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Process Testing for Improving and Optimizing the Solvay Sodium Carbonate Production Process with Filter Press Technologies

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Introduction

The Solvay process, also known as the ammonia-soda process, produces sodium carbonate and calcium chloride. The two raw materials are limestone (calcium carbonate) and brine (sodium chloride).

The limestone is thermally decomposed to produce calcium oxide and carbon dioxide. The carbon dioxide passes through an ammonia-brine resulting in precipitation of sodium bicarbonate which is then calcined to sodium carbonate.

Figure 1 shows the existing Solvay process including the final products, recycled components as well as the waste product of calcium carbonate ($CaCO_3$)/calcium chloride ($CaCl_2$).

This article focuses on the calcium carbonate/calcium chloride waste product that was initially discharged into the ocean and the approach used by the client with the Diemme® Filtration filter press technology to eliminate the environmental problem and create a salable product.

Process Objectives and Filter Press Testing

From Figure 1, the calcium carbonate / calcium chloride is a waste product that was currently being discharged into the sea. This has created an environmental problem for the site. In addition, the calcium chloride waste is lost revenue as there is a value to the product for salt de-icing of roads, sidewalks, etc. in cold weather climates.
The client contacted Diemme® Filtration to conduct laboratory test work to determine the optimum solid-liquid filtration technology to produce high-quality calcium chloride.

There are two steps in the process. First, the calcium carbonate is filtered and dewatered. The dewatered cake is discharged to conveyor belts into a crusher and then into a downstream mix tank. In this mix tank, cake is mixed with water and dissolved with hydrochloric acid to produce the final calcium chloride, in liquid form. This calcium chloride liquid is filtered again before evaporation to produce the final product.

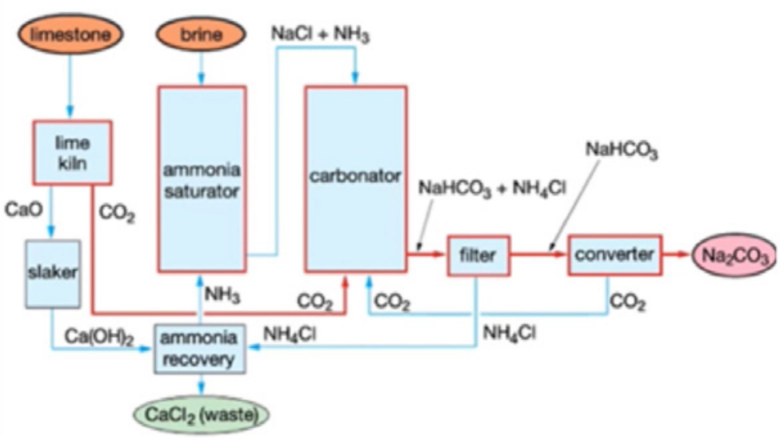

Figure 1: Existing Solvay Sodium Carbonate Production Process

Figure 2 shows the Diemme® Filtration Filter Press Laboratory Test Unit. The lab test unit consists of two feeding tanks with agitators, two sets of filtration chambers with varying cake thickness and two membranes for plate pressing.
The filter surface area is 0.008 m^2.

The first slurry to be filtered is 36% solids consisting of magnesium hydroxide, calcium sulphate and various silicates in a calcium carbonate solution at 60° C.

The particle size distribution (PSD) of the solids is a D50 of 29 µm and a D90 of 93 µm. The client's objective is to filter this suspension at a concentration of 80 g/l to the lowest possible residual moisture and be able to discharge completely from the filter cloth.
Figure 3 shows the test results.

	Test 1	**Test 2**
Chamber thickness (mm)	20	25
Chamber volume (l)	0.156	0.196
Filter surface area (m^2)	0.008	0.008
Max. feed pressure (bar)	5	7
Feeding time (min)	5	7
Volume of turbidity filtered (l)	1.21	1.57
Average feed rate (l/hm^2)	1815	1682
Final feeding speed (l/hm^2)	30	30
Final squeezing time (min)	5	5
Squeezing pressure (bar)	12	12
Filtered	Immediately clear	Immediately clear
Cake	Compact	Compact
Detachment	Good	Good
RH (% by weight with salt)	35.11	35.11
Cake thickness (mm)	14	14
Reduction in (m^3sludge/ m^3cake)	11	11

Figure 2: Diemme® Filtration Filter Press Laboratory Test Unit – Filtration Test Results

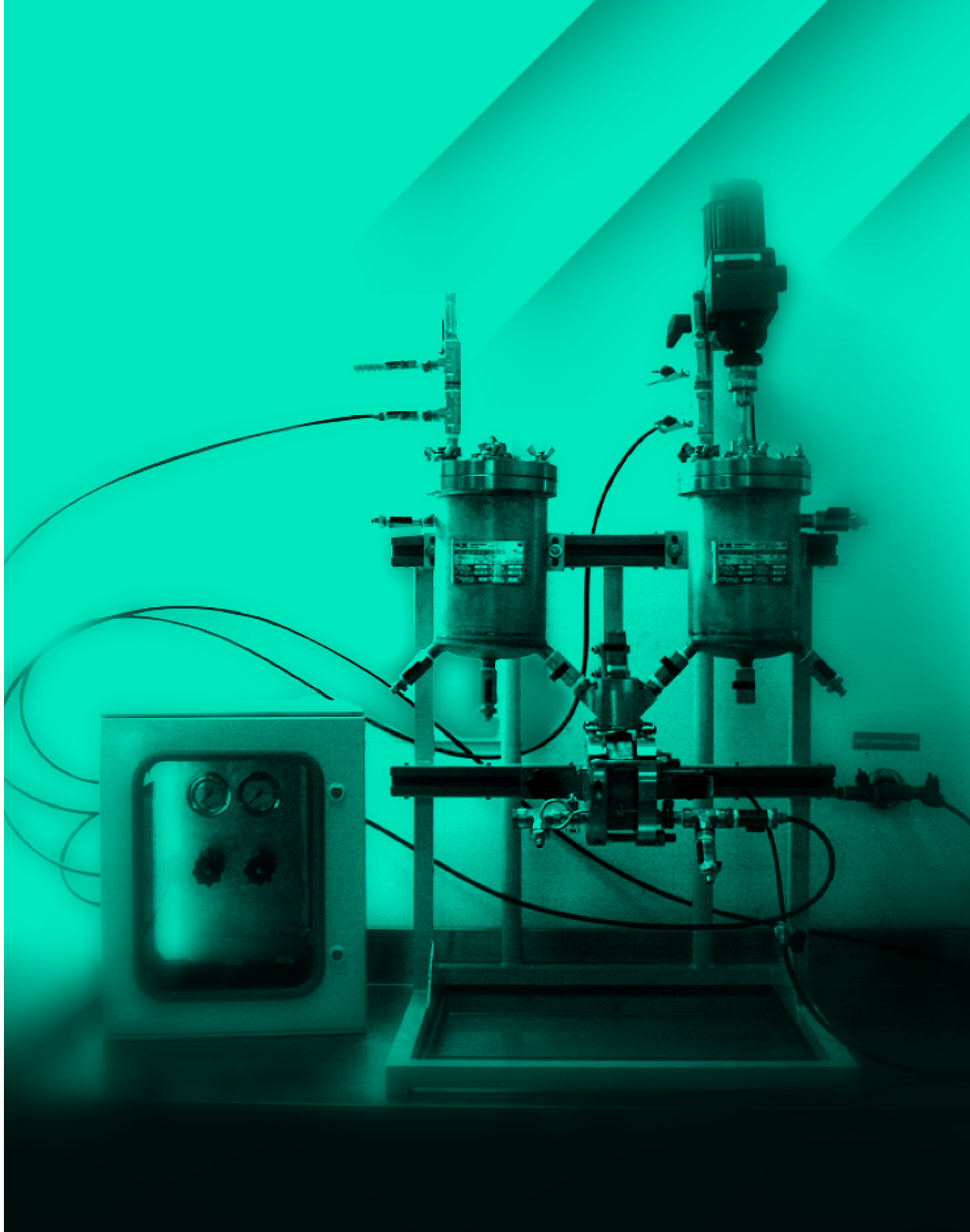

Figure 3: Diemme® Filtration Filter Press Laboratory Test Unit

For the first slurry, the testing showed excellent filterability with the granular crystals forming a compact and homogenous cake. The well-formed cake discharged completely from the polypropylene filter cloths. The scale-up is based upon test number 2 using membrane plates with a chamber/cake thickness of 50 mm. The sizing results in two (2) GHT 1200 x 1200 overhead beam-high performance filter presses with a filtration area of 210.24 m² each. The GHT 1200 has 46 membrane plates with 50 mm chambers and a total volume of 4428 liters.

For the second final product slurry, additional testing was conducted. In this case, the slurry contained magnesium, as a precipitated magnesium-hydroxide. This slurry is filtered to remove the hydroxides from the $CaCl_2$ liquid before it is evaporated, concentrated and stored in big super sack bags. The scale up for this part of the process resulted in one (1) ME 1000 x 1000 side beam-versatile performance filter press with a filtration area of 36 m². The ME 1000 has 24 membrane plates with 20 mm chambers and a total volume of 1000 liters.

Figure 4 shows the improved Solvay process with the Diemme® Filtration solid-liquid filtration technologies to produce a final clean and saleable calcium chloride.

Figure 5 shows the GHT 1200 x 1200 installed at the plant.

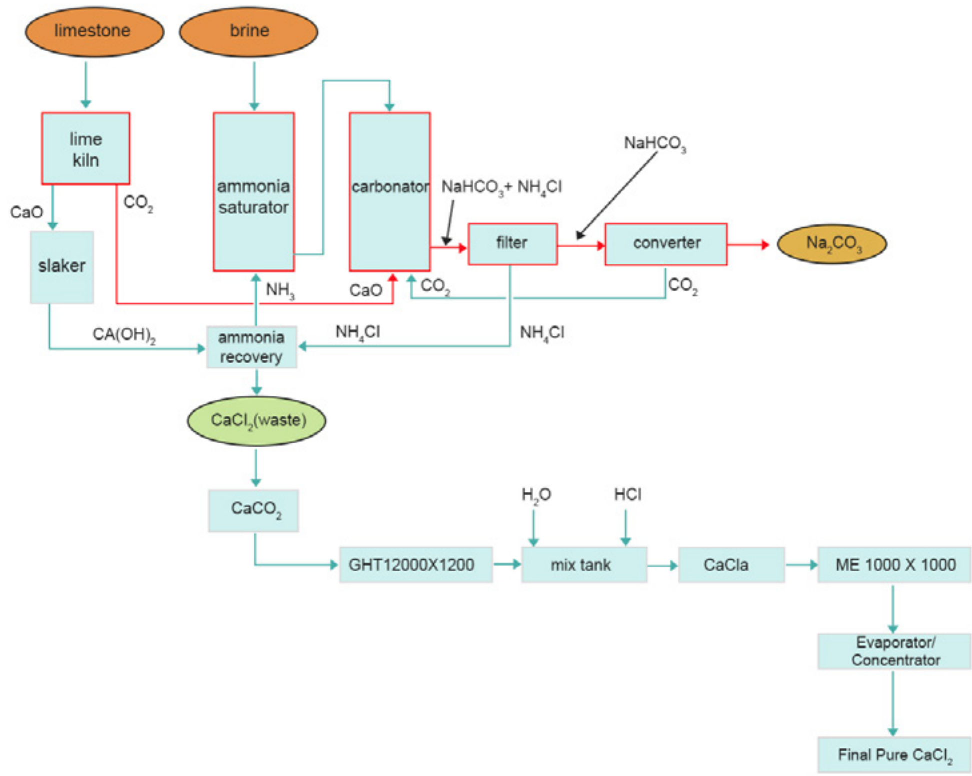

Figure 4: Improved Solvay Sodium Carbonate Production Process for Producing Final Pure $CaCl_2$ for Sale with the Diemme® Filtration Filter Press Technology

Figure 5: Diemme® Filtration GHT 1200 x 1200 Installed on the Top Floor of the Structure

Figure 6: Diemme® Filtration GHT 1200 x 1200 Shown in the Process Step with Plate Stack Closed

Diemme® Filtration Filter Press Technology

The Diemme® Filtration filter press technology consists of a plate stack which forms a series of chambers. Each plate is covered with a filter cloth with plate-to-plate sealing. The slurry is pumped into the plate chambers, uniformly filling with solids while the filtrate is collected via a piping manifold.
The solids continue to fill the plate chambers until they are completely full of saturated filter cake. Cake washing follows, either counter-current or co-current followed by dewatering and drying by gas blowing and/or membrane squeezing. When the process cycle is completed, the plate stack opens, and the cake is discharged.

The GHT technology has important benefits for the client by providing for high productivity. The filter plates, which are hung from the upper beam, are moved automatically by a rapid shifting device equipped with a carousel system which reduces the opening and closing sequencing time. The membrane plates incorporate a laser control system ensuring correct plate pack alignment and automatically stops the filter press during upset conditions. Four hydraulic cylinders placed on the plate pack corners ensure perfect operation and limited structural stress, even in the most unfavorable working conditions for high reliability and high uptime. The pressure exerted by the closing device is uniformly distributed on each plate with an automatic control system that adjusts the cylinder length according to the exerted pressure, to ensure perfect closure of the plate pack without any leaking of process fluids.

Due to the harsh environment additional mechanical features have been included such as automatic cloth washing as well as liners to protect moving parts from dust and corrosive vapors and liquids. Finally, the GHT uses state-of-the art electronic interlock systems for maximum operator health and safety such that routine maintenance operations are simple due to easy access to the filter plates, both for inspection and for filter cloth replacement. The interlocks as well as load cells and other sensors are programmed into the Diemme® Filtration PLC control system.

The ME technology has additional process benefits of "variable-volume chambers" for the membrane plates. These variable plates are used when in-chamber cake washing or drying by air/gas blowing is required. Finally, the mechanical design of the ME provides for simultaneous plate pack opening and plate shifting. All plates are linked to the mobile header which, when opened, allows the plates also to open at short intervals resulting in the filter cakes being discharged in rapid succession. This minimizes the cake discharge time cycle for increased production.

Figure 7: Diemme® Filtration ME1000 used for the separation of Mg hydroxide from $CaCl_2$

Optimization Testing for Cake Washing

Diemme® Filtration provides continuing process support for their clients. As a result, after 1 ½ years of operation, the client wanted to upgrade the process and incorporate cake washing into step 1 of the process to reduce the chloride concentrations to below 5 g/kg of cake. Samples were shipped to the Diemme® Filtration laboratory and washing tests were performed. The tests included co-current and counter-current washing, air/gas blowing and squeezing. Many tests were performed and the Diemme® Filtration engineers developed a washing procedure based upon test 3 using a 50 mm thick membrane plate and washing the cake with water at a temperature of 50° C. The wash ratio used was a volume of water equal to 2 times the volume of the filter chamber and performing the blowing phase of the cake in a counter-current manner.

Figure 8 shows the test results and graphically in Figure 9. From the washing tests, it can be seen that the cake can be washed well and the chlorides in the cake (0.62 g of Cl per kg of cake) are significantly lower than the specification. Further, the testing showed that with a low wash ratio of only 2 chamber volumes the specification can be achieved.

	Test 3
Chamber thickness (mm)	25
Chamber volume (l)	0.196
Filtering surface (m²)	0.008
Maximum feeding pressure (bar)	6
Feeding time (min)	8
Volume of turbidity filtered (l)	0.66
Average feeding speed (l/hm²)	619
Final feeding speed (l/hm²)	20
Volume reduction (V sludge/V cake)	3.38
Washing time (minutes)	14
Membrane pressure during washing (bar)	3
Washing water pressure (bar)	3
Volume of wash water (l)	0.4
Final wash water conductivity (mS/cm)	6.5
Air blowing time (minutes)	=
Membrane pressure during blowing (bar)	=
Blow air pressure (bar)	=
Average air consumption during blowing (Nl/h)	=
Final squeezing time (minutes)	3
Final squeezing pressure (bar)	12
Filtered	Clear after 2`
Cake	thixotropic
Detachment	discreet
Cake R.H. (% weight) 105 °C	29.64
Cake thickness (mm)	20
Cl- (g of Cl per kg of cake)	0.62
Washing efficiency (%)	**98.7**

Figure 8: Diemme® Filtration Filter Press Laboratory Test Unit – Washing Test Results

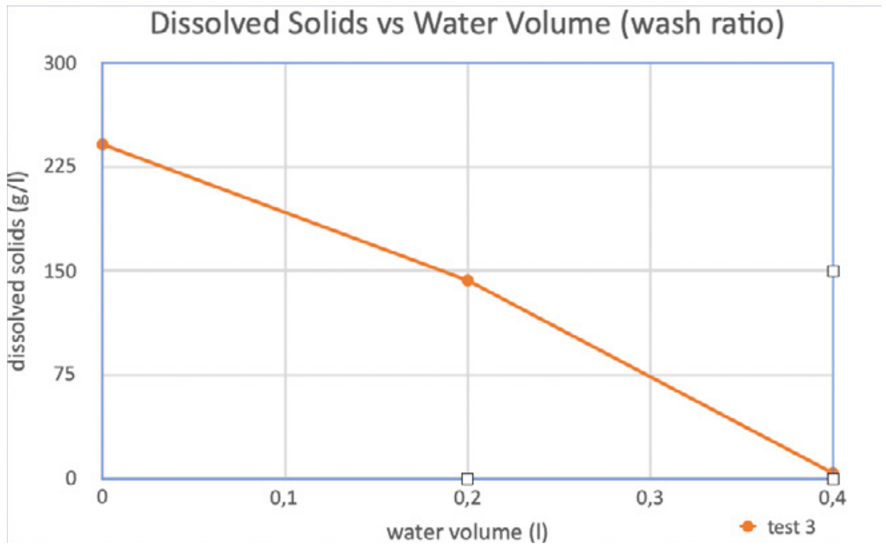

Figure 9: Dissolved Solids (Chloride Concentration) versus Wash Ratios

Process Benefits and Summary

The Diemme® Filtration Filter Press technology provided significant improvements for the client for the Solvay process. The most important is making the process more sustainable and environmentally-friendly. Diemme® Filtration was able to eliminate the calcium chloride as a waste product and produce a high-quality salable product.
The GHT and ME designs produce a consistent quality and cake dryness. The units have low maintenance and operating costs based upon the Diemme® Filtration PLC controls system, instrumentation and automatic operations.

Diemme® Filtration and the client engineers have worked together to analyze the process, conduct testing and collect the data necessary to make correct decisions for process optimization. Close cooperation allowed for creative problem solving for an technically efficient and commercially cost-effectiveness process solution.

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