Resource recovery from concentrates arising from industrial water reuse

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HighCon

Background

Every day, substantial amounts of valuable resources are lost through water reuse as part of industrial effluent treatment because they are disposed off with the Reverse Osmosis concentrate streams Both, from an economical and an ecological point of view, the loss of these resources should be avoided as well as their exposure into the environment Thus HighCon aims at the recovery of these resources If it takes place at all, in conventional treatment of concentrates, multi stage Reverse Osmosis is usually used in combination with an evaporation process in order to reduce the volume drastically The result is a solid mixture that contains almost all components of the industrial waste water and normally it has to be disposed for a fee.

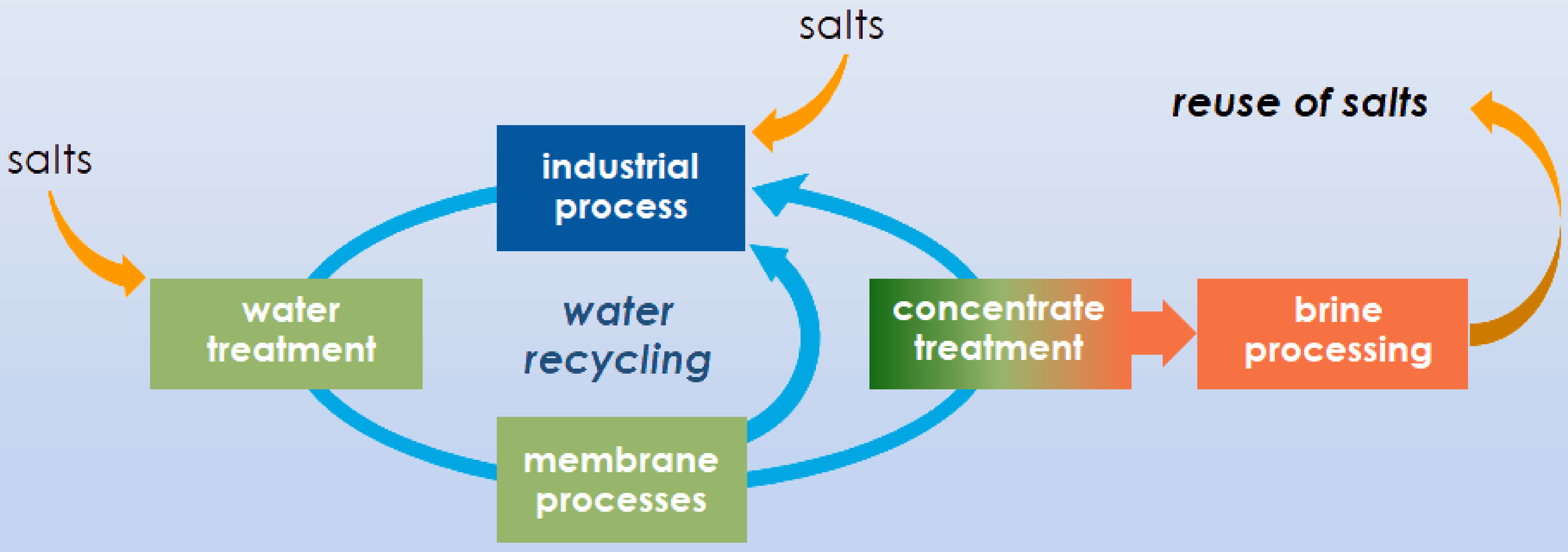

Fig. 1: HighCon concept for water recycling and concentrate treatment with salt recovery

Project Objectives

- Development of innovative, multi stage and selective processes for water reuse and concentrate treatment with the aim of generating high purity residuals, e.g. salts, which are suitable for reuse
- Reduction of energy consumption e.g. by further development and optimization of separation and evaporation processes
- Holistic optimization of water reuse supported by a simulation tool, based on investigation of different concentrate treatment processes at the HighCon demonstration sites

Project Concept

HighCon started with a review of existing technologies and the development of processes for concentrate treatment. Extensive laboratory tests with real wastewater build the basis for the planning of pilot plants. The resource recovery is made possible by innovative technology combinations and the further development and adaptation of innovative technologies such as Electrodialysis Metathesis, Membrane Distillation, and selective Low Temperature Distillation Crystallization, based on the requirements of selected fields of industries. Subsequently, the demonstration with reverse osmosis concentrates was carried out at DEK Berlin and Fraunhofer ISE (Freiburg) with L'Oréal concentrate from Karlsruhe.
The market analysis in combination with a technology roadmap ensures an optimal economic implementation. A simulation tool illustrates the complex interrelationships from raw water flows to the concentrate utilization, thus optimizing water recycling in a holistic way for the first time. Sustainability is included in the process evaluation as far as possible with currently available tools.

HighCon Developments

- Process for concentrate treatment (Fig. 2) with the aim of separating groups of substances (organic and salts)
- New plate module for Membrane Distillation enables use as a "one-time pass" process (SolarSpring)
- Electrodialysis Metathesis for the production of two concentrates with different ion composition and one diluate (DEUKUM)
- Low maintenance "self-cleaning" cooling tower for the evaporation and crystallization plant (Terrawater)
- Method for in-situ visualization, quantification and evaluation of inorganic coating formation using optical coherence tomography (DVGW-EBI)

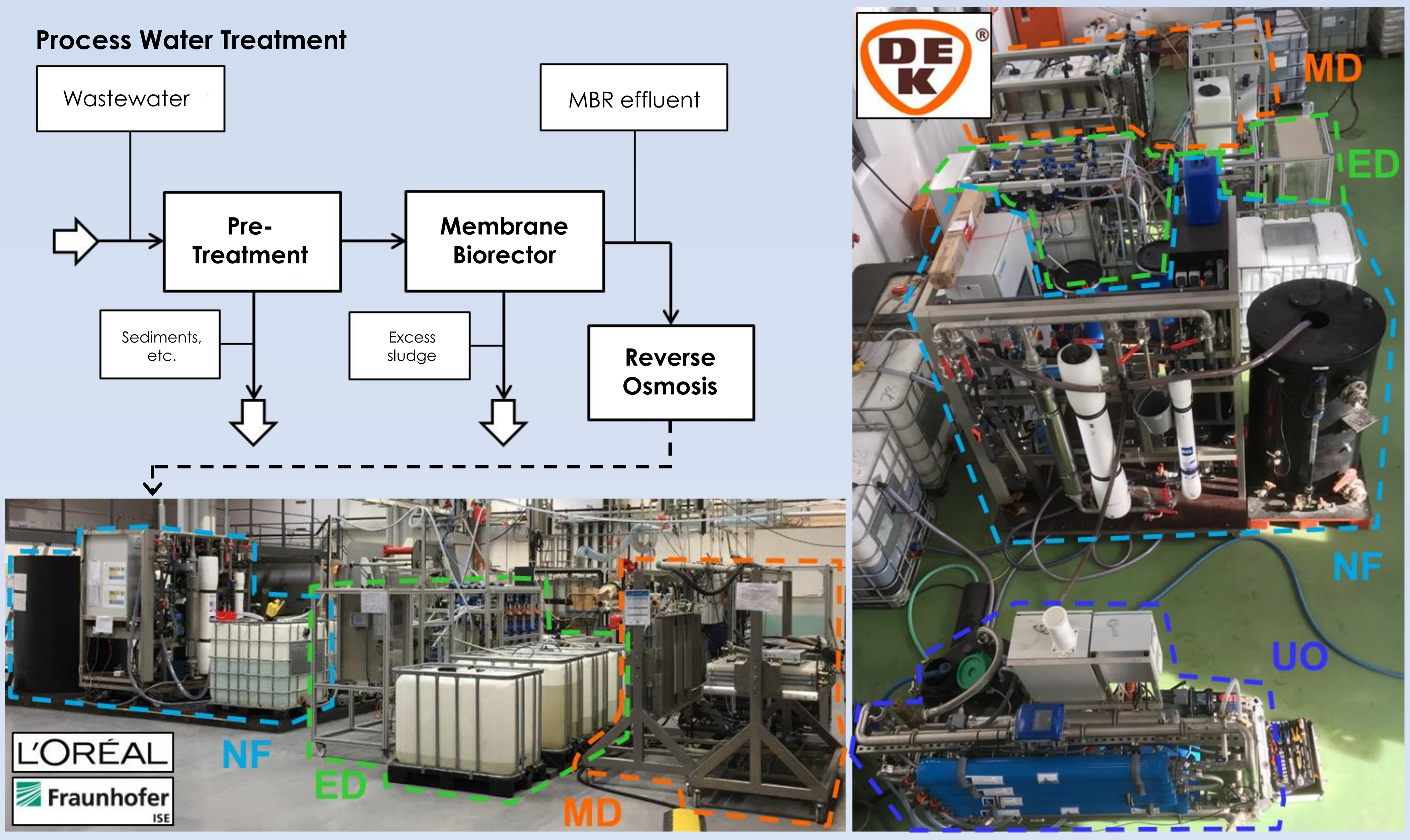

Fig. 2: HighCon demonstration plant in Freiburg at Fraunhofer ISE (left) and DEK Berlin (right); Nanofiltration (NF), Electrodialysis (ED), Membrane Distillation (MD)

Projektdaten

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Project Term: September 2016 – December 2019

Homepage: http://www.highcon.de

Research Partners	Industry Partners	Demonstration Sites	Funding Agencies
Technische Universität Berlin; DECHEMA Gesellschaft für Chemische Technik und Biotechnologie e.V.; DVGW; Fraunhofer ISE	terra water; SolarSpring membrane solutions; DEUKUM; WEHRLE	MEWA Textil-Management; CLARIANT; L'ORÉAL; DEK Berlin	Gefördert vom Bundesministerium für Bildung und Forschung; Betreut vom PTKA Projektträger Karlsruhe, Karlsruher Institut für Technologie

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Resource recovery from concentrates

Recycling options and cost calculation

Recycling Options

Within the HighCon project, different ways for recyclable substances and residues from concentrates are examined. Three aspects of concentrate treatment have to be considered:

- Selective recovery of a valuable substance with high purity (salts)
- Water recovery for reuse
- Savings in disposal costs by reducing the amount of residues and adjusting the composition

While a monetarization of recovered salts is currently not very lucrative, the HighCon process for concentrate treatment with regard to the closing of recoverable substance cycles up to Zero Liquid Discharge (ZLD) becomes interesting when considering the listed aspects. With an expectable scarcity and increasing competition for resources as well as further developments of separation technologies for residuals, the recovery of valuable substances will become increasingly important in the future.

Tab. 1: Overview of average costs for different disposal options; costs are always dependent on the concentrate composition and the site; transport costs are not included

Disposal options for concentrates	Average costs
Discharge into the sewage system	Usually the most cost-effective variant
Deposit as utilization in mine backfilling	~ 150 €/t
Deposit in storage tanks (liquid)	up to 420 €/t at 2% chloride content +22€/t each additional 2% chloride
Deposit as hazardous waste	up to 600 €/t
Combustion	~ 175 €/t

Recource Recovery & Cost Comparison

In HighCon, resource recovery is enabled by innovative technology combinations. Organic components and polyvalent ions can be separated from the RO concentrate by nanofiltration (NF). Monovalent ions such as Cl^- and Na^+ are contained in the NF permeate and further concentrated by electrodialysis (ED) and membrane distillation (MD). Using an evaporation process (Multi Effect Humidification - MEH) and crystallization, salts can be recovered selectively and with very low organic impurities.

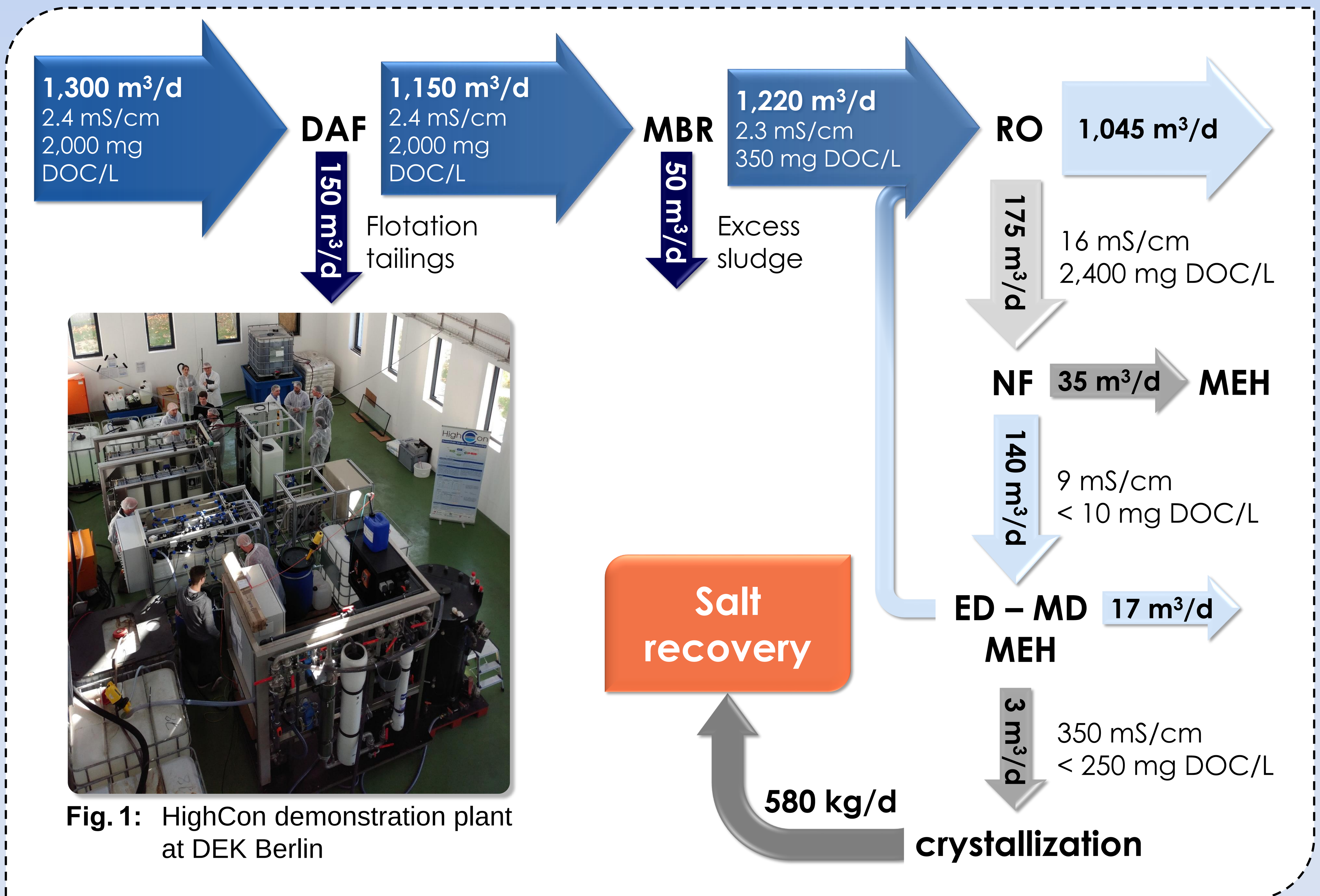

Fig. 1: HighCon demonstration plant at DEK Berlin

Fig. 2: HighCon process for concentrate treatment - Balance example for food industry

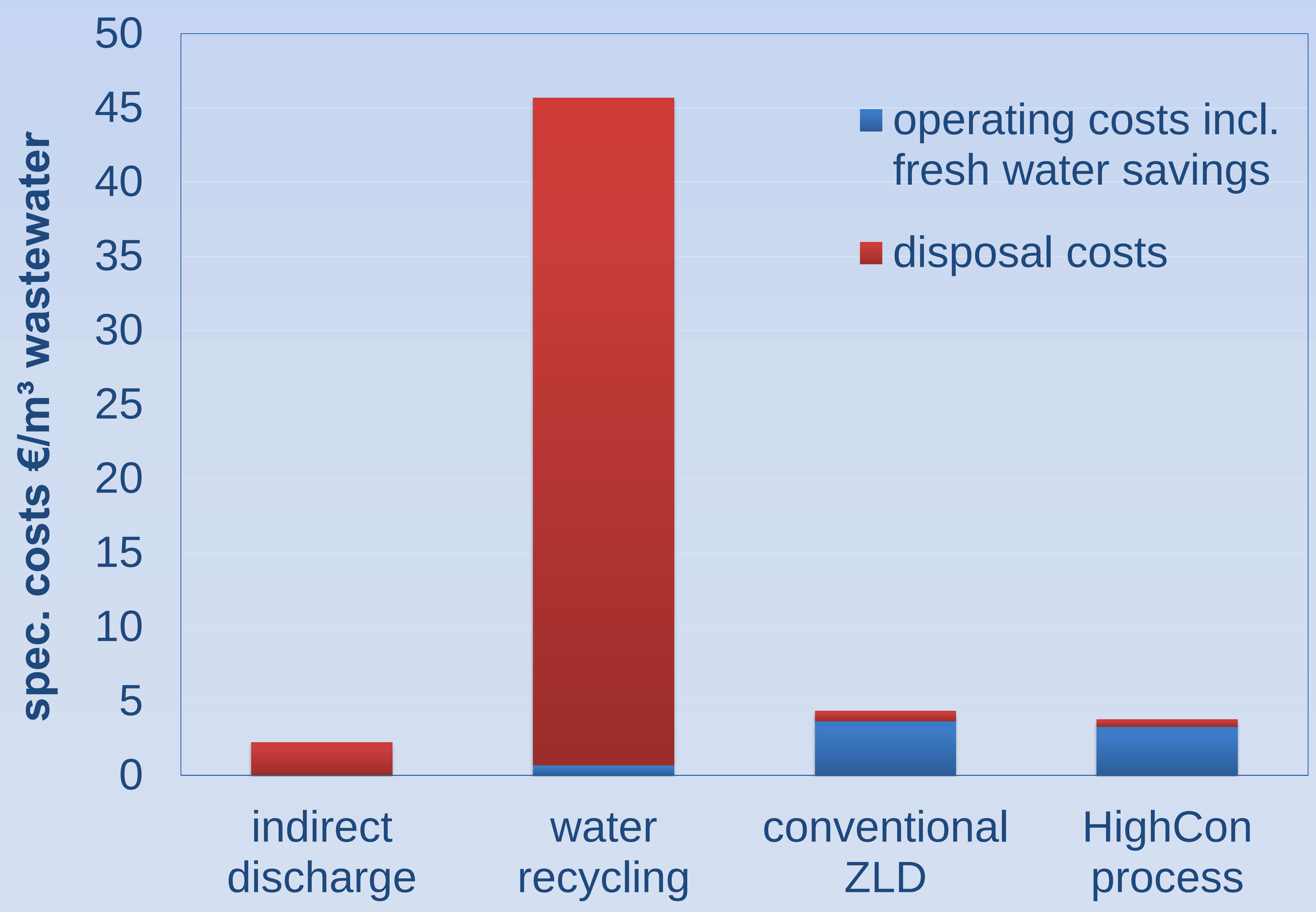

Fig. 3: Cost calculation for an example site

The indirect discharge of the MBR effluent (see Fig. 2) and water recycling without concentrate treatment are compared with conventional ZLD and the HighCon process. In this case, water recycling is only economically reasonable in combination with a concentrate treatment.

Prospects for concentrate treatment

- In the long term, the effects of nonbiodegradable organics on concentrate treatment must be considered – especially with regard to the recovery of salts.
- Regulatory requirements for industrial discharges are becoming stricter, so concentrate treatment is becoming increasingly attractive, not only against the background of resource recovery.
- Production and recycling processes must be considered in a holistic way in order to replace so-called "end-of-pipe" solutions including avoidance strategies.

Research Partners	Industry Partners	Demonstration Sites	Funding Agencies
Technische Universität Berlin, DECHEMA Gesellschaft für Chemische Technik und Biotechnologie e.V., DVGW, Fraunhofer ISE	terra water, SolarSpring membrane solutions, DEUKUM, WEHRLE	MEWA Textil-Management, CLARIANT, L'ORÉAL, DEK Berlin	Gefördert vom Bundesministerium für Bildung und Forschung; Betreut vom PTKA Projektträger Karlsruhe, Karlsruher Institut für Technologie

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Karlsruher Institut für Technologie

Super-concentration of wastewater in circulation by membrane distillation

HighCon

Experimental studies for technology adaptation

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*** DVGW Research Center at the Engler-Bunte-Institut, Karlsruhe Institute of Technology (KIT), Karlsruhe; **SolarSpring GmbH Membrane Solutions, Freiburg in Breisgau; *** Fraunhofer Institute for Solar Energy Systems ISE, Freiburg in Breisgau**

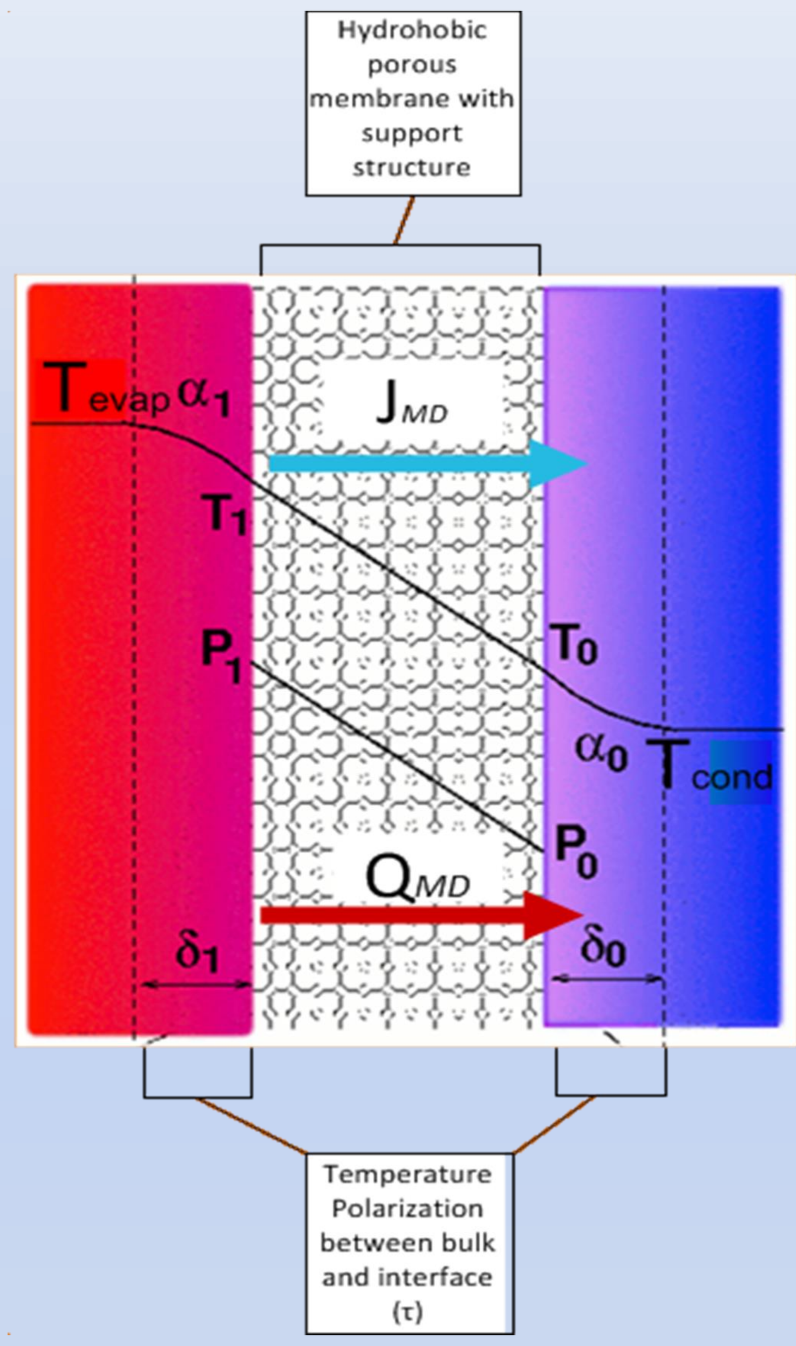
Fundamentals of membrane distillation

Mass and heat transfer in membrane distillation (MD) is defined as:

$$J_{MD} = C\,\frac{dp}{dT}(T_1 - T_0)$$

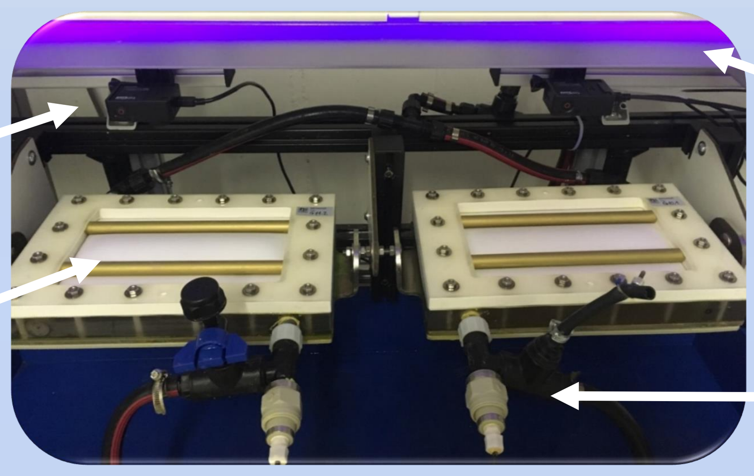
$$Q_M = \left(C\,\frac{dp}{dT}\,\Delta H_v + \frac{\lambda_M}{\delta_M}\right)(T_1 - T_0)$$

The mass transport coefficient C represents a superposition of molecular and Knudsen diffusion and viscous flow; dp is the change in vapor pressure over temperature dT and T_1 and T_0 are the temperatures at the membrane interface.
ΔH_v is the latent heat, λ_M is the thermal conductivity of the material and δ_M is the thickness of the membrane.



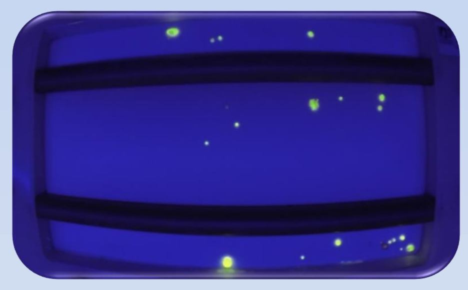
Investigation of the wetting behavior of MD-membranes

With the newly developed test setup it is possible to observe and optically analyze the polymer membrane during the distillation process. Since the feed water is enriched with the colorant uranine, which fluoresces under ultraviolet light, any liquid passages and the distillate can be distinguished from each other.



By varying the combination of operating parameters and the use of polymer membranes from different manufacturers, the influence on pore wetting is investigated and suitable materials are selected.

Test result without pore wetting



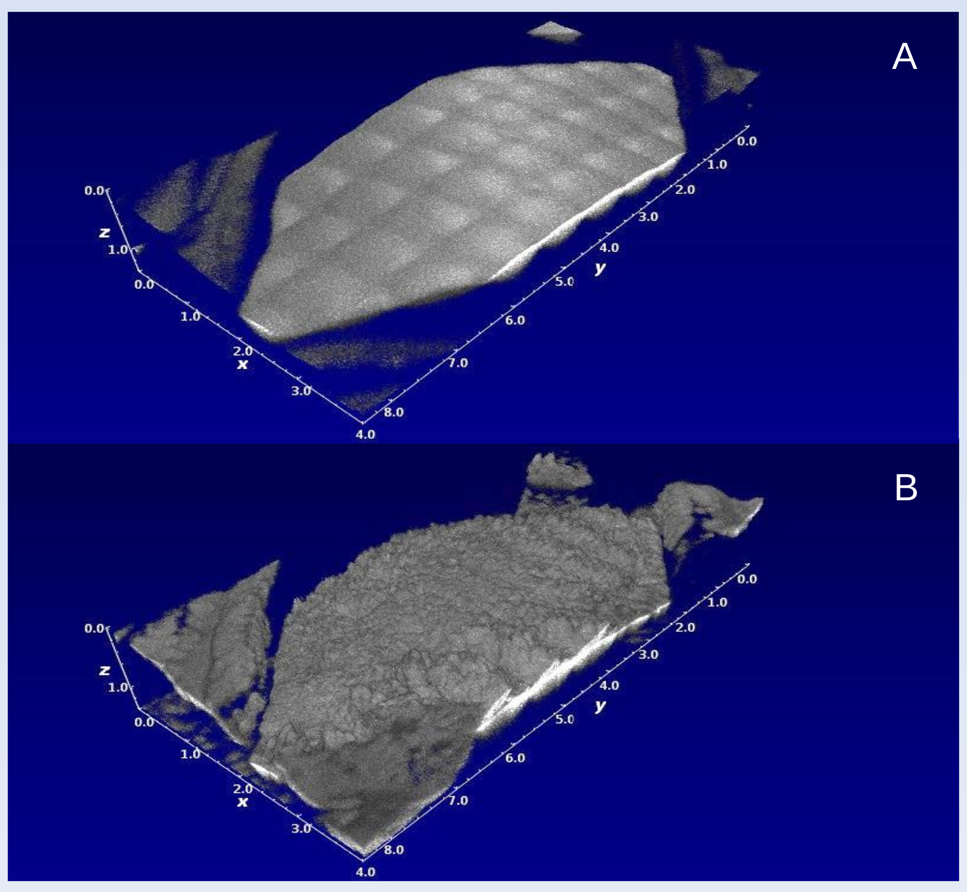
Test result with pore wetting

For a successful adaptation of the MD technology to the requirements of highly concentrated saline wastewater different investigations are necessary. Within the HighCon project, experiments with the following **goals** are pursued with three project partners:

1. Investigation of the wetting behavior of MD membranes (SolarSpring GmbH)
2. Investigations on scaling and fouling in the MD with real feed water (DVGW-EBI)
3. Thermodynamic investigations with saline waste water (Fraunhofer ISE)

Online in-situ quantification of scaling using OCT

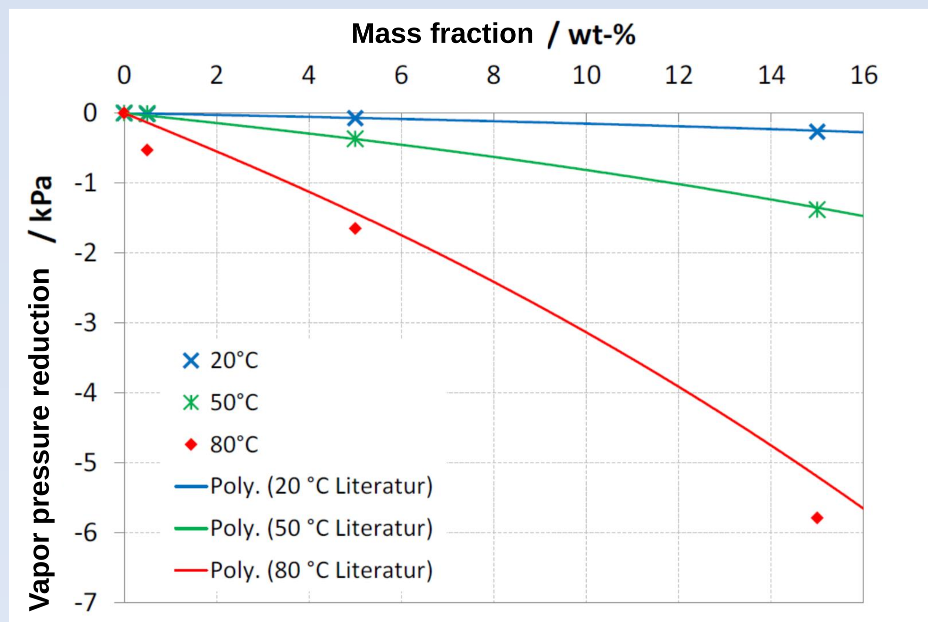
Fouling increases the temperature polarization in MD and thus reduces the driving force. In addition, the available membrane area is reduced. Optical coherence tomography (OCT) enables the formed top layer to be visualized in-situ via 3D images and quantified by means of adapted image processing.
For this purpose, a Direct Contact Membrane Distillation (DCMD) test cell was equipped with a frame, which allows a precise placement of the OCT scan head at different positions. The image of the fouling layer was taken in a 4-hour interval and shows promising results.



3D-OCT image of the clean (A) and the fouled membrane (B)

Determination of the characteristics saturation limit and vapor pressure of unknown salt solutions

High salt concentrations in the feed reduce the vapor pressure above the solution, so that due to this thermodynamic effect a significant reduction of the driving force can be observed. A VPXpert-L from Grabner Instruments was used to determine the vapor pressure of different salt solutions.



Vapor pressure reduction plotted above mass fraction NaCl

With this instrument the reduction of vapor pressure could be measured in dependence of the salt content. In a further experimental setup, the saturation limit of unknown salt solutions is determined on the basis of conductivity.

DVGW EBI
SolarSpring membrane solutions
Fraunhofer ISE

Translation of the poster from German into English by TU Berlin, Chair of Environmental Process Engineering