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Testing of a novel IFAS-MBR process with co-precipitation

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ABSTRACT

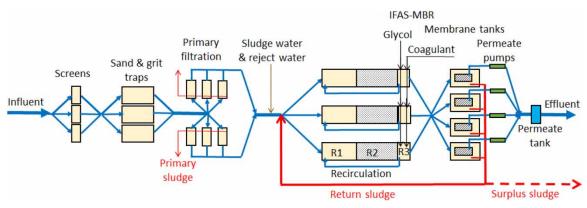
IFAS-MBR with co-precipitation, not yet commonly used in practice, will result in a very compact process for nutrient removal. The process, based on a combined pre- and post-denitrification IFAS process with membrane separation (IFAS-MBR), was tested in two parallel small-scale plants. Train A was operated with co-precipitation in order to achieve high removal of total P (TP). Train B, without co-precipitation, served as a control. Due to the coagulant (Al) addition, the concern was precipitation on the biofilm carriers in the aerobic reactor in Train A. A small internal air-lift pump proved to be very efficient in controlling biofilm thickness and removing excess biofilm mass as needed. A coagulant dose equivalent to an Al/TP molar ratio of 1.9 was necessary to achieve 99% TP removal and 0.10 mg TP/l in the effluent of Train A. Very good removal of total N was achieved in both trains. Train A had a biofilm nitrification rate of 0.65 g NH_4 - N/m^2 d at 12–13 °C and 5.2–5.6 mg O_2 /l. The tests demonstrated that an IFAS-MBR process with co-precipitation and an aerobic suspended biomass SRT of 5–10 days is feasible, and that all the performance goals set up for the full-scale plant were achieved.

Key words: biofilm control, co-precipitation, IFAS-MBR, municipal wastewater, nitrogen removal, phosphorus removal

HIGHLIGHTS

- IFAS-MBR with co-precipitation and combined pre- and post-denitrification for removal of P and N from wastewater.
- Removal of 95% COD, 80% N, 100% SS and 99% P at a dose of 1.9 mol Al/mol P.
- Air-lift pump to control the biofilm biomass and optimize nitrification on the biofilm carriers in the IFAS reactor.
- IFAS-MBR process worked well at suspended biomass aerobic SRTs of 5–10 d.

GRAPHICAL ABSTRACT



INTRODUCTION

The Gardermoen wastewater treatment plant (WWTP), Ullensaker, Norway, needs to expand capacity and meet new discharge requirements calling for removal of >70% total N (TN) and >98% total P (TP) as annual averages,

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and thermotolerant coliform bacteria (TCB) <100 CFU/100 ml. Site restrictions require a very compact design and the stringent P-removal requirement calls for filtration of the final effluent. The primary treated wastewater in the existing full-scale plant has a low C/N ratio and an external carbon source (glycol) is used, supplied free of charge from the nearby airport (Rusten & Ødegaard 2007).

One choice for the upgrade would be a traditional activated sludge based membrane bioreactor (MBR), but since the existing plant is based on moving bed biofilm reactors (MBBRs), it was of interest to analyse the potential of an IFAS-MBR process where the MBBR-based IFAS (integrated fixed-film activated sludge) would result in a compact bioreactor and the membrane separation would give a good as well as compact biomass separation step (Ødegaard 2018). A pre-feasibility study indicated, indeed, that an IFAS-MBR process, shown in the simplified process flow-sheet in Figure 1, was a potential option.

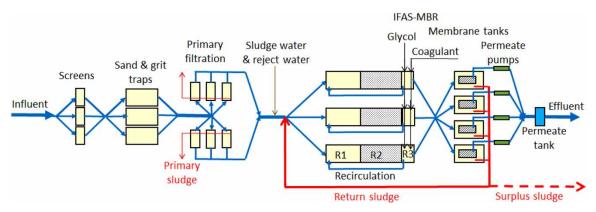


Figure 1 | Simplified flow sheet for the proposed IFAS-MBR process at Gardermoen WWTP.

Historically, the use of activated sludge processes with membrane separation generated a cost and energy penalty that was only worth paying if the discharge standards were very tight, water reuse was the goal and/or a low footprint was needed (Jefferson *et al.* 2018). However, in recent years membrane prices have decreased and fully optimized MBR energy consumptions of less than 0.4 kWh/m³ treated wastewater have also been demonstrated (Krzeminski *et al.* 2017; Yamashita *et al.* 2019). MBR processes with activated sludge and co-precipitation is an established technology, and for an effluent TP limit of 0.1 mg/l comparisons have shown MBR with co-precipitation to have favourable total lifecycle costs (capital + O&M) (Young *et al.* 2014).

Most of the studies comparing pure moving bed biofilm reactors (MBBRs) followed by membrane separation (MBBR-MBR) and MBBR-based IFAS processes with membrane separation (IFAS-MBR) go in the favour of IFAS-MBR (Leyva-Díaz et al. 2017). Most studies that compare the traditional activated sludge-based MBR and the IFAS-MBR are also in favour of the IFAS-MBR (Duan et al. 2015; Mannina et al. 2018). The IFAS-MBR is claimed to have the advantage of operating at higher fluxes, being more compact, having better energy efficiencies and better membrane fouling control (in most papers) than the traditional MBR. Mannina et al. (2018) also demonstrated lower nitrous oxide emissions. Hence the IFAS-MBR should be the most interesting system, taking advantage of the best qualities of suspended biomass, attached biomass and membrane separation.

One of the benefits of IFAS is that nitrification can be achieved at less than half of the aerobic solids retention time (SRT) that is used in traditional activated sludge plants, when the SRT is based on mixed liquor suspended solids (MLSS) (Ødegaard et al. 2014). Moreover, several studies have shown that, due to the lower SRT, the specific pre-denitrification rate in IFAS systems is up to twice as high as in conventional activated sludge systems (Rusten et al. 2003; Onnis-Hayden et al. 2011). Since both the nitrification zone and the denitrification zone can be smaller, there is potential for the bioreactors in an IFAS-MBR to be significantly smaller than in the conventional MBR.

A comparison by Ødegaard (2018) of a conventional MBR and an IFAS-MBR, both with co-precipitation of phosphorus, showed that at 10 °C the bioreactor volume for the IFAS-MBR was about 60% of the total bioreactor volume for the conventional MBR. This made the IFAS-MBR a very attractive process for the expansion of the Gardermoen WWTP. However, chemical co-precipitation of phosphorus in an IFAS-MBR system had never been tested. Unpublished observations from previous short-term testing of co-precipitation in MBBR systems showed

precipitation on the biofilm carriers, which made the carriers so heavy that they were no longer suspended in the reactor. Therefore, co-precipitation of phosphorus has not been practiced in full-scale MBBR systems. This led to legitimate concerns about possible negative interactions from chemical precipitation also on the biofilm carriers in an IFAS reactor. Consequently, the Ullensaker municipality decided to test this concept in small scale, before seriously considering an IFAS-MBR process with co-precipitation.

The primary objectives of this research were to:

- Investigate the effect of co-precipitation of phosphorus on the biofilm carriers in an IFAS-MBR process, by running in parallel one train with co-precipitation and one train without.
- In the case of precipitation on the biofilm carriers, to develop a method for controlling the biofilm thickness and thus mitigate any negative effects on the nitrifying biofilm carriers from co-precipitation of phosphorus. Secondary objectives were to:
- Document feasibility and conditions for achieving low effluent TP concentrations (>98% removal) in an IFAS-MBR. This can only be achieved by adding a coagulant.
- Document membrane fouling and removal of nitrogen at aerobic, suspended biomass SRTs of 5–10 days (based on biomass in bioreactor).
- Document removal of organic matter, nitrogen, SS and TCB in relation to the effluent criteria for the full-scale plant.

METHODS

The small-scale test plant (Figure 2) had two parallel trains, where an aluminium based coagulant (Ekoflock 90) was added downstream from the IFAS-tank in Train A to achieve very low final effluent TP. Train B served as a reference without coagulant. Spent aircraft de-icing fluid (monopropylene glycol) from the nearby airport was used as an external carbon source for the post-denitrification (DN) in both trains.

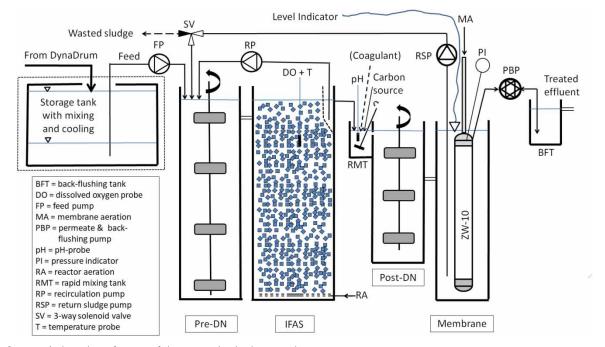


Figure 2 | Flow sheet for one of the two trains in the test plant.

Influent wastewater was primary treated with a drum filter (DynaDrum, $100 \,\mu m$ filter cloth) to protect the membranes (module: ZeeWeed-10, $0.93 \, m^2$ membrane area, $0.04 \,\mu m$ average pore size). The membranes were operated on a cycle of 9.5 min permeate flow followed by 0.5 min of backflush at twice the permeate flow. Forward membrane flux averaged 11.5 LMH (l/m^2 -h), with a maximum of 14.2 LMH. Membranes were chemically cleaned about every 80–90 days.

The IFAS reactors had biofilm carriers (BWT 15, Biowater Technology, Tønsberg, Norway) with a bulk specific surface area of $828 \text{ m}^2/\text{m}^3$ and a filling fraction of 48.9%. In Train A, the IFAS reactor had a small air-lift pump, which could be turned on to control biofilm thickness in case of excessive precipitation on the carriers due to the coagulant addition. The air-lift pump had an inner diameter of 46 mm and a total length of 1.6 m. Both the position and air-flow of the air-lift pump were adjustable, with normally 55-60% of the air-lift pipe submerged in the IFAS reactor.

Total empty bed hydraulic retention time (HRT) in the biological system (excluding membrane tank) averaged 9.3 h (7–10 h range), based on influent flow (Q_{in}). Both recirculation and return sludge pumping were normally at $2.0 \times Q_{in}$, but recirculation was reduced to $1.0 \times Q_{in}$ for the last four weeks of the test period in order to reduce the amount of oxygen returned from the IFAS reactor to the pre-DN reactor. Average design flow rates and reactor sizes for the test plant are shown in Table 1.

Table 1 | Normal flow rates, biofilm carrier filling and reactor volumes for one of the two identical trains

Influent flow	9 l/h; 216 l/d
Recirculation flow	18 l/h
Return activated sludge flow	18 l/h
Pre-denitrification reactor	29 l wet volume
IFAS reactor	45 l wet volume
IFAS biofilm carriers	48.9% (22.0 l) filling fraction of BWT 15
Rapid mixing tank for external carbon source (and coagulant in Train A)	0.66 l wet volume
Post-denitrification reactor	9.2 l wet volume
Membrane tank	9.3 l water volume with membrane installed
Permeate and backflushing tank	1.9 l wet volume

The storage tank for the primary treated wastewater was filled twice a week and initially the temperature in the tank gradually increased to room temperature after each filling. A cooler was then installed to reduce the temperature in the storage tank to about 4 °C, and after insulating the pilot-plant reactors the temperature in these reactors gradually came down to between 11 and 12 °C towards the end of the experiments. However, a few days in the last week of June and first week of July 2019 had higher temperatures than planned, due to problems with the heat exchangers (see temperatures in Figures 3, 7 and 8).

Monitoring, control, sampling and analyses

Samples of water and suspended biomass were taken as grab samples twice per week, 2 or 3 days after the storage tank was last filled, so that the samples were representative for the pilot-plant performance with the wastewater composition in the storage tank. Water samples from each individual reactor were pipetted directly from the reactor to a gravity pre-filter (Whatman 597 $^{1}/_{2}$, 4–7 μ m pore size) and then immediately filtered again with syringe filters (Whatman GF/C, 1.2 μ m average pore size). This was done to minimize the biological activity prior to analyses, which were performed as soon as a sufficient volume of filtered sample was available.

Influent samples to the pilot-plant (after DynaDrum primary treatment) were analysed for total COD (TCOD), filtered COD (FCOD), total nitrogen (TN), total nitrogen on filtered sample (FTN), NH₄-N, NO₂-N, NO₃-N, total phosphorus (TP), total phosphorus on filtered sample (FTP), PO₄-P, suspended solids (SS) and alkalinity. Filtered samples from the individual biological reactors and the membrane reactors were analysed for FCOD, FTN, NH₄-N, NO₂-N, NO₃-N, FTP and PO₄-P. The final effluent samples were analysed for TCOD, TN, NH₄-N, NO₂-N, NO₃-N, TP, PO₄-P and SS. Whatman GF/C filters were used for filtration of samples and measurement of SS.

Biomass in each reactor were analysed for total solids (TS) and volatile solids (VS) and were sampled at the same time as the water samples. Wasted sludge was collected automatically from the return sludge lines, using solenoid valves. TS and VS were measured on flow proportional weekly samples of wasted sludge. The return sludge from the membrane tanks was so concentrated that it did not thicken in standard 1,000 ml cylinders.

Therefore, a diluted sludge volume index (DSVI) was measured by taking 200–300 ml of return sludge and diluting it up to 1,000 ml with permeate from the respective train.

Attached biomass on the biofilm carriers was measured as TS at least every two weeks and sometimes more frequently, using an internal Aquateam COWI laboratory procedure. In this procedure, the dry weight of the carriers is measured before and after all biomass has been removed from the carriers. Biomass is removed by soaking the carriers in a 50% domestic chlorine solution and then rinsing with hot water until no trace of biomass can be spotted. If necessary, the cleaning process is enhanced by applying sonication and/or using small pipe brushes on all surfaces of the carriers, followed by additional rinsing.

All analyses were done in the on-site laboratory. Merck Spectroquant Cell Test kits, Merck Spectroquant Reagent Test kits, a Merck Spectroquant Thermoreactor TR 620 and a Merck Spectroquant Prove 100 Spectrophotometer (Merck/Millipore-Sigma, USA and Canada) were used for chemical analysis. SS, TS and VS were analysed according to Standard Methods (2005).

Specific nitrification rate for the biofilm on the biofilm carriers was measured in separate batch tests. From each IFAS reactor 100 biofilm carriers were carefully removed and placed in separate beakers, which were then filled with 500 ml effluent from Train B. Train B effluent was chosen because it contained sufficient amounts of phosphorus. NaHCO₃ was added as a buffer and NH₄Cl was added to have excess NH₄-N concentrations. The beakers were placed on magnetic stirrers in water baths to provide proper turbulence and a temperature close to that in the pilot reactors. Air diffuser stones were used to supply oxygen. The tests typically lasted for 3 h and the decrease in NH₄-N concentrations was measured by frequent sampling. Dissolved oxygen (DO) concentrations, temperature and pH were monitored.

In order to compare specific biofilm carrier nitrification rates, measured at different temperatures and DO concentrations, the model presented in Rusten *et al.* (1995) was used. For temperature correction, a temperature coefficient (θ) of 1.09 was used. The reduction in the oxygen concentration available for nitrification (due to consumption in the outer, heterotrophic layer of the biofilm) was set at 1.0 mg O_2/I . At IFAS reactor concentrations above 2.5 mg NH₄-N/I the rate limiting factor was always the DO concentration. With a fully developed nitrifying biofilm and a fixed temperature, the nitrification rate will then, as an example, increase by 27% if the DO is increased from 6 mg O_2/I to 8 mg O_2/I .

All flow rates were manually measured daily and adjusted if needed. DO, temperature and pH were also measured daily in every reactor. The test plant was PLC controlled. Settings and run-times for all pumps and open-shut times for solenoid valves were logged and could be changed over the internet. Values from DO and temperature probes in IFAS tanks and pH probes in rapid mixing tanks were automatically logged, together with water levels in membrane tanks and trans-membrane pressure (TMP) over the membranes. The level indicator would automatically stop the feed pump or the permeate pump if the water level in the membrane tank got too high or too low, and automatically start the pump again when the water level was inside the pre-set limits.

Denitrification loads and removal rates were calculated for NO_3 -N equivalents, where oxygen (0.35 g NO_3 -N_{eq}/g O_2) and nitrite (0.6 g NO_3 -N_{eq}/g NO_2 -N) were converted to equivalent amounts of NO_3 -N. Calculations for pre-denitrification were based on influent flow, internal recycle flow (from IFAS to pre-DN), return sludge flow (from membrane tank to pre-DN); concentrations of NO_2 -N, NO_3 -N and DO in these three streams; TS, VS and DO in the pre-DN reactor; and NO_2 -N and NO_3 -N out of the pre-DN reactor. Calculations for post-denitrification were based on influent flow, return sludge flow (from membrane tank to pre-DN); NO_2 -N and NO_3 -N out of the IFAS reactor and out of the post-DN reactor; DO in the IFAS reactor; TS, VS and DO in the post-DN reactor; and the flow and concentration of the external carbon source.

RESULTS AND DISCUSSION

The pilot plant was started on 24 September 2018, with seed sludge from one of the nitrifying biofilm reactors in the full-scale plant. Chemical precipitation of phosphorus was introduced in Train A on 17 December 2018. Data presented in this paper are from 2 February to 13 November 2019.

Operating conditions and overall influent concentrations, effluent concentrations and removal efficiencies for the two parallel trains are shown in Tables 2 and 3, respectively. Both recirculation flows and return sludge flows were sometimes lower than the intended set-point due to either partially clogged tubes or problems with the pumps. DO concentrations in anoxic reactors were always low. The aerobic IFAS reactors had DO concentrations in a suitable range for a nitrifying biofilm process (Rusten *et al.* 1995). The membrane reactors had very high DO concentrations

Table 2 | Operating conditions for test plant from 2 February to 13 November 2019

	Train A (co-precipitation)			Train B (control)		
Parameter	Average \pm St. Dev.	Median		Average \pm St. Dev.	Median	
Influent flow, l/h	9.3 ± 0.7	9.0		9.3 ± 0.8	9.0	
Recirculation flow, l/h	16.4 ± 3.8	18.0		16.9 ± 2.9	18.0	
Return sludge flow, l/h	17.6 ± 0.9	17.7		17.5 ± 1.5	18.0	
DO in pre-DN, mg/l	0.04 ± 0.01	0.03		0.04 ± 0.09	0.02	
DO in IFAS, mg/l	7.3 ± 1.1	7.1		6.5 ± 1.3	6.3	
DO in post-DN, mg/l	0.06 ± 0.24	0.02		0.03 ± 0.05	0.02	
DO in membrane tank, mg/l	8.8 ± 0.6	8.9		7.7 ± 0.8	7.8	
Temperature in IFAS, °C	15.7 ± 3.6	14.4		15.9 ± 3.6	14.6	
Activated sludge in IFAS, mg TS/l	6779 ± 1675	7053		5429 ± 857	5647	
Activated sludge in IFAS, mg VS/l	4556 ± 1057	4630		4503 ± 697	4720	
Biofilm in IFAS, mg TS/l	4499 ± 1792	4740		3515 ± 1493	3880	
Activated sludge in membrane tank, mg TS/l	10160 ± 2744	10531		7883 ± 1733	8047	
Activated sludge in membrane tank, mg VS/l	6850 ± 1650	7195		6647 ± 1170	6770	
Suspended aerobic SRT, da	7.3 ± 1.5	7.2		9.3 ± 1.8	9.8	
Total aerobic SRT, d ^b	13.2 ± 4.2	11.4		15.7 ± 2.8	15.4	
Total SRT, d ^c	23.8 ± 5.8	21.9		28.7 ± 4.3	29.6	
External C-source, g COD/g TN _{in biology}	2.3 ± 0.5	2.3		2.4 ± 0.6	2.4	
	Average	Min	Мах	Average	Min	Мах
pH in pre-DN	7.4	6.0	8.3	7.3	6.4	8.3
pH in IFAS	7.0	5.0	8.2	6.9	5.5	8.2
pH in rapid mixing tank	6.6	4.6	7.5	6.9	5.6	7.9
pH in post-DN	6.9	5.2	7.9	7.1	5.5	8.0
pH in membrane tank	7.2	5.3	8.4	7.4	5.3	8.4

^aSuspended aerobic solids retention time (SRT), based on activated sludge in IFAS reactor only.

due to vigorous aeration being necessary to keep the activated sludge in suspension and create enough turbulence to avoid sludge cakes on the membrane fibers. Temperatures were similar in the two trains.

Activated sludge concentrations in the reactors were changed over the course of the test to cover different SRTs. Measured as VS, the concentrations were similar in the two trains. However, due to the addition of coagulant in Train A, the TS concentrations were significantly higher in Train A than in Train B. The average VS/TS ratios were 0.67 in Train A and 0.83 in Train B. Suspended biomass aerobic SRTs, based on the IFAS reactor only, were most of the time about 2 days lower in Train A than in Train B (caused by the extra sludge production from precipitation). The lowest suspended biomass aerobic SRT was only 3.8 days in Train A and 4.7 days in Train B. Biomass in membrane tanks are normally not included in the aerobic SRT calculations for MBR plants, even though the membrane tanks are aerated. Any nitrification in the membrane tanks is considered as an additional safety factor in the design of these systems. Somewhat higher effluent NH₄-N concentrations out of Train A than Train B were observed in the second half of the test period, as seen in Figure 3. This happened at the same time (1 July to the middle of August) as the influent NH₄-N concentrations increased and the temperatures in the bioreactors dropped. Average effluent concentrations in Figure 3 were 4.7 mg NH₄-N/l for Train A and 1.4 mg NH₄-N/l for Train B. Median effluent concentrations were 2.1 mg NH₄-N/l for Train A and <0.2 mg NH₄-N/l for Train B. This difference between the two trains was caused mainly by the lower suspended aerobic SRT in Train A compared to Train B as the temperature dropped, and this will be discussed further in the section on nitrification. A lower minimum pH in the IFAS reactor in Train A (pH 5.0) vs. Train B (pH 5.5) may also have been a contributing factor. However, due to the high influent concentrations, the difference in the amount of nitrified nitrogen was only 4% based on average effluent concentrations and 2% based on median effluent concentrations.

^bBased on both suspended biomass and biofilm biomass in IFAS reactor only.

clincludes biomass in membrane tank.

Table 3 | Influent concentrations (after primary treatment), effluent concentrations and removal efficiencies for the test plant from 2 February to 13 November 2019

Parameter			Train A (co-precip	itation)	Train B (control)		
		^a Influent, mg/l	Effluent, mg/l	Removal, %	Effluent, mg/l	Removal, %	
Total COD	Median	366	15.0	95.8	25.5	93.4	
	Min	126	5	90.3	5	85.8	
	Max	597	44	98.8	41	98.6	
Filtered COD	Median	184	-	-	-	-	
	Min	78	-	-	-	-	
	Max	354	-	-	-	-	
Suspended solids	Median	115	0.00	100.0	0.10	99.9	
	Min	36.0	- 1.5	99.1	- 1.4	99.5	
	Max	315	0.90	101.6	0.60	101.5	
Total P	Median	8.3	0.080	99.1	5.5	32.9	
	Min	3.6	0.016	85.4	3.7	8.4	
	Max	12.4	1.30	99.8	7.8	53.2	
PO ₄ -P	Median	5.6	0.060	98.9	5.4	5.2	
	Min	2.8	<0.005	73.0	3.7	- 27.3	
	Max	9.6	1.27	>99.9	7.7	21.7	
Total N	Median	89.0	16.0	83.1	11.7	86.9	
	Min	44.0	0.30	42.7	0.50	27.1	
	Max	126	55.0	99.4	70.0	99.1	
NH ₄ -N	Median	69.0	2.12	-	<0.20	-	
	Min	35.7	<0.20	-	<0.20	-	
	Max	97.0	25.9	-	18.1	-	
NO ₂ -N	Median	0.03	0.11	-	0.03	-	
	Min	0.02	0.01	-	0.01	-	
	Max	0.06	2.87	-	2.06	-	
NO ₃ -N	Median	0.50	11.1	-	10.4	-	
	Min	0.20	0.50	-	0.50	-	
	Max	1.30	39.4	-	48.0	-	

^aInfluent to test plant, after primary filtration.

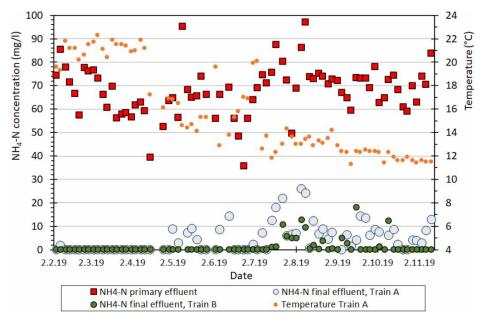


Figure 3 | Influent and effluent NH₄-N concentrations for the two trains, together with temperatures in Train A IFAS reactor.

The influent to the IFAS-MBR had relatively high concentrations of TN and TP but was low in soluble organic matter. Hence, influent C/N ratios were low, with averages of 4.3 g TCOD/g TN and 2.1 g FCOD/g TN. The overall removal of organic matter was excellent in both trains, but slightly better in Train A due to the coagulation of some of the colloidal organic matter that would pass through the membrane in Train B. As expected, average removal of SS was 100% in both trains since the membranes had an average pore size of only $0.04~\mu m$. Individual effluent SS values different to zero were due to the error in the analytical precision of these measurements, resulting in some negative values. Phosphorus removal will be discussed later, but removal of TP was excellent in Train A (co-precipitation) with a median removal efficiency of 99% compared to 33% in Train B (control).

Performance of each biological reactor will also be discussed later, but influent and effluent TN concentrations are shown in Figure 4. For the entire period, average TN removal efficiencies were 80% for Train A and 83% for Train B, well above the required annual average removal. Spikes in effluent concentrations around 1 March can be explained by the plant being stopped for chemical membrane wash shortly before sampling. Increased effluent concentrations and lower removal efficiencies on some days from July onwards were due to a combination of lower temperatures (down to 11 °C), increased hydraulic load and insufficient dosing of external carbon source. The slightly lower nitrification in Train A compared to Train B, previously mentioned, accounts for the difference in average TN removal efficiencies between the two trains.

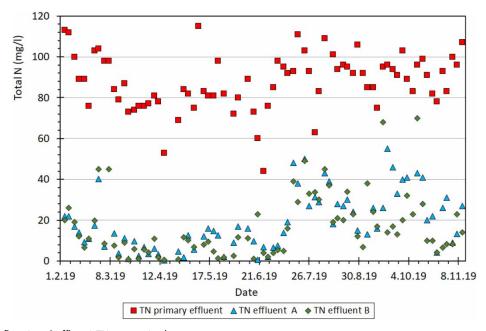


Figure 4 | Influent and effluent TN concentrations.

Multiple grab samples showed TCB to always be $\leq 20 \text{ CFU}/100 \text{ ml}$ in the final effluent of both trains.

Phosphorus removal

In Train B (control) removal of TP was always low since only particulate P was removed. On some days, the concentrations of PO_4 -P in the effluent were higher than in the influent due to hydrolysis of organic P. Removal of TP in Train A (co-precipitation) was dependent on the amount of coagulant added. Figure 5(a) shows influent and effluent concentrations, removal efficiencies and Al/TP molar ratios from day to day. Al/TP molar ratios are based on the Ekoflock 90 dose and the TP concentration in the influent to the IFAS-MBR pilot plant. From the beginning of February 2019, the molar ratios were ≥ 2.0 and effluent TP concentrations were very low with removal efficiencies above 98%. Coagulant doses between 3 and 4 mol Al/mol TP were obviously higher than necessary and resulted in removal efficiencies well above 99% and effluent concentrations well below 0.1 mg TP/l. From the end of June 2019, the coagulant dose was reduced to give removal efficiencies for TP between 98 and 99%. Close to the end of the testing period, the coagulant dosing was significantly reduced and at an Al/TP molar ratio below 1.5 there was a sharp increase in effluent TP concentrations and the removal

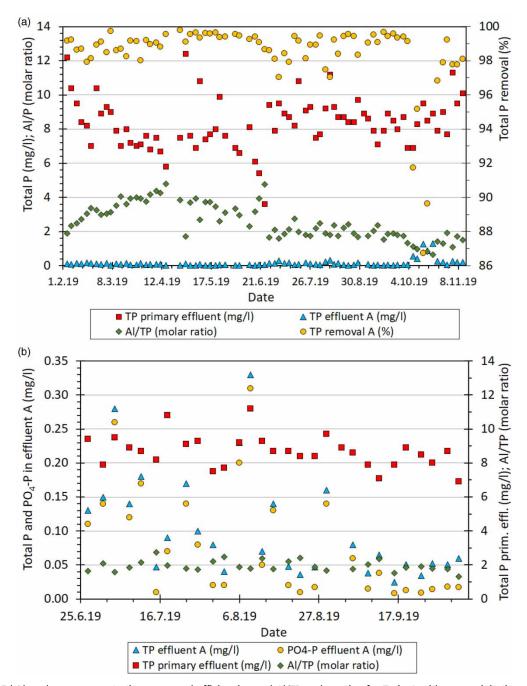


Figure 5 | Phosphorus concentrations, removal efficiencies and AI/TP molar ratios for Train A with co-precipitation.

efficiency decreased to about 85% for TP after a few days with Al/TP molar ratios from 0.7 to 1.1. When the coagulant dose was increased to above 1.5 mol Al/mol TP the removal efficiency again reached 98% for TP.

From the end of June to the beginning of October 2019, we had a period with very stable coagulant dosing and an Al/TP molar ratio of 1.9 ± 0.3 and this period is highlighted in Figure 5(b). Except for a couple of days, we had very low effluent concentrations with averages of 0.10 mg TP/l and 0.08 mg PO₄-P/l and median values of 0.08 mg TP/l and 0.04 mg PO₄-P/l. Average and median removal efficiencies for TP were 98.8% and 99.1%, respectively.

Pre- and post-denitrification

Conditions and results for pre-DN reactors and post-DN reactors are shown in Table 4. Loads, rates and concentrations are given as NO_3 -N equivalents (NO_3 -N_{eq}). Due to a higher fraction of inert biomass in Train A (coprecipitation), average and median specific pre-DN rates, based on biomass as TS in the reactors, were slightly

Table 4 | Pre-DN and post-DN conditions and results for Train A (co-precipitation) and Train B (control), from 2 February to 13 November 2019

		Pre-denitrifica	Pre-denitrification		Post-denitrification	
Parameter		Train A	Train B	Train A	Train B	
DN reactor load, mg NO ₃ -N _{eq} /g TS-h	Average Median Min Max	3.2 2.6 0.5 12	4.7 3.8 1.6 12	9.8 7.2 3.8 29	13 12 6.6 30	
DN reactor rate, $mg\ NO_3\text{-}N_{eq}/g\ TS\text{-}h$	Average	1.2	1.4	5.4	7.7	
	Median	1.1	1.3	5.2	7.3	
	Min	0.3	0.2	3.6	3.7	
	Max	2.9	3.3	8.7	11	
DN reactor load, $mg\ NO_3\text{-}N_{eq}/g\ VS\text{-}h$	Average	4.6	5.6	14	16	
	Median	3.7	4.5	10	14	
	Min	0.8	2.0	5.6	7.7	
	Max	17	14	40	36	
DN reactor rate, $mg \ NO_3\text{-}N_{eq}/g \ VS\text{-}h$	Average	1.8	1.7	7.7	9.1	
	Median	1.7	1.6	7.6	8.8	
	Min	0.4	0.2	4.8	4.2	
	Max	4.0	3.9	12	14	
C/N-ratio in to the DN reactor, g FCOD/g NO_3 - N_{eq}^a	Average	3.6	3.3	3.6	3.0	
	Median	3.3	2.4	3.6	3.1	
	Min	0.8	0.7	2.2	0.2	
	Max	8.5	11	6.2	4.9	
Reactor effluent concentration, mg NO_3 - N_{eq}/l	Average	7.9	11	8.8	12	
	Median	4.7	9.7	5.6	10	
	Min	<0.5	<0.5	<0.5	<0.5	
	Max	25	22	35	42	
Temperature, °C	Average	15.7	15.9	15.9	16.0	
	Median	14.4	14.5	14.6	14.6	
	Min	11.2	11.4	11.2	11.5	
	Max	22.4	22.6	22.4	22.6	

^aBased on FCOD in the primary effluent for the pre-DN reactor and COD in the added external carbon source for the post-DN reactor.

higher in Train B than in Train A. Based on VS, however, the specific pre-DN rates were slightly higher in Train A than in Train B. Several factors influence the pre-DN rates, to an extent that they can mask the effect of temperature. This is demonstrated in Figure 6, where high DN rates were observed at the lower temperatures. Detailed examination of the data showed that in both trains the C/N ratios and the reactor effluent NO_3 - N_{eq} concentrations had the most impact on the pre-DN rates and were more important than the temperature. With reactor effluent concentrations around 3 mg NO_3 - N_{eq} /l, typical pre-DN rates in Train A were 1.5–2.0 mg NO_3 - N_{eq} /g VS-h, and with reactor effluent concentrations from 12 to 18 mg NO_3 - N_{eq} /l, pre-DN rates were 2.5–3.5 mg NO_3 - N_{eq} /g VS-h. From 5 July to 13 November 2019, at the low average temperature of 12.7 °C, the average pre-DN rate in Train A was 2.1 mg NO_3 - N_{eq} /g VS-h.

The pre-DN rates observed in this study are in the upper end of corresponding rates on conventional activated sludge MBR systems (Judd 2006), and even more so considering the very low influent C/N-ratios in this study.

Specific post-DN rates were significantly higher than the pre-DN rates. The main reasons for this were the easily biodegradable external carbon source and the high NO_3 - N_{eq} loads. Median C/N ratios based on the added monopropylene glycol were low, with 3.6 g FCOD/g NO_3 - N_{eq} in Train A (co-precipitation) and 3.1 g FCOD/g NO_3 - N_{eq} in Train B (control). Median post-DN rates were 7.6 and 8.8 mg NO_3 - N_{eq} /g VS-h in Train A and Train B, respectively. This difference was mainly due to the much lower median NO_3 - N_{eq} load and effluent NO_3 - N_{eq} concentration in Train A, and thus more data points with very low effluent concentrations. For the entire test period, the average carbon source consumption was 5.3 g FCOD/g NO_3 - N_{eq} removed in Train A and 4.9 g FCOD/g NO_3 - N_{eq} removed in Train B. Similar to the pre-DN rates, other factors masked the temperature effect on the post-DN rates as well.

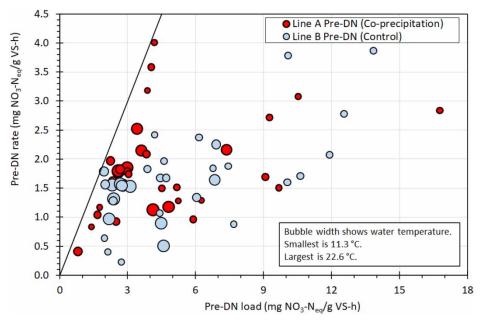


Figure 6 | Pre-DN rates versus loads for Train A and Train B. Bubble width indicates temperature.

Even though the specific post-DN rates were 4–6 times higher than the pre-DN rates, about 42% of the nitrogen removed by denitrification was removed by pre-DN in Train A (co-precipitation) and about 37% was removed by pre-DN in Train B (control). The reason for this was the much larger volume of the pre-DN reactor compared to the post-DN reactor.

Nitrification

Effluent NH₄-N concentrations and amount of NH₄-N removed are shown together with suspended biomass aerobic SRTs and IFAS-reactor temperatures in Figure 7(a) for Train A and Figure 7(b) for Train B, respectively. Train A showed some breakthrough of NH₄-N in May, at 14-15 °C and 7 d suspended biomass aerobic SRT. This coincided with some days of higher nitrogen loads, and the amount of NH₄-N removed did not drop compared to April. Train B showed no breakthrough in this period, due to a suspended biomass aerobic SRT above 10 d in April and which got back up to 10 d SRT in mid-May. As the temperature continued to drop and the ammonium-load increased in July (ref. Figure 3), both trains saw a breakthrough of NH₄-N. In Train A, the removal of NH₄-N actually increased, even though the suspended biomass aerobic SRT was as low as between 3.8 and 4.8 d in mid-July at a temperature of 12-13 °C. Looking at the beginning of October, with a suspended biomass aerobic SRT below 5.5 d and 11-12 °C, NH₄-N removal was steady in the 13-15 g NH₄-N/d range. We believe almost all this NH₄-N removal to be due to nitrification on the biofilm carriers in the IFAS reactor, and that only minimal nitrification took place in the suspended biomass. Train B had suspended biomass aerobic SRT of 10 d on 1 July, compared to 6 d SRT for Train A. Train B had also significantly shorter periods at low SRT. Train A was below 6 d suspended biomass aerobic SRT for about 2 months, while Train B was below this value for only 1-2 weeks. Thus, suspended biomass definitely contributed significantly to the nitrification in Train B, also at the lowest temperatures, maybe with an exception for the few weeks with suspended biomass SRT below 7 d at 12-13 °C.

The specific biofilm nitrification rates for biofilm carriers from the IFAS reactors are shown in Table 5. Both temperature and DO have a huge impact on the nitrification rates (Rusten *et al.* 1995, 2003). Compensating for the difference in temperature and DO, using the equations in Rusten *et al.* (1995), the specific biofilm nitrification rates in Train A were identical for the batch-tests on 29 March and 24 May. For the subsequent tests, the temperatures were even lower. Nevertheless, the specific biofilm nitrification rates increased in Train A, showing that a robust nitrifying biofilm had developed on the carriers.

For Train B the specific biofilm nitrification rates were very low for the first two sets of tests. The third test (16 July), however, showed significantly higher nitrification rates in Train B than in Train A. This was mainly due to the much higher DO in Train B and partly due to the higher biomass and thicker biofilm in Train A. For the last

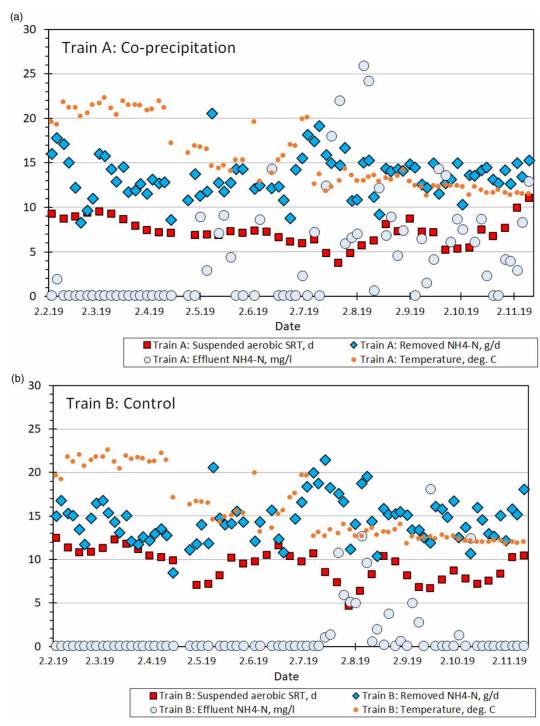


Figure 7 | Effluent NH_4 -N concentrations and amount of NH_4 -N removed, shown together with suspended biomass aerobic SRTs and IFAS-reactor temperatures for Train A (a) and Train B (b), respectively.

three sets of tests the nitrification rates were higher in Train A than in Train B, in spite of slightly higher DO in Train B. The reasons for these observations are difficult to explain, but it may have to do with the coagulant agglomerating colloids and fine particles that may influence the biofilm activity.

The specific biofilm nitrification rates were about the same as expected in conventional MBBRs at similar temperatures and DO concentrations (Rusten *et al.* 1995).

In an IFAS system for nitrogen removal there will always be some nitrification in the activated sludge, either due to sloughed nitrifying biofilm circulating in the system or because the aerobic suspended growth SRT is sufficiently long to sustain nitrification. Looking at the NH₄-N reduction over the membrane tank on days with

Table 5 | Results from batch-tests of nitrification rates on biofilm carriers

	Train A (co-precipitation)					Train B (control)				
Date, 2019	Temp., °C	DO, mg/l	рН	Biofilm, g TS/m²	Nitrif. rate, g NH ₄ -N/m ² -d	Temp., °C	DO, mg/l	рН	Biofilm, g TS/m ²	Nitrif. rate, g NH ₄ -N/m ² -d
29.03	23.6	5.6	7.9	5.5	1.18	23.4	5.5	8.1	4.1	0.38
24.05	15.5	4.9	7.8	8.5	0.51	15.5	5.3	7.9	7.8	0.31
16.07	12.6	5.5	7.6	14.1	0.56	12.6	6.4	7.7	6.1	0.69
29.08	14.8	5.1	7.7	17.9	0.56	14.9	5.2	7.8	12.9	0.48
13.09	12.4	5.6	7.6	15.5 ^a	0.65	12.2	6.0	7.8	13.8	0.48
24.10	13.5	5.2	7.8	13.5	0.65	13.5	5.6	7.9	10.0	0.57

^aAir-lift pump used 6 h/d on work-days for two weeks prior to test, to reduce excessive biomass on carriers

surplus NH_4 -N concentrations in the effluent will give an indication of the suspended growth nitrification rate. A mass balance approach was then used to find out how much of the NH_4 -N that was nitrified by the biofilm by subtracting the amount that was estimated to be nitrified by the suspended growth. Examples of this are shown in Table 6 for Train A. Only minimal nitrification was taking place in the activated sludge and more than 90% of the nitrification happened in the biofilm in the IFAS reactor. Compensated for the differences in temperature and DO, the specific biofilm nitrification rates in Table 6 agreed very well with the batch-test biofilm nitrification rate in Train A on 24 October.

Table 6 | Examples of distributions of nitrification in activated sludge and biofilm, respectively, for train A (co-precipitation)

			Activated sludge		Biofilm		
Date, 2019	Temp., °C	IFAS-reactor DO, mg/l	Aerobic suspended SRT, days	Specific nitrification rate, mg NH ₄ -N/g VS-h	Nitrification in activated sludge, %	Nitrification in biofilm, %	Specific nitrification rate, g NH ₄ -N/m ² -d
4.11	11.6	7.55	10.0	0.12	9.3	90.7	0.75
7.11	11.5	6.61	11.0	0.11	6.9	93.1	0.69

At first sight it is surprising that the suspended biomass is not fully nitrifying at an aerobic SRT of 10 d and 11–12 °C. However, from Figure 7(a) it can be seen that the suspended biomass aerobic SRT was between 5 and 7.5 days for the eight weeks prior to the data shown in Table 6. Temperatures were between 11.3 and 12.3 °C. These SRTs and temperatures were obviously too low to sustain suspended biomass nitrification. Even though the SRT increased to 10 and 11 days, respectively, over the next week and the next two weeks, this was not a long enough period to grow a significant amount of suspended nitrifying bacteria.

The amount of NH_4 -N removed from mid-July and onwards in Train A indicates that almost all nitrification took place on the biofilm carriers. Looking specifically at the data from the last six weeks of testing, the removal rate was fairly steady, between 13 and 15 g NH_4 -N/d. Using the measured biofilm nitrification rate in Train A from October 24 (Table 5) and adjusting to the average temperature of 11.7 °C and average DO of 7.9 mg O_2 /l over this six-week period, we get a specific biofilm nitrification rate of 0.79 g NH_4 -N/m²-d. With a biofilm surface area of 18.2 m² this equals an expected removal of 14.4 g NH_4 -N/d, according to the model developed by Rusten *et al.* (1995), thus confirming that almost all nitrification in this period took place on the biofilm carriers.

Biofilm development and control

The development of the biofilm biomass in the IFAS reactors, as g TS/m², is shown in Figure 8 for both Train A (co-precipitation) and Train B (control). There was only marginally more carrier attached mass (biomass plus precipitated mass – here called biomass for simplicity) in Train A than Train B until the middle of June. Then the biofilm biomass increased a lot in Train A, until it was more than twice the biomass in Train B. However, use of the air-lift pump in the IFAS reactor for a few hours on regular workdays (shown by the triangles in Figure 8)

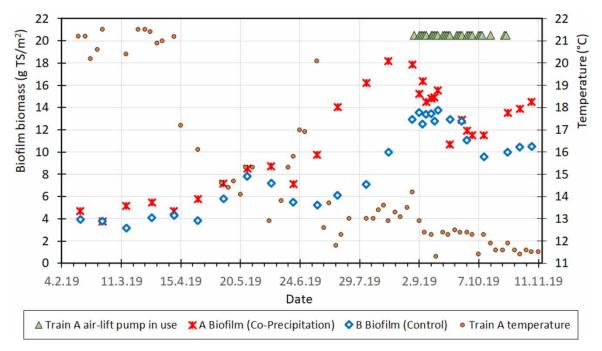


Figure 8 | Biofilm biomass in IFAS reactors in both Train A and Train B, together with corresponding temperatures in Train A.

brought the biofilm biomass in Train A down to the same level as in Train B within a few days. The turbulence and shear when the biofilm carriers were transported through the air-lift pipe proved to be an efficient tool for controlling the biofilm biomass. A biofilm that is too thick may reduce the specific nitrification rate due to the consumption of oxygen in the outer heterotrophic layer before it can reach the nitrifying part of the biofilm.

Figure 9 shows a photo of a single biofilm carrier from Train A (co-precipitation) and Train B (control), respectively. The carriers were photographed when submerged in water, to prevent the biofilm from collapsing. In these photos, the biofilm biomass was 13.9 g TS/m^2 for Train A and 10.4 g TS/m^2 for Train B. These are typical values for nitrifying biofilms on MBBR carriers. The biofilm thickness was typically 0.2–0.3 mm and slightly thicker in Train A than Train B.

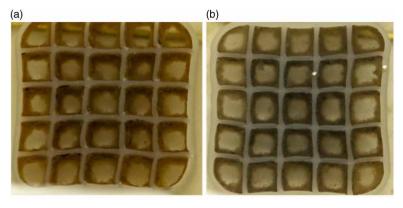


Figure 9 | Nitrifying biofilm. Biofilm carriers are 14.5 mm wide. (Photo: Nina Værøy, COWI.).

Sludge production and sludge volume index

For the same period as covered in Figure 5(b), with a very stable dose of coagulant added to Train A, the specific sludge productions in the biological reactors were as listed in Table 7. The higher sludge production in Train A was caused by the addition of coagulant in this train. Due to the high total SRT and the easily biodegradable external carbon source, the specific sludge productions were very low, based on the total TCOD in the influent to the biological stage plus the COD in the external carbon source.

Table 7 | Average specific sludge productions for the period from 27 June to 3 October 2019

	Train A (co-precipitation)	Train B (control)
Sludge production, g TS/g TCOD in influent to biological stage	0.36	0.22
Sludge production, g TS/g TCOD supplied, incl. external C-source	0.25	0.15
Chemical sludge production, g TS/g Al added	4.0	_

Diluted sludge volume index (DSVI) measurements on the return sludge showed that the sludge from Train A (co-precipitation) had DSVI from 114 to 151 ml/g TS and settled much better than the sludge from Train B (control) that had DSVI from 353 to 462 ml/g TS. However, with the membrane separation, the high DSVI in Train B was not a problem for the performance of the IFAS-MBR process.

Flux, chemical wash of membranes and pressure loss

The median value for the membrane flux was 11.4 LMH, and the highest membrane flux tested was 14.2 LMH. These fluxes are normal for conventional MBR systems.

Chemical wash of membranes was initiated when the TMP reached 0.2–0.3 bar, corresponding to an in-situ permeability of about 40–55 LMH/bar. Periods between each chemical wash varied from 78 to 91 days. Washing with 1000 ppm NaOCl at 35 °C was followed by washing with 2000 ppm citric acid (at between 10 and 35 °C). The washing solution was slowly pumped through the membranes to ensure good contact with the membrane pores. After chemical washing, the membrane permeability was tested in clean water at ambient temperatures between 18 and 24 °C. Permeabilities (average \pm standard deviation) were 623 \pm 213 LMH/bar for the membrane in Train A (co-precipitation) and 662 \pm 207 LMH/bar for Train B (control). This shows that the chemical wash was very efficient and resulted in similar permeabilities, as reported by Fleischer *et al.* (2005) after successful chemical wash of the same type of membrane. The membrane in Train A always had slightly lower permeability than the membrane in Train B, even when the clean water permeability was measured on the new membranes before they were put into service.

An example of TMP over the membranes is shown in Figure 10. This was 28 days after the last chemical wash and shows that the IFAS-MBR was operated at very low TMPs. During permeate production, the average pressure drop over the membrane was 0.030 bar (0.30 m water column) for both trains, at a flux of 11.2 LMH and 13 °C. This corresponds to an in-situ permeability of about 370–375 LMH/bar. It looks like a lot of noise on the logged pressure signals, but the difference between the minimum and maximum pressure drop during permeate pumping was only 0.008–0.011 bar (0.08–0.11 m water column), which is actually quite low considering the forceful aeration and high turbulence in the membrane reactors. During backflushing the pressure was between 0.040 and 0.044 bar.

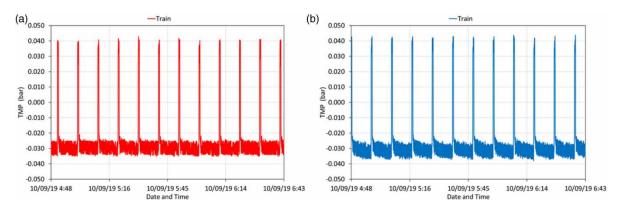


Figure 10 | Example of TMP over the membranes, 28 days after the last chemical wash. Train A (co-precipitation) on the left and Train B (control) on the right.

The experience from this test is that it seems as if fouling of the membrane in an IFAS-MBR (at aerobic SRT of 5–10 days – based on suspended biomass in the IFAS reactor) is not more pronounced than in an ordinary MBR (with typical aerobic SRT of 15–20 days (Judd 2006)). In future studies, one should investigate whether this holds also for even lower SRTs (<5 days aerobic suspended biomass SRT), since conventional IFAS systems with enough biofilm surface area have been shown to nitrify even at such low SRTs.

CONCLUSIONS

A combined pre- and post-denitrification IFAS process with membrane separation (IFAS-MBR) was tested in two parallel small-scale plants, treating primary filtered (by fine sieves) wastewater. The main objective was to evaluate the IFAS-MBR process and investigate the possible negative effect of co-precipitation of phosphorus on the biofilm carriers. Train A had coagulant addition for co-precipitation and Train B served as a control with no coagulant addition.

The IFAS-MBR process worked well at aerobic SRTs as low as 5–10 days, based on the suspended biomass in the biological reactors. Removal of organic matter, nitrogen and suspended solids were very good in both trains. In Train A (co-precipitation), average removal efficiencies were 95% for TCOD, 80% for TN and 100% for SS.

In Train B (control) average removal of TP was less than 35% since only particulate P was removed. In Train A (co-precipitation), at an Al/TP-molar ratio of 1.9, effluent concentrations were very low with averages of 0.10 mg TP/l and 0.08 mg PO₄-P/l, and median values of 0.08 mg TP/l and 0.04 mg PO₄-P/l. Average and median removal efficiencies for TP were 98.8% and 99.1%, respectively.

The specific biofilm nitrification rates in the IFAS reactors were about the same as expected in conventional MBBRs at similar temperatures and DO concentrations. Controlling the biofilm biomass and thickness by intermittent use of an air-lift pump was an efficient tool for optimizing the nitrification on the biofilm carriers in Train A. With biofilm control specific nitrification rates were typically 0.65 g NH₄-N/m²-d at 12–13 °C and 5.2–5.6 mg O_2 /l in Train A.

C/N ratios and reactor effluent NO_3 - N_{eq} concentrations had the most impact on the pre-DN rates and were more important than the temperature. Specific post-DN rates were significantly higher than the pre-DN rates. The main reasons for this were the easily biodegradable external carbon source and the high NO_3 - N_{eq} loads.

Specific sludge production, including the COD added as external carbon source, was $0.25 \, \mathrm{g}$ TS/g TCOD supplied for Train A (co-precipitation) and $0.15 \, \mathrm{g}$ TS/g TCOD supplied for Train B (control). The difference was due to the chemical sludge production in Train A, which was $4.0 \, \mathrm{g}$ TS/g Al added. Average DSVI was $130 \, \mathrm{ml/g}$ TS for Train A and $390 \, \mathrm{ml/g}$ TS for Train B.

Membranes were operated at a flux of 11.5 ± 0.9 LMH. Chemical wash of membranes was initiated when the TMP reached 0.2–0.3 bar. Clean water permeabilities after chemical wash were typically 600–700 LMH/bar, which shows that the chemical wash was very efficient and the membranes were fully recovered.

The tests demonstrated that co-precipitation in an IFAS-MBR process is feasible, and that all the performance goals for the planned full-scale plant were achieved, and hence that this process might be a viable option.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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