. This soil was chosen based on prior experience and its ability to allow rapid degradation of organic additives (Henriksen and Breland, 2002). Additional information on the incubation soil, including selected chemical properties, is given in Table S3.

Prior to the incubations, fish sludge samples equivalent to 320 kg N ha^{-1} were mixed with the soil thoroughly by hand for 1 min, and then subportions of 20 g DM soil were transferred to 100-mL glass jars. There were in total 21 sub-portions per treatment, allowing three replicates for seven sampling dates (day 0, 2, 5, 10, 20, 40 and 80). The sub-portions were watered to 60 % of water holding capacity (WHC, 100 % WHC = 460 g H_2O kg^{-1}) and kept in a climate chamber at 15 °C with a lid loosely placed on top to keep the environment aerobic. The samples removed on the different sampling dates were subjected to extraction with 80 mL 2 M KCl for 1 h. The extracts were filtered (Whatman filters 589/3, pore size 2 μm) and kept in a freezer before analysis of NH_4-N and NO_3-N using a Konelab Aqua 60 analyser. Mineral N content in the No N control treatment was subtracted from the sample value at all time points (11.5, 8.3, 11.8, 10.3, 15.1 and 22.5 mg N kg^{-1} soil at day 0, 2, 5, 10, 20, 40 and 80, respectively).

Fractionation of organic N in fish sludge products into quickly and slowly plant-available pools was determined by adjusting the N-release model developed by Henriksen et al. (2019) to the incubation data by minimising the sum of squared residuals. The model is based on the assumption that organically bound N in samples can be divided into two pools, and that N release will follow first-order kinetics, with rate constant $k_1 = 0.15$ day^{-1} for the quickly available organic N and $k_2 = 0.0008$ day^{-1} for the slowly available organic N (rate constants taken from Henriksen and Breland (1999)).

Nitrogen release from Fish sludge 4-c has been studied previously, in an incubation experiment described by Henriksen et al. (2019), and those data were also used in the model.

2.4. Data analysis

Relative agronomic efficiency (RAE) in the field of the fish sludge products was calculated based on grain yield:

$$\mathrm{RAE} = 100 \times \frac{X_1}{\mathrm{N\ applied}}$$

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

where N applied is amount of N applied with fish sludge product (kg N ha^{-1}), Y_1 is grain yield (ton ha^{-1}) as an effect of fish sludge product, X_1 is amount of Min N (kg N ha^{-1}) resulting in equally high grain yield as application of fish sludge product at 120 kg N ha^{-1}, and a and b are slope and intercept obtained from linear regression with Y = grain yield (ton ha^{-1}) after Min N application and X = Application rate of Min N (0, 60 and 120 kg N ha^{-1}). The slope and intercept for Rakkestad were: a = 0.02, b = 4.47, R^2 = 0.58 (2019); a = 0.03, b = 4.59, R^2 = 0.67 (2020). At Øsaker, crop growth was uneven and grain yields low, and therefore RAE measured at Øsaker are not reported.

For the field experiments, P balance was calculated as:

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

where P applied is P amount applied with mineral fertiliser or fish sludge product in 2019 and 2020 and P removed is P removed with the harvested grain in 2019 and 2020.

Analysis of variance (ANOVA) with replicates as block was applied to study the effect of the fertiliser treatments on grain yield, RAE, protein content and hectolitre weight. The data were checked for normal distribution (normal quantile plots) and homogeneity of variance (residual vs fitted plots). To perform multiple comparisons, Tukey's multiple comparison test was used (α = 0.05). Relationships between single parameters were studied by simple linear regression. *t*-tests (α = 0.05) were conducted to compare particular groups of samples. JMP® Pro 15.2.0 and Minitab Statistical Software 20.2.0.0 were used for statistical analysis.

3. Results

3.1. Nutrient composition in fish sludge products

Nitrogen concentration in the dried fish sludge products varied between 27 and 70 g N kg^{-1} DM (Table 1). There was wide variation in total N concentration even for dried fish sludge products treated by the same technology, e.g. Fish sludge 4-b and 4-c (Flatanger hatchery) had significantly higher N concentrations than Fish sludge 4 (Sisomar hatchery). Nitrogen concentration varied also between fish sludge products sampled at the same hatchery at various time points, e.g. Fish sludge 2 and 2-b (70 and 67 g N kg^{-1} DM, respectively) and Fish sludge 2-c (42 g N kg^{-1} DM). The C/N ratio in dried fish sludge products varied between 6.0 and 15.5, with significantly higher C/N ratio in Fish sludge 4 than in the other dried fish sludge products according to *t*-tests. The C/N ratio in feed samples was lower (5.9–7.2, results not shown) and there was no significant relationship between C/N ratio in fish sludge and that in respective feed samples. The ratio of NH_4-N to total N was low (0–8 %) in all dried fish sludge products. In Digestate 63 and 76 % of total N was present as NH_4-N, whereas in Dried digestate (+ fish sludge) only 7 and 8 % of total N was present as NH_4-N. The P concentration in the dried fish sludge products varied between 15 and 40 g P kg^{-1} DM. The N/P ratio was low in all dried fish sludge products, between 0.8 and 3.8, whereas the N/P ratio in Digestate was 13 in 2019 and 19 in 2020. The K concentration was very low in all fish sludge products (0.2–3.5 g K kg^{-1} DM in the dried fish sludge products, 0.02–0.03 kg ton^{-1} in Digestate).

3.2. Nitrogen effects in field experiments

At Øsaker, crop growth was uneven and grain yields were low, especially in 2020 (0.4–1.8 ton ha^{-1}) (Fig. S3 in SM). Plants did not respond to increasing rate of mineral fertiliser above 60 kg N ha^{-1} (½ Min N), in 2019 or 2020. According to a *t*-test, the six fish sludge products had on average a small positive effect on yield compared with the unfertilised control (No N), in both 2019 and 2020. In 2020, Fish sludge 1, 2 and 3 and Dried digestate + fish sludge significantly increased grain yield compared with No N. Thus the very low yields at Øsaker indicate that plant growth was limited by factors other than N fertilisation, e.g. poor soil structure. In the following, therefore, only N effects observed at Rakkestad are reported.

The field experiment at Rakkestad showed that fish sludge can have equally good effect as mineral N fertiliser, but the N availability in the fish sludge varied considerably between the products (Fig. 1). In 2019, when barley was grown at Rakkestad, the effect of Fish sludge 3 and Digestate on grain yield was not significantly different from No N or Min N. Fish sludge 1, 2 and 4 had no significant effect on grain yield compared with No N. The protein content in grain varied between 10.7 and 13.2 %, with the lowest content in unfertilised grain (No N) and the highest in grain fertilised with Min N (Table S4 in SM). None of the fish sludge products significantly increased protein content in grain compared with the unfertilised No N treatment. Digestate resulted in significantly increased hectolitre weight compared with the unfertilised No N treatment (Table S4). In 2020, when oats were grown and grain yields showed the combined residual effect of fertiliser applied in 2019 and same-year effect of fertiliser applied in 2020, both Digestate and Dried digestate + fish sludge resulted in equally high grain yield as Min N. The effect of Fish sludge 1, 2 and 3 was comparable to that of ½ Min N. Fish sludge 4 showed

Table 1
Selected characteristics of the fish sludge products used in the field and incubation experiment and in four additional fish sludge products (Fish sludge 2-b, 2-c, 4-b, 4-c). DM = dry matter.

Product	Sampling year	Dry matter	pH	Organic matter	Carbon	Total N	C/N	NH_4-N	P	K	S	Ca	Cl	Na
		%		% of DM	g kg^{-1} DM	g kg^{-1} DM		% of total N	g kg^{-1} DM					
Fish sludge 1	2019	91	6.0	81	461	56	8.5	0.6	20	0.8	4.1	39	1.4	2.7
	2020	92	6.3	77	423	50	8.8	0	40	1.0	4.2	71	0.8	3.0
Fish sludge 2	2019	98	5.4	75	451	58	7.7	3.3	28	1.0	4.9	63	0.9	1.7
	2020	95	5.2	79	468	70	6.8	6.4	32	0.7	6.3	51	0.9	1.8
Fish sludge 2-b	2018	83	5.3	–	453	67	6.8	5.0	37	1.1	0.1	84	–	2.4
Fish sludge 2-c	2020	89	5.2	77	395	42	9.4	8.3	28	1.0	3.6	62	1.6	1.8
Fish sludge 3	2019	96	6.0	86	489	57	8.4	0.5	15	2.3	3.5	23	3.0	4.1
	2020	91	5.5	86	492	58	8.8	0.4	26	3.5	5.4	36	5.0	7.2
Fish sludge 4	2019	92	–	80	423	35	12.2	3.0	17	0.5	2.1	33	2.2	2.0
	2020	91	6.4	80	411	27	15.5	0	36	0.9	3.1	62	2.3	4.3
Fish sludge 4-b	2018	95	5.8	82		63		0.5	26	–	4.0	45	–	–
Fish sludge 4-c	2018	96	5.1	86	496	70	6.9	0.9	19	–	4.2	–	0.1	–
Dried digestate (+ fish sludge)	2019	94	7.5	63	372	58	6.4	7.9	38	0.3	7.3	86	1.4	1.2
	2020	74	7.0	64	349	60	6.0	7.0	30	0.2	3.9	66	0.7	1.0
Product	Sampling year	Dry matter	pH	Organic matter	Carbon	Total N	C/N	NH_4-N	P	K	S	Ca	Cl	Na
		%		% of DM	g kg^{-1} DM	g L^{-1}		% of total N	g L^{-1}				mg L^{-1}	
Digestate	2019	1.8	8.4	70		3.8		63	0.3	0.03	0.09	0.4	400	35
	2020	0.5	7.8	52	371	1.7	4.9	76	0.09	0.02	0.04	0.3	620	25

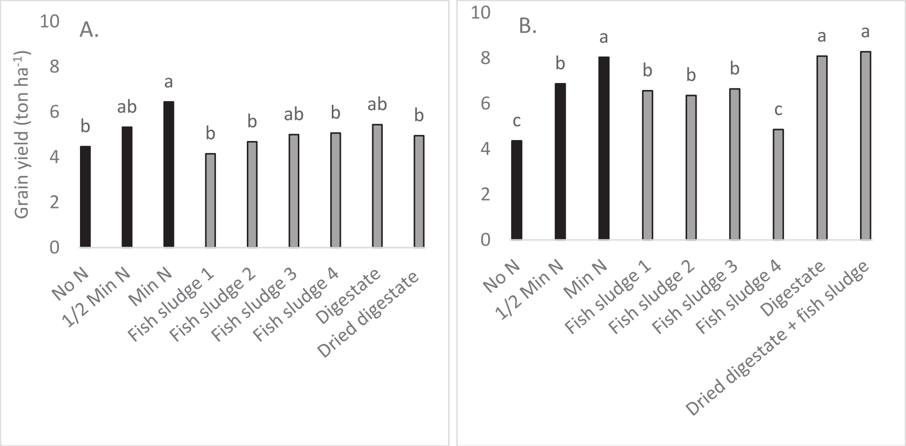

Fig. 1. Grain yield (85 % dry matter; ton ha^{-1}) during the field experiment at Rakkestad as an effect of the different fertiliser treatments in (A) 2019 and (B) 2020. Different letters indicate significant differences between fertiliser treatments (one-way ANOVA with $p < 0.05$, followed by Tukey's *t*-test).

no effect compared with No N and resulted in significantly lower grain yield than the other fish sludge products. In 2020, protein content in grain varied between 9.7 and 11.7 %, with the lowest content in unfertilised grain (No N) and the highest in grain fertilised with Min N and Dried digestate + fish sludge (Table S4). In 2020, there were no significant effects of fertiliser treatment on grain hectolitre weight.

3.3. Nitrogen effects in incubation experiment and distribution in N fractions

The fish sludge products used in the field experiment in 2020 were studied in the incubation experiment. Measured and simulated N release from fish sludge products during incubation is presented in Fig. S4 in SM. Distribution of N fractions (NH_4-N, quickly and slowly available organic N) in the fish sludge products is presented in Fig. 2.

In the Digestate, 76 % of total N was present as NH_4-N (Table 1) and this was recovered on day 0 of the incubation (Fig. S4). Almost half of the organic N in the Digestate was quickly mineralised, while the remaining organic N was not released during the course of the incubation experiment.

For the Dried digestate + fish sludge, only 36 % of total N was estimated to be either directly plant-available as NH_4-N or as quickly available organic N (Fig. 2), even though the product apparently gave equally good effects as Digestate and Min N in the field experiment at Rakkestad in 2020 (Fig. 1).

In Fish sludge 1, 2 and 3, between 22 and 27 % of total N was present as NH_4-N or quickly available organic N. Fish sludge 4 did not contain any N as NH_4-N and, after incubation in soil, 10 % of the total N applied was first immobilised before it was released again at around day 10 (Fig. S4).

3.4. Environmental pollutants in fish sludge products

Cadmium concentration was <1.5 mg kg^{-1} DM in all fish sludge products except Digestate (2020), and Zn concentration was <800 mg kg^{-1} DM (Table 2), meaning that both were below the EU maximum limit for organic fertilisers (European Union 2019/1009). A *t*-test revealed that the Cd and Zn concentrations were higher in fish sludge products sampled in 2019 than in the respective feed samples (Table S5 in SM). Further, there was a positive linear relationship between the concentration of P (g P kg^{-1} DM) and Zn (mg kg^{-1} DM) in the dried fish sludge products ($y = 14.0x + 92.9$; $R^2 = 0.60$; $p < 0.05$), and between P and Cd (mg kg^{-1} DM; $y = 0.02x - 0.001$; $R^2 = 0.35$; $p < 0.05$), indicating poor digestibility of both P and heavy metals.

Mean, minimum and maximum concentrations of PCB_7, $PBDE_7$ and PCDD/F + DL-PCB in undigested fish sludge (Fish sludge 1, 2, 3 and 4) and anaerobically digested fish sludge (Digestate and Dried digestate (+ fish sludge)) are presented in Table 3. The concentrations of these contaminants in digested fish sludge (although single samples) were 2–8 times

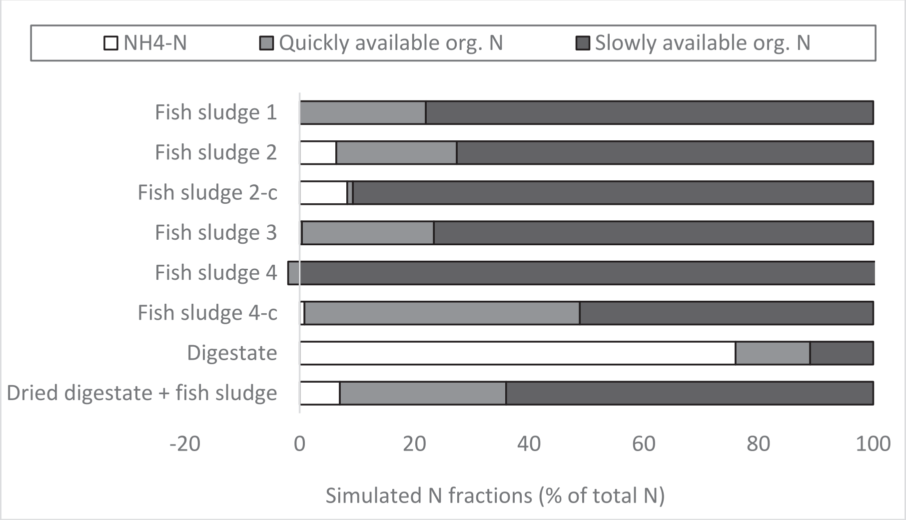

Fig. 2. Distribution of total nitrogen (N) fractions in fish sludge products (2020), determined by analysis (NH_4-N) followed by application of a first-order kinetics N release model to incubation data (quickly and slowly available organic N).

Table 2
Concentrations (mg kg^{-1} dry matter) of cadmium (Cd), zinc (Zn), copper (Cu), chromium (Cr), lead (Pb) and nickel (Ni) in the fish sludge products used in the field experiments and in four additional fish sludge products (Fish sludge 2-b, 2-c, 4-b, 4-c).

Product	Sampling year	Cd	Zn	Cu	Cr	Pb	Ni
Fish sludge 1	2019	0.43	350	16	4.8	0.59	7.7
	2020	0.65	500	25	4.1	0.74	6.7
Fish sludge 2	2019	0.74	510	21	8	0.80	4.6
	2020	0.55	540	17	4.5	< 0.5	5.6
Fish sludge 2-b	2018	0.98	560	26	16	2.4	4.1
Fish sludge 2-c	2020	0.95	490	27	14	< 0.5	11
Fish sludge 3	2019	0.32	340	18	3.1	< 0.5	1.0
	2020	0.34	420	22	2	< 0.5	1.5
Fish sludge 4	2019	0.40	390	13	3.7	< 0.5	1.7
	2020	0.68	740	25	4.1	< 0.5	2.6
Fish sludge 4-b	2018	0.37	270	8.2	3.4	< 0.5	< 0.5
Fish sludge 4-c	2018	0.23	340	14	1.3	0.7	< 1.5
Digestate	2019	0.32	200	9.4	1.8	0.1	4.3
	2020	2.1	696	35	7	0.7	17.2
Dried digestate (+ fish sludge)	2019	0.80	760	18	2.8	< 0.5	7.1
	2020	1.30	570	31	8	0.58	7.1

higher than in the four dried, undigested fish sludge samples. The levels of PCB_7 and $PBDE_7$ were of the same order of magnitude in undigested fish sludge and in feed samples, whereas the levels of PCDD/F + DL-PCB were higher in undigested fish sludge than in feed (*t*-test, $p < 0.05$).

3.5. Environmental pollutants in grain and soil

After two growing seasons, a significant linear relationship was detected between total Zn application with fish sludge products in 2019 and 2020 (kg ha^{-1}) and Zn concentration in grain (g kg DM^{-1}) at Rakkestad ($y = 0.9x + 31$; $R^2 = 0.46$; $p < 0.05$). Already in 2019, Fish sludge 1 and 3 significantly increased Zn concentrations in grain at Rakkestad compared with the ½ Min N treatment, while in 2020, Fish sludge 4, Dried digestate + fish sludge and Min N increased Zn concentrations in grain compared with the ½ Min N treatment (Table S6 in SM). At Øsaker, equivalent effects on Zn concentrations in grain were not detected. Cadmium concentrations in grain were below the detection limit (< 0.2 μg Cd g DM^{-1}) for all fertiliser treatments at Øsaker and Rakkestad in both years (Table S6).

The screening of organic pollutants in individual grain and soil samples showed PCB_7 concentrations in grain of 0.6–0.9 ng g^{-1} DM, which were slightly higher than the concentrations in soil (0.5–0.6 ng g^{-1} DM). There were differences in congener patterns, with higher concentrations of congeners PCB-52 and PCB-101 in grain than soil, and higher concentrations of PCB-28, PCB-138 and PCB-153 in soil than grain (Table S7 in SM, appendix B). $PBDE_7$ concentrations in grain (0–0.05 ng g^{-1} DM) were slightly higher than in soil (0.02 ng g^{-1} DM). PBDE-99 was the only dominant congener in soil, while PBDE-47 was the dominant congener in grain (Table S7). PCDD/F + DL-PCB concentrations in grain (0.006–0.009 TEQ pg g^{-1} DM) were lower than concentrations in soil (0.3–0.4 TEQ pg g^{-1} DM).

Table 3
Mean (standard deviation), minimum and maximum concentration of PCB_7 (PCB-28, -52, -101, -118, -138, -153, -180), $PBDE_7$ (PBDE-28, -47, -99, -100, -153, -154, -183) and PCDD/F + DL-PCB (lower bound, LB) in feed, undigested fish sludge (Fish sludge 1, 2, 3 and 4) and digested fish sludge (Digestate and Dried digestate (+ fish sludge)) sampled in 2019 and 2020. n = number of samples, SD = standard deviation, DM = dry matter. Concentration of each congener is given in Table S7 in Supplementary Material (appendix B).

Sample		PCB_7 (LB)	$PBDE_7$ (LB)	PCDD/F + DL-PCB (LB)
		ng g^{-1} DM	ng g^{-1} DM	TEQ2005 pg g^{-1} DM
Feed ($n = 5$)	Mean (SD)	3.09 (1.32)	0.21 (0.12)	0.25 (0.08)
	Minimum	1.18	0.07	0.14
	Maximum	5.23	0.41	0.34
Undigested fish sludge ($n = 8$)	Mean (SD)	3.12 (1.19)	0.27 (0.09)	0.58 (0.16)
	Minimum	1.74	0.18	0.34
	Maximum	5.71	0.44	0.78
Digested fish sludge ($n = 3$)	Mean (SD)	9.76 (2.65)	0.85 (0.49)	1.92 (0.97)
	Minimum	6.33	0.20	0.58
	Maximum	12.77	1.38	2.87

3.6. Effects of fish sludge products on soil phosphorus and pH

Two years after application, calculated P balances revealed that all dried fish sludge products resulted in large amounts of residual P in the soil at both sites, with between 65 and 197 kg P ha^{-1} at Øsaker and between 41 and 178 kg P ha^{-1} at Rakkestad (results not shown). The surplus was significantly highest for Fish sludge 4 at Øsaker and Rakkestad. The P balance for Digestate was comparable with that for Min N at both sites (3.3 kg P ha^{-1} at Øsaker and -33 kg P ha^{-1} at Rakkestad).

When the field experiment was established in spring 2019, the soil at Øsaker had an optimum content of extractable P (69 mg P-AL kg^{-1} soil; Table S3), indicating a need for P application. After two growing seasons, Fish sludge 4 significantly increased the P-AL level in the soil compared with the Min N treatment to a high P-AL level (Table S8 in SM; NIBIO, 2021). At Rakkestad, the P-AL level in the soil was already high when the field experiment was established, but with some variation between the plots (Table S3). After two growing seasons, the P-AL level was not significantly affected by the fertiliser treatments (Table S8).

Fig. 3 shows the relationship between P balance (kg P ha^{-1}) and change in P-AL (converted to kg P ha^{-1} in upper 20 cm) as an effect of the different fertiliser treatments at Øsaker during two consecutive seasons (spring 2019 to autumn 2020). Fertiliser treatments with data points below the 1:1 line resulted in a smaller increase in P-AL than indicated by the P surplus, meaning that part of the P surplus was bound so strongly that it was not released in the P-AL analysis. This was the case for all fish sludge products except Digestate. At Rakkestad, no such relationship was identified, due to lack of effects of fertiliser treatments on P-AL levels in the soil (Table S8).

There was no significant effect of fertiliser treatments on soil pH at Øsaker or at Rakkestad (Table S8). The significant difference in pH between plots fertilised with Fish sludge 1 and 4 at Rakkestad can probably be explained by soil variation at the site, since pH in the fish sludge products was comparable (Table 1).

4. Discussion

4.1. Nitrogen effects

This study showed that fish sludge has potentially good effects as N fertiliser to spring cereals. However, there was large variation between different fish sludge products in terms of N concentration and quality.

The good effects of liquid Digestate as N fertiliser in the field experiment at Rakkestad (Fig. 1) were in good agreement with most of the N in Digestate being immediately plant-available as NH_4-N (Fig. S4). In practical agriculture, however, the high water content of digestate is a major obstacle, resulting in high economic and environmental costs related to transport. Attempts have therefore been made to overcome this challenge, but the present study showed that energy-intensive dewatering and drying of Digestate caused NH_3-N loss and hence reduced both the content and quality of the remaining N in Dried digestate (+ fish sludge) (Table 1; Fig. 2). We have no explanation for the surprisingly good fertilisation effect of Dried digestate + fish sludge in the field experiment in Rakkestad in 2020 (Fig. 1). Based on these results, other dewatering technologies, e.g. sorption, precipitation etc., should be explored to preserve NH_4-N in the Digestate and exploit its good potential as fertiliser.

The variable effect of the fish sludge products as N fertiliser in the field experiment in 2020 was well reflected by the simulated quickly available N fractions based on the incubation experiment (i.e. NH_4-N and quickly

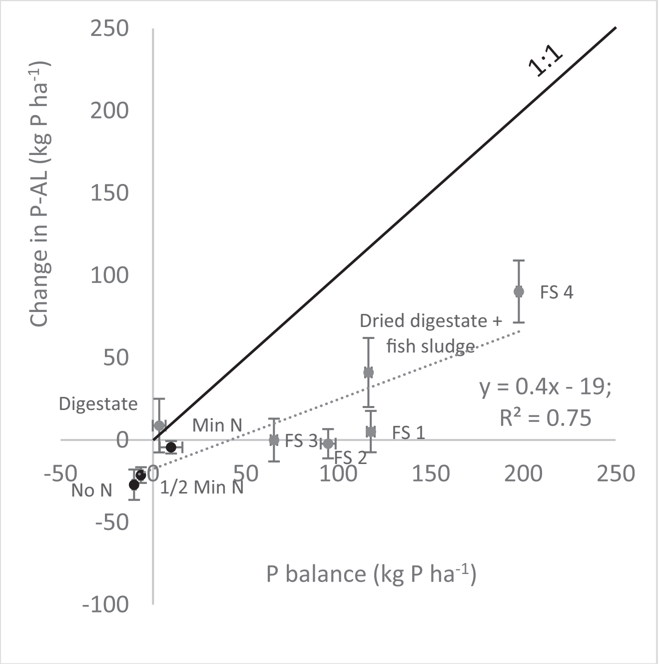

Fig. 3. Relationship between phosphorus (P) balance (kg P ha^{-1}) and change in P-AL (kg ha^{-1}) as an effect of the different fertiliser treatments during two consecutive seasons at Øsaker (spring 2019 to autumn 2020). Black points indicate control treatments (No N, ½ Min N and Min N). FS = fish sludge. Error bars represent standard deviation within each treatment.

available organic N; Fig. 4A). These results suggest that soil incubation experiments in combination with modelling can give a good indication of the N quality of fish sludge products with unknown fertilisation effects.

In the dried fish sludge products including Fish sludge 1, 2, 3 and 4, the NH$_4$-N content was originally low (Table 1), and the largest N fraction was present as slowly available organic N (Fig. 2). In 2020, Fish sludge 1, 2 and 3 still resulted in yields comparable with ½ Min N in the field experiment at Rakkestad (Fig. 1). However, Fish sludge 4 did not increase grain yields compared with the unfertilised control treatment (No N) in either year. In accordance, in the incubation experiment, Fish sludge 4 resulted initially in immobilisation of N (Fig. S4).

Nitrogen concentration and quality varied not only between fish sludge products treated by various technologies, but also between products treated by the same technology, sampled at various locations (Fish sludge 4 and 4-b/4-c) and/or time points (Fish sludge 2/2-b and 2-c). Similarly, Madariaga and Marín (2017) observed great variation in total N concentration, from 28 to 89 g kg^{-1} DM, for 18 fish sludge samples from RAS hatcheries for smolt in Chile. A previous Norwegian study showed that fish sludge from salmon hatcheries contains on average 50 % feed residues in addition to fish excrement, but the variation is high (Aas et al., 2016). Therefore the N fertilisation effect of a fish sludge appears to be determined to a greater degree by its composition than by the treatment technology applied.

We found a negative relationship between C/N ratio in fish sludge products and RAE in the field experiment at Rakkestad in 2020 (Fig. 4B), and a significantly higher C/N ratio in Fish sludge 4 compared with the other dried fish sludge products. This suggests that C/N ratio in dried fish sludge products can function as a simple indicator of its N quality.

4.2. Environmental pollutants

The heavy metal concentrations detected in fish sludge products (Table 2) were at levels previously reported for Norwegian land-based fish sludge samples (Brod et al., 2017; Brod and Øgaard, 2021). However, they were lower than levels reported for 15 Chilean fish sludge samples analysed by Madariaga and Marín (2017) (e.g. 1084 mg Zn kg^{-1} DM and 3.6 mg Cd kg^{-1} DM on average). After two growing seasons, fish sludge application resulted in elevated Zn concentration in grain at the Rakkestad site (Table S6).

Average PCB$_7$ concentration in undigested fish sludge (Table 3) was lower than the median level in 95 Norwegian sewage sludges (11 ng g^{-1} DM; range < 1–66 ng g^{-1} DM) analysed in 2017/2018 by Blytt and Stang (2019), whereas average PCB$_7$ concentration in digested fish sludge (Table 3) was comparable to the median level in Norwegian sewage sludges (Blytt and Stang, 2019). The higher concentrations of persistent organic contaminants in anaerobically digested fish sludge than undigested fish sludge (Table 3) were expected, based on organic matter reduction (Muñoz et al., 2014), as also reported during composting (Needham and Ghos, 2019). Average concentrations of PBDE$_7$ in fish sludge products were considerably lower (Table 3) than median values for total PBDE previously reported for sewage sludge, e.g. 372 ng g^{-1} DM in 95 Norwegian sewage sludges (Blytt and Stang, 2019) and 1814 ng g^{-1} DM in eight Italian sewage sludges (Cincinelli et al., 2012). In sewage sludge, however, PBDE-209 dominates the congener profile, accounting for up to 97.0 % and 99.8 % of total PBDE in the studies by Blytt and Stang (2019) and

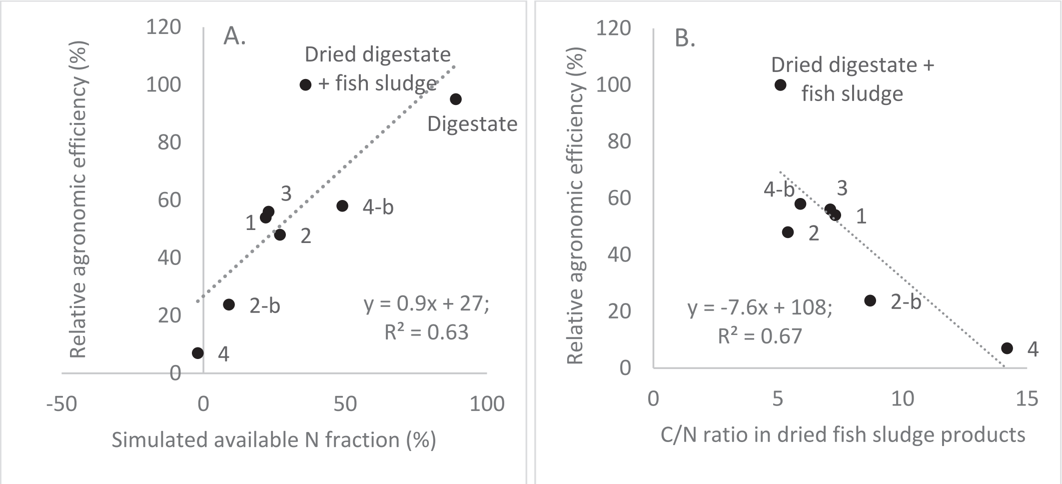

Fig. 4. Relationship between relative agronomic efficiency (RAE %, Table S4) of fish sludge products during the field experiment at Rakkestad in 2020 and (A) Simulated available N (NH$_4$-N and quickly available organic N) based on the incubation experiment and (B) carbon/nitrogen (C/N) ratio in the dried fish sludge products (Digestate not included). RAE % for samples 2-b and 4-b are unpublished data based on a separate field- and a pot experiment with spring cereals, respectively.

Cincinelli et al. (2012), respectively, despite many discharge sources with particularly high PBDE-209 concentrations having been eliminated or reduced (Blytt and Stang, 2019). In comparison, PBDE-209 accounted for between 29 and 70 % of total PBDE analysed in the fish sludge products sampled in 2020 (Table S7). Average concentrations of PCDD/F + DL-PCB in the fish sludge products (Table 3) were lower than the median concentration in 16 Belgian sewage sludge samples (5.6 ng TEQ kg^{-1} DM) and in 15 compost samples (5.5 TEQ ng kg^{-1} DM) analysed by Elskens et al. (2013), and lower than concentrations reported for Spanish sewage sludge compost (7.4–300.5 ng TEQ kg^{-1} DM) (Muñoz et al., 2018) and for Swiss compost and digestate (6.2 ng TEQ kg^{-1} DM) (Brändli et al. 2007). The maximum level for PCB and PCDD/F stipulated in the relevant EU directive (European Union, 1986) is 800 ng g^{-1} DM and 100 ng TEQ kg^{-1} DM, respectively. Measured concentrations in fish sludge (Table 3) were hence below the applicable EU limits established in 1986. Application of PCDD/F via sewage sludge in field experiments in Turkey was shown to result in accumulation in soil over time (Koyuncu, 2022). Accumulation in soil likely occurs for all persistent organic pollutants when applied at higher rates than removed from soil.

4.3. Unbalanced nutrient composition

In all fish sludge products except the liquid Digestate, the N concentration relative to P was too low to match cereal requirements (NIBIO, 2021) (Table 1). Since the field experiments were designed to meet crop N demands, all dried fish sludge products, including the Dried digestate, resulted in excess P application (Table S2). During dewatering of fish sludge, mineral N (NH_4^+- and NO_3^--N) will follow the liquid phase and NH_4-N may volatilise to the atmosphere as NH_3-N, especially during drying under high-temperature conditions. All fish sludge products studied here, including the liquid Digestate, also lacked K in relation to N and crop requirements, since K^+ mainly occurs in soluble form and will therefore also accompany the liquid phase during mechanical dewatering. To allow for efficient blue-green nutrient recycling in practice, imbalances in fish sludge products will have to be counteracted by adding mineral fertiliser components, either separately or mixed with the fish sludge. Teuber et al. (2005), too, suggested that sea salmon sludge should be complemented with additional N and K before application as fertiliser to land.

Of the excess P applied to soil with fish sludge products, after two growing seasons only a minor fraction was recovered as AL-soluble P, an estimate for plant-available P in soils (Fig. 3). Low plant availability of P in fish sludge is in agreement with findings by Brod and Øgaard (2021) of significantly lower P uptake in barley after fertilisation with dried fish sludge products compared with manure solids or mineral P fertiliser. Insufficient P fertilisation effects can be explained by P mainly being present as stable calcium phosphates, e.g. apatite, as also suggested by Brod et al. (2015). If fish sludge is to be transformed from waste into a valuable fertiliser product, great efforts will have to be devoted to optimising the P fertilisation effects of the sludge.

5. Conclusions

This study examined the quality and effect of fertiliser products based on fish sludge (i.e. feed residues and faeces) from farmed smolt. Chemical characterisation showed unbalanced nutrient composition in fish sludge, with low N/P ratio and low K content relative to crop requirements. Field and soil incubation experiments showed that fish sludge can have good effects as N fertiliser to spring cereals. However, there was wide variation in total N concentrations and N quality, even in fish sludge products treated by the same technology but sampled at different locations and/or times. When fertilisation plans with fish sludge products are to be established, soil incubation tests in combination with modelling and analysis of C/N ratio in dried fish sludge products can be used as indicators of N quality. To utilise P, imbalances in fish sludge products will have to be counteracted e.g. by combination with mineral fertiliser components. Zinc and cadmium concentrations were below the EU maximum limit in all but one of the fish sludge products studied, but Zn concentration in grain was elevated after only two growing seasons with fish sludge fertilisation. The persistent organic contaminants PCB_7, $PBDE_7$ and PCDD/F + DL-PCB were detected in fish sludge, indicating a need for monitoring of these potential environmental and human toxins in fish sludge.

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CRediT authorship contribution statement

Eva Brod: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Writing – original draft, Visualization, Project administration, Funding acquisition. **Trond Maukon Henriksen:** Methodology, Writing – review & editing. **Robin Ørnsrud:** Resources, Writing – review & editing, Funding acquisition. **Trine Eggen:** Writing – review & editing.

Data availability

Data will be made available on request.

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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