

WP4

Promoting circular economy in the chemical sector: an innovative approach to recover resources from wastewater generated in the silica industry



eurecat



GrupoTYP SA

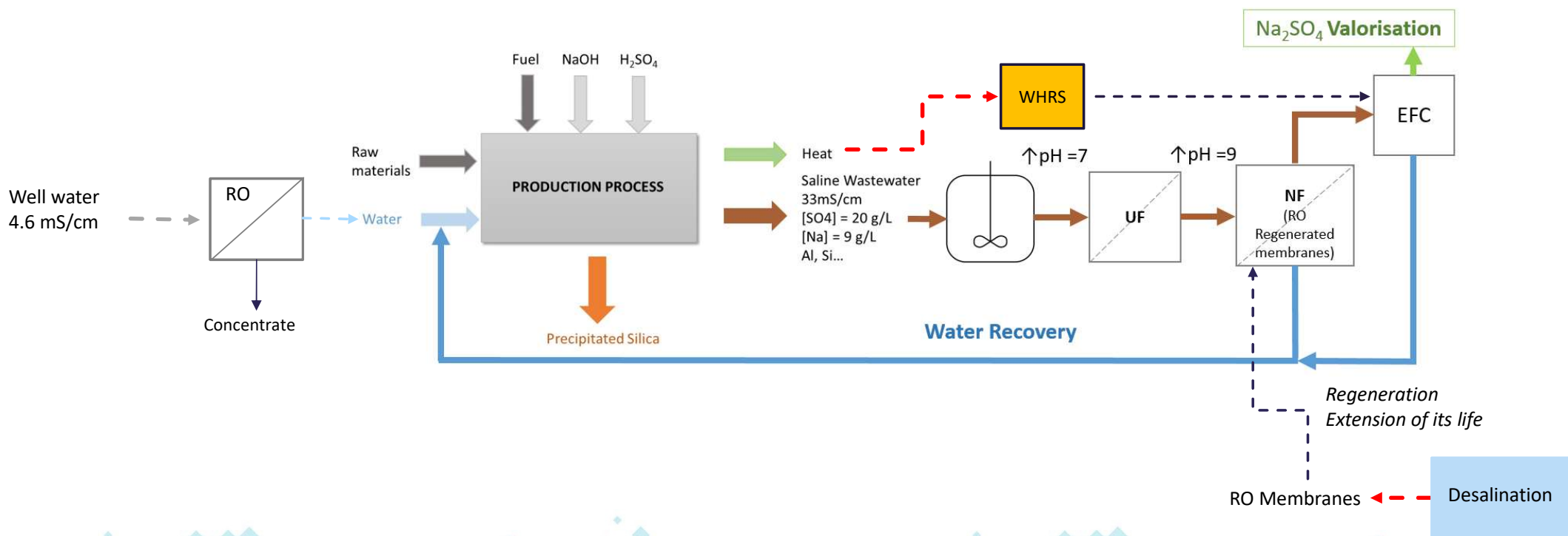
SEALEAU

TU Delft



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Circular economy scheme





OBJECTIVES

- ▶ To demonstrate the technical and economic feasibility of implementing a circulars economy scheme in the precipitated silica industry to recover water, sodium sulphate, waste heat, acids and alkalis.
- ▶ To demonstrate the technical feasibility of using recovered nanofiltration (NF) membranes for concentrating effluents with high salinity.
- ▶ To evaluate eutectic freeze crystallization (EFC) and forward-feed evaporation technologies to crystallize Na_2SO_4 from concentrated effluents.
- ▶ To demonstrate the technical and economic feasibility of the waste heat recovery system (WHRS) to reduce energy requirements of the circular economy scheme.
- ▶ To demonstrate the technical and economic feasibility of electrodialysis with bipolar membranes (EDBP) to recover NaOH and H_2SO_4 from silica industry effluents



Subtask 4.3.2. Operation and optimization

- Start-up on September – December 2018
- RO/NF stage operation until end of 2019
- Development of data treatment tool
- Evaluation of Type I membrane: January – March 2019
- Evaluation of Type II membrane: April – June 2019
- Evaluation of commercial SWRO membrane: September – October 2019
- November 2019 production of concentrate for EFC
- Crystallization stage: treatment of concentrate from RO/NF
- Tentative schedule
 - EFC pilot plant October – December 2019?
 - Evaporation pilot plant?





Subtask 4.3.2. Operation and optimization

Ultrafiltration



Nanofiltration
(regenerated membranes)



Regenerated membranes properties

The properties of the regenerated membranes were set in order to produce a permeate with a suitable quality to be reused and reducing the working pressure of the RO process.

Different reuse strategies within the production process can be considered depending of permeate conductivity:

- Low conductivity, similar to permeate from RO used to treat well water: direct reuse.
- Conductivity around 4.6 mS/cm: permeate could be mixed with well water to be treated in the RO and/or used as washing water in the production process
- Conductivity >4.6 mS/cm, water used as washing water

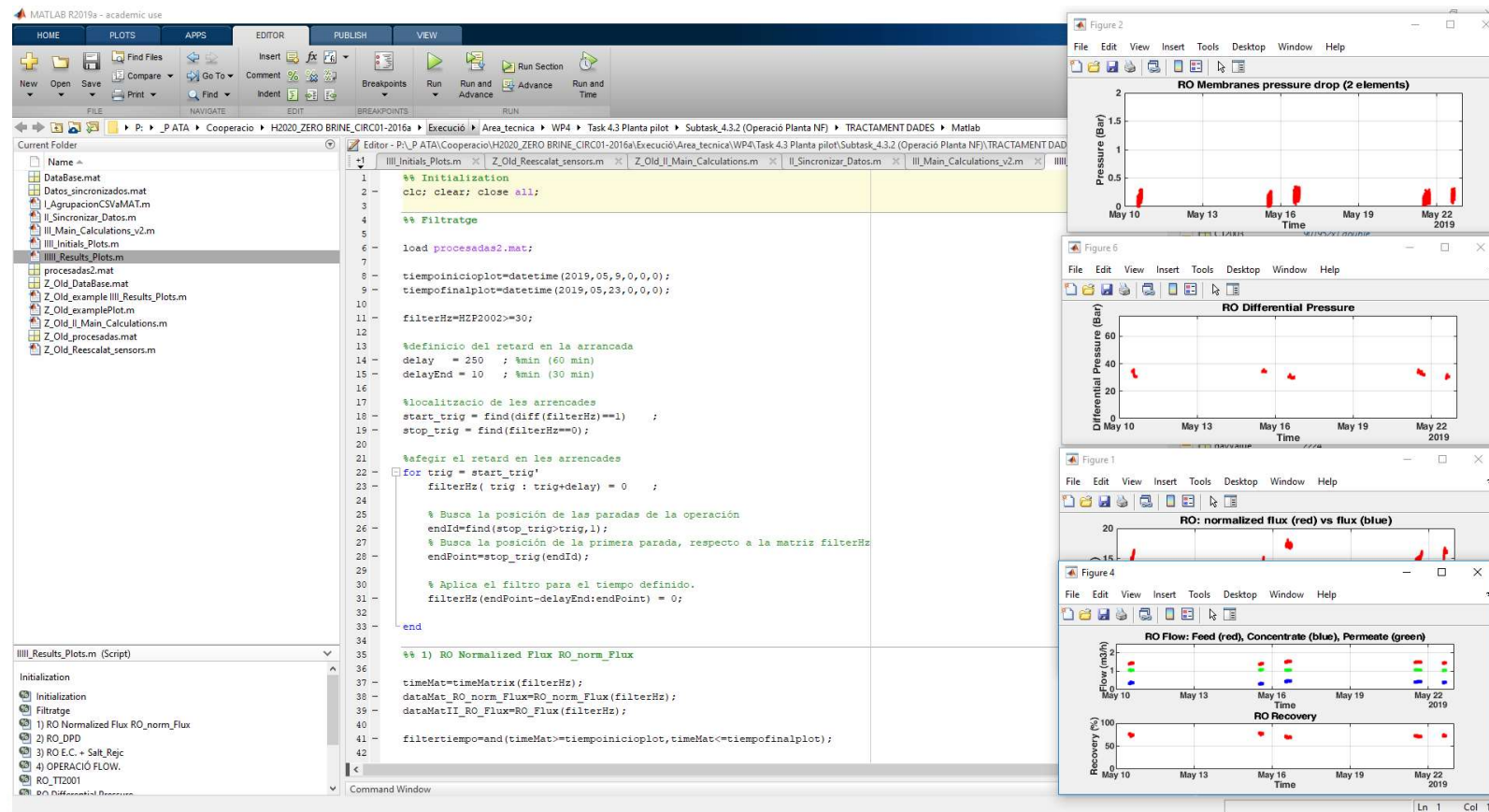
Regenerated membranes properties

The targeted regenerated membrane should present the following properties:

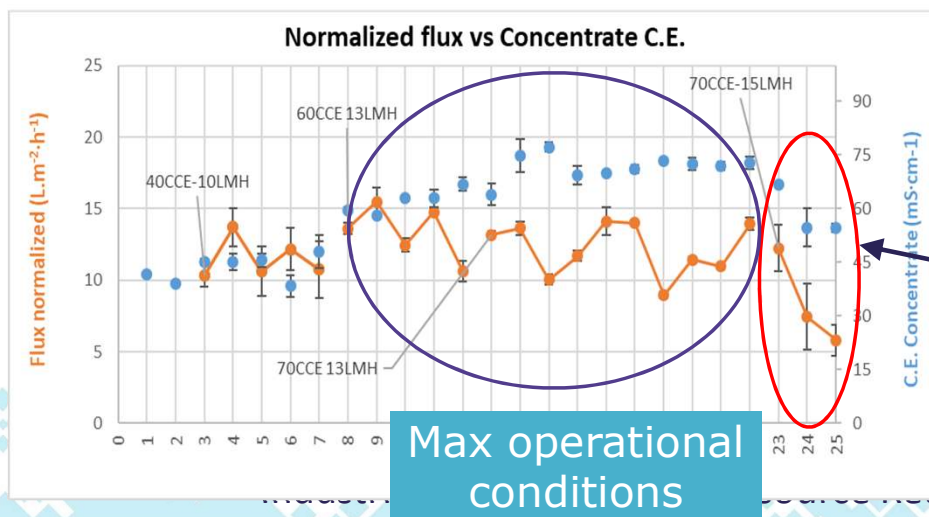
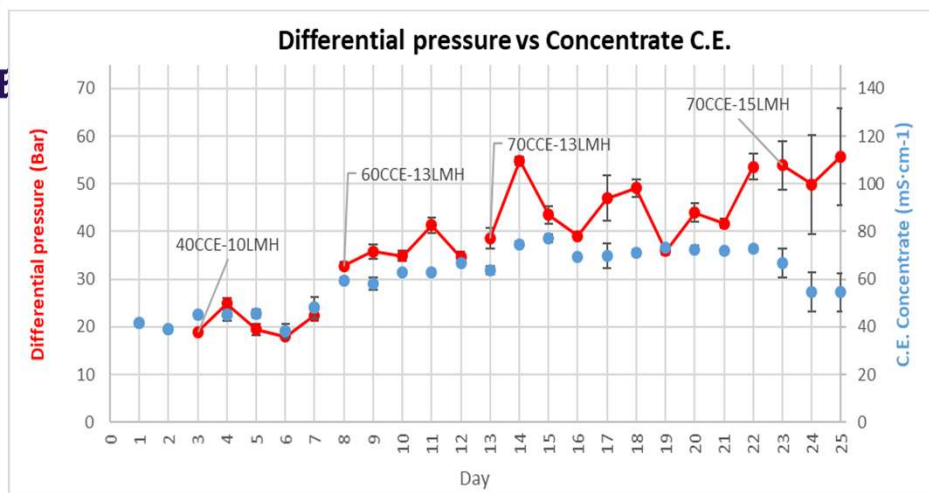
- Higher permeability than commercial SW-RO membranes: its increase will allow to reduce working pressure and energy consumption.
- Permeate quality adapted to the water uses at IQE. For that, a minimum rejection of 85% has been defined.

	NaCl		MgSO ₄	
	Permeability (L/m ² ·h·bar)	Rejection NaCl (%)	Permeability (L/m ² ·h·bar)	Rejection MgSO ₄ (%)
Type I - 1	1.8	98.9	3.1	98.7
Type I - 2	1.7	98.4	3.0	99.2
Type II - 1	3,7	91,1	3.9	94.3
Type II - 2	4,2	94,4	3.8	97.5

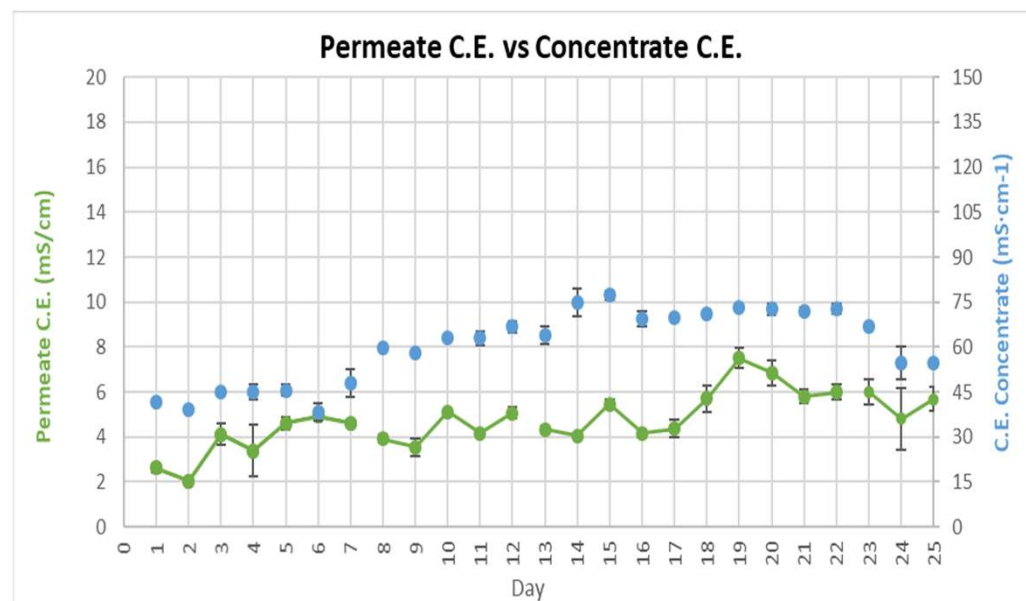
Data treatment tool using Matlab



Pilot plant Operation Type I



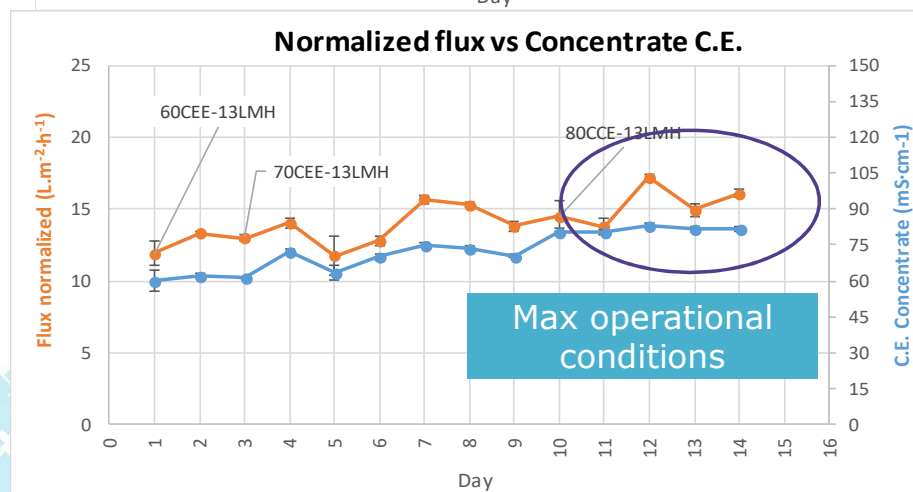
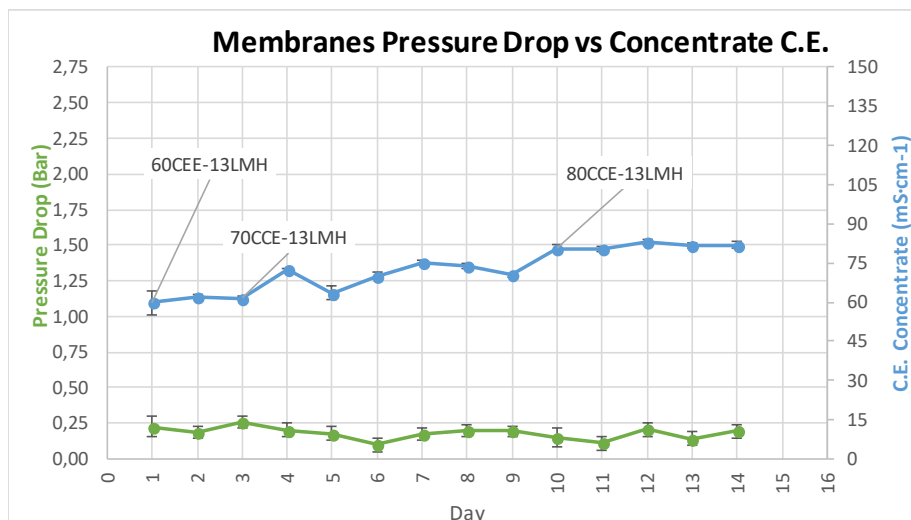
Recovery increase until maximum operation pressure



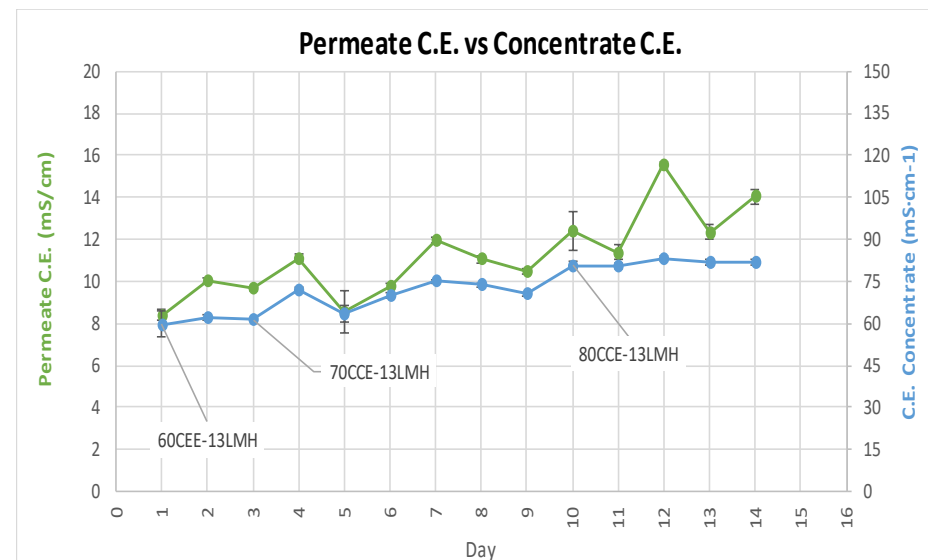
Scaling: nLMH drop & sudden differential pressure increase >60 Bar.

Recovery ♦ Circular Economy

Pilot plant Operation Type II



Recovery increase until a concentrate of 80mS/cm is produced



Pilot plant operation conditions

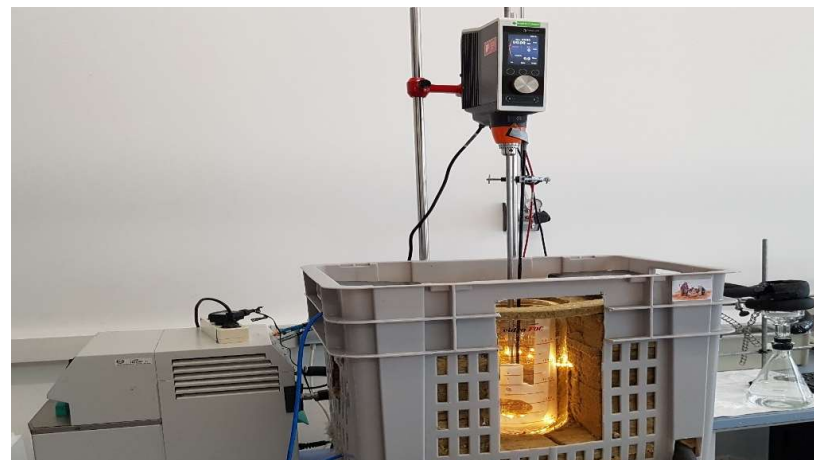
Criteria for operation conditions optimization:

- Maximum concentrate conductivity (High recovery): important for the crystallization stage
- Permeate quality suitable for reuse
- Low working pressure

	TYPE I	TYPE II
C.E. Feed ($\text{mS}\cdot\text{cm}^{-1}$)	30 ± 4	36 ± 3
C.E. Concentrate ($\text{mS}\cdot\text{cm}^{-1}$)	72 ± 3	82 ± 1
C.E. Permeate ($\text{mS}\cdot\text{cm}^{-1}$)	5 ± 1	13 ± 2
C.E. Rejection (%)	82 ± 5	64 ± 2
Recovery (%)	67 ± 5	73 ± 3
Pressure (bar)	45 ± 6	32 ± 2
Norm Flow (LMH)	12 ± 2	15 ± 1

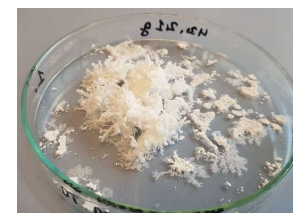
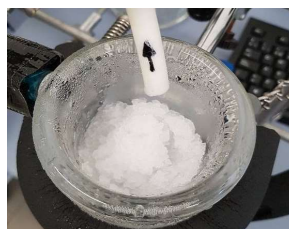
Design and optimization of Na_2SO_4 crystallization

- Comparison of EFC and Forward Feed evaporation
- Bench scale evaporation not available. Comparison will be made at pilot scale.
- Thermodynamic simulations
- Bench-scale experimental system construction at Eurecat
- Experiments using synthetic samples



Eutectic point
-1,27°C

Ice Recovered ($0.48 \text{ mS}\cdot\text{cm}^{-1}$)



Na_2SO_4 Recovered

Industrial Wastewater ♦ Resource Recovery ♦ Circular Economy



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Industrial Wastewater ◆ Resource Recovery ◆ Circular Economy