

# Design of Eutectic Freeze Crystallization for the recovery of pure Na<sub>2</sub>SO<sub>4</sub> salts from RO brines

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## Introduction

Resource recovery and recycling are among the main elements that contribute to the increase of societal sustainability. ZERO BRINE promotes a circular economy approach and demonstrates new configurations to recover resources from saline impaired effluents (brines) generated by process industries, while eliminating wastewater discharge and minimizing environmental impact of industrial operations.

Eutectic Freeze Crystallization (EFC) is considered as an innovative approach to recover high quality end-products, with good market value from wastewater effluents. EFC is a fairly recent technology that has the potential to separate and recover salts and ice of high purity, with low energy requirements [1]. Within the scope of ZERO BRINE, a complex Reverse Osmosis (RO) concentrate stream from a silica production industry in Spain (Industrias Químicas del Ebro S.A, IQE) and an industry water production facility in The Netherlands (Evides Industriewater, Rotterdam Port) studied for the possibility of recovering sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>) crystals.

## Methods

### 1. Design of EFC equipment

In order to conduct the bench-scale EFC trials, a 15L setup was designed and developed from scratch [Figure 1]. This process involved several trials reaching eutectic conditions with a synthetic solution of sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>). Several modifications were implemented to improve scraping and thus, avoid ice scaling and enhance heat transfer.

### 2. Eutectic Temperature / EFC bench-scale trials

A characterization of the wastewater of the precipitated silica production process indicated that sodium, sulfate, magnesium and chloride are the dominating ions (~99 wt%) in the brine stream, with sodium and sulfate being the most abundant (~92 wt%). In order to assess the effect of impurities (such as calcium and silica) on the eutectic temperature of the binary sodium sulfate-water solution, 4 synthetic solutions with different compositions were prepared and tested in the 15L EFC crystallizer. After successfully reaching eutectic conditions, the temperature of each solution was measured and registered.