

WP4

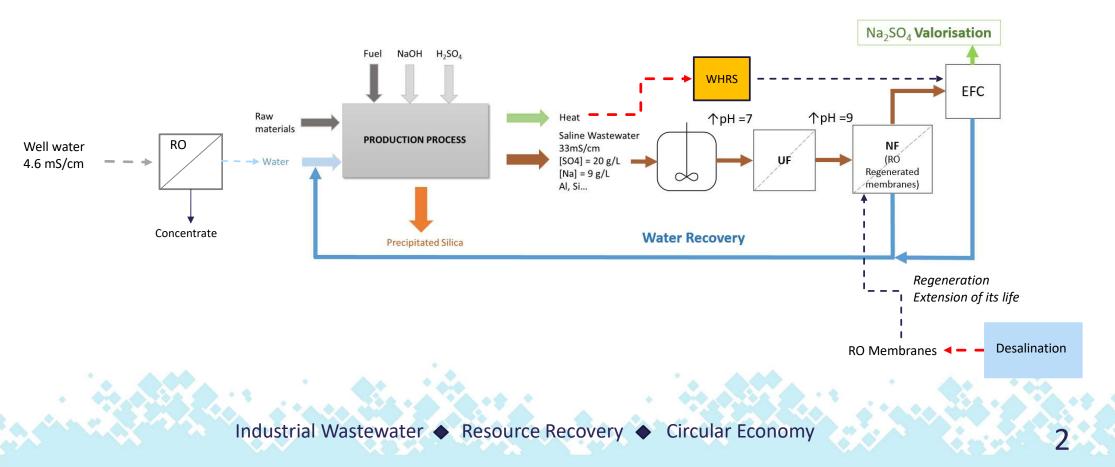
Promoting circular economy in the chemical sector: an innovative approach to recover resources from wastewater generated in the silica indutsry



The ZERO BRINE project (www.zerobrine.eu) has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 730390.



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 Na₂SO₄ 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 electrodyalisis with bipolar membranes (EDBP) to recover NaOH and H₂SO₄ from silica industry effluents



- 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)



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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	Rejection	Permeability	Rejection
	(L/m²·h·bar)	NaCl (%)	(L/m²·h·bar)	MgSO ₄ (%)
Type I - 1	1.8	98.9	3.1	98.7
Туре 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

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Data treatment tool using Matlab

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	<pre>19 - stop_trig = find(filterHz==0); 20</pre>	Time
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	<pre>26 - endId=find(stop_trig>trig);</pre>	20 20
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una car a c	34	RO Flow: Feed (red), Concentrate (blue), Permeate (green
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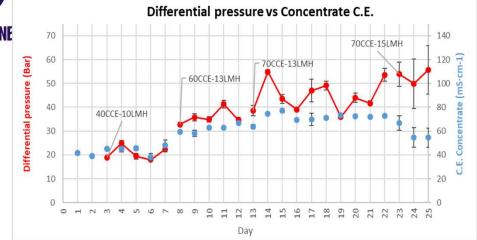
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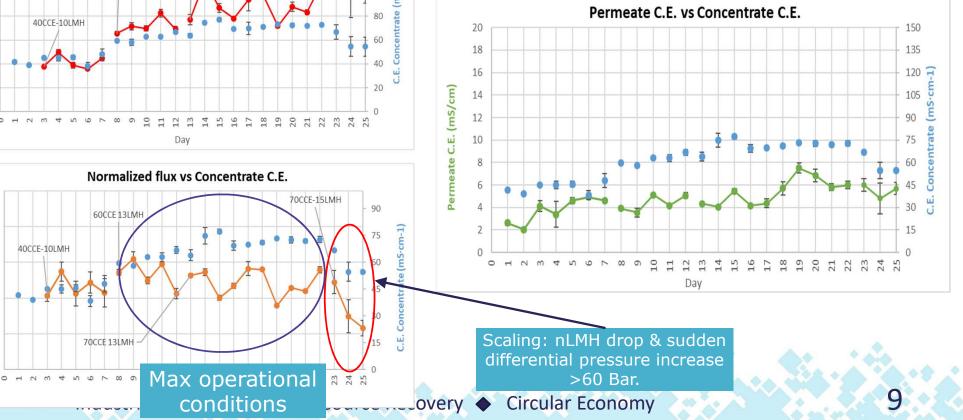
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Flux normalized (L.m⁻²·h⁻¹)

Pilot plant Operation Type I

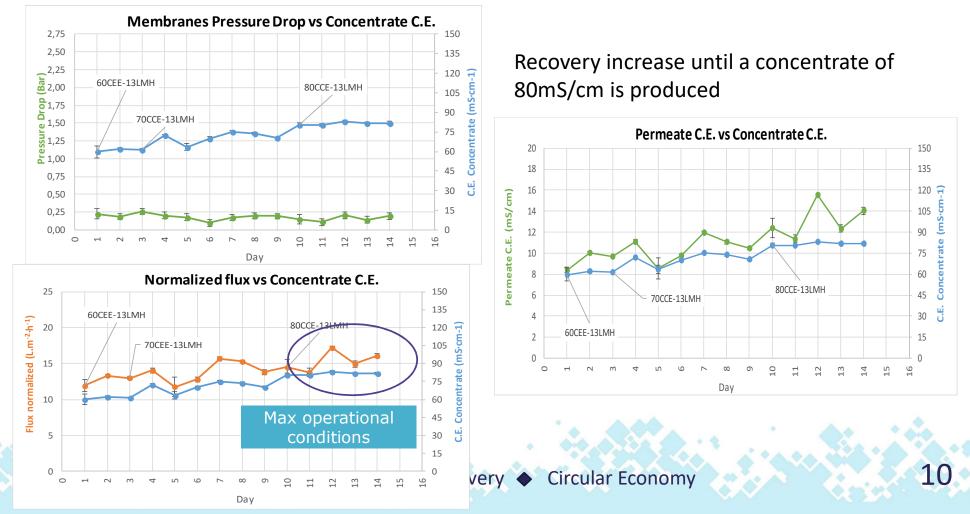


Recovery increase until maximum operation pressure





Pilot plant Operation Type II





Pilot plant operation conditions

Criteria for operation conditions optimization:

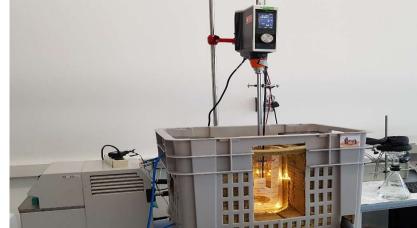
- Maximum concentrate conductivity (High recovery): important for the crystallization stage
- Permeate quality suitable for reuse
- Low working pressure

	ΤΥΡΕ Ι	
C.E. Feed (mS·cm⁻¹)	30 ± 4	36 ± 3
C.E. Concentrate (mS·cm ⁻¹)	72 ± 3	82 ± 1
C.E. Permeate (mS·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 Na₂SO₄ 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 -







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