Ministry of Environment and Food of Denmark
Environmental Protection Agency

DOGAS

Direct concentration of wastewater for biogas production

MUDP report

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.

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1. Introduction

Developing the key infrastructure for a circular bio economy

The report in hand is the final report for the project DOGAS – direct concentration of wastewater for biogas production", financed by the MUDP program under the Ministry of Environment and Food of Denmark.

The aim of the project was to prove that it is possible to filtrate raw wastewater with new membrane technologies in order to bring the concentration of organic matter in the water up to a level that makes it possible to digest it directly in a biogas reactor. At the same time the nutrients will pass through the membrane to the water fraction, which then can be used as fertilizing irrigation water.

This way the suggested DOGAS solution represents a new approach to wastewater treatment. A solution that instead of using energy for processing the organic matter produces energy doing in so, and in the process avoids emission of not only CO2, but also methane and dinitrogenoxide, which are many times more potent climate gasses. As such the approach has the potential of turning the business model for wastewater treatment plants up side down, from being problem solvers to becoming resource providers for the local community.

The project has the Island of Samsø as test location. Samsø has the ambition of becoming a model island for a circular bio economy, where all the biological resources are kept in close loops, among them the resources in the wastewater. As the proposed solution can make it possible to recirculate both carbon, water and nutrients it can become a key infrastructure for such a future circular bio economy if successful.

Circular island

The project was conducted from November 2016 until February 2018 at the locations of the different partners and at Samsø Spildevand, Nordby Plant during the pilot test phase.

It was carried out in conjunction with another MUDP project on Samsø *The Irrigation Symbiosis*, which solely focused on producing nutrient rich irrigation water by running existing treatment technologies differently. The DOGAS idea sprang out of the initial investigations in that project, and was established as a supplement to it. As such the pilot test of the DOGAS solutions was run parallel to the tests in *The Irrigation Symbiosis* at the same pilot plant facilities at Samsø.

The following project partners were involved in the project:
Alfa Laval A/S
Samsø Spildevand, A/S
Minor Change Group Aps
Danish Technological Institute
AL-2 Teknik A/S

2. Summary

Direct concentration of wastewater for anaerobic digestion in biogas reactors is possible and will turn traditional wastewater treatment upside down – when the method is fully developed.

2.1 New approach to wastewater treatment verified

If it by concentrating raw wastewater with use of membranes is possible to process the concentrated wastewater directly in a biogas reactor and thereby gain energy from the organic matter in the water, the proposed DOGAS method will open up new paths for wastewater treatment. After several test runs at a pilot plant at Samsø late summer/early autumn 2017, it is clear, that it is possible to make this direct concentration with membrane technologies.

Two types of membranes were tested, a microfiltration membrane with a porosity of 0,2 µm and an ultrafiltration membrane with as little as 0,01 µm porosity. Surprisingly the very tight ultrafiltration membrane performed best on all parameters, and did not as feared face clogging problems. Both membranes hold back almost all the organic matter in the wastewater, and with that the majority of the organic xenobiotics and heavy metals as well as viruses and most bacteria. On the contrary nitrogen pass through the membrane in form of ammonium and the same is the case for soluble phosphorous, making the permeate from the filtration valuable as fertilizing irrigation water. With concentration levels of polluting substances far below threshold values, it is also suitable for this purpose. As such the pilot test runs at Samsø verified the method as promising new approach to wastewater treatment.

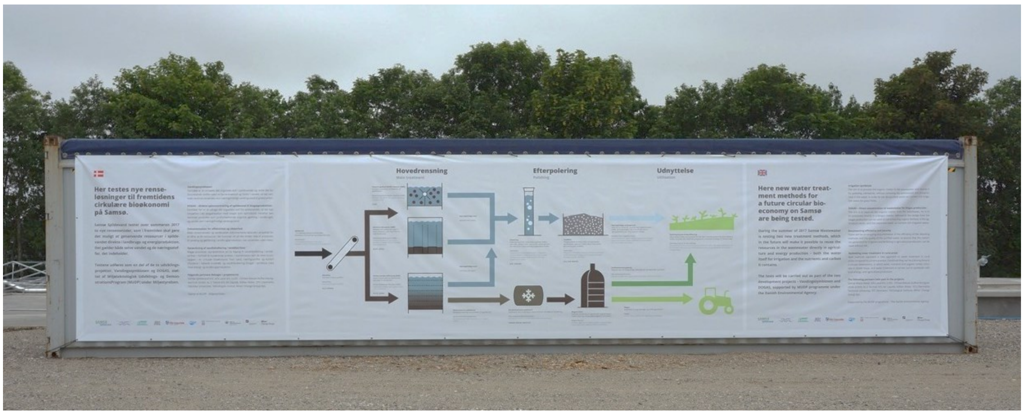


Figure 1. Display of combined processes in the *DOGAS* and *Irrigation Symbiosis* projects on pilot container at Samsø.

2.2 Further development for realizing potential

Due to difficulties avoiding biological activity and other challenges during the test runs, further research and development of the process will have to be done before the method is ready for full-scale implementation. In the closing phase of the project it therefore was decided to establish a follow up project to complete the development of the DOGAS method for making it ready for full-scale operation – and at the same time develop the full integration with biogas production and agricultural production. This compete system solutions can become a key infrastructure in a future circular bio economy – at Samsø and elsewhere in the world.

3. Dansk resumé

Det er muligt at foretage en direkte opkoncentering af spildevand mhp. anaerob omsætning i biogasreaktorer. Når metoden er færdigudviklet, vil den betyde et nybrud med den måde man hidtil har tænkt og praktiseret spildevandsrensning på.

3.1 Formål og perspektiv

Formålet med *DOGAS – direkte opkoncentrering af spildevand til biogasproduktion* var at vise, at det ved hjælp af nye membranteknologier er muligt at filtrere det organiske stof (COD) fra spildevandet, så der kan opnås en så høj koncentration, at det kan omsættes direkte i en biogasreaktor med meget kort opholdstid. Herefter kan det afgassede slam anvendes som jordforbedrende organisk gødning i landbruget. Samtidig vil kvælstof og fosfor passere gennem membranen ved filtreringen, mens forurenende stoffer tilbageholdes, hvorved vandfraktionen kan anvendes som sikkert næringsholdigt vandingsvand.

Den foreslåede DOGAS metode repræsenterer herved en ny tilgang til vandrensning, hvor man i stedet for at bruge energi på at omsætte det organiske stof producerer energi. Udover at spare atmosfæren for CO2, undgår man også udslip af de mange gange mere potente klimagasser metan og lattergas, som der er risiko for ved den traditionelle aerobe omsætning i åbne tanke. Tilmed kan metoden potentielt vende forretningsmodellen for Spildevandsanlæg på hovedet ved at gå fra en omkostning til rensning af vandet for organisk indhold til at genere profit ved at levere energi og ressourcer til landbrugsproduktion.

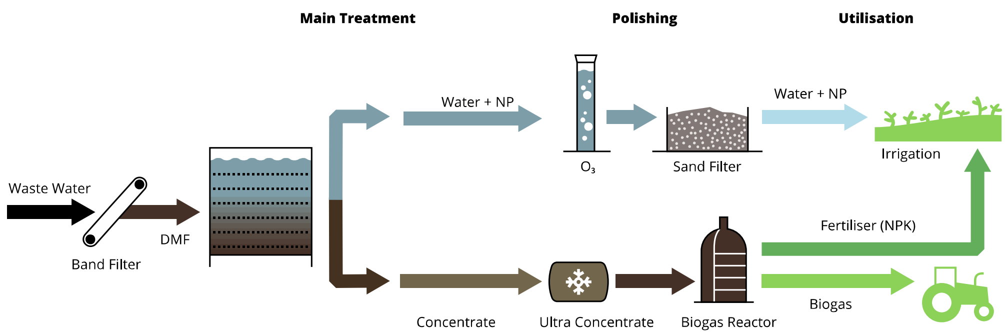
3.2 Baggrund

Ideen til projektet er udsprunget af at lægge et cirkulær økonomisk tankesæt ned over spildevandsrensning i byområder omgivet af landbrugsland. Projektet har derfor haft Samsø som omdrejningspunkt, der i tråd med sin profil som grøn ø, har sat sig for at blive en modelø for en cirkulær bioøkonomi. En økonomi, hvor anvendelsen af de biologiske ressourcer sker i lukkede kredsløb, så de bliver på øen og der kan skabes værdi af dem igen og igen. Det gælder ikke mindst vandet og de næringsstoffer, som transporteres rundt med vandkredsløbene.

Der var derfor allerede blevet igangsat et andet MUDP projekt på Samsø - *Vandingssymbiosen*, hvor det skulle undersøges, om det et muligt at køre eksisterende vandrenseteknologier på en ny måde, så næringsstofferne kunne bibeholdes i vandet så at det kan genanvendes som næringsholdigt vandingsvand i den intensive landbrugsproduktion på øen. I de indledende undersøgelser kom DOGAS i spil som en ny rensemetode, som kunne være interessant at udvikle og teste parallelt med de eksisterende. Partneren Alfa Laval, som leverede en membran bioreaktor (MBR) til Vandingssymbiose-projektet, havde et år forinden gennemført forsøg på Lunds Universitet med direkte opkoncentrering af spildevand vha. deres membraner, med lovende resultat. Det var dog i meget lille skala, som det var vanskeligt at vurdere potentialet ud fra. Når membranerne alligevel skulle stilles op til pilotkørsel i Vandingssymbioseprojekter, var det oplagt i forlængelse af disse forsøg, at lave testkørsler med Alfa Lavals membraner i DOGAS drift, for at få viden om, hvordan metoden fungerer i større skala og dermed vurdere potentiale i at udvikle den til færdigt stadie. DOGAS projektet blev derfor etablereret som et supplement til *Vandingssymbiose* projektet og gennemført parallelt med pilotfasen i dette projekt i løbet at 2017.

3.3 Projektforløb

Projektet blev indledt med undersøgelser af sammensætningen af spildevandet, som tilflyder små og mellemstore renseanlæg i landområder og dernæst en specifik analyse og karakterisering af spildevandet ved Nordby Renseanlæg på Samsø. På baggrund blev det samlede pilotanlæg til testkørslerne konfigureret og opbygget i containere som blev flyttet til Samsø. Som vist i figur 1 nedenfor var der før membranfiltreringen installeret et båndfilter (AL-2 teknik), som fjernede de grovest partikler fra spildevandet, så de ikke tilstoppede membranerne. Efter membranfiltreringen blev vandet fra processen ledt gennem en to-trins efterpoleringsproces. Første trin var en ozonering for at nedbryde eventuelle rester af organiske MFS'er. Ozoneringen hygiejnisererede samtidigt vandet. Derefter blev det ledt gennem et sandfilter, som fanger eventuelle rester af tungmetaller, som måtte være tilbage i vandet efter hovedrenseprocessen.



Figur 1: Set up for pilotanlæg på Samsø

Pilotanlægget ved Nordby Renseanlæg kørte i perioden 15. september – 22. oktober med direkte membranfiltrering til opkoncentering af spildevandet. Der blev her kørt med 2 forskellige membrantyper. En ultra filtreringsmembran (UF) på ~0,01µm, og en mikrofiltreringsmembran (MF) på 0,2 µm.

Gennem hele testforløbet er der blevet taget prøver af vandet, som er blevet analyseret for COD reduktion, og fjernelse af organiske miljøfremmede stoffer (oMFS) og tungmetaller – og bevarelse af næringsstoffer (N og P) i vandet. Det er blevet gjort ved såkaldte koncentrationsprofiler, hvor man følger en vandprotion gennem systemet ved at time prøvetagningstidspunkterne efter de hydrauliske opholdstider ved hvert trin.

3.4 Resultater og observationer fra DOGAS-pilot driften

Driften af DOGAS set-uppet i godt en måned gav en klar indikation af, at det er muligt at fortage en direkte opkoncentrering af spildevandet vha. membraner - kort sagt at metoden virker efter hensigten. Meget overraskende viste den tætte UF membran at performe bedst. Pga. den forholdsvis korte driftsperiode, var de ikke muligt at få et klart billede af driftseffektiviteten over tid.

Analyserne viste en høj tilbageholdelse af COD med lavere COD koncentrationer i permeatet end typisk ved biologisk rensning. Samtidig passerede næsten al kvælstof i form af ammonium samt det vandopløste fosfor igennem membranen. Desuden var der en lige så høj tilbageholdelse af oMFS'er og tungmetaller, som ved den biologiske rensemetode, dvs. tæt på 100%.

Udfordringen ved driften af DOGAS metoden er at undgå biologisk aktivitet i membranreaktoren. Det var vanskeligt ved pilotanlægget pga. svingende COD og ammonium koncentrationer i spildevandet, og for lang opholdstid i buffertanken ved renseanlægget i Nordby, hvorved den biologiske omsætning allerede var igangsat. Fremadrettet skal der køres med meget kort opholdstid – max 1 time og en kontinuerlig tilførsel af frisk spildevand. Ved et opfølgende projekt vil det derfor være bedre at køre driftsforsøgene ved det større renseanlæg i Ballen. Forholdet mellem temperatur og opholdstid er også afgørende for den biologiske aktivitet. Det kan derfor overvejes at køle den luft der pumpes ind over membranerne for at modvirke fouling, til max 15. °C for at begrænse den biologiske aktivitet. Der kunne også anvendes andre luftarter, som hæmmer biologisk aktivitet, f.eks. CO2 eller Ozon.

En anden driftsudfordring er fouling på membranerne, som gradvis nedsætter kapaciteten, indtil de bliver renset. Membranerne kan under normale omstændigheder køre i ca. 3 uger mellem rensninger (CIP – clean in place). Renseintervallerne afhænger bl.a. af vandets hårdhed, idet der er større kapacitet ved blødt vand. UF membranerne kan renses med kaustisk soda (natriumhydroxid), hvilket er at foretrække, da det er langt mere miljøvenligt end klor (natriumhypoklorit), som MF membranerne er nødt til at blive renset med. Membranerne kan evt. også renses løbende ved at tilsætte enzymer eller anvende ozon. For at få et mere klart billede af rensebehovet, vil det som optakt til en efterfølgende test af driften i fuld skala, være interessant at lave en driftskørsel i mindre skala, hvor man undersøger sammenhængen mellem flow og opkoncentreringsrate over tid.

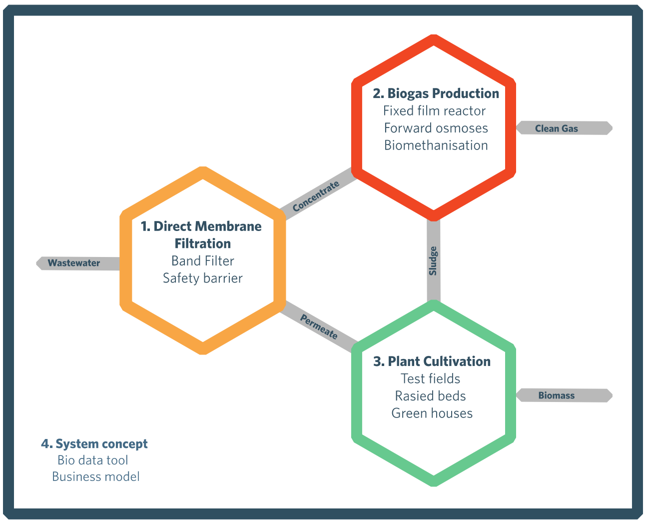
3.5 Konklusioner på DOGAS pilotdrift

MF og UF membraner kan anvendes til direkte opkoncentrering af spildevand – UF membranen performer bedst. Næsten al ammonium og opløst forfor trænger igennem membranerne til permeatet, mens stort set alle oMFS'er og tungmetaller tilbageholdes i koncentratet. For at få optimal drift af DMF teknologien, skal den biologiske aktivitet begrænses mest muligt – vha. kort opholdstid (max. 1 time) og køling af luft (max 15 °C) eller anvendelse af en luftart, som hæmmer biologisk aktivitet. For at kunne teste driftsikkerheden af DOGAS metoden bør der være et flow på 1 m^3 i timen ved et forsøgsanlæg i et evt. opfølgende projekt.

3.6 Opfølgende Innovationsfondsprojekt - FERM

I slutfasen af projektet (februar 2018) blev der udarbejdet og indsendt en ansøgning til Innovationsfonden om sådant opfølgende projekt - *FERM – Fertigation Water from Direct Membrane Filtration of Wastewater* - hvor DOGAS metoden skal færdigudvikles og kobles tæt sammen med hhv. biogasproduktion og dyrkning med det næringsholdige vand i en tæt symbiose. Et samlet systemkoncept (se figur 3), som kan blive krumtappen i en fremtidig cirkulær bioøkonomi, hvor de biologiske ressourcer anvendes og regenereres i tætte kredsløb. Ansøgningen blev dog ikke imødekommet pga. dens kompleksitet med 3 elementer i spil. Men konsortiet blev anmodet om at genindsende projektet i en forsimplet form med fokus alene på færdigudvikling af DMF teknologien. Det vil ske i august 2018.

Figur 3:
De 3 elementer og det samlede koncept i FERM projektet



4. Designing solution for concentration of municipal wastewater

4.1 Wastewater composition in small municipal WWTPs

In order to design and run the pilot plant according to the conditions at Nordby, Samsø a literature study of the wastewater composition in small municipal wastewater treatment plants (under 5000 PE) was carried out in and compared with analysis of the wastewater flowing to the plant at Nordby. The main part of this work was carried out in *The Irrigation Symbiosis* project and is summarized in the tables below. They give an overview of the available data for 14 small municipal WWTPs compared with measurements for a period of 5 months (test period) at Nordby WWTP on Samsø:

Nutrients

Nutrient salts have been shown as average values for Total-N (nitrogen) and Total-P (phosphorus) in the following Table 1. The standard deviation has been indicated in brackets after the value.

Table 1. Average nutrient salt concentration in small municipal WWTPs.

	Total-N [mg/l]	Total-P [mg/l]
Average 14 plants < 5000 PE	46 (20)	7.9 (3.1)
Nordby (Samsø)	83 (25)	9.6 (2.8)

The Total-N level at Nordby WWTP is thick, while the average level of small WWTPs and is classified moderate. Total-P content is classified between thin and moderate both in Nordby WWTP and the average of the inlet water in all small WWTPs.

Organic micro pollutants

Standard deviation has been indicated in brackets after the value. Inlet values for Nordby WWTP are based on 4 measurements divided across a period from May to October 2017.

Table 2. Average concentration of organic micro pollutants in small municipal WWTPs

	LAS (Linear alkylbenzene sulfonate) [µg/l]	Σ PAH (Polycyclic aromatic hydrocarbons) (11 substances) [µg/l]	Σ NPE (Nonylphenol ethoxylates) [µg/l]	DEHP (Di(2-ethylhexyl)phthalate) [µg/l]
Average 14 plant < 5000 PE	2757 (2031)	0.3 (0.3)	2.8 (2.6)	14.4 (10.7)
Nordby (Samsø)	2725 (263)	0.6 (1.0)	1.6 (1.2)	3.5 (2.4)

The average wastewater for all 14 plants < 5000 PE is classified according to Henze[1] in relation to organic micro pollutants as very thin, except PAH, which is between very thin and thin. The same may be attributed to inlet water from Nordby.

Heavy Metals

The following Table 3 shows the average concentration of heavy metals in the influent of small WWTPs

Table 3. Average concentration of heavy metals in small municipal WWTPs

	Cadmium [ng/l]	Quicksilver [ng/l]	Lead [µg/l]	Nickel [µg/l]	Chrome [µg/l]	Zinc [µg/l]	Copper [µg/l]
Average 14 plants < 5000 PE	178 (168)	85 (106)	7 (7)	6 (3)	5 (3)	145 (95)	42 (26)
Nordby (Samsø)	186 (157)	21 (41)	3 (2)	7 (4)	4 (3)	169 (168)	36 (38)

The average wastewater from 14 WWTPs < 5000 PE is classified in relation to heavy metals as very thin, except copper (thin) and zinc (between thin and moderate). The same is valid for inlet water in Nordby.

4.1.1 Conclusions on Nordby wastewater composition

Nordby wastewater is generally comparable to the average indicators of wastewater received by the small Danish WWTPs < 5.000 PE.

However, the content of nitrogen is higher and is classified as thick wastewater according to literature. Thus, there is an even higher potential in keeping nutrients in water in order to use it as nutrient-rich field irrigation water.

The impact of organic organic micro pollutants in Nordby was small in all 4 analyses over a period of 4 months. The impact of heavy metals was also very small and corresponds to the average impact of small WWTPs.

Low COD can also been explained due to low flow generating a long residence time in sewer and/or in buffertank. High ammonia is somewhat unusual for pure municipal water.

4.2 Legislative framework for concentration of wastewater solution

A desk top survey the legislation regarding reuse of treated wastewater in agricultural production Concentration of Wastewater by using membrane also carried out in *The Irrigation Symbiosis* project, concluded that it will not conflict with the actual legislation to reuse of the treated wastewater for irrigation. But it would be subject to an approval by the authorities. It has to be guaranteed, that bacteria and virus are removed, eventually with post treatment after the membrane filtration (UV, Ozone). And for it to become a common praxis, impact of possible presence of micro pollutant on crops when irrigating with the water has to be investigated and safety documented, even though the majority of organic micro pollutant as well as heavy metals are removed in direct filtration.

[1] Teoretisk Spildevandsrensning – biologiske og kemiske processer, Mogens Henze, Poul Harremoës, Jes la Cour Jansen, Erik Arvin, Polyteknisk Forlag, 3. udgave 2006, ISBN 87-502-0942-6

4.2.1 Specifik legislative framework in Denmark EU

According to the reviewed legislation, it may be concluded, that treated wastewater can be used as field irrigation water in Denmark upon the permission by Danish Nature Agency and local authority. First, the Nature Agency must consider whether the treated wastewater has an agricultural value. If this is the case, the threshold values specified in Sludge ministerial act may be applied, and the local authority can give a permit.

4.2.2 Regulative framwork in EU regarding reuse of water

At EU level, a new Directive is being drafted that will regulate the minimum requirements for water quality for reuse in agriculture (Development of minimum quality requirements at EU level for water reuse in agricultural irrigation and aquifer recharge). The directive is being drafted, and it is not yet announced, when the directive is expected to be implemented.

Recently (on 10 May 2017) a public consultation analysis report prepared for the European Commission - ”Policy options to set minimum quality requirements for reused water in the EU - analysis of open public consultation”[2] was published. The report summarizes the perception of different countries on both safety of reused water as well as specific aspects to be covered by EU minimum quality requirements on a very general level.

4.2.3 Relevant legal requirements in the USA

The water related legislation differs from state to state. Therefor the scope/possibilities for reuse of water are very different.

For drinking water two overall relevant law texts exist on the federal level
- “Safe drinking water act”[3]
- “Clean water act”[4]

The purpose of “Safe drinking water act“ is the securing of the drinking water quality, comprising the protection of the ground water resources for pollution. The ”Clean water act” is the basis for effluent regulation to recipients in the USA. Requirements for the quality of surface water are part of this act.

There is no federal legislation, that comprises water for reuse for non drinking water applications. Furthermore the regulation for reuse of water in the different states is not based on scientific risk analyses. The ”Guidelines for water reuse: EPA 645-R-04-108” from the USAs Environmental Protection Agency specify a number of recommendations with regard to quality requirements, if the water is to be used for field irrigation. These quality requirements cover number of typical wastewater parameters (BOD, dry matter and turbidity), pathogenic microorganisms and several organic micro pollutants including pharmaceutical active substances. Generally it is not prohibited to use treated wastewater for field irrigation in the USA.

[2] http://ec.europa.eu/environment/water/pdf/WaterReuse2ndConsultation-Report-and-Annex-COM.pdf

[3] https://www.epa.gov/sdwa

[4] https://www.epa.gov/laws-regulations/summary-clean-water-act

4.3 Design of pilot scale plant for Samsø

The DOGAS pilot scale plant at Samsø consisted of the elements shown in Figure 1 below. Before the membranes a belt filter was installed in order to remove large particles from the wastewater. After filtration the permeate was lead to an ozonation step to remove eventual remaining organic xenobiotics and a sand filter to capture residues of heavy metals. Test of freeze drying of the concentrate for concentrating it further was carried out as well.

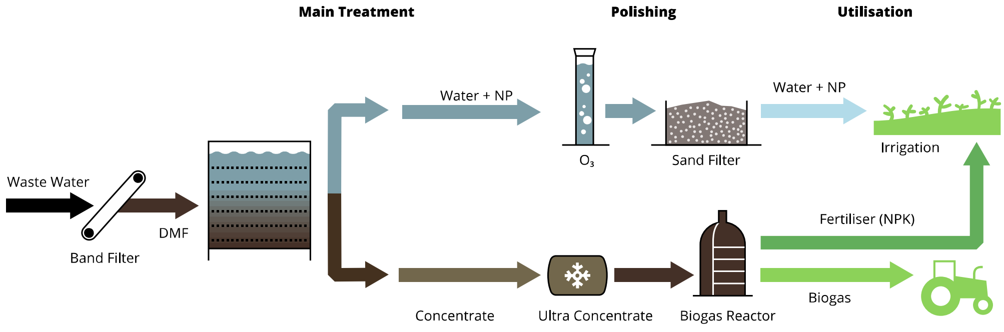


Figure 1: Set up for the pilot plant at Sam

The pilot membranes used in *The Irrigation Symbiosis* project was modified from a MBR setup to a direct filtration unit. In the first period the two lines were operated with PVDF membranes with a porosity of 0,2 µm. In a second phase one of the line with PVDF membrane was modified and an ultrafiltration membrane type UFX (hydrophilic membrane) with porosity of 10 K Dalton was installed (about 0,01 µm), to evaluate the improvement in retention.

4.3.1 Belt filter

An AL-2 belt filter with a filter band, pore size 150 µm (see

Figure) was used as a prefiltration step to prevent membrane clogging in membranes. The belt filter was dimensioned to continuously produce a flow of minimum 260 l/h to feed the tanks. The water was pumped from the full scale SBR reservoir tank containing water after sand/grease treatment of the Nordby WWTP. The belt filter operated very stable during the

whole experimental period.

Figure 2. Belt filter installation at Samsø

4.3.2 Membrane setup

Alfa Laval MBR pilot scale used for membrane test is shown in
Figure was used for normal MBR treatment of wastewater. The system was mounted in a 20' container with the membranes (two similar lines) in one end. The roof at that part of the container could be lifted for access to the tanks.

Figure 3. MBR pilot scale system seen from inside the container, with two lines. One equipped with a PVDF membrane 0,2 µm and a ultrafiltration membrane type UFX of hydrophilic type with a porosity of about 0,01 µm. Each module had a membrane area of $9m^2$.

The pilot plant consisted of components and prefabricated piping ready for operation when installed and connected to the local water streams and power supply systems. The container system included a feed section, two anoxic/aeration-MBR tanks with an aerated volume of approximately 0,9 m^3 in each of them. In each membrane tank, a hollow sheet membrane filtration module was installed as well as an incorporated PLC/MCC. The pilot plant was intended for semi-automatically controlled MBR production in non-Ex classification zones.

The plant was operated in direct filtration mode and with the lowest possible retention time to reduce microbial activity as much as possible:

Figure 4: The pilot container at Nordby, Samsø

5. Pilot scale test for concentration of wastewater

Evaluation of operation and results from analysis program during pilot test runs at Samsø.

5.1 Operation of pilot

The pilot scale tests were conducted at the Nordby Wastewater plant (Samsø Spildevand A/S. The used water was pumped from a reservoir behind the grit and fat removal step and pre-treated on a belt filter equipped with a cloth of 150 micron (picture 1). The pilot plant with 2 parallel lines used in *The Irrigation Symbiosis* project was modified in the beginning of July 2017 to reduce the retention time in the membrane part, and in beginning of September 2017 each line was equipped with new membrane filtration modules. One line was equipped with a microfiltration membrane of PVDF type 0,2 micron and the other with an ultrafiltration membrane of hydrophilic Polysulfone membrane type UFX of 10 K Dalton (0,01 micron).

The pilot plant was operated in continuously mode for concentration of wastewater. During 2 months each line was operating continuously for evaluating following factors:

Belt filtration operation

- Belt filtration unit worked perfectly and a good reduction of suspended solids.was acchived

Capacities on membranes over time.

- An average flux of 6 l/m^2/h in gravity operation was obtained on the two membranes.

Concentration vs volume factor.

- The volume vs concentration factor was rapidily limited due to bacteria development in membrane filtration tank, which reduced COD over time.

COD reduction, Nitrogen reduction, P reduction as well as suspended solids.

- As mentioned above it was difficult to concentrate COD in the membrane system due to bacteria development biodegrading the organic part. Nitrogen in form of ammonia had a high permeability through the membrane as expected, as well as soluble phosphorous (orthophosphate).

Fouling tendency on membrane

- Compared to MBR operation had a higher fouling tendency of membrane but still on an acceptable level for having an economical operation and membrane life time. Organic fouling was probably heavier due to the bacteria activity generating EPS material. It can be expected that fouling will be different if bacteria activity can be avoided.

Cleaning procedure on membrane

- Microfiltration membrane was cleaned with a sodium hypochlorite solution the same way as a traditional MBR. Cleaning was successful and water flux was recovered.
- Ultrafiltration membrane was cleaned with a caustic soda solution at pH 11 but cleaning efficiency was not perfect probably due to low pH. UFX membranes can be cleaned at pH 13, which probably will improve the efficiency.
- It will be an advantage to use caustic soda instead of sodium hypochloride to avoid the formation of chloramine.

Figure 5:
One of the two membrane lines in operation

5.2 Results from analysis program

During the test runs of the membranes, an analysis program was carried out, testing for the performance on the parameters: COD concentration, nutrient retention in the permeate and removal of heavy metals and organic xenobiotics from the water.

5.2.1 Common wastewater parameters

The following figures show the common wastewater parameters analyzed in the inlet and permeate of the two membrane tanks with microfiltration membrane (MF) and ultrafiltration membrane (UF) respectively.

Figure 6 shows the COD concentration in the inlet and permeate (out) of both filtration tanks. The absolute largest part of the COD was particle bound and was retained in the concentrate. The permeate had a very low COD concentration with the ultrafiltration membrane having been more effective (< 55 mg/l) than the microfiltration membrane (<73 mg/l) as expected.

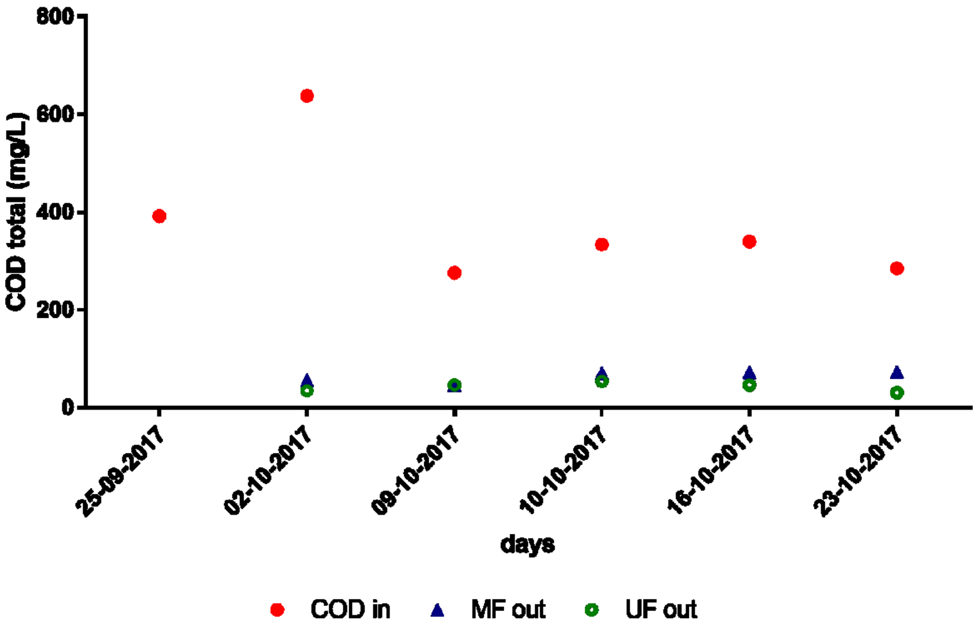


Figure 6: COD in the inlet and permeate (out) of microfiltration and ultrafiltration tanks.

Figure 7 shows the total nitrogen in the inlet water and the ammonium in the permeate. In the beginning of the experiment the nitrogen was kept efficiently in the permeate. Due to microbial decomposition of the ammonia afterwards, the ammonium in the permeate was lower than in the inlet water. This can be prevented with a different reactor design, allowing very low retention time for the water, so that sludge formation is prevented.

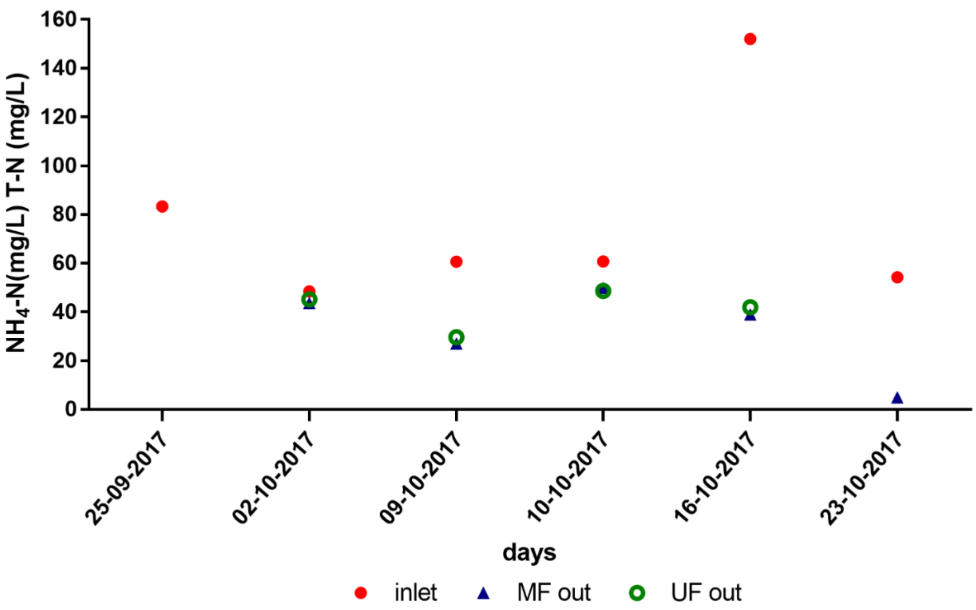


Figure 7. Total nitrogen in the inlet and ammonium nitrogen in the permeate of the microfiltration and ultrafiltration tanks

5.2.2 Removal of organic micro pollutants

The membranes potential to remove organic micro pollutants from the permeate was tested with spiking profile experiments, where inlet water spiked with organic micro pollutants was sampled in the outlet after one hydraulic retention time. The spiking concentration for the organic micro pollutants was 10 times the naturally abundance in small WWTPs. LAS was not spiked, as a trial in laboratory scale lead to heavy foam formation, which was undesirable in pilot scale.
Figure 8 and 9 on the following page show the removal of organic micro pollutants and heavy metals from the permeate.

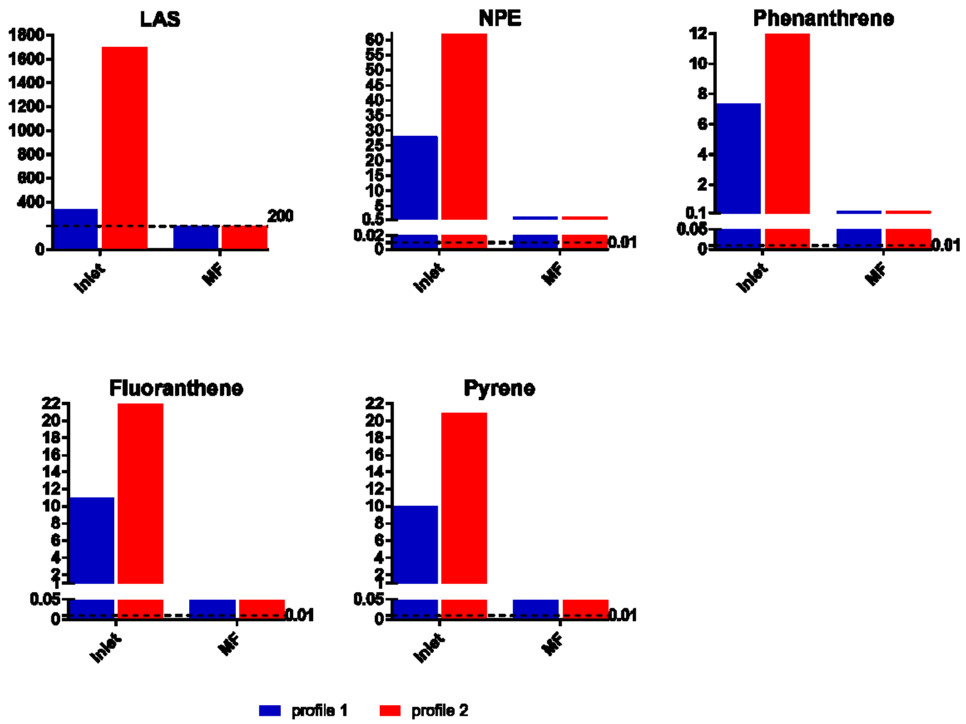


Figure 8: Spiking profiles of organic micro pollutants. The dotted lines correspond to the detection limit for each compound.

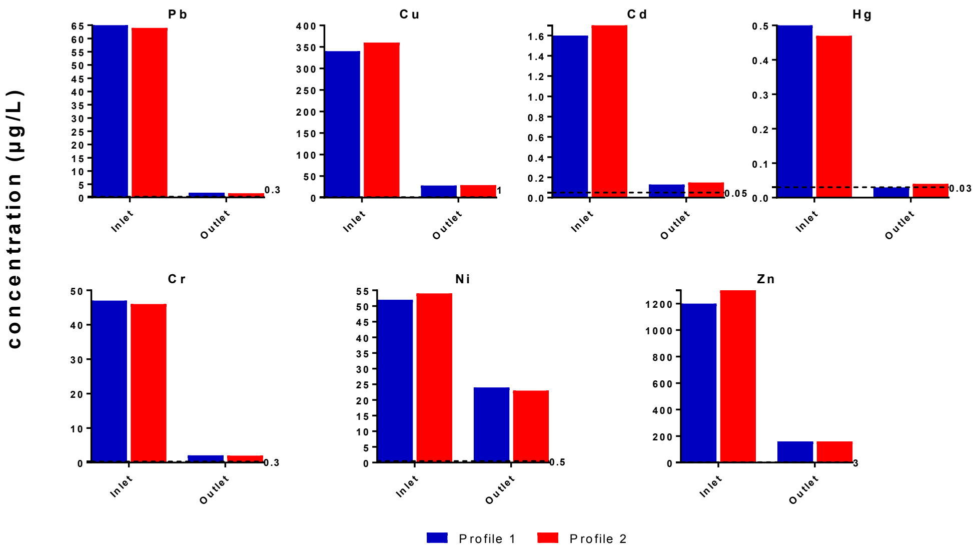


Figure 9. Spiking profiles of heavy metals. The dotted lines correspond to the detection limit for each compound.

Both the organic micro pollutants and the heavy metals were almost completely removed from the permeate even at peak concentrations in the inlet water (the 10 times higher than normal). A comparison of similar results from *The Water Symbiosis* tests, with tress hold values stated in Sludge Act, which regulates the use of treated wastewater for irrigation, shows that's these concentrations are a factor 10-1000 below. As such the permeate is suitable for irrigation.

5.3 Freeze concentration

A freeze concentration step was designed after the membrane concentration to further concentrate the wastewater. The following test was conducted as a proof of concept and estimation of the cost and benefit of such a technique.

5.3.1 Freeze concentration technique

In the freeze concentration process the incoming waste water stream is divided into further COD concentrated water and less COD containing "clean water".

The vacuum technology used is shown in figure 9.

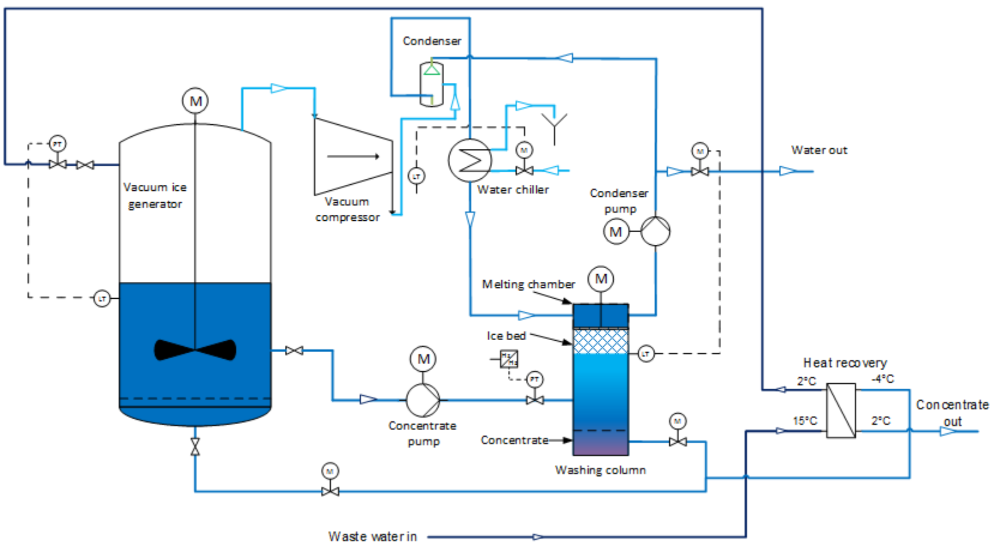


Figure 10. Vacuum technology for freeze concentration of wastewater

The vacuum freeze concentration unit contains following equipment:

- Vacuum ice generator.
- Vacuum compressor
- Condenser
- Washing column (including the concentrate part, ice bed and melting chamber)

The wastewater flows into a heat recovery heat exchanger before entering the vacuum ice generator. The heat exchanger recovers heat from the concentrate flowing out of the washing column and use it to cool down the waste water flow into the freeze concentration equipment. The part of the water that evaporates in the vacuum ice generator is compressed by the compressor and discharged to the condenser. The condenser uses a direct heat exchange, which could be the water vapour from the compressor outlet, which is condensed directly into the water flowing from the washing column.

Ice and concentrate is pumped from the ice generator to the washing column. In the washing column, the ice crystals float up due to gravity to the top and are separated and washed on their way to the melting chamber. In the melting chamber the ice crystals are melted to produce water. The water is then discharged from the system. By keeping a pressure on the pure water circuit a water front can be maintained in the washing column where ice will pass through on its way to the melting chamber. In this way, the concentrate is washed out of the ice. The concentrate is removed from the bottom of the wash column through the heat recovery heat exchanger on its way out of the freeze concentration unit.

The system can be constructed energy producing and thereby reduce the payback time by upgrading the heat from the condenser through a heat pump to the district heating system and by using the cold water from the ice melting for chilling purposes as shown.

5.3.2 The test setup for freeze concentration

The applicability of the freeze concentration technique on wastewater was tested with the following test setup (se figure 10). The test was conducted with ten times concentrated synthetic OECD wastewater as this concentration was expected after the membrane concentration step. The waste water sample was placed in a barrel.

Figure 11. Test setup for freeze concentration

The freezing plate containing a cavity with cold refrigerant was dipped into the barrel and froze the water out of the sample to the exterior of the plate. After two hours, the plate was moved into the ice container. Hot refrigerant gas was pushed through the plate to release the ice, which dropped into the ice container. The ice was weighted and a sample analyzed for COD. Thereafter the freezing plate was lowered into the barrel again and refrigerant pumped through the plate to repeat the process.

5.3.3 The results

A wastewater sample was taken prior to the test. During the test samples of the ice were taken periodically as well as a sample of the concentrate at the end of the test. All samples were kept in the freezer for subsequent COD analysis.

Figure 12 shows the concentration of the waste water as well as the purity of the ice frozen out of the wastewater

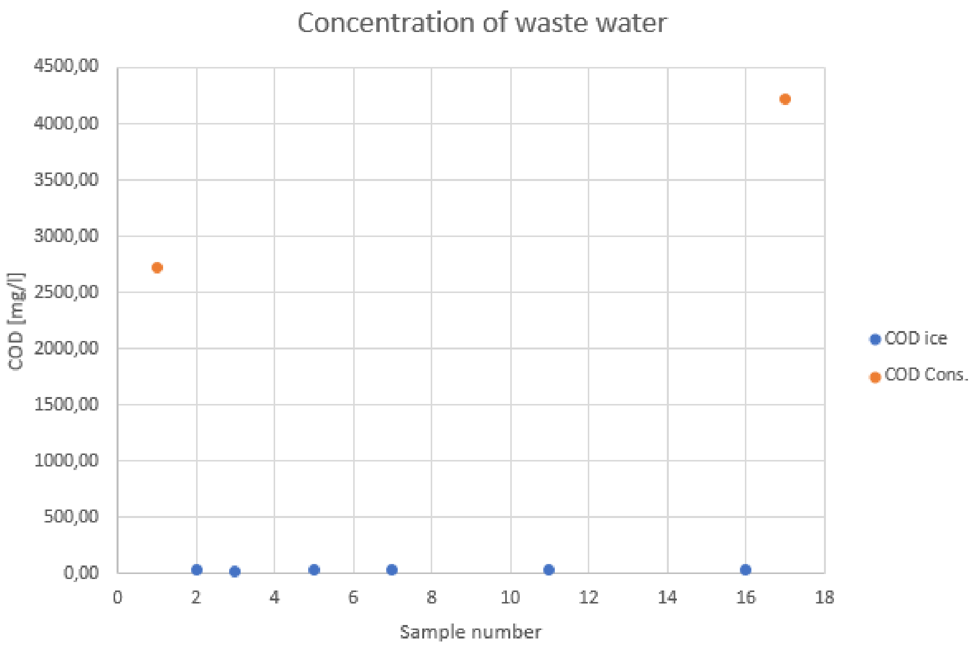


Figure 12. COD concentration in the ice (blue dots) and the concentrate (orange dots)

The COD start concentration of the wastewater was 2718 mg/l. The separated ice had a very high purity that ranged from 24.4 mg/l COD to 40.05 mg/l with an average of 32.03 mg/l. The concentrate at the end of the test had a COD concentration of 4525 mg/l corresponding to a concentration factor of 1.7 during the test.

The test as a proof of concept showed the applicability of the freeze concentration technique for concentration of wastewater in a system described in section 5.3.1. It is technically possible to scale up the process for a fullscale application that performs a concentration factor 10.

5.3.4 Estimated price of equipment

For the estimate of a price for the equipment and the operation of a freeze concentration the following assumptions were made:

- Average wastewater amount 75 m³/day (calculation based on 10 times concentrated water after a membrane step of a 5000 PE WWTP; 1 PE = 150 l/day).
- Average wastewater temperature 15°C.
- Concentration factor 10.

The price is a rough estimate based on the system shown in

without upgrading of the system for energy production as described in section. The initial cost of the equipment is estimated to 1.8 million DKK and the running cost per year 0.19 million DKK.

The suspended sludge concentration in Nordby WWTP after the belt filter was in average 0,15 g/l, resulting in a concentration of 15 g/l after the membrane concentration step and the freeze concentration step (concentration factor 100). With the assumption, that suspended sludge corresponds to volatile solids (SS=VS), a methane production of 350 ml CH4/g VS and a price of 4 kr./m3 methane, the concentrated wastewater after the freeze concentration step is worth 21 kr./m3.

5.3.5 Conclusion

The test showed that freeze concentration can be a feasible solution for concentrating wastewater for biogas production, both technically and financially.

5.4 Biogas potential of different fractions in the process

The biogas potential of the following fractions in the process was measured:

- Screening refuse from band filtration
- Sludge from belt filter
- Residue from grease separator

Due to the occurring biological activity in the direct filtration tanks, which presumable was started already in the buffer tank of the Nordby WWTP because of to long retention time, no concentrate was available for measurement of biogas potential.

The biogas potential was determined by using Bioprocess Control-equipment (Measurement protocol for biogas potential measurements for verification tests (ETV, CBMI), v6, 16/5-2011), methode 2: Measurement using the bioprocess control system).

The following approach was used for the tests:

- Thermophilic anaerobic digestion at 51 °C
- Triplicates of the samples plus blind sample
- Dry matter measurement: 105 °C, 48 hours
- Volatile solids: 550 °C, 24 hours
- The samples were added with a concentration of 7.5 or 10 gVS/l respectively
- Triplicates of a cellulose reference

The data for the accumulated methane production are presented as measured values in nml CH_4/gVS after fully completed anaerobic digestion

5.4.1 Results – dry matter and volatile organic substances

The amount of dry matter (DM) and volatile organic substances (VS) was determined in the sample. The results are shown in Table 4.

Table 4. Dry matter and volatile organic substances in samples

Sample	DM [%]	Standard deviation (DM) [%]	VS [VS-%]*	Standard deviation (VS) [%]

Screening refuse from band filtration	35.71	1.94	81.06	0.30
Belt filter sludge	5.94	0.58	75.64	1.10
Residue from grease separation	18.93		78.01	

*calculated as 100 – loss of volatile organic substances on the base of dry matter

5.4.2 Results – Biogas

The anaerobic digestion was completed after a period of approximately 32 days, where the accumulated methane yield was constant. The result for the accumulated methane production after 30 days is shown in table 5.

Table 5. Accumulated methane production after 30 days

Sample	Acc. methane production [Nml CH_4/gVS]	Standard deviation [CH_4/gVS]	Acc. methane production [Nml CH_4/gDM]	Acc. methane production [Nml CH_4/g sample]
Screening refuse from band filtration	247	159	200	71
Belt filter sludge	475	7	359	21
Residue from grease separation	430	25	335	64

The data from the anaerobic digestion are shown figures 12-16. The methane production is shown in normal ml methane/g VS corresponding to nm^3/kg VS (VS= organic matter based on dry matter). The calculated methane production is calculated as methane production per g material as well as methane production per g dry matter

The cellulose reference resulted in a methane production of 369 Nml/g VS with a standard deviation of 6 Nml/g VS, which exactly was the expected (375 Nml/g VS +/- 25 Nml/g VS).

Residue from band filtration 10gVS/L

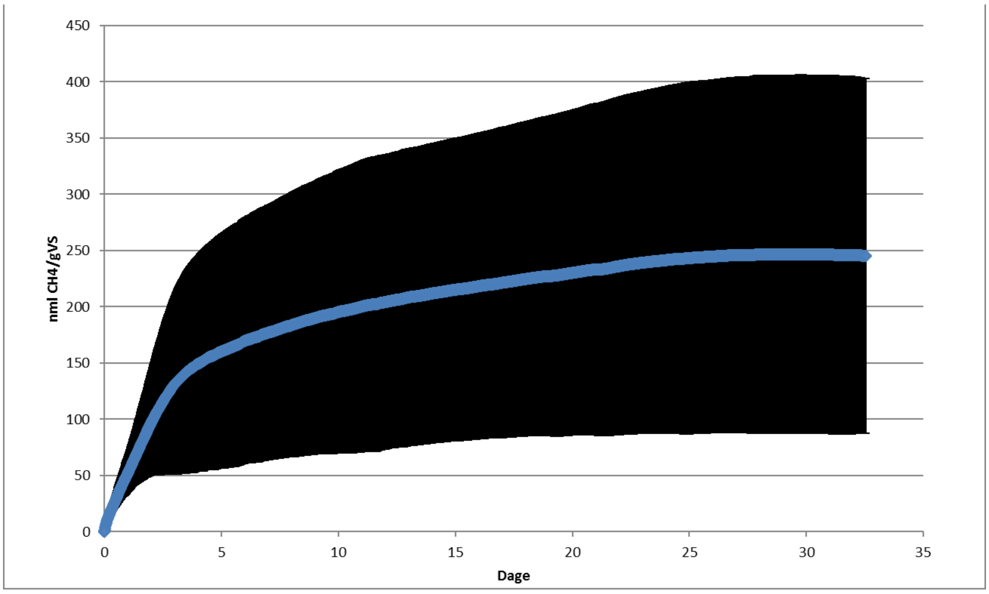


Figure 13. Residue from band filtration accumulated methane production (the black bars are the standard deviation). Thermophilic anaerobic digestion with an organic load of 10 gVS/l.

AS shown in figure 13, the standard deviation of the screening refuse was very high due to the very heterogenous sample, which made a representative sampling for the test sample impossible.

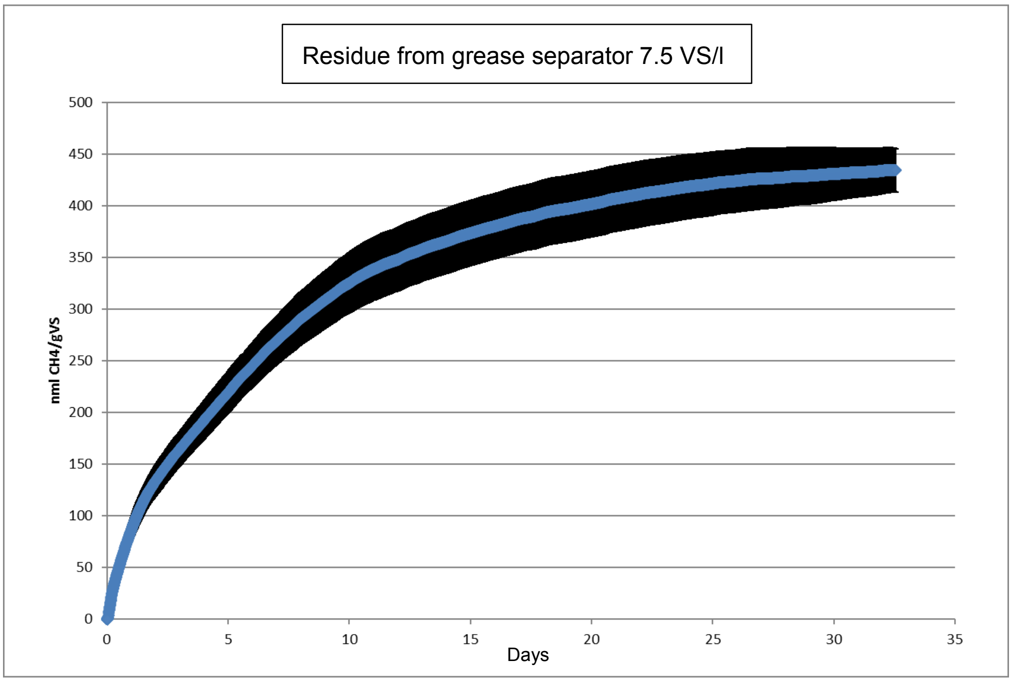


Figure 14. Residue from grease separator - accumulated methane production (the black bars indicate the standard deviation). Thermophilic anaerobic digestion with an organic load of 7.5 gVS/l

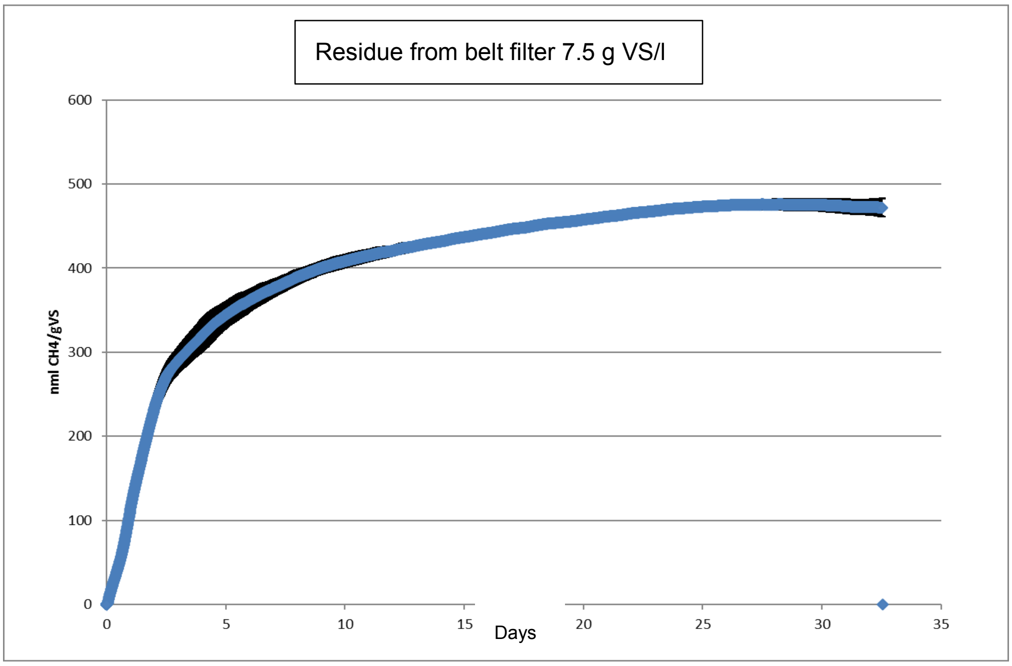


Figure 15. Residue from belt filter - accumulated methane production (the black bars indicate the standard deviation). Thermophilic anaerobic digestion with an organic load of 7.5 gVS/l

The residue from the belt filter was digested relatively fast. The major part of the organic matter was digested after 9 days, which indicates, that the biomass from that fraction was easily digestible.

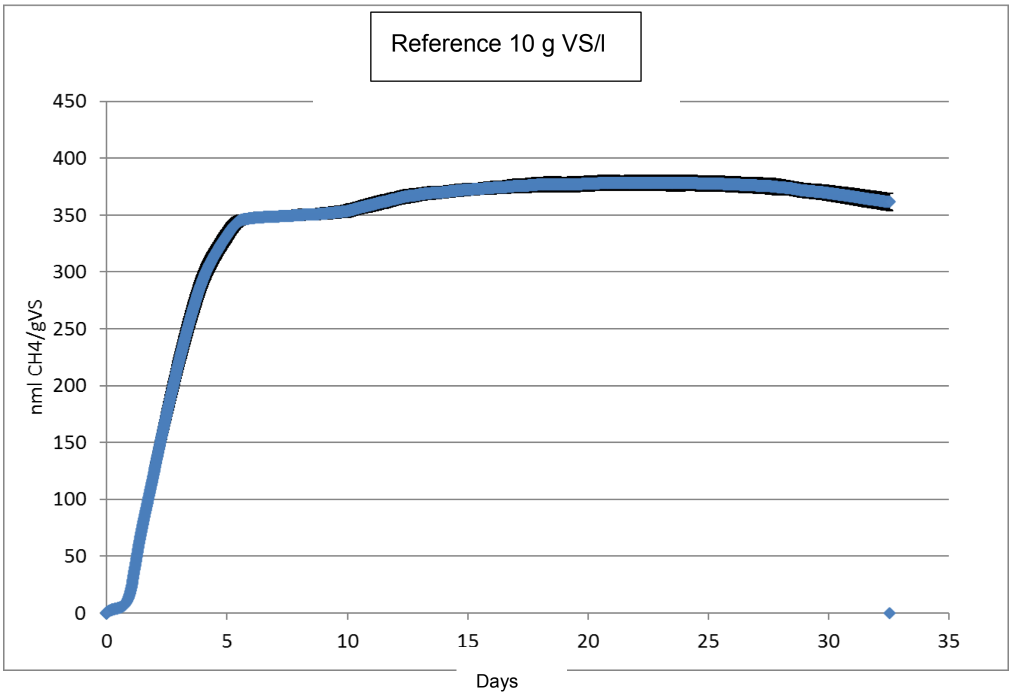


Figure 15. Cellulose reference - accumulated methane production (the black bars indicate the standard deviation). Thermophilic anaerobic digestion with an organic load of 10 gVS/l

The anaerobic digestion of the cellulose reference resulted in a methane production of 369 Nml/g VS with a standard deviation of 7 Nml/g VS, which was perfectly in the expected range of 375 Nml/g VS +/- 25 Nml/g VS. This indicates, that the inoculum used and the data logging system functioned properly.

6. Conclusion and perspectives

The DOGAS approach to wastewater treatment is possible and feasible. It has the potential of turning wastewater plants into energy and bio resource providers. Though more research and test on optimizing the operation has to be done, before this potential can be realized.

6.1 Process aspects

Concentration of wastewater with membranes has shown to be a feasible process. The main challenge is to keep retention time as low as possible to avoid biological activity, which should be overcome by a correct plant design. Short retention time in buffer tank before the filtration is also crucial to avoid the biological activity getting started. Ultrafiltration has shown to have a capacity performance very close to an open membrane with a porosity of 0,2 µm. The fouling aspect can probably be improved by better control of biological activity. With a tight UF membrane other cleaning procedure can be used as the pH range for membrane cleaning agents is 1 to 13. The energy consumption is very low and is only linked to air scouring.

6.2 Results

The two membranes have shown a good COD retention nevertheless retention was higher with the tight Ultrafiltration membrane. COD values in outlet are comparable to COD out of a biological treatment plant. N ammonium permeability is very high, which means that N ammonium is present in the treated water. Phosphorus will also pass through the membrane in form of orthophosphate and be kept in the treated water. When biological activity is well controlled there will be no COD biodegradation or nitrification. Organic xenobiotics as well as heavy metals had a good retention on both membranes.

6.3 Investment prospect for full scale plant

With conservative estimation of cost for a plant of 20.000 PE (3.600 m3/day) the figures for a full scale plant will be as follows:

Investment without biogas installation:
Pre-treatment equipment : Dkk 3.5 mill
Membrane and equipment: Dkk 14 mill.
Labour: Dkk 8 mill.
Total Dkk 25 mill.
Energy consumption: between 0.2 and 0.3 kWh/m^3
No N_2O nor CO_2 emission

A similar plant with traditional process will sum up to approx. Dkk 30 mill., and an energy consumption of minimum 1 kWh/m^3.

When biogas production from concentrated wastewater is added to the plant it will become a net producer of clean energy. N-ammonium and orthophosphate will be in the treated water making it a valuable resource in agricultural production.

6.4 Value creation - occupation, export, patent options

The economical value created by the DOGAS approach to wastewater treatment can be estimated on 2 parameters:

1) Improved economy in WWTPs
2) Increased sale of technologies

1) Samsø Spildevand's plant in Ballen, can be used as an example for estimating the value created by implementing the DOGAS system in a WWTP. It will realistically neutralize the cost for energy consumption and potentially generate revenue from sale of energy and irrigation water. Furthermore environmental taxes for discarding treated wastewater to recipient can be avoided.
For the Ballen plant the figures are:
Capacity: 10.000 person equivalents (PE)
Cost energy consumption: 200.000 dkk
Environmental taxes: 100.000 dkk
Total annually savings: **300.000 dkk**.
Irrigation water: 250.000 m3 + 10T N + 1,5T P, can fertigate approx. 250 ha
Estimated income: 5 dkk. per m3 water = **1.25 mio. dkk**. + potential income from sale of energy.

In places abroad where it would be relevant to install the DOGAS system, it would typically be in plants of 100.000 PE or more and in areas with water shortage and hence a much higher price on fertigation water. This will make the business case very favourable indeed.

2) The export potential for the DOGAS system is foreseen to be substantial. For Alfa Laval and the other technology providers behind this will result in a significant value creation both in terms of revenue and employment. Below the potential is estimated with Alfa Laval as example.
A sale of membranes to a 100,000 PE capacity per year will mean additional earnings of 30 mio. dkr. and employment of approx. 8 extra employees of which 75% might come in their plant in Nakskov, which is characterized by declining business activity and low employment. If a capacity of 1 mio. PE was sold, further 300 mio. dkk. in revenue is generated and estimated another 20 employees will be needed

In addition to this direct value creation there will be an indirect local in terms of new bio economical business opportunities.
Furthermore there will be an option of taking out a patent on the DMF process, when fully developed – in DK/EU.

6.5 Application of solution - at Samsø and around the world

As Samsø has limited fresh water resources and will not go for desalination of sea water, it makes sense to consider the reuse of water. At the same time it is considered to establish biogas plant for agricultural waste. By concentrating wastewater there is potential for use of concentrate for this local biogas production. This way a combined WWTP and biogas plant will form a key infrastructure for a circular bio economy at the island.

However the DOGAS solutions will be even more relevant to apply in places around the world suffering from much more severe water scarcity than Samsø, like Southern Europe, California, South Africa and other emerging markets. Being able to both cleanse water and deliver energy and water and nutrients, it will have a special potential on locations in developing countries lacking the basic infrastructure for a viable agricultural production.

During the course of the project this potential was communicated locally, nationally and internationally, and evoked interest from a vide variety of stakeholders. In the late phase of the pilot test run a delegation from the Turkish Ministry of Water and Environment visited the pilot site at Samsø to learn about the DOGAS approach.

Figure 16. Turkish delegation visiting the test site at Nordby, Samsø

6.6 Outline development project for full scale plant

As we are talking about a completely turnaround of wastewater treatment process it will be wise to install a semi industrial plant with biogas to get the complete picture about the process before building a full-scale plant. Pilot tests have to be followed by larger tests inclusive the biogas step for being able to make a complete mass balance of the complete process and a final economical evaluation. A semi full-scale plant specially designed for the purpose with very short retention time for controlling bacteria growth can provide this data and knowledge on how to operate the membranes optimally for realizing the potential of the DOGAS method.

It was therefore decided to establish a follow up project to complete the development of the DOGAS method for making it ready for full-scale operation – and at the same time developing the full integration with biogas production and agricultural production. In the closing phase of the project an application was sent to the Innovation foundation of Denmark (Innovationsfonden) for funds for this work.

The applied project is called *FERM – Fertigation Water from Direct Membrane Filtration of Wastewater* and figure 18 illustrates the 3 elements that are to be merged into a complete system concept for a circular bio economy infrastructure.

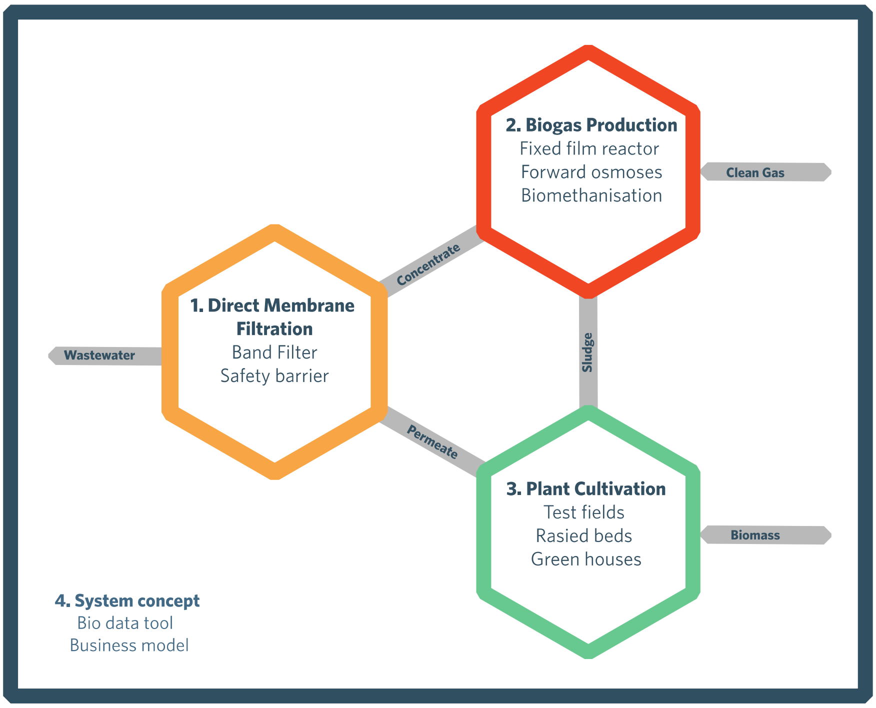


Figure 17: The elements of the FERM project.

Apart from the current partners in the DOGAS project it will have participation of a number of additional companies and research institutions, among Technical University of Denmark, who will lead the project. The FERM project has a total budget of 44 mill. Dkk.(28 mill. Dkk. funding) and is planned to last 3 1/2 year. It will provide the ideal framework for finishing the work creating this groundbreaking new approach to wastewater treatment, which was initiated in the DOGAS project.

Unfortunately the application was not granted due to its complexity with 3 technological elements in play. But the consortium was encouraged to send in a revised application with a sole focus on the final development of the DMF technology. This will be done by August 2018.

DOGAS - Direct concentration of wastewater for biogas production

Direct concentration of wastewater for anaerobic digestion in biogas reactors is possible and will turn traditional wastewater treatment upside down – when the method is fully developed.

If it by concentrating raw wastewater with use of membranes is possible to process the concentrated wastewater directly in a biogas reactor and thereby gain energy from the organic matter in the water, the proposed DOGAS method will open up new paths for wastewater treatment. After several test runs at a pilot plant at Samsø late summer/early autumn 2017, it is clear, that it is possible to make this direct concentration with membrane technologies.

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