Reduction of Internal Phosphorus Load in New Lakes by Pretreatment of the Former Agricultural Soil—Methods, Ecological Results and Costs

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Abstract: New lakes are established or reestablished to provide ecosystem services such as limiting floods and nutrient discharge and to improve biodiversity. New lakes are often established on fertilized land formerly used for agricultural purposes, thereby posing a risk of issues such as phosphorus (P) release when inundated. Release of P from agricultural soil affects both the developing ecosystem of the new lake and may increase downstream eutrophication. To decrease P release following inundation, three simple and cost-effective soil pretreatments were tested through laboratory soil–water fluxes from the test sites in the new Lake Roennebaek and the fluxes of P, nitrogen (N), and iron (Fe) were compared. The pretreatments compared were sand-capping, depth-plowing, and addition of the commercial iron product CFH-12® (Kemira). Untreated agricultural soil incubated under laboratory conditions released 687 ± 88 mg P·m⁻² over 207 days and 85% was released within 60 days from inundation followed by low soil–water P exchange during the remaining incubation period. However, P was still released from the untreated soil 180 days after inundation within the lake. The cumulated P flux of the three pretreatment methods was in comparison negative, between −12 ± 3 and −17 ± 4 mg P·m⁻² over 207 days incubation and showed negative P fluxes from cores collected within the lake 180 days after inundation. This study showed that the release of P when establishing new lakes on former agricultural land could be minimized using these simple and cost-effective methods, which may improve the ecological status of future lakes and enable the establishment of new lakes without threatening vulnerable downstream ecosystems.

Keywords: newly established lake; internal P loading; pretreatment; agricultural soil; sand-capping; iron oxyhydroxide; depth-plowing; topsoil removal; reservoir

1. Introduction

Extensive land reclamation due to urban development and agricultural expansions have led to a reduction of wetlands, where ponds and lakes especially have disappeared in intensively cultivated lowland regions during the last 200 years [1–3].

A large part of the areas that were diked and drained in Denmark during the 1800s land reclamation are today marginalized and provide low economic value for agriculture [4]. Today, the interest in restoring these former lakes has increased, with a focus on regaining ecosystem services and reducing eutrophication and loss of biodiversity in downstream freshwater and marine ecosystems [5,6]. During the last few decades, more than 50 large shallow lakes have been reestablished on recently cultivated land in Denmark [4]. By reestablishing wetlands and lakes, nutrient transport can be reduced by methods such as nitrogen (N) removal through denitrification and phosphorus (P) retention through adsorption, sedimentation, and incorporation into biomass [6–9]. However, in lakes reestablished on
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P saturated soil, inundation can mobilize P, leading to eutrophication of the new lake and increased transport of nutrients to downstream ecosystems [10–12]. Reported P mobilization from inundated agricultural soil varies from less than 1% of the total P content [13–15] to a massive loss of up to 40% [12,16]. The mobilization of P can be very fast, with the majority of the release progressing within as little as two days after flooding [12,13,16,17], while other studies have demonstrated P release several years and even decades after reflooding [10,18–20].

Fertilized land accumulates P in the topsoil due to the accumulation of organic matter and strong association with metals, e.g., iron (Fe) and aluminum (Al) hydroxides and calcium (Ca) minerals [21–23]. Degradation of organic matter and changes in the biogeochemical properties of the soil are the main reasons for the P release induced by flooding [16]. Organogenic soils and the use of organic fertilizers can result in most of the P content being bound in organic forms [24,25]. Mineralization of these P forms can be increased by processes such as elevating pH or regular resuspension, rapidly releasing vast amounts of P [10,12,25–27]. Anaerobic conditions follow the inundation and mineralization of organic matter and may lead to a reduction of Fe, decreasing the P binding capacity of the soil [14,28–30]. Thus, the mobilization of P from the dissolution of Fe$^{3+}$ (hydr-)oxides is important for the release of P and the ratio between the content of Fe-bound P and reducible Fe is important for the P release to the oxygenated water column [28,31–33].

Restoring lakes in a eutrophic state due to internal loading can be an expensive and cumbersome endeavor [34]. The outcome of lake restoration methods depends on many factors and the efficiency of chemical products relies on certain redox and pH conditions which can result in the release of toxic compounds if these are not met [35–37]. Elevated Fe concentrations due to lake restoration measures can lead to toxic effects in both plants and animals [37]. Thus, it is desirable if immediate eutrophication of reestablished lakes can be prevented by pretreatments of the soil prior to inundation, reducing P release from the new lakebed. Hence, accumulated P can be directly removed by topsoil removal or made unavailable in situ using either physical capping barriers or chemical immobilization [38–41].

Removal of topsoil can be an effective method to restore terrestrial habitats to a nutrient-poor state. Dredging can also improve the ecological state of lakes, however, the costs of excavation and transport of the removed soils are high [26,39,42–44]. In addition, deeper soil layers can have a P content too high for removal of topsoil to effectively reduce P release [39,44]. In this study, depth-plowing was tested as an alternative to topsoil removal. Such soil reworking prior to lake establishment, aiming to translocate P-rich topsoil beneath the soil with a lower P content and higher P-binding capacity have been suggested to decrease the P release [10,18] but have to our knowledge not been directly tested. Agricultural soil in Denmark has a homogeneous P-rich plow layer to a depth of approximately 30 cm and a generally elevated P content down to 75 cm deep, however, with increasing Fe:P and Al:P ratios [45], emphasizing the importance of reaching an adequate depth.

Physical and chemical barriers of capping materials can reduce P release from lake sediments, which is demonstrated with various materials such as sand, P-adsorbing natural soils, calcite, modified clays, and zeolites [46–50]. Physical barriers reduce P release by separating chemical and microbial processes in the sediment and water column by decreasing the diffusive flux of dissolved compounds, preventing resuspension and elevated exchange through bioturbation [38,44]. Sand-capping is a widely used in situ capping technique that has been found to decrease the release of various contaminants from sediments and improve visibility and growth conditions for plant and animal communities [38,44,46,51]. The diffusive P release through the sand-cap layer decreases as the cap thickness increases and as grain size decreases. Changes from mobile P to non-mobile apatite associated P have been found in sand-capped sediments, due to extended diffusion time and increased sorption to Ca species [46,49,52–54]. The P binding capacity of sand depends on free adsorption sites of metal compounds and is generally not regarded as high, but specialized materials that immobilize P through specific chemical reactions can decrease the amount of product needed [44,47,53]. Chemical immobilization of P can be accomplished by increasing the P adsorption capacity through the addition of compounds containing elements such as Fe, Al, Ca, or lanthanum (La) [41,55–58]. The addition of
commercial products and industrial by-products has been found to reduce sediment P release and reduce diffuse P runoff from agricultural soils [59–61]. The P adsorption stability of Fe compounds are generally redox sensitive [23,37,55,62], however, the commercial amorphous Fe product, CFH-12® (Kemira), consisting of poorly ordered Fe oxides containing magnesium (Mg) and Ca carbonates, has been demonstrated to be less redox sensitive, reducing P efflux even under anoxic conditions [63,64]. Thus, CFH-12 was found to bind up to 70% of the P adsorbed by amorphous Fe(OH)₃ [63], effectively reduce P leaching from agricultural soil [61,64], and reduce P release from eutrophic lake sediments [63] at environmentally relevant concentrations. Chemical immobilization of P as a pretreatment to reduce release and P availability from inundated agricultural soil by addition of La-modified clays (LMC) and liming have, however, previously been demonstrated to be ineffective [40], even though LMCs have been found to effectively reduce P efflux from eutrophied lake sediments, e.g., reference [58], and limestone has been found to effectively reduce P mobility under reduced conditions typically prevalent in flooded soils [65].

In this study, we compared the effect of depth-plowing, sand-capping, and CFH-12 pretreatments on the exchange of P, Fe, and N from an inundated agricultural soil. Hence, experimental plots treated with either depth-plowing, sand-capping, or CFH-12 were established on agricultural soils prior to the flooding and establishment of Lake Roennebaek. These pretreatments were applied to decrease P release following flooding and investigate if these methods constitute tools able to alleviate rapid eutrophication of new lakes and possibly avoid or postpone the need for lake restorations after establishment. Intact soil cores of each treatment collected prior to the flooding of the lake were inundated for 207 days in the laboratory and the fluxes between pretreatments and untreated agricultural soil were compared. In addition, intact soil cores were collected from the experimental plots 180 days after flooding in order to compare the laboratory experiments with the flux under natural conditions. Finally, the major binding forms of P and the potentially mobile P pool were identified by conducting sequential extraction on pre-inundated soil samples.

2. Materials and Methods

2.1. Study Site

The newly established Danish Lake Roennebaek was flooded during winter 2017 as part of an afforestation project implemented by the municipality of Naestved [66]. Lake Roennebaek had a surface area of 7.9 ha divided into two basins with a total volume of 118,000 m³. The Lake received water from 3 inlets. Inlet 1 and 2 (I1 and I2) collected drainage water from the catchment and the third inlet (I3) from a small spring [67], see Figure 1. The max depth was 3.5 m (mean 1.5 m), however, the water level was expected to seasonally fluctuate with more than 1 m. A pipe-system ensured a continual outlet to Roennebaek stream of 6–13 L·s⁻¹ [67]. The purpose of establishing Lake Roennebaek was to protect downstream watercourses against erosion and desiccation by maintaining a steady year-round water flow reducing N-emissions and improving local biodiversity and recreational value [66,68]. The 270 ha catchment area contained 8% urban area and, prior to the afforestation, 70% agricultural areas and 18% forest and protected nature, see Figure 1.

2.2. Experimental Site and Soil Treatments

The experimental site was established in the northwest corner of the lake, see Figure 1, on former agricultural soil, characterized as clay soil [69]. Soil samples were collected and analyzed to assess the homogeneity of the experimental site prior to lake establishment, see Section 2.3. and Table 1. Four treatment plots of 3 × 3 m spaced 15 m apart were established at depths corresponding to 1.5 ± 1 m after flooding. Four pretreatments were applied to the plots: sand-capping, addition of CFH-12, depth-plowing, and an untreated control, see Figure 1.
Depth-plowing was simulated by removing the topsoil layer to a depth of 30–40 cm and digging up the deeper soil layer to a depth of 80 cm, then burying the topsoil upside down in the hole beneath the soil. Due to the small size of the plots, depth-plowing by a tractor was not possible. Depth-plowing by a tractor was not possible.

Hence, 2.9 kg·m⁻² of the granular product, with grain size of 0.85–2 mm and a specific surface area of 32 ± 10 m²·g⁻¹ [63,64], was homogenously distributed with a garden fertilizer spreader on the undisturbed soil. Due to the small size of the plots, depth-plowing by a tractor was not possible. Depth-plowing was simulated by removing the topsoil layer to a depth of 30–40 cm and digging up the deeper soil layer to a depth of 80 cm, then burying the topsoil upside down in the hole beneath the soil originating from the deeper layer.

The sand-capping pretreatment consisted of sand with a grain size of 1–8 mm and a density of 1.65 g·cm⁻³. The sand was added directly on top of the undisturbed soil, without removing the groundcover. A sand-cap of approximately 6 cm, corresponding to 99 kg·m⁻², was applied since P release has been found not to decrease significantly by further increasing cap thickness [49].

CFH-12 was added in a molar Fe:P ratio of 10:1 to the potentially mobile-P content in the plowing layer, in accordance with Fuchs et al. [63], assuming that 44% of CFH-12 consists of Fe [64]. Hence, 2.9 kg·m⁻² of the granular product, with grain size of 0.85–2 mm and a specific surface area of 32 ± 10 m²·g⁻¹ [63,64], was homogenously distributed with a garden fertilizer spreader on the undisturbed soil. Due to the small size of the plots, depth-plowing by a tractor was not possible. Depth-plowing was simulated by removing the topsoil layer to a depth of 30–40 cm and digging up the deeper soil layer to a depth of 80 cm, then burying the topsoil upside down in the hole beneath the soil originating from the deeper layer.
Table 1. Lake morphometry, water-chemical parameters of Lake Roennebaek, and soil characteristics of the experimental site. Standard deviations are stated following ±.

| Lake Characteristics | Soil Characteristics |
|----------------------|----------------------|
| Lake area (ha)/Catchment (ha) | 7.9 \(^1\)/270 | Dry weight (%) | 81.7 ± 2.6 \(^5\) |
| Volume (m\(^3\))/Residence time (month) | 118,000 \(^1\)/6 \(^1\) | Loss on ignition (%) | 5.1 ± 0.7 \(^5\) |
| Max depth (m)/Secchi depth (m) | 3.5 \(^1\)/1.3 ± 0.4 \(^2\) | Density (g·cm\(^{-3}\)) | 1.97 ± 0.06 \(^5\) |
| O\(_2\) (mg·L\(^{-1}\))/Color (mgPt·L\(^{-1}\)) | 8.6 ± 5.6 \(^3\)/99 ± 29 \(^2\) | TP (mg·gDW\(^{-1}\)) | 0.62 ± 0.09 \(^5\) |
| pH/Alkalinity (meq·L\(^{-1}\)) | 8.2 ± 0.6 \(^3\)/3.7 ± 0.6 \(^2\) | P-number (mgP·100·gDW\(^{-1}\)) | 1.9 ± 0.5 \(^5\) |
| Chl-a (µg·L\(^{-1}\))/DOC (mg·L\(^{-1}\)) | 12.6 ± 15.3 \(^3\)/16 ± 3 \(^2\) | TFe (mgFe·gDW\(^{-1}\)) | 11.9 ± 0.5 \(^5\) |
| SO\(_4\)\(^2-\) (mg·L\(^{-1}\)) | 15.4 ± 5.4 \(^4\) | TAl (mgAl·gDW\(^{-1}\)) | 11.9 ± 0.6 \(^5\) |
| TP (µg·L\(^{-1}\))/DIP (µg·L\(^{-1}\)) | 80 ± 70 \(^2\)/10 ± 10 \(^2\) | Molar TFe:TP | 10.8 ± 1.5 \(^5\) |
| NO\(_3\)\(^-\) (µg·L\(^{-1}\))/NH\(_4\)\(^+\) (µg·L\(^{-1}\)) | 40 ± 80 \(^2\)/50 ± 40 \(^2\) | Soil type | Clay soil \(^6\) |

\(^1\) From Rasmussen \[66\]. \(^2\) Annual mean values collected at the deepest part of the lake (W), see Section 2.6.; standard deviation between the 7 months is stated. \(^3\) Measured near lakebed simultaneously with \(^2\), see Section 2.6. \(^4\) Mean from 3 water samples, one from each inlet (I1, I2, and I3) collected 25 June 2018. \(^5\) Mean and standard deviation from (n = 16) pre-flooding soil samples of the plowing layer, see Section 2.3. \(^6\) From Greve & Møller \[68\].

2.3. Pre-Flooding Soil Sampling

To assess the heterogeneity of the experimental site, 16 pre-flooding soil samples evenly distributed within the experimental site were collected in September 2017, each consisting of 5 kg of soil from the plow layer. The soil was homogenized and dry weight (DW) was measured by drying 30 g of soil for 24 h at 105 °C. The organic content was measured as loss on ignition (LOI) by combusting 5 g of dried subsamples for 5 h at 520 °C. The total content of P, Fe, and Al (TP, TFe, TAl) were measured on 0.1 g incinerated soil by ICP-OES (inductively coupled plasma with optical emission spectroscopy; Optima 2100 DV, Perkin Elmer) following 1 h autoclaving at 120 °C \[70\] in 8 mL 1 M HCl. Plant available P (Olsen-P) was analyzed according to Rubæk and Kristensen \[71\] by extracting 5 ± 0.1 g dry soil in 100 mL 0.56 M NaHCO\(_3\) solution at pH 8.50 ± 0.02 for 30 min. The extracts were filtered through 1.2 µm Whatmann GF/C filters, degassed using 4 M H\(_2\)SO\(_4\), and measured spectrophotometrically as dissolved inorganic P (DIP) at 880 nm as a molybdate blue complex \[72\] after conservation by addition of 200 µL 2 M H\(_2\)SO\(_4\), 10 mL sample.

2.4. Laboratory Experiments on Undisturbed Soil Cores Collected Prior to Flooding

2.4.1. Soil Core Sampling

Immediately before flooding of Lake Roennebaek, December 2017, 16 undisturbed soil cores were collected with acrylic Kajak tubes (\(\Theta= 5.2 \text{ cm}, \ h = 40 \text{ cm}\)), four cores from each pretreatment plot. The cores were stored outside with a bottom stopper and protected from rain to avoid P leaching. No top stopper was applied prior to analysis, thus exposing the cores to the natural fluctuations in temperature and humidity. To estimate the potential efflux of P, N, and Fe after flooding, three cores from each pretreatment plot were incubated in a 207 days soil-water flux experiment, see Section 2.4.2. The remaining soil core was sliced in depth intervals and extracted sequentially, according to a modified Psenner extraction \[73\], see Section 2.4.3.

2.4.2. Flooding Experiment

Three cores of each pretreatment plot were incubated for a 207 days soil–water flux experiment (termed “flooding experiment”). Each core was inundated with 350 mL of mixed water from the inlets I1 and I2, see Figure 1. The cores were equipped with stirring magnets placed 5 cm above the soil surface to ensure mixing of the overlying water and the cores were placed in an incubation chamber in darkness and a water bath of 15 °C, to approximate temperatures prevailing during the summer season. Soil–water fluxes of nutrients were measured at regular intervals 21 times during the experiment, initially measured 2–3 times a week, gradually extended to approximately once every second week at
the end of the experiment. The fluxes were calculated as the difference between initial amounts and amounts present at the following sampling, with each end amount serving as the initial amount of the following flux interval, constituting a continual flux corrected for amounts removed and added to keep the volume of water constant during samplings. The fluxes between each sampling were presented as cumulated fluxes of each core. Fluxes of DIP, total dissolved P (TDP), NO₃⁻, NH₄⁺, and dissolved Fe (DFe) were measured on filtered (1.2 µm) water samples. Water samples were collected with a syringe and the removed water was replaced by a known volume of mixed inlet water with known nutrient concentrations (DIP, TDP, NO₃⁻, NH₄⁺, and DFe). TDP was measured as DIP on filtered, acidified (2 M H₂SO₄) samples after wet oxidation with potassium-peroxy-disulfate and autoclavation at 120 °C for 1 h. DFe was measured spectrophotometrically at 562 nm on filtered (1.2 µm) acidified (2 M H₂SO₄) samples using the ferrozine method [74]. NO₃⁻ and NH₄⁺ were analyzed spectrophotometrically on a flow injection system, Skalar sanplus, on water samples filtered on 1.2 µm GF/C filters and stored frozen at −18 °C immediately after filtration until analysis. NO₃⁻ was analyzed as an azo-dye from nitrite after reduction through a cadmium column. NH₄⁺ was analyzed as a green complex measured at 630 nm formed from a modified Berthelot reaction with monochloramine reacting with phenol.

2.4.3. Sequential Extraction of Soil

Sequential extraction was conducted on cores from each of the four treatment plots, collected prior to flooding, see Section 2.4.1. Due to a time-consuming procedure and high homogeneity of P, Fe, and Al, see Table 1, only one replicate of each treatment was studied to minimize the workload related to extraction. Cores were sliced in one cm intervals down to five cm depth and in five cm intervals at depths up to fifteen cm, see Figure 1. For the sand-capping treatment, the entire sand layer (6.5 cm) was considered as one additional depth interval, see Figure 2.

Using a modified sequential extraction scheme [73,75] seven operationally defined P pools were characterized. One g of fresh soil was sequentially extracted in a series of solutions starting with H₂O extracting loosely adsorbed P (P-H₂O), after which a bicarbonate-dithionite (BD) solution was used to extract reducible Fe (Fe-BD) and the associated P (P-BD) [76]. Then, addition of NaOH extracted P associated with e.g., Al (P-NaOH) and P bound in humic acids (HA-P-NaOH), separated by acidification and filtration on GF/C filters (1.2 µm) subsequently combusted and determined as TP. Finally, an extraction in HCl dissolved P bound to minerals, such as Ca-bound P (P-HCl). Remaining residual P (P-res) in the soil pellet was quantified by autoclavation (1 h at 120 °C) in HCl following incineration. TP in all extracts, as well as Fe-BD, were measured by ICP-OES and inorganic P was measured as DIP on the H₂O, BD, and the filtered NaOH extracts, with the difference between DIP and TP constituting the non-reactive P (nrP). The potentially mobile P pool was constituted by DIP-H₂O, DIP-BD, and nrP. For a more detailed description of the sequential extraction procedure, see Reitzel [73]. A subsample of each sediment slice was used for determination of DW, LOI, and TP, see Section 2.3.

2.5. Nutrients and Fe Flux from Inundated Soil

Three soil cores were collected, from each treatment plot in June 2018, 180 days after flooding, to follow the soil–water exchange of P, N, and Fe (termed “inundated soil experiment”). The cores were sampled with Kajak tubes, kept inundated and without top stopper to allow gas exchange.

The cores were submerged in incubation chambers containing water collected directly above the experimental site with a heart valve sampler, to secure a water composition like the water overlying the lakebed. Following 72 h acclimation to laboratory conditions of 15 °C, aeration, darkness, and magnetic stirring, DIP, TDP, NO₃⁻, NH₄⁺, and DFe were sampled before and after 24 h incubation, see Section 2.4.2.

2.6. Lake Water Parameters

TP, DIP, NO₃⁻, and NH₄⁺ along with supporting water parameters (chlorophyll-a (Chl-a), total alkalinity, dissolved organic carbon (DOC) and color) were monitored 7 times: monthly from May
to September in November 2018, and in February 2019 to characterize the water quality of Lake Roennebaek, see Table 1. Water samples were collected with a heart valve water sampler immediately above the lakebed from the deepest part of the lake (W) at 3–3.5 m, see Figure 1. Chl-a was determined spectrophotometrically on ethanol extractions of 1.2 µm GF/C filters following water filtration [77]. Total alkalinity was measured by Gran titration [78]. DOC was measured on filtered (1.2 µm) samples using infrared spectrophotometry on a TOC 5000 Total Organic Carbon Analyzer. SO$$_4^{2-}$$ was measured once in each inlet (I1, I2, and I3) on water samples collected 25 June 2018, determined by the turbidimetric method [79]. Secchi depth was obtained using a 30 cm Secchi disc and vertical profiles of O$_2$ concentration, temperature, and pH were measured by YSI probes, see Table 1.

2.7. Statistical Analysis

Differences between the four treatments at the end of the flooding experiment were compared by a one-way ANOVA followed by a Holm–Sidak post hoc test. This test was also used to compare differences of the daily fluxes within the flooding experiment at 180 days after flooding and the fluxes of the inundated soil experiment 180 days after inundation between the treatments. An $\alpha = 0.05$ was used as cutoff for significant differences. All statistics were performed with SigmaPlot 14.0.

3. Results

3.1. Soil Phosphorus Content

The TP content decreased slightly with depth, from 0.78, 0.68, and 0.94 to 0.62, 0.48, and 0.58 mg P·g DW$^{-1}$ in the control, CFH-12 treated soil, and in the soil beneath the sand-cap, respectively, see Figure 2. The Olsen-P in the untreated soil of the plow layer was 0.013–0.029 mg P·g DW$^{-1}$ within the ES. The TP content of the depth-plowed soil was lower than the other treatments, varying between 0.42 and 0.64 mg P·g DW$^{-1}$ with the lowest content at the surface. The TP content of the sand layer was 0.37 mg P·g DW$^{-1}$. The P pools in the control, CFH-12 treated soil, and soil beneath the sand-cap showed similar sizes and limited changes with depth. The general contribution to TP was of 1% for the DIP-H$$_2$$O pool, 18% for DIP-BD, 12% for nrP, 9% for P-NaOH, 26% for HA-P-NaOH, 21% for P-HCl, and the P-res pool contributed 13% of TP. The potentially mobile P pool constituted 32%, 29%, and 30% of TP within the upper 10 cm of the control, CFH-12 treated soil, and soil beneath the sand-cap, respectively, see Figure 2.

The depth-plowed soil had a higher content of HCl-P, compared to the other treatments, contributing 38% of TP while HA-P-NaOH contributed 19%, nrP 8%, and P-BD 9% of TP, respectively. The mobile P pool of the depth-plowed soil varied between 0.09 and 0.16 mg P·g DW$^{-1}$ constituting between 17% and 29% of TP and without a tendency of change with depth. In the sand-cap material, P-HCl contributed to 48%, P-H$_2$O to 21%, and P-BD to 19% of TP while P-res, nrP, and HA-P-NaOH were insignificant. The potentially mobile P pool in the sand layer was 0.16 mg P·g DW$^{-1}$ constituting 43% of TP and making up less than half of the mobile P pool in the topsoil just beneath, see Figure 2. LOI decreased with depth from between 12% and 18% in the surface to 6–7% at 10 cm depth of the control, CFH-12 treated soil, and soil beneath the sand-cap. LOI of the depth-plowed soil was 3–4% and only 0.6% in the sand-cap. The molar Fe-BD:DIP-BD ratios were generally low in the surface soil (0.6, 5.6, 2.0, and 0.8 for the control, CFH-12, depth-plowing, and sand-capping treatment, respectively), but with considerably lower concentrations of both DIP-BD and Fe-BD, of 0.11 mg Fe·g DW$^{-1}$, in the sand layer, see Figure 2.
Within the first 90 days after flooding, 93% of the N removed as NO$_3^-$ was 2.6 ± 2020 CFH-12 treated soil was very high immediately after inundation. After 14 days, the cumulated flux significantly decreased and 1595 mmol Nm$^{-2}$d$^{-1}$ flux of the flooding experiment was positive for the control and the CFH-12 treated soil, 309 mmol Nm$^{-2}$d$^{-1}$ respectively, showing a similar tendency as the flooding experiment. The removal of NO$_3^-$ was 30% more NO$_3^-$ respectively, showing a similar tendency as the flooding experiment. The control removed significantly less N in comparison with the pretreated soils and significantly lower (p = 0.016) than the depth-plowed soils removing 10.8 mmol Nm$^{-2}$d$^{-1}$, see Table 2. The NH$_4^+$ flux of the flooding experiment was positive for the control and the CFH-12 treated soil, 309 ± 327 and 1595 ± 442 mmol Nm$^{-2}$d$^{-1}$, respectively, while negative for the sand-capped and depth-plowed soil, −26 ± 4 and −34 ± 10 mmol Nm$^{-2}$d$^{-1}$, respectively. Only the flux from the CFH-12 treated soil was significantly different from the control soil (p = 0.015). The NH$_4^+$ flux of flooding experiment from the CFH-12 treated soil was very high immediately after inundation. After 14 days, the cumulated flux reached 1602 ± 80 mmol Nm$^{-2}$ followed by a negative flux until day 21 and a relatively stable release of,

3.2. Soil–Water Flux

The control soil released significantly more P (p < 0.001), 687 ± 88 mg Pm$^{-2}$d$^{-1}$, during the 207 days flooding experiment, compared to the CFH-12 treated, sand-capped, and depth-plowed soils all showing negative fluxes of −12 ± 3, −13 ± 13, and −17 ± 4 mg Pm$^{-2}$d$^{-1}$, respectively. P was primarily released as DIP, representing 99% of the TDP flux of the control treatment, and 85% of the accumulated DIP flux was released within the first 60 days of inundation. The DIP release was initially close to linear with a release of 10 mg Pm$^{-2}$d$^{-1}$ and after 100 days of incubation almost no exchange was observed, see Figure 3.

The DIP flux of the inundated soil experiment measured 180 days after flooding released 1.8 ± 1.9 mg Pm$^{-2}$d$^{-1}$ from the control soil whereas the CFH-12 treated, sand-capped, and depth-plowed soil had a negative flux of −0.4 ± 0.6, −0.4 ± 1.2, and −1.0 ± 0.4 mg Pm$^{-2}$d$^{-1}$, respectively, showing a similar tendency as the flooding experiment. The control removed significantly more NO$_3^-$ ((p < 0.001), 16 ± 1 g Nm$^{-2}$) in the flooding experiment, compared to the depth-plowed, sand-capped, and CFH-12 treated soils which only removed 3 ± 1, 4 ± 2 and 5 ± 1 g Nm$^{-2}$ respectively. Within the first 90 days after flooding, 93% of the N removed as NO$_3^-$ from the control cores took place, initially with a close to linear removal of 150 mg Nm$^{-2}$d$^{-1}$ and a low removal for the remaining incubation period. The removal of NO$_3^-$ from the control soil in the inundated soil experiment was 2.6 ± 3.2 mgm$^{-2}$d$^{-1}$, and was lower compared to the pretreated soils and significantly lower (p = 0.016) than the depth-plowed soils removing 10.8 ± 1.5 mgm$^{-2}$d$^{-1}$, see Table 2. The NH$_4^+$ flux of the flooding experiment was positive for the control and the CFH-12 treated soil, 309 ± 327 and 1595 ± 442 mg Nm$^{-2}$, respectively, while negative for the sand-capped and depth-plowed soil, −26 ± 4 and −34 ± 10 mg Nm$^{-2}$, respectively. Only the flux from the CFH-12 treated soil was significantly different from the control soil (p = 0.015). The NH$_4^+$ flux of flooding experiment from the CFH-12 treated soil was very high immediately after inundation. After 14 days, the cumulated flux reached 1602 ± 80 mg Nm$^{-2}$ followed by a negative flux until day 21 and a relatively stable release of,
on average, 2 mg N·m⁻²·d⁻¹ during the remaining period. The DFe flux of the flooding experiment was positive from the control and CFH-12 treated soils amounting to 427 ± 407 and 531 ± 508 mg Fe·m⁻², respectively. In contrast, the sand-capped and depth-plowed soils had negative fluxes of −37 ± 28 and −71 ± 29 mg Fe·m⁻², respectively, however, without significant differences between treatments, see Figure 3. Compared to the flooding experiment, the NH₄⁺ and DFe fluxes of the inundated soil experiment were positive for all treatments. Opposite to the flooding experiment, the highest inundated soil flux of both NH₄⁺ and DFe was observed from the depth-plowed soil (75 ± 3 mg N·m⁻²·d⁻¹ and 36 ± 8 mg Fe·m⁻²·d⁻¹, respectively), and was significantly higher than the flux after 180 days (p < 0.001 and p = 0.002 respectively) of the flooding experiment. Both the control and sand-capped soils released significantly lower amounts of NH₄⁺ (p = 0.006 and p = 0.027 respectively) and DFe (p = 0.008 and p = 0.030 respectively) than the depth-plowed soil. The NH₄⁺ and DFe fluxes of the CFH-12 treated soil in the inundated soil experiment were not significantly different from the other treatments, see Table 2.

Figure 3. Cumulated soil–water fluxes of the flooding experiment, a and b designate significant differences between treatments 207 days after flooding. (A) Dissolved inorganic phosphorus (DIP) scaling on y-axis change at 10 mg P·m⁻² to allow visualization of fluxes of both control and pretreated soil; F = 196.3, DF = 11, P < 0.001. (B) Dissolved iron (DFe); F = 2.7, DF = 11, P = 0.114. (C) NO₃⁻; F = 60.7, DF = 11, P < 0.001. (D) NH₄⁺; F = 23.7, DF = 11, P < 0.001. The marks indicate the average cumulated flux (n = 3) and the error bar indicates the standard deviation between soil cores. TDP was not included since 99% of TDP was constituted by DIP and no extra information would follow from including TDP in the figure.
4. Discussion

4.1. Overview

Our results showed that sand-capping, addition of CFH-12, and depth-plowing were suitable pretreatments to reducing P release from inundated agricultural soil. This was demonstrated both from the flooding experiment and the inundated soil experiment. Assuming that the control plot represented the entire 7.9 ha Lake Roennebaek, 54 kg P or 2% of the potentially mobile P pool in the upper 15 cm of the plow layer was released during the first 200 days of inundation. Within the first 60 days of the experiment, 85% of the released P was mobilized and the exchange was low for the remaining period, according to the flooding experiment. However, the P release took place for a longer period under the conditions within Lake Roennebaek, where P was still released from the control soil after 180 days inundation.

4.2. Effect of Pretreatments

4.2.1. DIP Flux

The low P release of the untreated control soil compared to the potentially mobile P pool in Lake Roennebaek may be explained by the high molar TFe:TP and TAl:TP ratios (of 11 and 22 respectively) indicating the presence of free adsorption sites at least under aerobic conditions [28,80,81]. However, the initial release of 10 mg P m⁻² d⁻¹ from the untreated control soil was high compared to the release from lakebeds of both new and natural Danish lakes with TFe:TP ratios above 8:1 [82,83]. The low Fe-BD:DIP-BD ratio also indicated that both the size of the mobile P pool and the capacity of the sediment to control P release could be overestimated. The nrP pool identified in the inundated soils likely contributed to the overestimation of the potentially mobile P pool within the timeframe of this study, since fractions of lake sediment nrP showed slow mineralization rates [84] and since organic content of terrestrial soils has been found to be refractory and only mineralized slowly in inundated soils [85]. The low Fe-BD:DIP-BD indicated that a part of the P extracted by BD may not be associated with redox-sensitive Fe, or that dissolved Fe was not being released proportional to P in the ratio of 8:1 found by Jensen et al. [28]. This suggests that the majority of TFe in the former agricultural soil may have consisted of Fe associated to silicates, incorporated in organic compounds or complexes such as with SO₄²⁻ and CO₃²⁻, insoluble and unable to sequestrate P in amounts comparable to amorphous or

| Flux     | Treatments (mg m⁻² d⁻¹) | Control | CFH-12 | Sand-Capping | Depth-Plowing |
|----------|-------------------------|---------|--------|--------------|---------------|
| DIP      | Inundated               | 1.8 ± 1.9 a,x | -0.4 ± 0.6 a,x | -0.4 ± 1.2 a,x | -1.0 ± 0.4 a,x |
|          | Flooding                | -0.1 ± 0.2 a,x | <−0.01 a,x | <−0.01 a,x | <−0.01 a,x |
| NO₃⁻     | Inundated               | -2.6 ± 3.2 a,x | -10.9 ± 6.4 ab,x | -5.0 ± 6.8 ab,x | -10.8 ± 1.5 b,x |
|          | Flooding                | -7.8 ± 2.1 a,x | -8.3 ± 5.7 a,x | -9.6 ± 0.3 a,x | -7.3 ± 1.9 a,x |
| NH₄⁺     | Inundated               | 4.7 ± 14.0 a,x | 39.6 ± 42.9 ab,x | 18.7 ± 28.2 a,x | 74.9 ± 3.3 b,x |
|          | Flooding                | 0.3 ± 0.5 ab,x | 2.8 ± 2.3 ab,x | -0.1 ± 0.1 a,x | <−0.01 b,y |
| DFe      | Inundated               | 3.7 ± 8.3 a,x | 18.4 ± 15.1 ab,x | 5.4 ± 13.9 a,x | 36.2 ± 8.2 b,x |
|          | Flooding                | 0.2 ± 0.2 a,x | 3.8 ± 6.8 a,x | -0.1 ± 0.1 a,x | -0.1 ± 0.1 a,y |

Table 2. DIP, NO₃⁻, NH₄⁺, and DFe fluxes from the different pretreatments. The soil–water flux (n = 3) of the inundated soil experiment measured 180 days after inundation compared to the calculated daily fluxes from the flooding experiment (n = 3) 180 days after inundation. Statistically significant differences between sediment–water fluxes of the different pretreatments and control are indicated with a and b and statistically significant differences between the fluxes of the inundated soil experiment and the fluxes of the flooding experiment with the same treatment are indicated with x and y. TDP was not included since 99% of TDP was constituted by DIP and no extra information would follow from including TDP in the table.
crystalline Fe-(hydr)oxides [32,33,86,87]. If the Fe-BD:DIP-BD ratio was reflecting the amount of free P absorption sites, Fe was saturated with P and would not be expected to control the P release under aerobic conditions [28,32]. Anoxic conditions were found to emerge quickly after the inundation of agricultural soil and only leave a thin oxidized surface [14]. However, in our study, the amount of P released from the reduction of Fe would most likely be lower than estimated from the DIP-BD pool, where a Fe-BD:DIP-BD ratio below 8.3 was observed. This indicates that a part of the P extracted by BD would not be associated with reducible Fe [28]. Thus, from the Fe extracted by BD, no more than 590 mg P m$^{-2}$ could be expected to be released from the uppermost 5 cm soil, closely resembling the actual P release observed from the untreated control soil. A low number of free binding sites in the soil was further supported by the fact that the addition of additional P binding sites in the form of CFH-12 completely stopped the P release.

Both the sand-capping and depth-plowing treatments completely stopped the release of DIP, DFe, and NH$_4^+$, as well as decreased the NO$_3^-$ removal, most likely by constituting a physical barrier separating the water from nutrient-rich soils [54]. Limited oxygen availability decreased the P released from mineralization of the buried organic matter and the diffusion through the barrier extended the time before mobilized P would be released to the water [38,44].

P released from the reduction of Fe or mineralization in the soil beneath the sand-cap did not diffuse through the sand-cap within the timeframe of this study, indicating that the grain size and cap thickness were sufficient to prevent a fast P release following inundation. The sand used in this study had a low content of Fe but may have contained free adsorption sites able to sequester P from e.g., Al or Ca [53], which was supported by the high proportion of P-HCl found in our study. If the P adsorbing capacity of the sand was too low to sequester the P mobilized from the soil beneath, P would at some point be expected to diffuse through the sand-cap [49,88]. The effect of depth-plowing was expected to depend on the distribution of P through the soil profile and to work best on layered soils with P-rich topsoil and deeper layers of mineral soils with low P content and high content of metals with P-binding capabilities, e.g., Fe. As for the sand-capping, the depth-plowing treatment was expected to work by decreasing mineralization and diffusive release of the buried P rich topsoil, which worked within the timeframe of the study, considering no P efflux was observed. However, the surface of the depth-plowed soil contained a potentially mobile P pool, although half the size of the control, and a low Fe-BD:DIP-BD ratio, suggesting that Fe would not be expected to control the P release [28,32,33]. The lack of P efflux may be explained by an overestimation of the mobile P pool by the extraction procedure. The depth-plowed soil had a low organic content and nrP pool compared to the control soil and the quality and degradability of the organic compounds could be expected to be lower, considering they originated from deeper soil layers [84,85,89]. Hence, this would lead to a lower P release from the mineralization of organic matter. As for the control soil and the sand layer, the low Fe-BD:DIP-BD indicated an overestimation of P adsorbed to reducible Fe and the low organic content possibly increased the depth of oxygenated soil, mitigating mobilization of P from the reduction of Fe. Even though the content of BD-extractable Fe was low, the negative P flux of the inundated soil experiment indicated a P adsorption capacity of the depth-plowed soil that may be caused by crystalline Fe or Al, indicated by the high TAl:TP ratio [33,80,90]. Plowing and soil reworking have been found to increase P leaching and loss of particulate P from resuspension [91–93], however, this could not be assessed from this study. Another possible disadvantage of the depth-plowing method could be the possible threat posed to undiscovered archaeological objects; archaeologic inspections of the location may be necessary prior to treatment.

4.2.2. DFe, NO$_3^-$, and NH$_4^+$ Flux

The DFe released from the control soil was lower than the P release, further supporting that only a minor part of the Fe pool was reducible. As observed by Grunth et al. [14], the DFe release was delayed relative to DIP, which together with the low release could be due to slow dissolution of Fe compounds under reducing conditions, precipitation following reoxidation and immobilization by reaction with
compounds such as \( \text{SO}_4^{2-} \) and \( \text{CO}_3^{2-} \) [33,94]. The decreasing \( \text{NO}_3^- \) removal and \( \text{NH}_4^+ \) release after the initial 100 days incubation could be explained by decreased ammonification and denitrification as the labile organic content in the water and soil surface was mineralized [95]. This also explains the higher rates of \( \text{NO}_3^- \) removal and \( \text{NH}_4^+ \) release observed in the inundated soil experiment where labile organic matter could be supplied from external sources and primary production within the lake.

The low release of \( \text{NH}_4^+ \) and removal of \( \text{NO}_3^- \) from the sand-capped and the depth-plowed soil under laboratory conditions could be explained by the low organic content and quality in the surface, decreasing ammonification and denitrification [85,89,95]. The \( \text{NH}_4^+ \) released from the buried topsoil was also expected to be slowed by the decreased degradation rate of organic content at reducing conditions [85,95]. The low DFe release from the sand-capped and depth-plowed soil could be explained by reduced mineralization allowing oxidized conditions to a greater depth and that only a minor part of Fe was present in reducible forms [94]. In contrast, the high release of both \( \text{NH}_4^+ \) and DFe from the depth-plowed soil in the inundated soil experiment may reflect an increased surface area and aerobic mineralization due to increased porosity and pockets formed from the treatment, increasing the diffusive release from deeper soil layers, as well [52].

The observed DFe release from the CFH-12 treated soil corresponded to a release of only 0.04% of the Fe added and was released immediately after inundation. However, the cumulated flux was not significantly different from the control soil, indicating no appreciable CFH-12 dissolution, as also found by Fuchs et al. [63]. The dissolved Fe could potentially lead to toxic effects, however, the low DFe release compared to control soil indicated no immediate risk of toxic effects to flora and fauna in the water column posed from pretreatment with CFH-12 [37]. The increased \( \text{NH}_4^+ \) release coincided with the initial release of DFe after inundation. This suggests that the high \( \text{NH}_4^+ \) release was caused by CFH-12 constituting a source of electron acceptors increasing the microbial ammonification and release of sorbed \( \text{NH}_4^+ \) directly by reduction of Fe (III) and by exchange with Fe\(^{2+}\) to cation sorption sites [96–98]. The decreased \( \text{NO}_3^- \) removal in the CFH-12 treated soil suggests that labile organic compounds were made unavailable, inhibiting denitrification by organo-Fe complexation or adsorption of DOC to Fe phases [99,100]. The low Fe release does, however, not indicate continuing microbial reduction of Fe and CFH-12 has previously been found resistant towards a microbial reduction and not to affect \( \text{NH}_4^+ \) or \( \text{NO}_3^- \) exchange of lake sediments [63]. Fe plays a key role in the N and C transformations in soils as an electron acceptor and ligand, thus, the addition of Fe products may also impact nitrification [101] and emission of climate gases [96,102]. Decreased amounts of \( \text{CH}_4 \) emitted from inundated soils could, therefore, be another positive effect of Fe addition worth considering when restoring aquatic systems, where increased climate gas emissions pose a disadvantage [96,103].

### 4.3. P Release from Agricultural Soil

Pretreating Lake Roennebaek may not be a financially reasonable solution considering the low amount of P and the short period of release from the untreated agricultural soil, as shown by the flooding experiment. However, previous studies have observed high P release from inundated agricultural soils and emphasize the necessity of mitigation. The P release from the control soil in this study rather resembled fluxes observed from natural uncultivated soils of 3–10 mg P·m\(^{-2}\)·d\(^{-1}\) than fluxes observed from agricultural soils of 15–600 mg P·m\(^{-2}\)·d\(^{-1}\) [13,16]. Compared to agricultural soils in Denmark, the content of Olsen-P was relatively low in Lake Roennebaek [45], suggesting that most Danish soils would probably release larger amounts of P when inundated.

The observed P release could be increased by reduced conditions at the soil surface [27,31]. In situ \( \text{O}_2 \) concentration in Lake Roennebaek was generally high throughout the monitoring period, but was as low as 0.12 mg·L\(^{-1}\) during a drought in July 2018, indicating that reduced conditions could occur locally. Even though the DIP-BD pool may not exclusively represent P associated with reducible Fe, the extraction procedure indicated that up to 8 g P·m\(^{-2}\), from the uppermost 5 cm soil, could be released under reducing conditions, which could severely impact the ecological state of Lake Roennebaek.
The high DIP, NH$_4^+$, and DFe fluxes of the control soil in the inundated soil experiment compared to the fluxes after 180 days of the flooding experiment suggest that the period of exchange between soil and water may be underestimated by the flux of the flooding experiment or that the conditions within Lake Roennebaek caused a slower exchange stretching over a longer period. A prolonged flux within the lake relative to laboratory conditions could be explained by decreased mineralization and slower diffusive release at lower temperatures, increasing from 2 °C to 17.5 °C within Lake Roennebaek during the 180 days compared to a constant 15 °C for the flooding experiment [52,104]. The fluxes from the inundated soil experiment could also be prolonged by the input of labile organic compounds from primary production, resuspension, and infauna bioturbation [105–108].

Assuming that the P release was representative of the entire lakebed and no water enters or leaves Lake Roennebaek, the flooding experiment predicted a lake water concentration of 0.465 mg P·L$^{-1}$ whereas the flux of the inundated soil experiment estimated a concentration of 0.220 mg P·L$^{-1}$, better reflecting the observed concentration of 0.191 mg TP·L$^{-1}$ measured in the lake six months after inundation. This does not indicate increased P efflux from reduced conditions or a large loss of particulate P, due to resuspension as found by references [10,12,27]. Assuming low water exchange during the summer, P concentrations could be expected to be high enough to classify Lake Roennebaek as eutrophic, from the short period of internal P release alone and possibly leading to internal P circulation [109]. However, the residence time and external P loading of the lake dictate the longevity of the impact of the released P [27,109].

### 4.4. Comparison of Costs of Pretreatments

Using Lake Roennebaek as a case study, estimates of the costs of sand-capping, depth-plowing, and CFH-12 addition, as well as topsoil removal, follow from Table 3. Compared to topsoil removal, the costs of these pretreatments were much lower. The costs of the pretreatment methods primarily depended on transport costs and costs of product acquisition. Removal of topsoil would be very expensive, since the method requires transportation of large quantities of soil. Assuming removal of a 30 cm plow layer with a volume expansion factor of 1.33 [110], 3990 m$^3$·ha$^{-1}$ soil would have to be transported. This could be decreased notably if the excavated soil was used on-site in the construction of things such as dikes, hills, and bird islands. This could, however, pose a risk of nutrient leaching into the new lake [93].

| Method            | Costs (EUR·ha$^{-1}$) |
|-------------------|------------------------|
| Depth-plowing     | 900                    |
| Sand-capping      | 40,200                 |
| CFH-12            | 67,000                 |
| Removal of topsoil| 502,800                |

1 Estimated from Espensen et al. [111]. 2 Personal communication with Danish contractors (Gammelskov Maskinstation and Møns Entreprenørfirma). 3 Kemira Oyj. 4 Estimated from Fuchs et al. [63].

The most cost-effective method tested was depth-plowing since the expenses were confined to equipment acquisition and execution of the treatment, already commonly practiced when establishing forests or hedgerows [111,112]. The cost of executing the depth-plowing depends on the soil conditions. Stony ground, wet soils, and soils with pronounced precipitation layers increase time expenditure and costs [111]. Possible costs in relation to archaeological investigations were not considered. The sand-capping method involves high transport expenses but only 1/5th the bulk needs to be transported compared to topsoil removal (990 tons sand·ha$^{-1}$ for a sand-cap of 6 cm) and acquisition costs of sand would be low compared to P-binding products, such as CFH-12 [49]. P-binding products
involve lower transport expenses and higher acquisition costs per unit of product. Adding CFH-12
dosed to the mobile P pool in the entire plow layer requires 29 tons CFH-12·ha$^{-1}$. Applying CFH-12 in
amounts customized to the P release of soils may reduce the expenses of the pretreatment considerably.
Assuming CFH-12 binding P in a Fe:P ratio of 10:1 [63], 0.3 tons CFH-12·ha$^{-1}$ should be adequate to bind
the P released from the control soil during the flooding experiment. Commercial Fe-based products
were comparable in price to Al-based products per unit bound P, both representing approximately 20%
of the costs of products using La as the P-binding component [40,63]. However, the acquisition costs of
chemical immobilization could be markedly reduced if P-sequestrating industrial by-products, such as
water treatment residues and ochre sludge [59–61], could replace commercial products.

5. Conclusions
Sand-capping, depth-plowing, and CFH-12 pretreatment could reduce P release when establishing
new lakes on former agricultural soil. The pretreatments represented cheaper alternatives compared
to topsoil removal and could enable nature reservation projects previously considered to pose too
high a risk of P loss to downstream ecosystems. Depth-plowing was very cost-effective, but the
effect depends on the P and Fe distribution through the soil profile and would be expected to be
most efficient on stratified soils with P accumulated in the topsoil. Sand-capping and chemical
immobilization by CFH-12 addition was found to be as effective but more expensive and should be
applicable independently of the soil P distribution. The P release of the untreated agricultural soil was
limited to the first 100 days of inundation and only a small fraction of the potentially mobile P pool
of the soil was released, indicating that some agricultural land could be inundated without posing
a threat of severe nutrient release and that the potentially mobile P pool may be overestimated by
the extraction procedure. Moreover, the dissolution of Fe from CFH-12 was limited, suggesting that
the product could be applied to alleviate P release from shallow lakes without immediate risk of P
desorption and toxic effects. Soil pretreatment of all three methods affected the N exchange between
soil and water; most likely by limiting the availability of labile organic compounds and CFH-12 may
initially constitute an electron source increasing ammonification.

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