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TITLE

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SAMMENDRAG:

Avløpsvann og mekanisk filtrert slam fra resirkulerende akvakultursystemer (RAS) inneholder betydelige konsentrasjoner av nitrogen og fosfor og kan utnyttes direkte som gjødsel, f.eks. i hydroponiske systemer. Når dette ikke er mulig, må næringsstoffene fjernes og brukes som gjødsel andre steder, men dette innebærer ofte kostbar transport. For å øke mengden næringsstoffer per kg materiale som skal transporteres, f.eks. til jordbruksarealer, kan slammet filtreres ved hjelp av forskjellige metoder. Kjemiske koagulanter og flokkuleringsmidler brukes ofte til å fjerne (oppkonsentrere) partikler og næringsstoffer. I et gjødselperspektiv kan dette være negativt fordi kjemikaliene gjerne inneholder aluminium som gjør fosforet i slammet mindre tilgjengelig for planter. Denne rapporten beskriver innledende forsøk med fiskeslam tilsatt de naturlige polymerene lignocellulose og cellulose som alternativ til kjemiske flokkulanter og koagulanter, utført ved Danmarks Tekniske Universitet (DTU) Hirtshals, og videre testing av filterkakene som substrat for kompostering utført ved NORSØK, Tingvoll.

Den optimale sammensetningen av lignocellulose og cellulose som brukes til filtrering av fiskeslam ble bestemt ved å filtrere 1 L slam med 4,85 g TCOD/L (COD= kjemisk oksygenforbruk, «chemical oxygen demand») med kombinasjoner av lignocellulose (20, 25, 30, 35 g/L) og cellulose (0,5, 0,75, 1, 2 g/L). En blanding av 0,75 g cellulose og 35 g lignocellulose per liter ga best filtrering. Denne kombinasjonen ga en filterkake med et tørrstoffinnhold på 24 - 29 %, som inneholdt mesteparten av næringsstoffene i slammet (nitrogen og fosfor, N og P). Vekten av materialet som inneholdt N og P ble redusert med mer enn 80 % ved filtreringen.

To typer filterkaker ble produsert ved DTU Hirtshals med disse naturlige polymerene, med og uten aluminium-basert koaguleringsmiddel. Kakene ble frosset og fraktet til NORSØK, Tingvoll hvor de ble blandet med fiskemel for å lage partier med et C/N-forhold på 55, 30 og 20. Den termofile nedbrytningen og CO_2-respirasjonen i de ulike filterkakene (totalt 6 behandlinger) ble undersøkt i Dewar-flasker. Blandingene uten kjemisk koaguleringsmiddel var i gjennomsnitt varmere og slapp ut mer CO_2. Blandinger med C/N-forhold på 30 hadde sterkest varmeutvikling, etterfulgt av 20 og 55. Blandinger med C/N forhold på 20 og 30 slapp ut mer CO_2 enn de med C/N 55. Det var imidlertid bare blandinger med koagulant (C/N 20 og 30) som nådde det termofile temperaturområdet, dvs. at temperaturen kom opp i over 55 °C. Respirasjonen som ble målt reflekterte temperaturutviklingen i behandlingene.

Bruk av lignocellulose og cellulose som filtreringsmidler kan øke næringsgjenvinningen fra RAS-avløp samtidig som vekten av det oppsamlede slammet reduseres. Dette kan redusere kostnader forbundet med transport. Bruk av slike naturlige polymerer til å filtrere slam fra RAS-anlegg gir videre en biologisk nedbrytbar filterkake, som egner seg til kompostering. Det vil være av interesse å videreføre forsøk i en større skala, og undersøke kompostens egenskaper som jordforbedringsmiddel.

SUMMARY:

Effluent water and mechanically filtered sludge from recirculating aquaculture systems (RAS) contain significant concentrations of nitrogen (N) and phosphorus (P) and may be applied directly as fertiliser, e.g. in hydroponic systems. However, where this is not possible the nutrients must be removed and should ideally be applied as fertiliser elsewhere, but this often involves costly transportation. To increase the amount of nutrients per kg of material to be transported, e.g. to agricultural fields, the sludge may be filtered using various methods. Chemical coagulants and flocculants are commonly applied to aid particle and nutrient removal. This report describes initial trials with fish sludge amended with the natural polymers lignocellulose and purified cellulose as filter materials, conducted at Denmark Technical University (DTU) Hirtshals, and further testing of the filer cakes as substrates for composting conducted at NORSØK, Tingvoll.

The optimal composition of lignocellulose and cellulose to be used for filtration of fish sludge was determined by filtering 1 L of sludge at 4.85 g TCOD/L (total chemical oxygen demand) with combinations of lignocellulose (20, 25, 30, 35 g/L) and cellulose (0.5, 0.75, 1, 2 g/L). A minimum ratio of 0.75 g of cellulose to 35 g of lignocellulose per L of sludge was required for filtration. This combination could produce a filter cake with a dry matter content of 24 - 29%, containing most of the nutrients (N, P). The weight of material containing N and P was reduced by more than 80% by the filtration.

Two types of filter cake were produced at DTU Hirtshals with these natural polymers, with and without aluminium coagulant. The cakes were frozen and transported to the Norwegian Centre for Organic Agriculture, Tingvoll where they were amended with fish meal to make batches with a C/N ratio of 55, 30, and 20. The thermophilic decomposition and CO_2 respiration of these batches were studied in Dewar flasks. The mixtures without chemical coagulant were on average warmer and emitted more CO_2, and mixtures with C/N ratios of 30 were the warmest, followed by 20 and 55. However, only mixtures with coagulant (C/N 20 and 30) reached the thermophilic range (> 55 °C). Respiration fluxes and rates generally reflected the thermodynamics of the treatments. The treatments without coagulant emitted more CO_2 than those with, and the treatments with C/N ratios of 20 and 30 emitted more than those with C/N 55.

Utilising lignocellulose and cellulose as filter materials may increase nutrient recovery from RAS systems, while reducing the weight of the collected sludge as compared with traditional methods. This may reduce the costs associated with transportation and produce a biodegradable filter cake that is conducive to composting. Further trials should be conducted to test composting on a larger (practical) scale, and the quality of such compost for soil amendment.

COUNTRY: Denmark and Norway

APPROVED	PROJECT LEADER
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Preface

This report is written as a deliverable from the project "Sustainable utilization of MARine resources to foster GREEN plant production in Europe" (MariGreen, 2021-2024). Seaweed, residues from captured fish, and blue mussel residues, in addition to "manure" from cultivated fish, are relevant for the trials in MariGreen, aimed at developing blue fertilizers for green horticulture. Whereas fish sludge and silage from dead fish (animal byproducts of category 2) are not permitted for use in certified organic growing, Denmark has a significant certified organic production of rainbow trout. It seems reasonable to test how sludge from such production could be treated to recover important nutrients, especially phosphorus and nitrogen, aimed at application as soil amendments or fertilizers applied in organic growing. The industry partner AlumiChem participates actively in MariGreen to develop innovative filtering materials for RAS (recirculating aquaculture systems) and contributed actively to producing filter cakes for testing at Denmark Technical University (DTU) Hirtshals in August 2023. The filter cakes were further tested at NORSØK, which has been responsible for establishing methods for the successful composting of marine-derived residual materials in MariGreen, such as seaweed and fishbones. A composting test with two types of filter cake was conducted from November 2023 -January 2024. This report presents our findings.

Thanks to initial efforts by Hui Ching Seow, the report includes an introduction describing the general characteristics of the applied materials and filter cakes, that she produced during an internship at DTU Hirtshals in August 2023.

Tingvoll, 16.05.24

Anne-Kristin Løes

Leader of MariGreen WP3: Blue biomass valorisation

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1 Introduction

1.1 Fish sludge valorisation

Sludge (composed of fish faeces and uneaten feed) collected from recirculating aquaculture system (RAS) plants in Denmark and Norway is commonly treated in biogas plants (Letelier-Gordo et al., 2020). However, as fish sludge has a high water content, the transportation is energy-intensive and costly (Brod & Øgaard, 2021) and techniques are required to reduce the water content, without losing nutrients.

The high nutrient content of fish manure also creates an opportunity to achieve nutrient circularity by recycling fish waste products for agriculture. In one Norwegian study, dried fish sludge had a relative agronomic efficiency of 50-80% compared with mineral fertiliser (Brod et al., 2017). Nitrogen (N) and phosphorus (P) fertiliser effects were also found with the application of sludge from salmon and catfish on ryegrass (*Lolium multiflorum* L.) and lettuce (Brod et al., 2015; Celis et al., 2008; Yogev et al., 2020).

The European Commission set a target of having at least 25% of the EU's agricultural land to be used for organic farming and to significantly increase organic aquaculture by 2030 under the European Green Deal Farm to Fork strategy (European Commission, 2021). In line with the objectives of the European Green Deal and the Circular Economy Package, Regulation (EU) 2019/1009 on fertilizers allows for more organic and waste-based fertilisers to be freely traded within the EU (European Commission, 2022). Increasing market access for such fertilisers encourages the recycling of materials derived from poorly utilised or wasted organic materials. This may reduce the dependency of the EU on raw material imports. It has been estimated that recycled bio-waste fertilisers could substitute up to 30% of inorganic fertilisers from the current 5% (European Commission, 2016). This will generate economic opportunities and improve the sustainability of the fertiliser industry.

Nutrient recycling of fish sludge may be achieved via anaerobic digestion, composting, and direct field application (Khiari et al., 2019). One bottleneck in the valorisation of fish sludge is the transportation to fields or the relevant recycling facilities. The high water content increases transport costs and energy demand. As an example, in a RAS fish sludge with 13% dry matter (after mechanical filtration by Salsnes filter), 79% of the dry matter was organic matter, whereas 8% was nitrogen (> 90 % organically bound), 4% calcium, and 2% phosphorus (Brod et al., 2017). Hence, the slurry contained only 11 kg N per ton (1.1%), which is little compared with slurries from animal husbandry or digestate from source-separated organic waste. Effective solid-liquid separation techniques such as drum filters, and filtration with filter aids coupled with flocculation and coagulation are needed to transform fish sludge into a lighter product with a higher concentration of nutrients per unit of volume (Brod & Øgaard, 2021).

1.2 Filtering materials in RAS

Whereas commercial RAS plants often apply various filtering systems to concentrate the sludge, filtering aids may enhance the trapping of nutrients and organic matter. Filter aids may form a "protective layer" on the surface of the filter mesh, where particles can be deposited on the surface and within its porous structure (Bächle et al., 2021). The resulting filter cake will have increased porosity as compared with a "cake" (sludge) without filter aid. The filter aids can be added to the suspension (e.g., RAS water, or RAS sludge after mechanical filtration) before filtering. The particles in the suspension should adhere to the filter aid and form a lower specific cake resistance increasing filtration and quality of the filtrate (Heertjes & Zuideveld, 1978). Alternatively, a precoat material is placed on the filter mesh, and particles from the fish sludge are separated on and in the precoat material to make a fish sludge cake. The precoat material should be as open-pored as possible to prevent clogging and utilize the full volume of the pre-coat material for making the fish sludge cake (Bächle et al., 2021).

One option for bio-based pre-coat materials is to use plant-based cellulose, hemicellulose, and lignin in various proportions. Plant biomass may be referred to as lignocellulose. Lignocellulosic materials (e.g., sawdust) are suitable as a precoat due to their fibrous structure which results in an open-pored cake. In addition, lignocellulose is readily available, inexpensive, biodegradable, and can be easily modified thermally to produce high-value products (Chakhtouna et al., 2022). The use of lignocellulose and its composites in wastewater purification applications has been examined (Chakhtouna et al., 2022). Lignocellulosic materials have a high carbon content, which facilitates further treatment by composting.

Purified cellulose may be added as a supplementary filter aid to mechanically reinforce the filter cake and improve the adhesion of the pre-coat material (Braun et al., 2011). Cellulose is a natural flocculant, which can remove metal ions and organic debris efficiently from water due to the abundance of hydroxyl groups (OH^-) on the linear polysaccharide chains, providing cellulose with a strong chelating impact (Fauzani et al., 2021). Traditionally, cellulose had limited use as a flocculant due to low solubility and chemical reactivity. However, studies using modified nano cellulose have shown to be effective in removing suspended matter (Fauzani et al., 2021). The addition of cellulose into the filter cake may assist in the flocculation of particles and improve overall filtration effects.

1.3 Composting of fish sludge

For successful composting, the feedstock mixture should have a C/N ratio between 15 and 35, a moisture content between 45 and 60% (corresponding to 40-55% dry matter), and a pore space between 5 and 15% (Roman et al. 2015). Mechanically filtered RAS sludge is not well suited for composting alone, as the C/N ratio is usually low, e.g. 2.8 in the sludge analysed by Brod et al. (2017), referred to above for P and N contents. High volumes of solid materials with high C content would be required to balance the C/N ratio. However, if cheap, carbon-rich biomasses can be found that are efficient at adsorbing nutrients from RAS effluent water and/or sludge, composting the filter cake may be an option to reduce its weight and convert it into a soil amendment. At the Conservation Fund Freshwater Institute in West Virginia, USA, aquaculture sludge composting and co-composting are currently being studied, using an in-vessel composting system (Choudhury et al., 2023). Dewar flasks are commonly applied to test the temperature development of substrates applicable for composting and have been used for composting various blue materials as a part of the MariGreen project (Cabell and Løes 2023).

1.4 Purpose of study

The aims of the studies described in this report were:

- to identify the optimal amount and composition of lignocellulose and cellulose needed for a filter cake to effectively filter RAS sludge
- determine the nitrogen and phosphorus concentrations in the resulting filter cakes
- assess the behaviour of filter cake produced with and without aluminium coagulant during composting

The first two bullet points were studied at DTU, whereas the third was studied at NORSØK, using filter cakes produced at DTU.

2 Material and methods

2.1 Precoat materials, fish sludge, and their optimal ratios

Fish sludge was collected at DTU Hirtshals from a RAS tank with young salmon smolt fed by certified organic feed (Picture 1). In a series of initial optimization trials, sludge with a total chemical oxygen demand (TCOD) of 4.85 g /L was filtered, using various combinations of amounts of precoat materials:

- lignocellulose 20, 25, 30, and 35 g per L sludge
- additionally cellulose 0.5, 0.75, 1, and 2 g per L sludge

The lignocellulose (LIGNOCEL®) is produced from hardwood and softwood and was obtained from J. Rettenmaier & Söhne GMBH + Co KG, Germany. The material resembled sawdust made from coniferous wood applied as bedding material for animals.

The cellulose (Arbocel®) is a highly purified cellulose with a particle size of 180 µm, made from perennial plants, developed by J. Rettenmaier & Söhne GMBH + Co KG, Germany.

The fish sludge that was applied for the optimization trials had 0.2 ± 0.04 g total N and 0.25 ± 0.01 g total P per L and contained 1.77 ± 0.3 % dry matter (DM, n=2).

Picture 1 From left: Sludge from a RAS tank with rainbow trout (body size ca. 150 g) fed by certified organic fish feed, compiled in a sludge cone, ready for production of filter cake. Middle: Filter cake from woody material (lignocellulose). Right side: Detail of filter cake. Photos: Carlos Letelier-Gordo.

To study the ability of the filter cake materials to accumulate nutrients and organic matter, the filtrates were analysed for concentrations of nitrate N and total P, as well as total and soluble chemical oxygen demand (COD). Filter cakes were weighed and analysed for dry matter, ash (by loss on ignition), organic C, total N, and total P. The analyses were conducted at DTU Hirtshals.

Cakes containing < 35 g lignocellulose and < 0.75 g cellulose were too moist and were not subject to further analyses. Hence, all results reported in chapter 3.1 refer to filter cakes where 35 g of lignocellulose combined with 0.75, 1 and 2 g of cellulose were applied to treat 1 L of fish sludge. The filter cakes will be referred to as C0.75, C1, and C2, with the number corresponding to the amount of cellulose used in the filter cakes.

2.2 Filter cakes prepared for compost tests

In the laboratory of DTU, Hirtshals, two types of filter cake were prepared on August 25, 2023. For both cakes, about 35 g lignocellulose material and 6 g cellulose were applied per L of sludge (see photo on the report's front page). For the first batch, we used 1 kg lignocellulose, 170 g cellulose, 3 ml coagulant, and 100 ml flocculant for 30 L of sludge. For the second batch treated without chemical coagulant and flocculant, the amount of cellulose was doubled for the same amount of sludge.

The sludge utilized for the first filter cake batch had 0.65% DM, 0.09 g N per L, and 0.11 g P per L.

The sludge utilized for the second filter cake with coagulant had 0.74 ± 0.25% DM, 0.21 ± 0.11 g N per L, and 0.125 g P per L (n=2).

To prepare the filter cakes, a 50 L tank with a mixer was used to blend the fish sludge, lignocellulose, and cellulose, and for the first batch of filter cake, a chemical coagulant and a flocculant. For this batch, the coagulant was added first, then the flocculant, and mixed with a mechanical mixer for about 5 minutes until large clumps formed. The cellulose was added next and finally the lignocellulose. These were mixed in by hand before running the mixer again for about 10 seconds. An air-driven pump displaced the mixture into a filter chamber originally designed to filter milk. The effluent was circulated back into the tank and after about 15 minutes of filtration, it became clearer. The effluent was then released into a drain and the pump was stopped when the tank was empty. The filter cake was removed from the filtration chamber by hand.

In batch two, the cellulose was added first, then the lignocellulose, while mixing. The sludge was then pumped through the filter as with the first batch. The sludge applied for the filter cake without coagulant contained somewhat more feed residues and hence had a slightly different chemical composition.

Filter cakes were wrapped in plastic bags and frozen before transport to NORSØK. Before testing, representative samples were sent for chemical analysis at Nemko Norlab, Namsos, Norway. Dry matter (%), total C, total N, and elemental composition (plant macro- and micronutrients, potentially toxic elements, etc.) were measured. We have presented results for P, K, Na, and Al (Table 2); further results are found in Appendix I.

2.3 Compost tests with filter cakes

Analytical results for C and N were used to assess the C/N ratios of the filter cakes (see Table 2):

- Filter cake without coagulant (FC) had C/N = 40/0.41 = 98 and a DM% of 28.
- Filter cake with coagulant (FCC) had C/N= 41/0.75 = 55 and a DM% of 25.

For FC, the amount of material allowed for a comparison of 3 treatments with 2 replicates (n= 6 Dewar flasks). For FCC, 3 treatments were compared with 3 replicates (n = 9). Altogether, 15 flasks were used in the test. For each filter cake, the treatments were C/N = 55, C/N = 30 and C/N = 20, with treatment codes FC55, FC30, FC20 and FCC55, FCC30 and FCC20. The C/N was adjusted by the addition of milled, dried cod ("kleppafisk"), which is comprised of fish that are too small to be processed as saltfish (< 2.5 kg). This material had 89% dry matter, 36% total C, and 10.2 % total N (of DM), giving a C/N of 4.

For the FC, portions of 2400 g of filter cake were amended with 21 g fish meal to derive C/N = 55, 66 g to derive C/N = 30, and 122 g to derive C/N = 20. This corresponds to 8.8 g of fish meal per kg of fish cake material for FC55, 27.5 g for FC30, and 51 g for FC20. To compensate for the increase in DM% caused by the addition of fish meal, a similar amount of tap water as the applied amount of fish meal was added to each material batch. In practice, this amount of water was a bit too high (Table 3), and caused suboptimal conditions for successful composting, especially for FC which was already wetter than FCC.

For the FCC, portions of 3400 g of filter cake were amended with 0 g of fish meal to derive C/N = 55, 71 g to derive C/N = 30 and 170 g to derive C/N = 20. This corresponds to 21 g per kg for FCC30 and 50 g per kg for FCC20.

The amounts of materials required per treatment were well mixed, before filling the filter cakes (with fish meal) carefully into Dewar test flasks (volume 2075 cm^3) until they were full, on November 21, 2023. The flasks were gently tapped on the palm of the hand 2 times during the filling, once when 1/3 full and again when 2/3 full, to standardize the compaction of the materials. The bulk density was recorded by weighing the filled flask and dividing it by the flask volume. As expected, the bulk density increased with a decreasing C/N ratio because of a much higher bulk density of the fish meal (805 g/L) than fish cake (500 g/L), and with the added water in the treatments with fish meal (Figure 1).

The DM (%) and loss on ignition (LOI) of the initial materials applied in the flasks, and of the contents of each flask on December 6, December 21, and January 15, were measured at NORSØK by drying samples of 5 g of material at 105 °C until constant weight, and then igniting them at 550 °C for 180 minutes. Weights were recorded before and after drying and ignition.

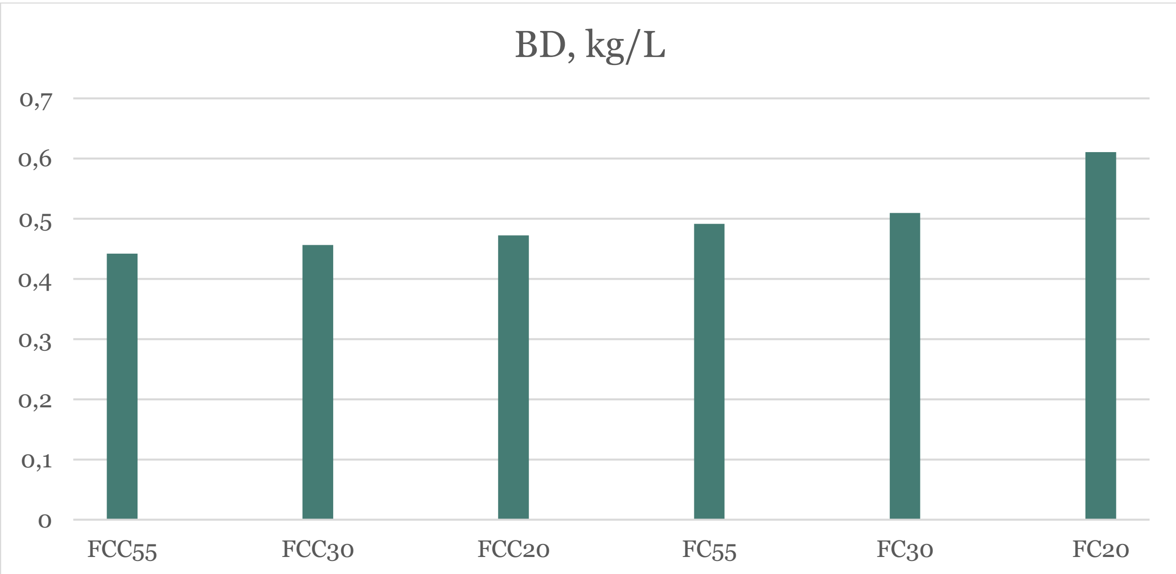

Figure 1. Bulk density (BD, kg/L) of fish cake with coagulant (FCC) and without (FC), amended with fish meal to obtain C/N ratios between 55 and 20, after filling the materials into Dewar flasks with a standardized compaction procedure.

The temperature was recorded every 6 hours by an automatic logger in each test flask. Technical details about the experimental setup are provided in Cabell & Løes (2023).

The flasks were emptied and filled again on December 6 and again on December 21, when the temperature in the flasks declined. After mixing the content of each flask thoroughly in a plastic tray, 5 g material per flask was sampled to determine dry matter and loss on ignition. The weight loss between each emptying/filling of the flasks was computed, correcting for the amounts of material removed for measurements and the small amounts of material sticking to the plastic tray. The plastic tray was rinsed with tap water, and this water was collected and sanitized by boiling before disposal. The experiment was completed on January 15, 2024.

The respiration of CO_2 was recorded just after the onset of the experiment on November 23, on December 6 (before emptying the flasks), on December 7, 8, 11,13, 18, 20, 22 (2023), and finally on January 2, 2024. The respiration in each flask was recorded using a wireless Pasco CO_2 sensor (Rittl et al., 2023). The sensor has a one-point calibration in the software with a default value of 400 ppm. The wireless CO_2 sensor was operated through a tablet (Picture 2) using the SPARKvue software. The measurements were done by gently pressing a plastic bottle where the bottom was removed about 1 cm down in the content of the flask and measuring the CO_2 emission every 15 seconds over 3 minutes per flask, giving a total of twelve measurements on each measurement date. The measuring bottle was thoroughly rinsed by moving it rapidly up and down in fresh air between each new measurement. CO_2 concentrations were calculated and expressed per g of dry matter measured near the respiration recording day (average value per treatment). Accumulated CO_2 emission was calculated by adding up daily CO_2 emissions over time.

Picture 2 Dewar test flasks for measurement of self-heating in fibre cake material from lignocellulose and fish sludge, with measurement of respiration in the bottle in the bottom left corner. The tablet shows the emission curve (respiration). Photo by AK Løes, December 6, 2023.

3 Results

3.1 DTU trial: Optimizing the ratio of precoat material to fish sludge

The test indicated that > 30 g of lignocellulose as a precoat was required to filter 1 L of fish sludge within 10 minutes (Figure 2, left side). 30 and 35 g of lignocellulose with 2 g cellulose could hold 150 g and 100 g of water and fish sludge particles, respectively (Figure 2, right side). This shows that the weight of the filter cake with 35 g lignocellulose was lower than for 30 g lignocellulose. This demonstrates that the 35 g treatment had a lower water content, suggesting that this amount was the most optimal amount to filter out 1 L of sludge at a total chemical oxygen demand (TCOD) of 4.85 g/L.

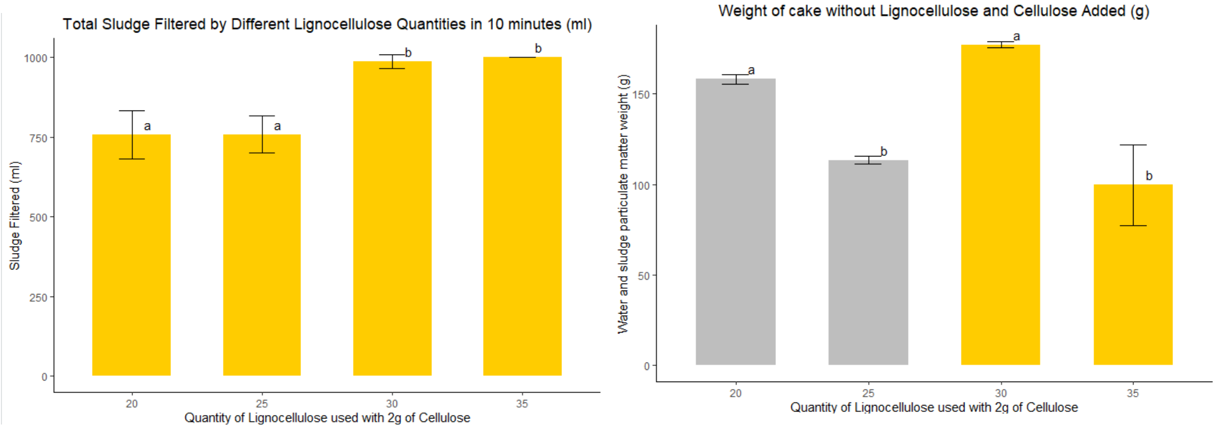

Figure 1 (Left) Amount of sludge filtered (max 1 liter) in 10 minutes for different lignocellulose quantities with 2g of cellulose (mean ± standard deviation, n=3). (Right) weight of the water and sludge particulate matter for different lignocellulose quantities.

All cake combinations using 35 g lignocellulose with an addition of 0.5 - 2 g of cellulose could filter out 1 L of sludge within 10 minutes (Figure 3, left side). However, filter cakes with 1 - 2 g of cellulose were lighter than filter cakes with less cellulose (Figure 3, right side). The average weight of the filter cake including 35 g of lignocellulose and 0.75 g of cellulose was 193 g.

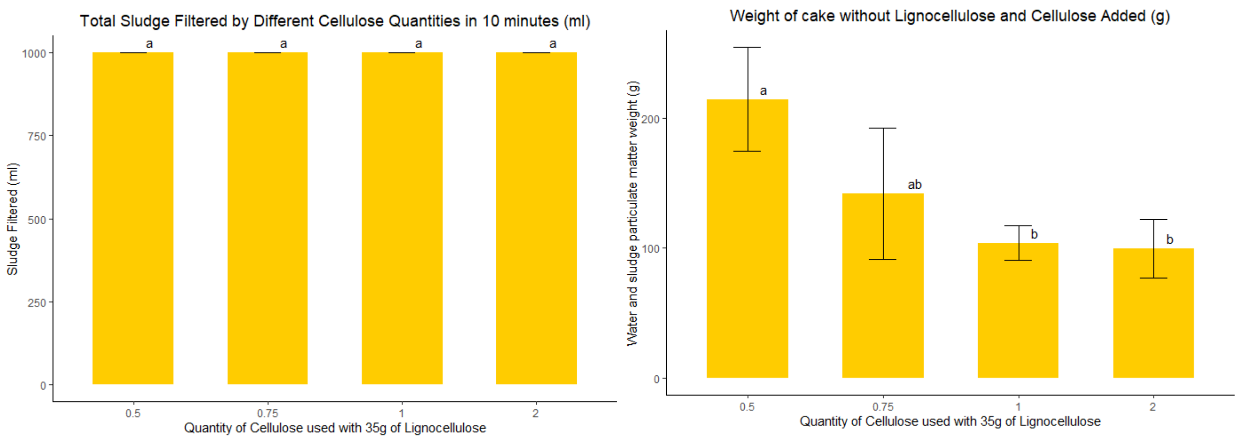

Figure 2 (Left) Amount of sludge filtered (max 1 L) in 10 minutes for different cellulose quantities with 35 g of lignocellulose (mean ± standard deviation, n=3). (Right) Weight of the water and sludge particulate matter for different cellulose quantities with 35 g of lignocellulose (mean ± standard deviation, n=3). Letters denote statistically significant differences between treatments ($p < 0.05$).

The filter cakes had 24-29% dry matter (DM) and there were no significant differences in DM, ash, or organic matter content with different additions of cellulose (Table 1). For total N (TN), there was a tendency for increased concentration with more cellulose (p=0.07). For total P (TP), the concentration was lower with 2 g cellulose.

Table 1. Proximate composition of filter cakes using 35 g of lignocellulose per L, and varying cellulose weights from 0.75 to 2 g per L (C0.75, C1, C2).

Characteristics	C0.75	C1	C2	p-value
DM%	24.1	29.0	29.0	0.307
Ash %	1.66	2.19	2.14	0.149
Organic matter %	22.4	26.8	26.9	0.328
Total N %	0.106	0.120	0.144	0.071
Total N g kg^{-1} DM	4.4	4.1	5.0	
Total P%	0.210	0.256	0.114	0.007
Total P g kg^{-1} DM	8.7	8.8	3.9	

The sludge contained 0.32 g total N, 0.65 mg P, and 14.5 g DM per L. The filtrate contained 0.05 g total N, 0.06 mg P, and 5.4 g DM per L. The mass balance for the C2 filter cake shows that more than 75% of the nitrogen and phosphorus were captured in the fish cake (Figure 4). This is similar to results from a larger scale test done with 30 L sludge, amended with 1 kg lignocellulose and 175 g cellulose (=33 g lignocellulose + 5.8 g cellulose per L), in which about 80% of the N and P was retained in the filter cake. This shows that this combination of filter aids could significantly lower the weight of sludge to be transported by 80%, i.e., less than 200 g compared with 1 kg.

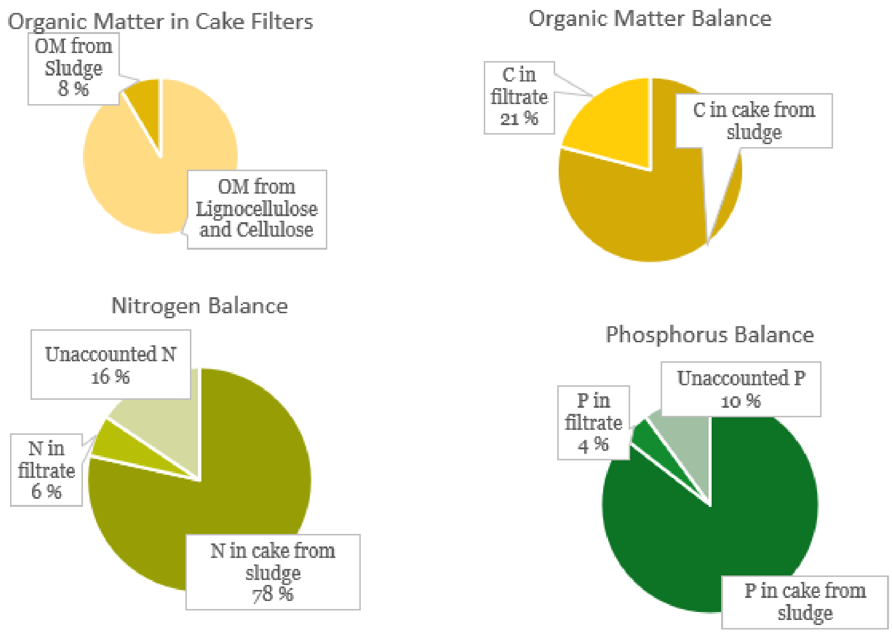

Figure 3. Ratios of organic matter, carbon, total Nand total P in sludge and filtrate with about 33 g of lignocellulose and 5.8 g cellulose per L as filter precoat.

3.2 Chemical composition of filter cake applied for compost tests

The chemical composition of the filter cakes is shown in table 2. Two different batches of sludge were applied to produce two types of filter cake: one without added coagulant (FC), and one with added coagulant (FCC). The sludge batches came from the same RAS tank, but the batch applied for FC contained significantly less feed residue. The FCC contained significantly more N and P than the FC per kg of dry matter. This may be a combined effect of higher nutrient concentrations and a higher retention effect with coagulant.

The concentrations of total N and P (g kg^{-1} DM) are roughly comparable in the fish cakes prepared for the compost tests (Table 2) and the cakes analysed at DTU Hirtshals to study the optimal proportions of lignocellulose and cellulose (Table 1), which used a sludge containing 0.2 g total N and 0.25 g total P per L.

The addition of coagulant seemed to increase the dry matter content of the cake by 3%. The C/N ratio of FC was 98, and of FCC it was 55. To achieve C/N ratios of 55, 30 and 25, the fish cake material was mixed with fish meal as described in Chapter 2.3.

Table 2. Concentrations of plant macronutrients, micronutrients, potentially toxic elements etc. in filter cake (FC) without (FC) and with coagulant (FCC), compared with the N and P concentrations in the pre-filtered sludge.

Characteristics	FC	FC sludge	FCC	FCC sludge
DM%	25		28	
Total C % (DM)	40		41	
Total N (g kg^{-1} DM)	4.3	0.09 g/L	7.5	0.21 g/L
Total P (g kg^{-1} DM)	2.9	0.11 g/L	4.2	0.125 g/L
Total K	0.16		0.14	
Total Na	3.9		6.8	
Total Al (mg kg^{-1} DM)	95		690	

3.3 Dry matter content, ash content and weight loss in compost flasks

The initial DM% of FC and FCC, as measured by Nemko Norlab (Table 2), were 25 and 28%. The addition of fish meal, with 89% DM, would in theory increase the DM% slightly, but since a similar amount of water was applied, the DM% of the batches put into the flasks on 21.11.2023 decreased with higher C/N ratio values (Table 3). The dry matter content in materials subject to thermophilic decomposition under aerobic conditions typically increases because water is evaporated due to heat generation. This is especially the case in windrows exposed to wind and sun. In our flasks, evaporation (and heat loss) was restricted by a fleece cover (Picture 1). During the decomposition of organic matter, the dry matter content may actually decrease because decomposition is linked with liquefaction. These processes may counteract each other, making the interpretation of changes in humidity during composting quite complicated.

At the onset of the experiment, the moisture content was 72-76% for FCC and 83-74% for FC treatments. This is well above the optimal moisture content level of 45-60% (Roman et al. 2015), but it did not hamper a significant heat development in all treatments (Figures 6-9).

After the first round of heat generation, marked by the mixing of the contents and re-filling of the flasks on December 6, the moisture content had increased by 4-5% in the C/N 55 fish cakes. In the C/N 30 fish cakes, the moisture content was almost the same as at the onset. In the C/N 20 fish cakes, the moisture content had decreased by 2-3%. If this decrease was due to high temperature, it should also have been found for C/N 30 cake materials since the temperature curves in C/N 20 and C/N 30 cakes were comparable (Figure 7). However, in C/N 30 the desiccation process with high temperature may have been counteracted by more decomposition. As shown in Figure 5, the total weight loss over rounds 1 and 2 was somewhat higher in the C/N 30 cakes than for C/N 20 cake materials.

During the second round of composting (from December 6 to 21), the moisture content did not change much in the flasks. By January 15 when the flasks were finally emptied, the moisture content had decreased slightly in all the flasks (Tables 3 and 4), possibly indicating that materials dried out when bacterial activity (which may increase humidity) levelled off.

Table 3. Dry matter content (%) in filter cake with and without coagulant (FC, FCC) with various C/N ratios after amendment with fish meal and water. Dry matter content in FCC55 was measured by Nemko Norlab (see Table 2); other values were measured at NORSØK.

Treatment	21.11.23	6.12.23	21.12.23	15.01.24
FCC55	28.0	23.4	24.6	27.4
FCC30	23.4	24.4	25.0	27.5
FCC20	24.6	25.8	27.0	29.4
FC55	26.0	20.7	21.1	24.1
FC30	24.0	22.6	22.6	23.9
FC20	17.7	21.2	21.1	22.3

The amount (mass) of dry matter in each flask was calculated at the start of the experiment, at the times when the flasks were emptied and refilled, and when the experiment was completed (Table 4). As is expected, the amounts of DM generally decreased from start to completion, though at different rates. The DM mass in the two treatments with C/N 55 decreased most during the first phase of composting and had almost no decrease during the final phase. The other treatments lost DM mass more evenly throughout.

Table 4. Average values of dry matter (DM), in g in filter cake with and without coagulant (FCC, FC) with various C/N ratios after amendment with fish meal, at the start of the experiment (21.11.2023), on the two dates the flasks were emptied and refilled, and upon completion (15.01.2024).

Treatment	21.11.2023	06.12.2023	21.12.2023	15.01.2024
FCC55	257	186	167	166
FCC30	221	196	169	164
FCC20	242	208	187	181
FC55	265	181	156	157
FC30	254	249	209	161
FC20	225	239	212	192

Also as expected, the ash content (% of dry matter) increased with the addition of fish meal (Table 5). It also increased slightly over time until December 21 (and for FC55, even until January 15), demonstrating the loss of organic matter in the flasks.

Table 5. Average values of ash (% of DM, measured as 100 - loss on ignition) in filter cake with and without coagulant (FCC, FC) with various C/N ratios after amendment with fish meal, at the start, on two dates of emptying and refilling, and at the completion of the experiment.

Treatment	21.11.23	6.12.23	21.12.23	15.01.24
FCC55	Not measured	3.8	4.6	3.3
FCC30	5.4	6.8	7.3	6.9
FCC20	9.2	9.9	10.7	10.6
FC55	4.1	4.5	4.8	10.0
FC30	5.7	7.7	8.0	8.1
FC20	4.4	10.7	11.0	10.7

A significant weight loss occurred in the flasks during composting rounds 1 and 2 and until the completion of the experiment on January 15 (Figure 5). This may be due to water evaporation, but also a significant loss of carbon as shown by an increased proportion of ashes in the dry matter (Table 5) and accumulated respiration (Figures 10 and 11).

Figure 4. Weight loss (average values per treatment) in Dewar flasks over time, measured between dates when flasks were filled and emptied (and filled again). Treatments = filter cake with and without coagulant (FC, FCC) with various C/N ratios after amendment with fish meal.

3.4 Temperature development in compost flasks

We aimed for a temperature around 20 °C in the room where the Dewar flasks were kept. During two periods the temperature decreased to 14-15 °C (Figure 6). Across the three treatments conducted with each material, there was a clear difference between FCC and FC (Figure 6). On average, the FCC treatments reached almost 60 °C in the first composting round, whereas the FC treatments on average did not reach 50 °C. In the second round (December 6-21), and after the second mixing and re-filling of flasks on December 21, the situation was the opposite with somewhat higher temperatures in FC treatments (Figure 6). The mean temperature for the FCC-treatments was 30.9 °C and for the FC-treatments it was 33.6 °C. However, despite having a higher mean temperature throughout the experiment, none of the FC-treatments reached the thermophilic phase (>55 °C). FCC20 and FCC30 both almost reached 70 °C.

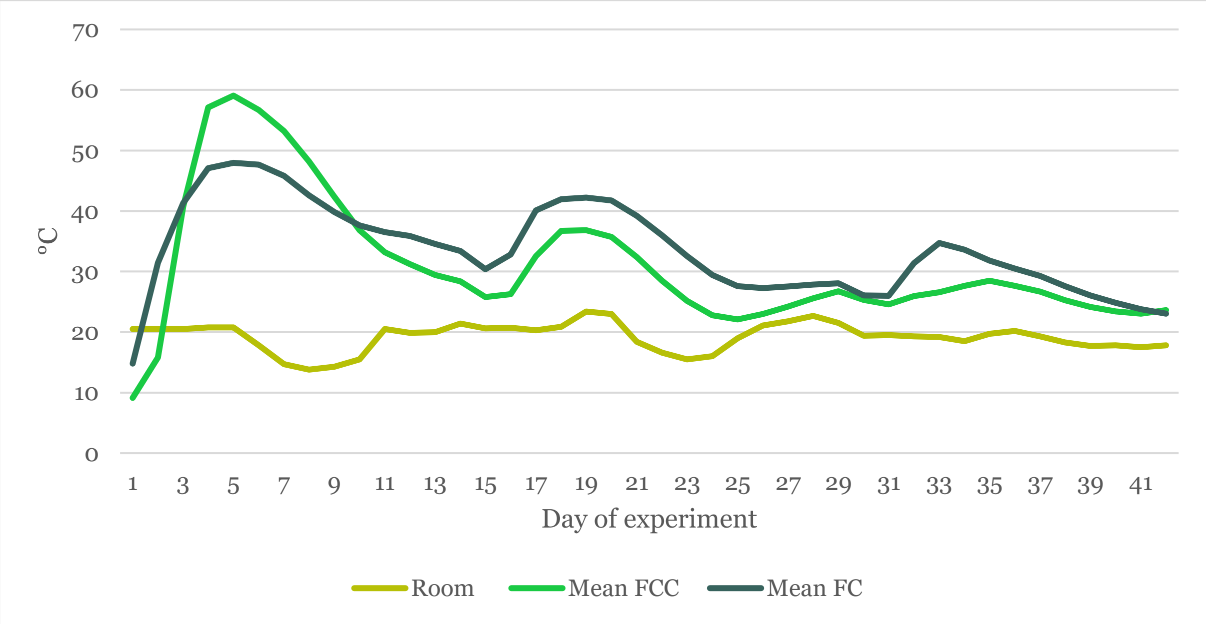

Figure 5. Mean values of daily temperature (°C) recorded 3 times per day in each Dewar flask, and in the room where the flasks were kept. Treatments = filter cake with and without polymer (FC, FCP). Average values across treatments with various C/N ratios after amendment with fish meal. Flasks were emptied, fish cake content mixed and filled into the flasks again on December 6 and December 21.

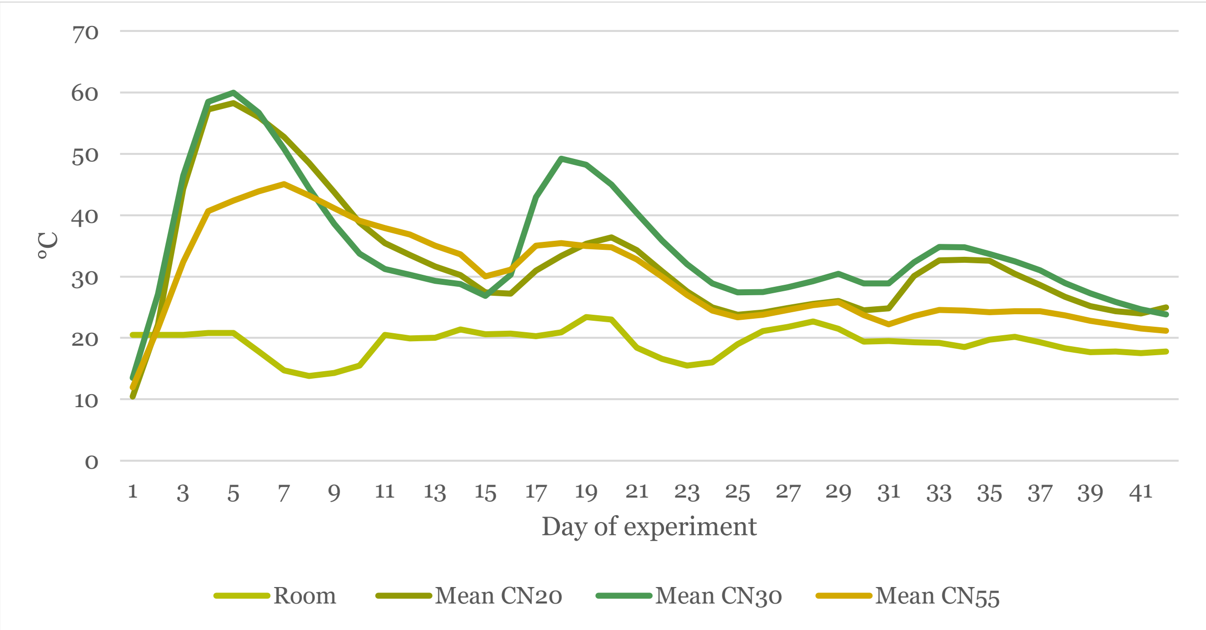

Figure 6. Mean values of daily temperature (°C) recorded 3 times per day in each Dewar flask. Average values for the three C/N ratios across treatments.

The thermodynamics was clearly affected by C/N ratio (Figure 7). For the FCC55, which was not amended with any fish meal, it seems that all available N was "consumed" quite rapidly during the first phase of heat initiation (Figure 8). Aeration during refilling did not cause significant new heat initiation, and the temperature was declining to become only 2-3 °C above room temperature. With C/N =30 and 20, significantly higher temperatures were reached in the first heating phase, but temperatures > 50 °C lasted only over 4-5 days. FCC30 had a stronger heat development than FCC20 in the second composting round. The explanation may be that a C/N ratio of 20 was too low to achieve good conditions for the thermophilic bacteria, and their activity may have been hampered by high concentrations of ammonia. There was indeed a strong smell of ammonia in these flasks during the emptying. The high moisture content can also be a factor. The mean temperatures for all treatments with a C/N ratio of 20, 30, and 55 were 32.3, 34.7, and 29.7 °C, respectively.

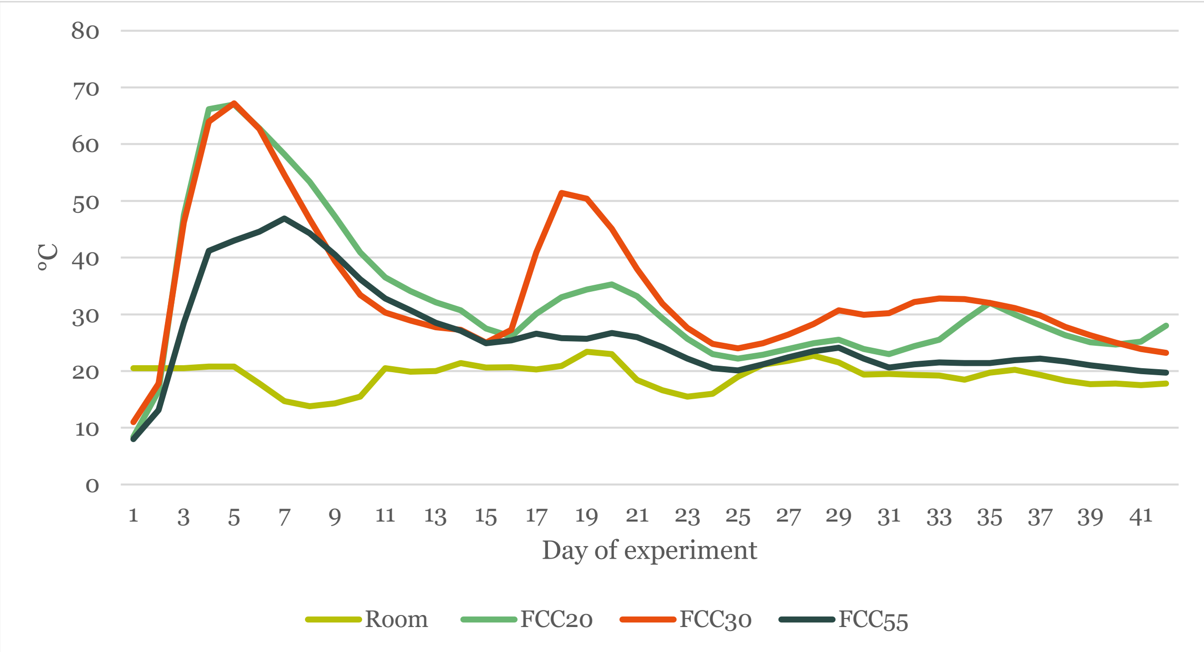

Figure 7. Mean values of daily temperature (°C) recorded 3 times per day in each Dewar flask, and in the room where the flasks were kept. Treatments = filter cake with coagulant (FCC). Treatments with various C/N ratios after amendment with fish meal.

In the fish cake with no coagulant, the pattern was a bit different. All treatments were amended with fish meal. This led to a long-term heat initiation in the FC55 treatment, which had temperatures > 40 °C until December 6 (Figure 9). The temperature was quickly back to the same level, but only for 6 days, whereafter the temperature rapidly declined, and the second refilling of flasks did not initiate a significant new heat development. Compared with FCC55, the temperature remained somewhat higher than the room temperature, about 7-8 °C. The temperature curves for FC20 and FC30 behaved comparably to FCP20 and FCP30. During the first round of heat initiation, they followed each other closely; during the second round there was significantly higher heat initiation with C/N=30, and after the second refilling, the curves were again quite similar for the two different C/N ratios. However, with the FC material, there was a clear phase of heat initiation after the second refilling which was not as evident for FCC. FC30 had the highest mean temperature of all treatments at 35.4 °C.

Figure 8. Mean values of daily temperature (°C) recorded 3 times per day in each Dewar flask, and in the room where the flasks were kept. Treatments = filter cake without coagulant (FC). Average values across treatments with various C/N ratios after amendment with fish meal.

3.5 Respiration

The respiration results are presented over the complete measurement period of 41 days (Figure 10), where day 1 = November 23, three days after the experiment was started on November 21, and day 41= measurements just before the final emptying of the flasks.

The treatment with the highest cumulative flux was FC20, followed by FC30, FCC30, and FCC20. Filter cakes with C/N 55 had significantly lower respiration rates. For each C/N level, the cakes without coagulant (FC) had a higher cumulative flux.

Another way of presenting respiration is per the mass of DM in the flasks on selected dates corresponding with filling, emptying, and refilling the flasks (Figure 11). The flasks were filled for the first time three days before the first respiration measurement on 23.11.2023. The measurement on 06.12.2023 was taken just before the bottles were emptied and refilled for the first time, and 07.12.2023 was the day after. 20.12.2023 was the day before flasks were emptied and refilled the second time, and 22.12.2023 was the day after. By this time there was little biological activity in the composts, except FC20, which also had a spike in the temperature following this refilling.

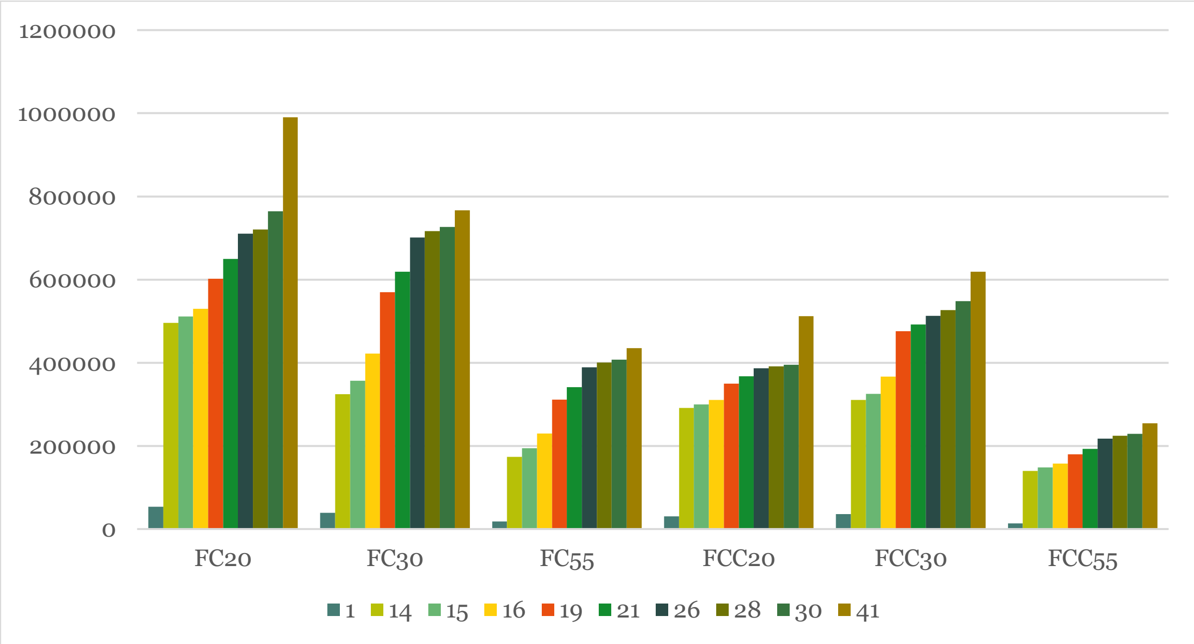

Figure 9. Respiration of CO_2 accumulated over time, presented as flux (mg CO_2 m^{-2}) from day 1 to 41, in Dewar flasks with filter cake without (FC) and with a coagulant (FCC) and for all C/N ratios.

Again, the results show that more carbon was emitted from the FC treatments compared with FCC treatments. This reflects the higher average temperatures in these treatments, but it may also be an effect of the higher moisture content in FC, the presence of coagulant in FCC, or possibly the greater amount of fish meal in FC treatments.

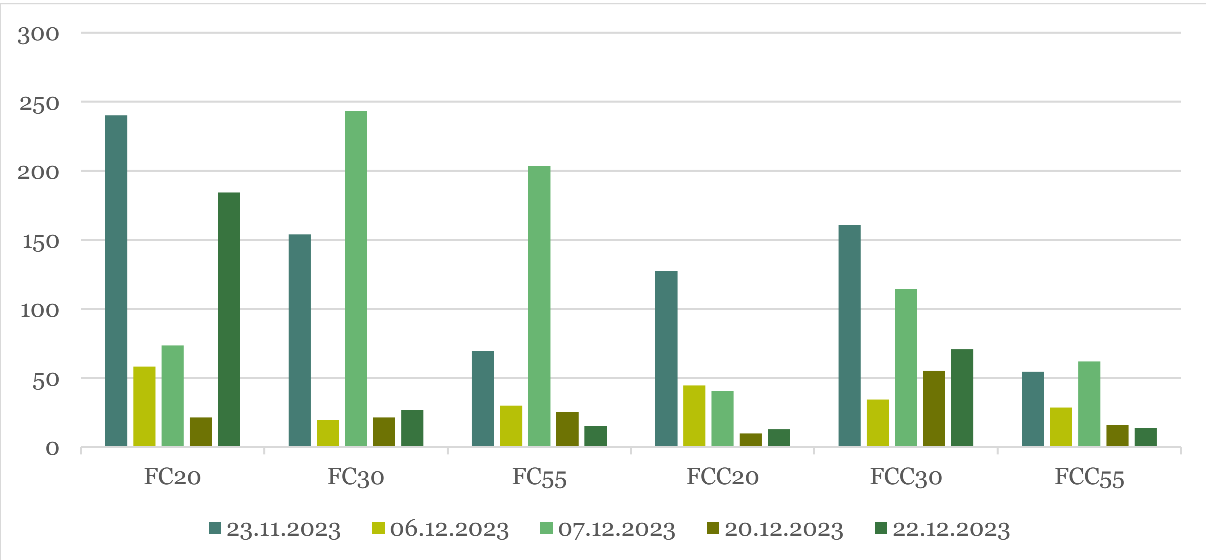

Figure 10. Respiration of CO_2 per gram of DM of feedstocks in the flasks on selected dates (mg CO_2 m^{-2} g^{-1} DM). Values are the mean of replicates for each treatment.

4 Discussion and concluding remarks

The results from both the filtration and compost experiments suggest that the combination of filtering RAS effluent with lignocellulose and cellulose with composting the filter cake can be an effective method to recover nutrients for returning them to the soil. Increasing the proportion of cellulose increased nitrogen recovery slightly but appeared to have had the opposite effect on phosphorus. While it is difficult to confirm due to differences in the effluent that was filtered, it appears that adding a coagulant also increases nitrogen and phosphorus recovery. Either way, the resulting filter cake is lighter than raw effluent and will significantly reduce transportation costs if nutrient recovery and recycling are the goals. While results from the filter cake experiments are promising, further experiments should be done to find the optimal combination of lignocellulose, cellulose, coagulant (alternatively with different types of coagulant), time, and flow for maximizing nutrient recovery.

The two filter cakes (with coagulant and without) behaved differently during the composting experiment, even when the C/N ratios were similar. Filter cakes with coagulant (FCC) were on average warmer during the first round of composting, while the ones without coagulant (FC) were warmer during the second and third rounds. C/N ratio was also a factor in the composting thermodynamics. The treatments with a C/N ratio of 20 and 30 were warmer than those with 55. C/N 30 maintained higher temperatures over a longer period. Results from respiration showed a similar trend for C/N ratios, with 20 and 30 releasing more CO_2 than 55. This comes as no surprise as respiration is closely associated with the thermodynamics of composting. As with the temperature for each C/N ratio level, the cakes without coagulant had a higher respiration rate than the treatments with coagulant. It is worthwhile to explore whether the N adsorbed from the RAS sludge behaves differently from the N in the fishmeal which was applied to produce C/N ratios of 20, 30 and for FC also 55, since the mineralization of N may significantly affect the thermodynamics during composting.

A natural follow-up to this composting experiment would be to conduct it at a larger scale and to test the finished compost as a soil amendment. Dewar flask trials give only an indication of which combinations of feedstocks generate heat, but they are not suitable for making fully cured compost as this requires contact with soil and soil organisms.

Recovering nutrients from RAS is an important first step in improving the sustainability and resource efficiency of aquaculture. Utilizing the recovered nutrients in plant production is the logical next step, and composting is a method that is relatively simple and effective to sanitize materials and reduce volume while maintaining nutrients. Since RAS facilities can be located almost anywhere, perhaps it would be practical to locate them near agricultural land where the distance from the nutrient source to nutrient application is short and where there are a variety of other organic wastes that can be used as feedstocks to optimize composting process parameters and plant nutrient balance. Agricultural land may also produce lignocellulose for filtering RAS sludge, e.g., in field margins.

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5 Appendix I: Analysis results of filter cakes used for Dewar flask experiment

Characteristic	Unit	With coagulant	Without coagulant
Dry Matter	%	28	25
Total Carbon	% of DM	41	40
Total Kjeldahl nitrogen	mg kg^{-1} DM	7500	4300
Boron	mg kg^{-1} DM	97	55
Sodium	mg kg^{-1} DM	6800	3900
Magnesium	mg kg^{-1} DM	410	480
Phosphorus	mg kg^{-1} DM	4200	2900
Sulphur	mg kg^{-1} DM	<1200	<1200
Potassium	mg kg^{-1} DM	140	160
Calsium	mg kg^{-1} DM	9000	7900
Chrome	mg kg^{-1} DM	0.93	0.86
Manganese	mg kg^{-1} DM	99	110
Iron	mg kg^{-1} DM	240	280
Cobolt	mg kg^{-1} DM	0.24	0.12
Nickel	mg kg^{-1} DM	0.36	0.25
Copper	mg kg^{-1} DM	3.7	3,6
Zink	mg kg^{-1} DM	85	67
Arsenic	mg kg^{-1} DM	0.39	<0.37
Molybdenum	mg kg^{-1} DM	0.27	<0.17
Silver	mg kg^{-1} DM	<0.085	<0.085
Cadmium	mg kg^{-1} DM	0.23	0.20
Tin	mg kg^{-1} DM	<3.8	<3.8
Mercury	mg kg^{-1} DM	<0.12	<0.12
Lead	mg kg^{-1} DM	0.47	0.45
Selenium	µg kg^{-1} DM	150	130
Vanadium	µg kg^{-1} DM	960	680
Aluminium	µg kg^{-1} DM	690000	95000
Silisium	µg kg^{-1} DM	240000	200000

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