# DHRTC Technology Conference 2021
# Book of abstracts

## Programme: Produced Water Management

**Contents**

## 3D-fluorescence spectroscopy associated with tensorial analysis for detection of crude oil in water

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Water produced during crude oil production undergoes cleaning processes to fit into a maximum limit of 30 mg $L^{-1}$ of oil in water, as required by OSPAR Agreement, before being discharged in the sea. Quantification by fluorescence spectroscopy of oil in produced water is substantially affected by inner filter effects and fluorescence resonance energy transfer, which leads to red-shift and quenching of fluorescence signal. Therefore, the conventional, single-wavelength sensors provide poor quantification since they are unable to consider variations in the signal that are not proportional to oil concentration. In this context, we aim to use 3D-fluorescence landscapes and handle inner filter effects and FRET by tensorial analysis to determine crude oil content in water, by a straightforward methodology exempt of sample treatment or additional steps prior to the fluorescence analysis. Oil-in-water (5-1000 mg $L^{-1}$) samples were prepared from an oil-in-isopropanol (1% m/v) stock solution and analyzed with a PerkinElmer FL8500 fluorescence spectrometer under different instrumental settings for the lower (5-100 mg $L^{-1}$) and upper (100-1000 mg $L^{-1}$) concentration ranges. Excitation-emission landscapes were measured within the following spectral ranges: excitation from 300 to 350 nm (10 nm intervals) and emission from 370 to 580 nm (0.1 nm resolution, which later was treated to keep only 1.3 nm-spaced points). The two datasets (one for each concentration range) consisted of 3-mode tensors with $S \times 163 \times 6$ dimensions ($S = 5$ and 9 for the lower and upper concentration ranges, respectively, 163 emission wavelengths and 6 excitation wavelengths), and no preprocessing was needed prior to data treatment since the spectral range measured does not comprise the areas where the Rayleigh scattering is expected, and the Raman scattering did not affect substantially the results. Each tensor was trilinearly decomposed into three loading matrices (one for each mode) with $S \times F$, $163 \times F$ and $6 \times F$ dimensions, in which F represents pure (linearly independent) components and was varied from one to five to decide the best rank for each tensor decomposition. The $S \times F$ matrix, containing relative concentrations, was correlated with the experimental concentrations by multiple linear regression. Five and two samples were used for calibration and test steps, respectively, for the lower concentration range, and nine calibration and five test samples were used for the upper concentration range. For both ranges, the best calibration models were obtained with $F = 2$, i.e., two pure components. Prediction errors of 2.1 and 44.8 mg $L^{-1}$ were achieved for the lower and upper ranges, respectively, which, together with other figures of merit considered, indicated that tensorial trilinear decomposition performed as a helpful tool to determine crude oil in water from 3D-fluorescence data.

## A chemical solution for produced water re-injection management

*Hamed Moosanejad, Hamid Nick*

Produced water reinjection (PWRI) is one of the methods of dealing with the produced water. Previous studies suggest that PWRI can decrease injectivity significantly due to formation of schmoo and filter cake. The loss of injectivity can be attributed to several effects, among them formation of resistant skins, clogging of the near well-bore area by ultrafine particles and schmoo-like scales likewise blocking the near well bore area. These studies suggested that acid treatments could restore the reservoir rock permeability. It is however not practical to consider frequent acid jobs for all the injectors. To circumvent this one could develop a method for continuous injection of ultra low concentration Acid/Salt. The additional chemical must be cost effective and environmentally friendly, and impose no damage to injectors. Here we illustrate the plan for finding the minimum concentration of additive required for preventing soft scale/filter cake formation/accumulation for PWRI schemes in chalk fields.

# Advanced control of produced water treatment processes using the online OiW measurement

*Stefan Jespersen (AAU), Zhenyu Yang (AAU) and John Bagterp Jørgensen (DTU)*

We strive to reduce the amount of oil discharged to the marine environment from offshore Oil & Gas water treatment facilities, without impacting the oil production. This will be accomplished through advanced control methods and the use of online Oil-in-Water monitors (OiW). We have already demonstrated that advanced controllers can stabilize the discharge concentration and the aim is to reduce the discharge concentration and the total discharge by using OiW monitor technology. Control-oritented grey-box models will be developed for the coupled three-phase separator and hydrocyclones, and sensor fusion techniques will be used to overcome the shortcomings of the current OiW technology.

The performance and potential pros and cons of different control methods and strategies will be tested both in simulation and on a lab-sized pilot-plant.

# A microfluidic approach towards studying near wellbore formation damage effects in carbonate reservoirs

## Evaluating the effect of produced water components on calcium carbonate with microfluidics

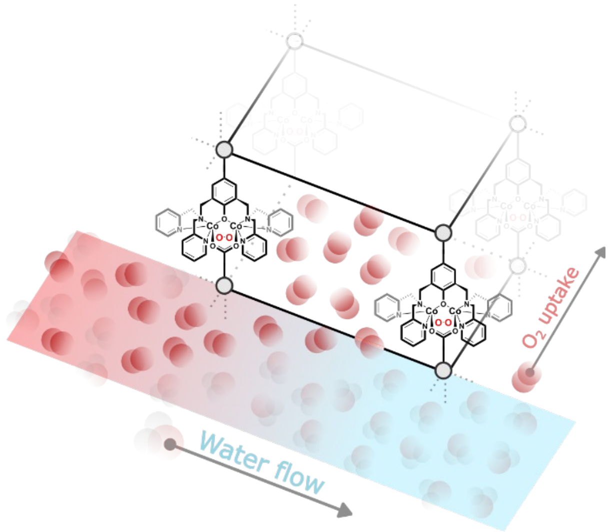
*Megan Heath, Marcin Dudek and Gisle Øye*

Reinjecting produced water into production wells is a cost effective, environmentally benign means to dispose of contaminated water produced by the oil industry. However, injectivity decline and formation damage due to deposits of various components in the produced water and seawater, such as solid particles, oil droplets or dissolved components, renders this process less effective. Therefore, film deposits and the transport of dispersed components in porous media need to be investigated to understand what happens at the pore-scale level. To study this, microfluidic methodologies will be developed in this project, specifically to study retention processes in calcium carbonate structures, mimicking reservoirs in the Danish part of the North Sea. This poster describes the proposed approach towards studying these phenomena.

## Biomimetic oxygen scavengers - From theory to field

*Ali A. Eftekhari and Jonas Sundberg*

Produced water (PW) is a by-product of oil extraction and contains a mixture of dispersed and dissolved oil. Reinjection of PW into the reservoir is an important tool to reduce the discharge of potentially harmful substances. For that, the PW is mixed with seawater containing high levels of dissolved oxygen which causes corrosion and bacterial growth. Currently, the removal of oxygen relies on the excessive use of chemical additives.




To mitigate this, our project aims to develop new types of metal-organic frameworks (MOFs) tailored for oxygen adsorption. MOFs are porous coordination polymers that form via the self-assembly of inorganic nodes and organic linkers. The surface area and chemical functionality can be tuned by careful choice of building blocks. This makes MOFs attractive materials for use as selective adsorbents. Specifically, the project aims to prepare MOFs based on linkers that integrate accessible secondary metal sites. Such sites can bind molecular oxygen via partial electron transfer similar to biological oxygen-transport proteins.

The project will combine experimental work with computational modelling. The materials will be implemented in a micro-scale reactor to obtain adsorption/desorption isotherms under simulated conditions. The experimental data will be used to develop computational models to support the design of pilot- and full-scale packed bed reactors. Ultimately, the goal is to transform theory into field operation.

## OAPID: BMED On-site Acid Production for In-line Descaling in the Whole PW Treatment-Injection Process

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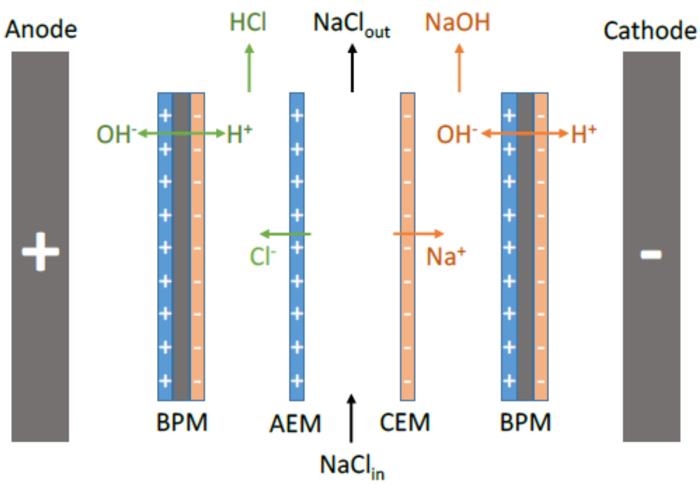
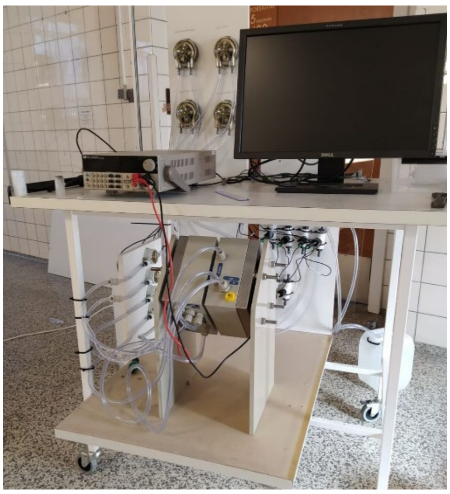
The oil industry is facing massive challenges in the coming years with achieving a green-transition to meet the zero-harmful-discharge from oil production. The re-injection of produced water (PW) for reducing the consumption of sea water and avoid harmful pollutants in the effluent is one way to go about it. Acid injection with the re-injected PW both hinders the growth of sulphate reducing bacteria in the oil reservoir and scaling inside the system and production pipelines. Since acid consumption can be a massive part of the expenses for producing oil, on-site production of acid can and will lower the expenses. One way to go about this is with the bipolar membrane electrodialysis (BMED) for simultaneously acid and base production, by separating salts and dissociate water into protons and hydroxyls.

As of now, the BMED setup has been shown to produce acid concentrations of 0.01M sulphuric acid with low feed concentration (0.3M sodium sulphate) and power consumption (45Wh), in only one hour of experiment. A system measuring all the important parameters (conductivity, flow, pH) with online controlling of the system pumps has already been established. Further studies on the BMED setup will investigate; (I) the optimization strategies for the current bipolar membranes for achieving faster and more acid production; (II) development of new bipolar membranes for improved water dissociation; (III) acid production on simulated sea water for "real world" simulation; (IV) testing the BMED acid production on an simulated descaling system, to demonstrate its efficiency and robustness for descaling; (V) Upscaling of lab-bench-setup for simulation of "real world" implementation.



# Coalescence of oil droplets in microchannels under brine flow

*Tian Wang, Simon Ivar Andersen, Alexander Shapiro*

We use microfluidics technique to study the coalescence of model oil droplets in microchannels. Different stages of the coalescence: convergence of the droplets and liquid film drainage, collision, interfacial film rupture, and merging are captured by high-speed imaging. The coalescence time is studied, defined as the merging time for the droplets after physical contact. Adding salts into the water phase may slow down the coalescence process. Cations $Na^+$, $Ca^{2+}$ and $Mg^{2+}$ do not significantly affect the coalescence time, while the presence of the anion $SO_4^{2-}$ produces much longer times than of $Cl^-$. The presence of a surface-active component, stearic acid, increases the coalescence time, in a clear correlation with the interfacial tension. At higher pH stearic acid is deprotonated into stearate; correspondingly, the interfacial tension decreases further and the electrostatic repulsion of the head groups of fatty acids increases. The interface between droplets and water is thereby stabilized and the coalescence time increases. A pH value higher than the pKa of stearic acid induces highly stable plastic-like oil-water interfaces. We discuss the implications of our results on the applications in the petroleum industry concerning e.g. enhanced oil recovery under smart waterflooding.

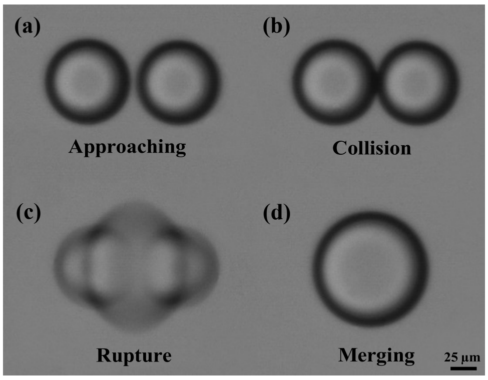



Figure 1. Four stages of the coalescence process: (a) approaching; (b) collision; (c) rupture of interfacial film and (d) merging.

# Fast evaluation of synergistic effects of combinations of production chemicals in produced water oil droplet stability

*Liridon Aliti, Alexander Shapiro, Simon I. Andersen*

Produced water is well-known to contain both dispersed and dissolved oil species which are hazardous to aqueous life. The efficiency of dispersed droplet removal is related to the droplet stability towards coalescence. Removal of dispersed droplets are normally easier when the droplets are bigger in size, hence it is desired to make them coalesce.

The performance of many oil field chemicals is related to the surface activity of these and the ease of dispersing these species into the oil-water system being produced. Optimization of the formulation of each product normally do not necessarily take into account the influence on the performance downstream the treatment processes. Therefore, the combination of chemicals e.g. a down-hole corrosion inhibitor and demulsifier (DE) may end up in the produced water stream and affect the performance of the droplet removal processes by either increasing or decreasing stability. In this work, we report a number of microfluidic studies of these effects and how blends of chemicals affect coalescence frequencies and droplet behavior.

This is associated with the performance of a produced water treatment system depending on oil-drop removal. The findings can be explained through the interfacial behavior and deviation from optimal oil-in-water instability conditions due to under- or overdosage of production chemicals. Another argument is the significant competition of chemicals to reach the droplet interface governed by differences in diffusion coefficients and the ability to adhere to the interface. The microfluidic approach has the advantage over bulk experiments (bottle tests) that mixing is fast and effects of diffusion is limited. Additionally, sample consumption is much lower which is advantageous when crude oil samples are in small quantity.

In the present work we present both model oil and crude oil systems with various chemicals. The results are explained using the Hydrophilic Lipophilic Deviation approach. The approach is a powerful tool for the optimization of the droplet stability. In one reported case, we observe that the combination of demulsifier and corrosion inhibitor leads to potential slugging which in the case of hydrocyclones or other separation devices could lead to a very poor performance, hence no droplet removal.

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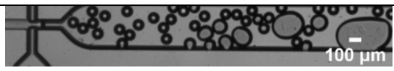
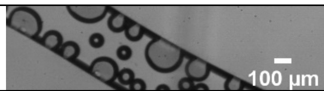
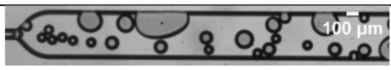
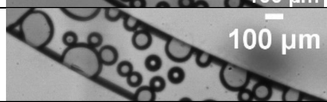
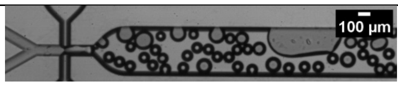
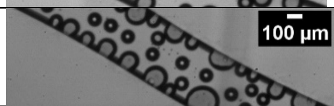
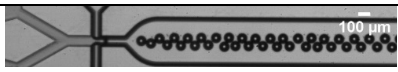
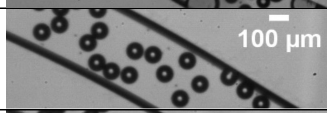
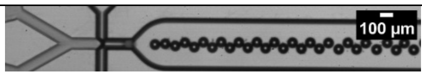
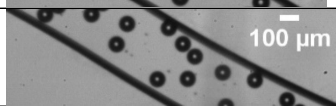
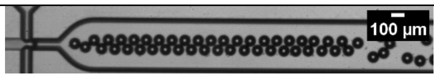
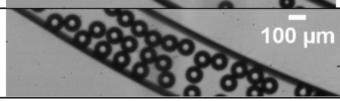
| 0 ppm |  |  |
| 2 ppm |  |  |
| 4 ppm |  |  |
| 10 ppm |  |  |
| 15 ppm |  |  |
| 20 ppm |  |  |

# Feasibility study on produced water oxidation as a pretreatment at offshore platform

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Produced water (PW) generated worldwide are steadily increasing and has lately become a significant issue of environmental concern. PW has a complex composition and suitable treatment at offshore platforms would be needed to achieve zero harmful discharge into the sea. In PW treatment, no single technology can meet regulation values, and combined physico-chemical with biological treatment of PW might be necessary. The feasibility of electrochemical oxidation (EO), heat-activated persulfate (SPS) and ozonation ($O_3$) was investigated as PW pretreatment step. The main goal was to remove organic species and improve biodegradability, and/or reduce toxicity to bacteria for the subsequent biological process. The experiments were performed with real PW sample from offshore platform.  A common treatment level of 5% and 10% equivalent removal of chemical oxygen demand (COD) were established in order to compare the methods performance. EO showed to be an efficient method for COD reduction in terms of partial conversion. The process was independent of the tested anode materials, and mainly controlled by current density. SPS reaction was time-consuming and largely dependent on temperature, as well as initial oxidant concentration. Both methods reduced COD and biochemical oxygen demand (BOD), but high oxidant doses might compromise any improvement in PW biodegradability due to by-products generation. Comparing the three methods, ozonation showed the most promising results applying ozone doses ranging from 3.5 to 151 mg $O_3$/L. The best results were achieved with 7.8 mg $O_3$/L corresponding to the energy consumption of 0.12 kWh/m$^3$. Benzene in PW was reduced up to 71% alongside with a significant toxicity reduction (>70%), and PW biodegradability (BOD/COD ratio) improvement (from 0.41 to 0.46). The results obtained indicate that ozonation might be an appropriate technology for PW pretreatment at offshore platform, but further research is required testing PW samples from different fields with different characteristics, e.g. lower initial BOD/COD ratio.

# Intelligent Testing Strategy of Produced Water Discharges fromOffshore Oil Extraction

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Produced water (PW) is an important waste stream of offshore oil extraction with regards to both volume and complex composition. This has raised concern regarding its ecotoxicological impacts on the receiving environment. For this reason, a risk-based approach (RBA) has previously been developed for PW management. This poster will present an overview of the key components of the RBA: 1. The exposure assessment consisting of either distance dependent dilution factors or grid-based exposure models, and 2. the hazard assessment consisting of toxicity analysis based on either the whole effluent toxicity (WET) or a combination of the individual compounds of PW. The methods and findings of RBA application on PW were evaluated based on a literature review of  studies analysing exposure and toxicity through other approaches. It was found that the predictions of PW dilution by complex models of the exposure assessment in RBA provide conservative estimates. However, the toxicity data of the hazard assessment has several shortcomings. These are related to the variable and only partly characterized composition of the effluent, the limited data on the toxicity of added production chemicals, as well as the behaviour of PW toxicity throughout the duration of testing. Further development of the RBA should therefore focus on increasing the quality of toxicity data as well as focused testing of drivers of whole sample toxicity.

# Model Predictive Control for Slug Flow Suppression and Water Treatment in Daily Operations of Oil Field Facilities

*John Bagterp Jørgensen, Steen Hørsholt, Zhanhao Zhang, Tobias K. S. Ritschel, Zhenyu Yang, Stefan Jespersen*

We develop a model predictive control (MPC) based advanced process control (APC) system for offshore oil and gas production processes that can significantly reduce oil concentration in discharged water while maintaining high oil and gas production. Using engineering domain knowledge, we model the flow line, the three-phase separator and the de-oiling hydrocyclones to simulate and control oil-in-water concentration in the produced water, while maximizing the oil and gas production. Figure 1 shows an illustration of the production system, from well to zero harmful discharge of produced water. We implement MPC technology for simultaneously stabilizing and optimizing the oil and gas production processes to maximize the oil production without violating legislative requirements of the discharged water. Novel sensors are combined with the APC system for monitoring oil-in-water concentration and providing data for modeling. The APC system will be tested using industry standard digital twins as well as in a pilot plant at Aalborg University in Esbjerg.

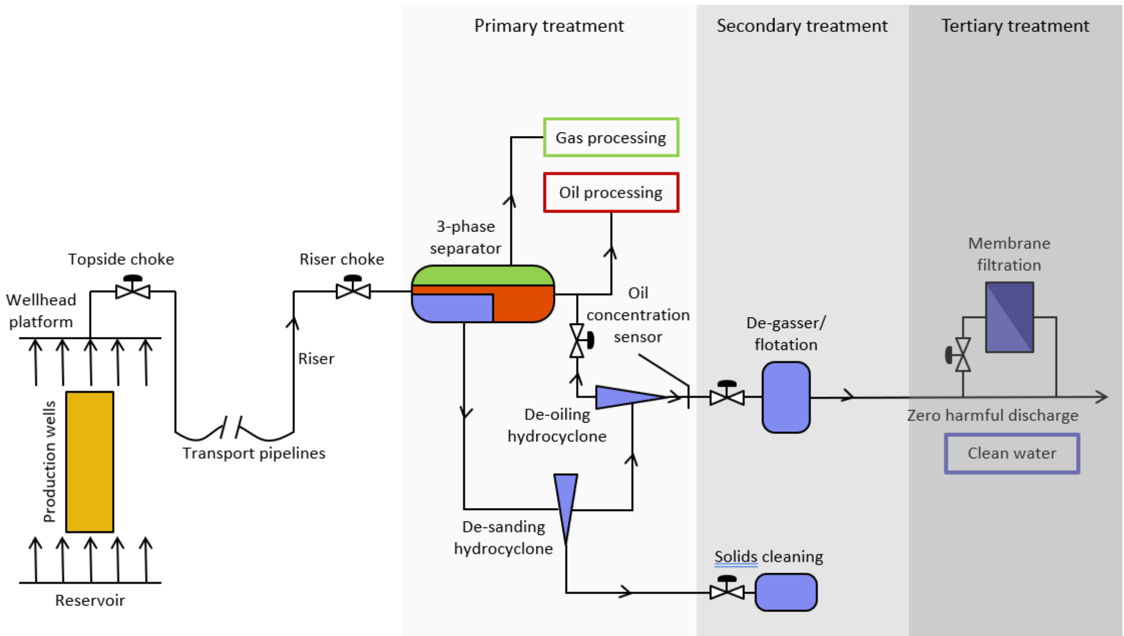


**Figure 1**: Units operations of offshore oil and gas production. The processes consists of a flow line and riser (where slug flow phenomenon can occur and cause large disturbances to the following separation processes), a 3-phase separator (oil, gas, water), de-oiling hydro-cyclones, de-gasser, and membrane filtration.

# Moving Bed Biofilm Reactor for produced water treatment on the seafloor

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Produced water (PW) is the largest waste stream generated in oil and gas industries. It has a complex composition, containing various toxic compounds that should be removed before discharge into the sea. A general biological treatment of PW would be a substantial improvement compared to current technologies, in terms of stability, extents and rates. The main goal of this research work was to provide a proof-of-concept of Moving Bed Biofilm Reactor (MBBR) for PW treatment that due to space constraints at offshore platforms would be placed on the seafloor (Figure 1). PW was treated by MBBR established with AnoxKaldnes™ K5 carriers with attached biofilm adapted to high salinity. The effect of MBBR performance at two different temperatures (10°C and 40°C), as well as the general design and operational parameters for MBBR were assessed in the present work. The experiments were carried out with real PW samples from offshore platform. The MBBR reduced large fraction of total organic carbon, and removed problematic chemicals such as BTEX and $H_2S$ scavengers, thus decreasing PW toxicity. Bio-kinetics studies showed that using the high temperature (40°C) of PW compared to 10 °C in the sea, promoted a faster removal (approx. 30% difference) of soluble chemical oxygen demand (sCOD) reaching up to 58% in 1 hour and 83% within 3 hours. After 8 hours treatment, there were no differences in sCOD removal between cold and hot reactors. Changes in feed composition such as salinity and HRT did not affect reactors performance. These results underline that MBBR is a robust technology able to cope with changes in both PW characteristics and operational parameters.

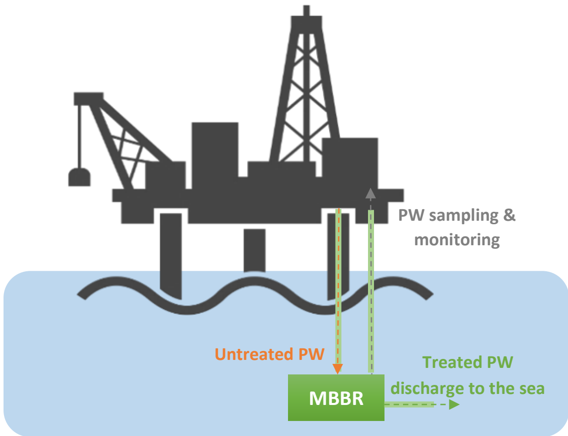


*Figure 1 - Project overview.*

# Potentially harmful hydrocarbon constituents in produced water polar and non-polar species

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Produced water (PW) is the water co-produce with oil and gas from oil wells and it represents the largest volume waste stream in oil and gas production operations on most offshore platforms. In 2017 there was an estimated 24 million tons of produced water produced from the Danish continental shelf alone [1]. Over the lifetime of an oil reservoir, the proportion of produced water increases, for the off-shore oil production in the Danish North Sea, several of the production wells have reached the water-to-oil ratio (water cut) of 80% in 2015 [2]. The currently most common methods for PW treatment are physical, e.g. hydrocyclones and gas flotation tanks which separate components based on centrifugal forces and gravity; these methods yield waste waters with acceptable levels of dispersed hydrocarbons for discharge to sea according to the current legal limit of 30 mg/L [3]. This limit does not address the polar species dissolved in the water which are potentially the more environmentally concerning constituents in produced water. It also does not include the production chemicals which are added during the production and treatment processes and their potential degradation products. There is a need to address the disposal of produced water to the marine environment from production operations, from a standpoint of environmental risk, because this emission is continuous during production and discharge volumes are increasing in most mature offshore fields.

The central aim of this study is a determination of Naphthenic Acids (NAs) in Produced Water (PW). Oil-derived mixtures of carboxylic acids are considered emerging contaminants with the potential to disrupt development of aquatic species [4]. NAs determination through FT-IR technique is a fast and cheap method and can facilitate the quantification of these class of compounds. Naphthenic acids are extract from PW using LLE and DLLME and measured in a FT-IR transmission cell. This study will provide detailed knowledge of the composition of dissolved organic pollutants in produced water for the evaluation of the environmental impact factor (EIF).

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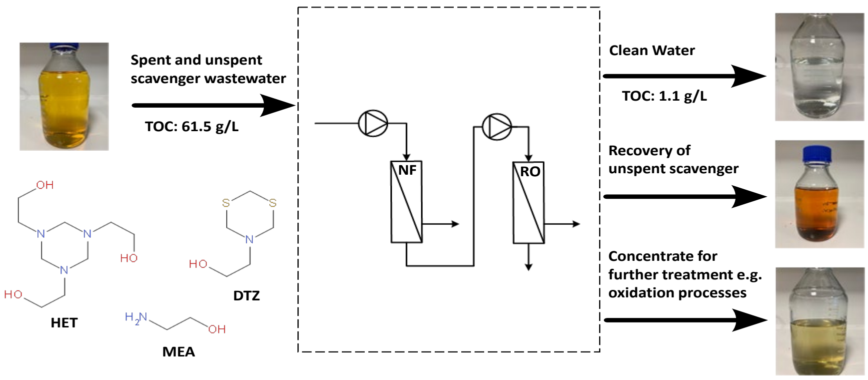
# Proof-of-concept of membrane-based recovery of unspent H2S scavenging chemicals

*Nikolaos Montesantos, Marco Maschietti and Jens Muff*

Section of Chemical Science and Engineering, Department of Chemistry and Bioscience, Aalborg University

After removing $H_2S$ from natural gas in offshore oil and gas installations, a triazine-based spent scavenger solution remains as a wastewater stream, which in some cases is discharged untreated into the sea. Three nanofiltration (NF) membranes (NF270, NF99HF, and DL), as well as one reverse osmosis (RO) membrane (XLE), were used for total organic carbon (TOC) reduction from spent and unspent scavenger (SUS) wastewater. The NF270 membrane reduced the TOC of SUS wastewater (61.5 g/L) by 65% while having a superior permeate flux compared to the other studied membranes and thus was subjected to elaborated study. In particular, this membrane showed a separation between the unreacted/unspent scavenger triazine (1,3,5-tris(2-hydroxy-ethyl)hexahydro-*s*-triazine, HET) and the main spent scavenger reaction product (5-(2-hydroxyethyl)hexahydro-1,3,5-dithiazine, DTZ) as it removed HET by 71% versus zero removal of DTZ. DTZ is a polar and hydrophobic molecule and consequently passes readily through the membrane despite an expected removal value of 50% based on a simple size exclusion pore flow model. The NF270 permeate was then used as a feed for RO filtration by the XLE membrane to further reduce the TOC of effluent stream by 98% (1.1 g/L). Lastly, both membranes did not demonstrate remarkable fouling when they were tested in 24 h preliminary lab-scale fouling experiments with a constant-concentration feed. The results of our work has recently been reported in a peer-reviewed paper [1], which is the first published study on SUS wastewater treatment using membrane technology. Our work so far proves the applicability of membrane technology for total organic carbon (TOC) reduction and simultaneously provides a promising route for further investigations on the recovery of unreacted scavenger from spent scavenger solutions.

The poster focuses on the results obtained in the first year of the project "Zero Discharge of H2S Scavenging Chemicals" which is part of the Produced Water Management programme.

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# TOWARDS ZERO DISCHARGE OF SPENT $H_2S$ SCAVENGERS: PROOF OF CONCEPT OF HYDROTHERMAL OXIDATION

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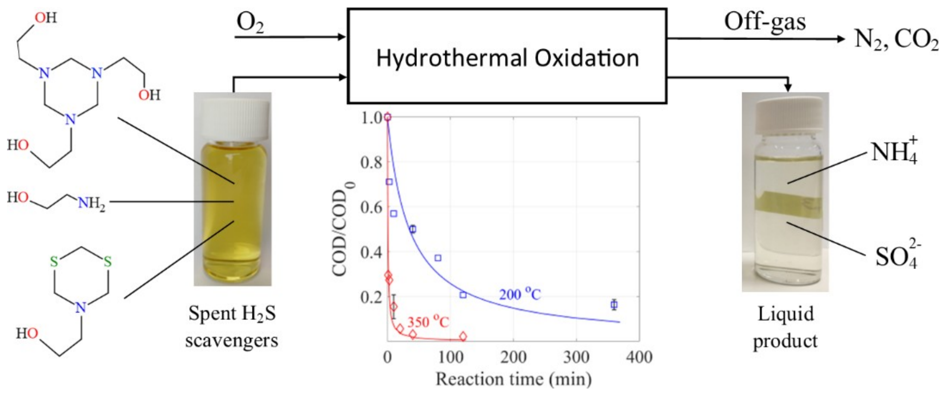
Triazine-based $H_2S$ scavengers are typically injected in large excess in offshore oil and gas installations in order to reduce $H_2S$ to acceptable levels. This practice results to unreacted triazine ending up together with the scavenging reaction products in a wastewater stream that is high in organic pollutants. Currently this wastewater is directly discharged in the sea, thus largely contributing to the environmental impact factor ofwater management offshore. This work provides a proof of concept for hydrothermal oxidation treatment on a typical spent stream of $H_2S$ scavenger. Unreacted triazine and the reaction products monoethanolamine and dithiazine were the main constituents of the wastewater. Oxidation experiments were performed in an injection batch reactor at 200 °C (low temperature, LT) and 350 °C (high temperature, HT) using an excess of oxygen as the oxidant. Reaction times of 3 to 360 min and 1 to 120 minwere investigated for LT and HT respectively. The three main compounds of the feed were fast converted and did not appear in the oxidation products. The COD of the spent scavengers was reduced significantly via the oxidation process, reaching 84% and 98% reduction at LT and HT, respectively. Intermediate reaction products included carboxylic acids, pyridines and pyrazines, while a clear trend of complete mineralization for organic nitrogen and sulfur to ammonium and sulphate was observed. Overall, the results indicate the feasibility of hydrothermal oxidation on $H_2S$ scavenger wastewater streams to reduce their environmental impact by converting toxic organic components to benign inorganic salts $CO_2$ and water. The results of our work have recently been reported in a peer-reviewed paper [1].

The poster focuses on the results obtained in the first year of the project "Zero Discharge of $H_2S$ Scavenging Chemicals" which is part of the Produced Water Management programme.




[1] N. Montesantos, M.N. Fini, J. Muff, M. Maschietti, Proof of concept of hydrothermal oxidation fortreatment of triazine-based spent and unspent $H_2S$ scavengers from offshore oil and gas production, Chemical Engineering Journal, 427, 131020 (2022). https://doi.org/10.1016/j.cej.2021.131020.

# VISUALISATION OF SIMULATED PRODUCED WATER RE-INJECTION BY MICROFLUIDICS

## Microfluidic study of droplet retention in porous media

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Uncontrolled permeability decline due to particles and droplets is one important limitation for implementing produced water re-injection (PWRI). Typically, core or packed bed flooding is used to investigate pore clogging phenomena, and the interpretation of retention mechanisms is limited to pressure readings and comparison of size distributions and concentrations of feed and effluent. Microfluidics is a method that involves control of fluids in confined microchannels, and it has been widely used in multiphase flow studies to visualize oil displacement. However, to date, microfluidics has been an unexplored field for PWRI. The poster demonstrates a microfluidic method for studying droplet capture in porous media. The method allows quantitative and qualitative analysis of spatial distribution of retained droplets over pore volumes injected.