Development of a pilot plant for the recovery of magnesium hydroxide from waste brines

Sviluppo di un impianto pilota per il recupero di idrossido di magnesio da salamoie di scarto

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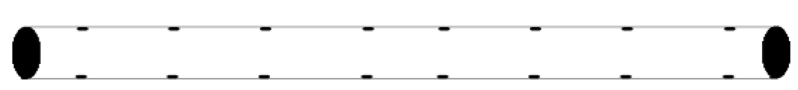
Waste brines from various industrial processes, mainly from saltworks, are an important source of minerals, such as magnesium, table salt and potable water. Magnesium is the third most abundant element in the sea salt, with a concentration between 1 and 1.5 g/L, but it can be up to 30 times larger in waste brines from saltworks. Magnesium can be recovered from waste brines by means of reactive crystallization, where:

$$Mg^{2+} + 2OH^{-} = Mg(OH)_{2\,(s)} \downarrow$$

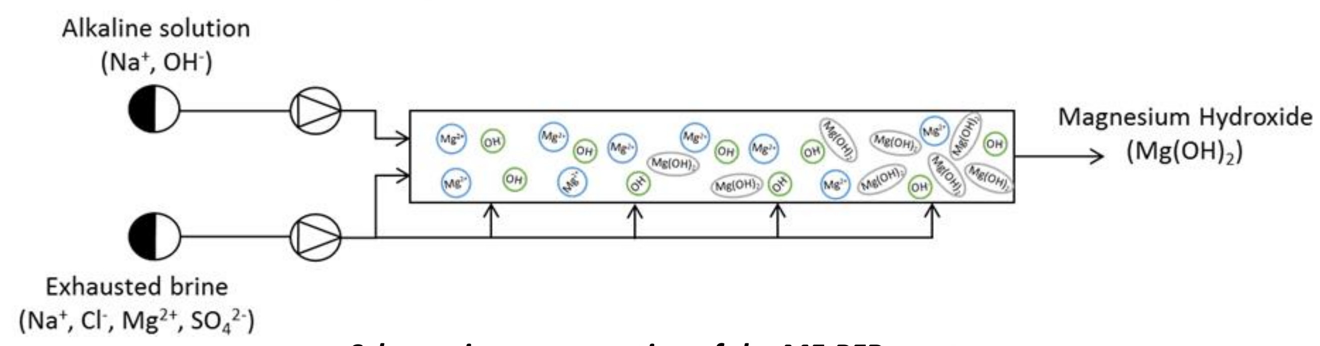
OH^- from: NaOH, $Ca(OH)_2$, NH_3, Na_2CO_3. In terms of purity, the best results are obtained using NaOH as alkaline solution. Once the magnesium hydroxide is collected, it is filtered and dried.

MF-PFR REACTOR

The reaction takes place in the MF-PFR, that is a PFR reactor with multiple feed inlets. It consists of two coaxial cylindrical tubes: the feed brine flows in the inner tube, while the alkaline reactant is forced to flow in the annular section.



Schematic representation of the inner tube

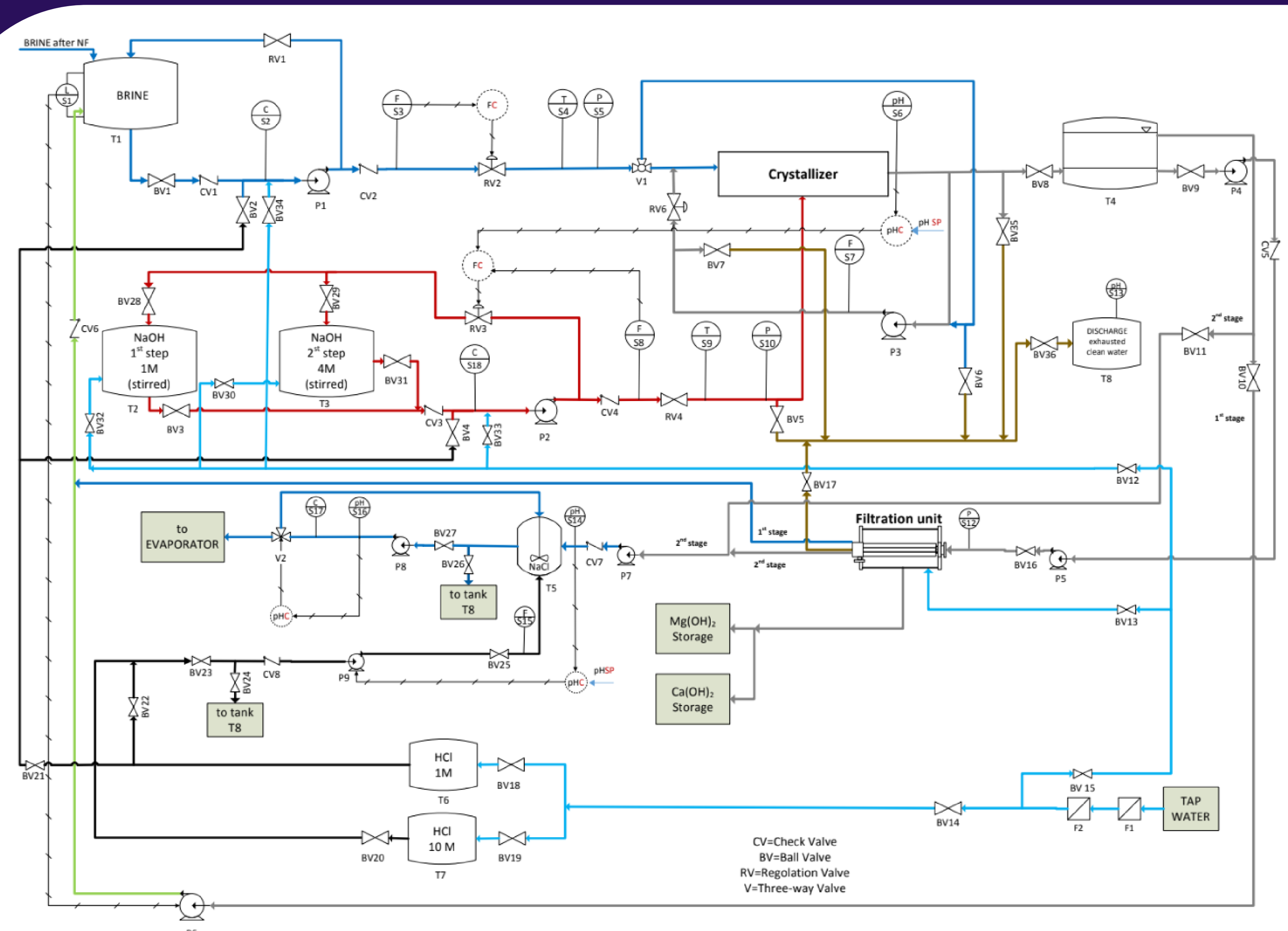


Schematic representation of the MF-PFR reactor

The inner tube is provided with 16 holes which allow the passage of the brine towards the annular section where it meets the alkaline solution.

The holes have a suitable geometry generating a vortex moving counter-clockwise around the tube axis thus allowing the process yield to increase.

P & I DIAGRAM



Piping and Instrumentation Diagram of the pilot plant

- After the MF-PFR reactor, the suspension containing magnesium hydroxide enters the filtration unit, where it is filtered and the solid is retained to form the cake.
- The last step concerning magnesium is the drying and the grinding of powders.
- The brine without magnesium must be treated before being discharged: it is sent to an agitated tank (T5) where a solution of hydrochloric acid is added to restore the pH value of the brine next to the neutral one.

PILOT PLANT

Pilot plant front view

Pilot plant front view with indication of the two feed tanks

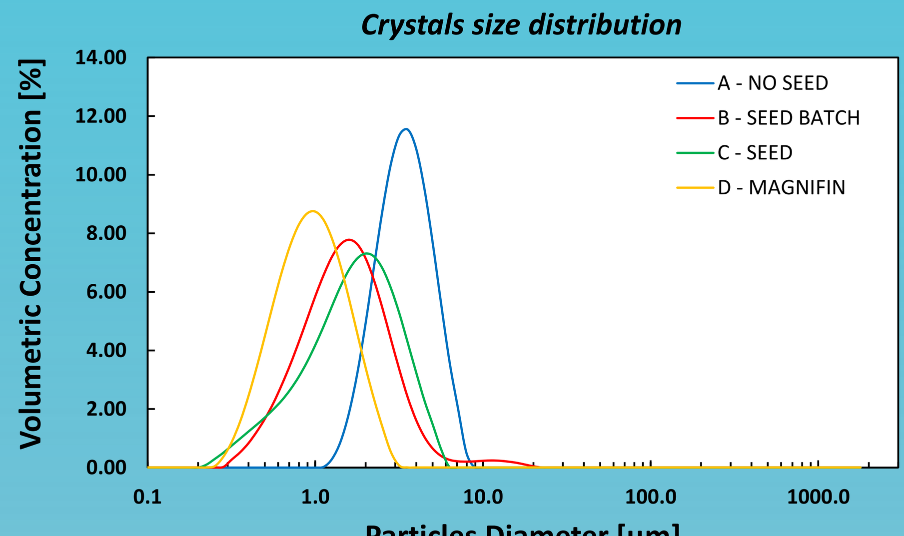
Reactant inlets in the MF-PFR

Pilot plant side view

HIGHLIGHTS

In order to evaluate the size distribution of crystal agglomerates collected, a test-run at different operative conditions is carried out.

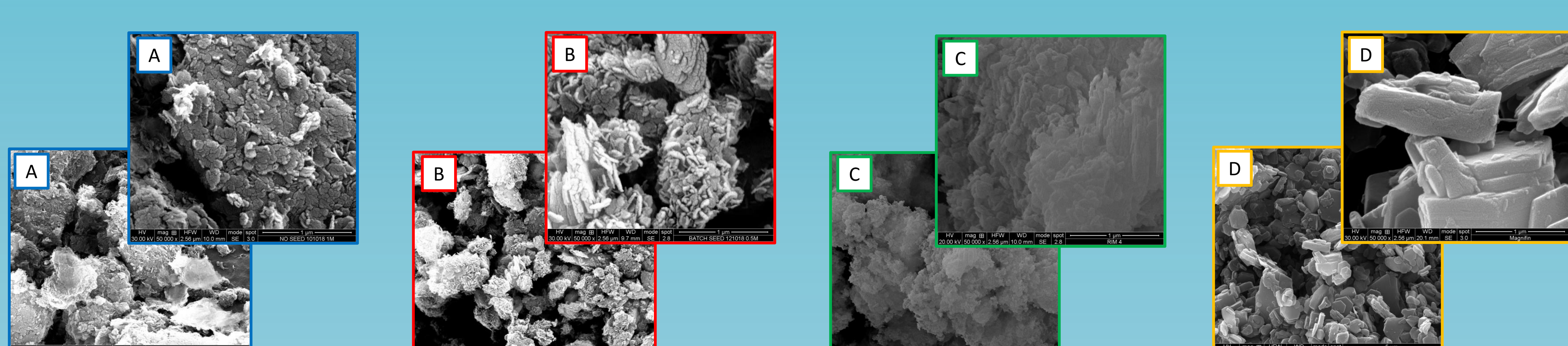
Crystals size distribution



Operative conditions of the test-run

	Sample	Brine Mg^{2+} Conc. [g/L]	Brine flow rate [L/min]	Base Conc. [mol/L]	Base flow rate [L/min]
A	NO SEED	4	1	1	0.33
B	SEED BATCH	4	0.5	0.5	0.33
C	SEED	4	4	4	0.33
D	MAGNIFIN	Market reference			

In the "A" case the brine flows in the MF-PFR without addition of magnesium hydroxide seeds, as opposed to both other conditions. In the "B" case the brine with seeds is fed until the reactor emptying time is reached (after this time the brine without seeds is allowed to flow in the MF-PFR) while in the "C" case it flows for the whole time of the test-run.



SEM pictures at different magnifications (10000 X and 50000 X) of the magnesium hydroxide (A, B, C) and of the market reference (D) samples

CONCLUSIONS

- The magnesium hydroxide crystals morphology obtained with the pilot plant is a globular and cauliflower-like agglomerates.
- The "B" and "C" distributions are larger than the "A" and their maximum volumetric concentrations are smaller because of the operative conditions: there is an accumulation of agglomerates on the seed surface.
- In all operative conditions a magnesium hydroxide purity higher than 99% is observed with a total conversion.

CONVEGNO GRICU 2019
30th June – 3rd July 2019, Palermo, Italy

Acknowledgements

The ZERO BRINE project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement N° 730390.