Carbon Mineralization by Aqueous Precipitation for Beneficial Use of CO_2 from Flue Gas

Final Scientific/Technical Report

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ABSTRACT

The objective of this project was to demonstrate an innovative process to mineralize CO_2 from flue gas directly to reactive carbonates and maximize the value and versatility of its beneficial use products. The program scope includes the design, construction, and testing of a CO_2 Conversion to Material Products (CCMP) Pilot Demonstration Plant utilizing CO_2 from the flue gas of a power production facility in Moss Landing, CA as well as flue gas from coal combustion. This final report details all development, analysis, design and testing of the project. Also included in the final report are an updated Techno-Economic Analysis and CO_2 Lifecycle Analysis. The subsystems included in the pilot demonstration plant are the mineralization subsystem, the Alkalinity Based on Low Energy (ABLE) subsystem, the waste calcium oxide processing subsystem, and the fiber cement board production subsystem. The fully integrated plant was proven to be capable of capturing CO_2 from various sources (gas and coal) and mineralizing it into a reactive calcium carbonate binder and subsequently producing commercial size (4ftx8ft) fiber cement boards. The final report provides a description of the "as built" design of these subsystems and the results of the commissioning activities that have taken place to confirm operability. The report also discusses the results of the fully integrated operation of the facility. Fiber cement boards have been produced in this facility exclusively using reactive calcium carbonate from captured CO_2 from flue gas. These boards meet all US and China appropriate acceptance standards. Use demonstrations for these boards are now underway.

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LIST OF ACRONYMS AND ABBREVIATIONS

ABLE – Alkalinity Based on Low Energy
AFLAS – trade name for a unique fluoroelastomer based upon an alternating copolymer of tetrafluoroethylene and propylene
ANSI – American National Standards Institute
ASTM – American Society of Testing and Measurement
BFD – Block Flow Diagram
CapEx – Capital Expenditure
CCMP – CO_2 Conversion to Material Products
CEM – Cation Exchange Membrane
CEMS – Continuous Emissions Monitoring System
DI – Deionized Water
EDITDA – Earnings before interest, taxes, depreciation and amortization
E-Chem - Electrochemistry
EDC – Ethylene dichloride
FCB – Fiber Cement Boards
FGD- Flue Gas Desulfurization
GC-MS – Gas Chromatograph Mass Spectrometer
HMI – Human Machine Interface
IEA – International Energy Agency
I/O – Input output
ISO – International Standard Organization
MAP – Mineralization via Aqueous Precipitation
OPC – ordinary portland cement
OSB – Oriented Strand Board
OSHA – Occupational Safety and Health Administration
PCC – Precipitated Calcium Carbonate
PCS –Partial Cement Substitute
PEL – Permissible Exposure Limit
PFA - Perfluoroalkoxy alkanes plastic
PFD – Process Flow Diagram
PLC – Programmable Logic Controller
Psi- pounds per square inch
PVC- Polyvinyl Chloride
PVDF – Polyvinylidene fluoride plastic
QMS – Quadrapole Mass Spectrometer
SEM – Scanning Electron Microscope
STEL – Short Term Exposure Limit
STY – Space Time Yield
XRD – X-ray Diffraction

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1. PROJECT OBJECTIVES AND EXECUTIVE SUMMARY OF THE PROJECT RESULTS

The primary objective of this project was to demonstrate an innovative process to mineralize CO_2 from flue gas directly to useful carbonates and maximize the value and versatility of its beneficial use as a commercial product. The project goal was to capture and convert at least 1 tonne/day of CO_2 from power plant flue gas. This objective was achieved by designing, constructing, and operating a CO_2 Conversion to Material Products (CCMP) Pilot Demonstration Plant using flue gas from the Dynegy power production facility in Moss Landing, CA. The pilot demonstration plant integrates several critical technologies into a system that captures CO_2 and converts it to commercially viable carbonate materials and other industrial chemicals. The conversion of CO_2 into carbonates requires alkalinity to absorb CO_2 from flue gas and deprotonate the absorbed CO_2 to form a carbonate. Through leveraging proprietary techniques, Calera has developed a new electrochemical platform called ABLE that integrates with CO_2 absorption and produces alkalinity for production of carbonate materials, but also coproduces a range of potentially valuable chemical products. Calera's proprietary electrochemical technology enables the production of these industrial chemicals at lower energy than conventional production methods resulting in a reduction in overall carbon emissions while at the same time directly capturing and converting CO_2 to carbonate material products. The program objective was also to expand the range of suitable feedstock to include waste oxides from other industrial production processes. A waste oxide processing subsystem was developed that allow the use of waste oxide materials such as carbide lime residues generated as a byproduct of coal to acetylene conversion.

The key metrics for this program included the following:

- Demonstrate an efficient integrated process that converts 1 tonne per day of CO_2 from the flue gas stream into a stable solid product available for sale and beneficial reuse.
- Demonstrate 12 months of the plant's operation including continuous operation for extended period of time.
- Demonstrate CO_2 removal from flue gas from a gas-fired power plant. The minimum percentage removal of CO_2 from a slipstream of the flue gas from the gas-fired plant is estimated to be 20% with a target removal rate of 80%.
- Integration of the CO_2 absorption into the Calera electrochemistry technology (ABLE) for low energy conversion to sodium carbonate for use in carbonate production unit.
- Conversion of produced sodium carbonate to calcium carbonate for cement applications (supplemental replacement of ordinary portland cement (OPC) and as a non-OPC binder material), high value decorative and architectural concrete applications, dry wall and other board type products such as cement board and as filler for paper and plastics.
- Testing of various process units to convert byproduct streams into chemical products
- Completion of ASTM and other testing requirements to prove product acceptance/usability of carbonate materials for cementitious substitutes for use in the construction industry, for fillers in papers and plastics, for architectural applications, for wall board applications, and for higher value added applications that take advantage of the unique properties of the material.

- Demonstrate the use of waste oxide materials from other industrial processes for the alkalinity and calcium feedstock to capture and convert CO_2 to beneficial carbonates.

The fully integrated plant has been proven to be fully capable of capturing CO_2 from various sources (gas and coal) at the target rates and mineralizing it into a reactive calcium carbonate binder and subsequently producing commercial size (4ft x 8ft) fiber cement boards. The subsystems included in the pilot demonstration plant were the following subsystems:

- Mineralization via Aqueous Precipitation - alkalinity is used to capture CO_2 from the flue gas and then the aqueous carbonated material is contacted with a high calcium brine to produce reactive calcium carbonate (vaterite) slurry. The slurry is then dewatered and the wet solid is dried to yield dry reactive carbonate powder. This reactive vaterite carbonate is a self-cementing material when mixed with water and certain additives where it recrystallizes to form calcium carbonate (aragonite) cement.
- Waste Calcium Oxide Processing – In this subsystem, waste calcium oxide sources such as carbide lime are processed to allow them to be used as the source of both alkalinity and calcium. Raw waste solid, which contains predominantly calcium hydroxide is dissolved using ammonia chloride and impurities are filtered out. The dissolved calcium hydroxide equivalent is then used to absorb CO_2 and form the reactive calcium carbonate vaterite slurry in an absorber reactor. The slurry is then dewatered and dried with the same equipment as in the MAP subsystem.
- Alkalinity Based on Low Energy (ABLE) – As an alternative to use of waste oxides, the subsystem produces alkalinity for capture and conversion of CO_2 in the form of sodium hydroxide (NaOH) using electrochemical splitting of salt (NaCl). The ABLE process also produces a useful chemical byproduct, ethylene dichloride (EDC) through the use of a chlorine shuttle reaction using copper chloride to transfer chlorine atoms where they are subsequently reacted with ethylene in a catalysis reactor. This technology can also stand alone as a lower energy alternative to the current state of the art technologies that produce sodium hydroxide and ethylene dichloride.
- Fiber Cement Board production – a complete continuous fiber cement board production line that uses the carbonate cement along with cellulosic fibers as input and produces commercial size 4-foot by 8-foot boards.

To date almost 30 tonnes of material has been produced by the MAP subsystem and converted to fiber cement boards. The MAP pilot plants continue to be used as a source of material for commissioning of the fiber cement board plant as well as production of fiber cement boards to be used in demonstration projects. Through testing on the waste oxide processing pilot plant, the program has also successfully demonstrated the technical industrial scale feasibility of recycling waste oxide materials to capture CO_2 and SO_2 pollutant gases, while also producing a valuable and novel cement product. While all of the pilot scale testing was done using carbide lime, lab testing has shown that the demonstrated process may be applied to other waste metal oxides, including various steel slags, with changes to only the initial oxide dissolution step. The versatility of the process creates a much more geographically broad range of applicability.

The ABLE pilot demonstration subsystem has been successfully operated in a full recycle manner where the copper reagent was chlorinated in the E-Chem cell and de-chlorinated in the catalysis reactor. The subsystem generated chemical grade sodium hydroxide and ethylene

dichloride sufficient to meet acceptance criteria. The sodium hydroxide produced in this subsystem was used in the MAP subsystem to capture CO_2 from flue gas.

The calcium carbonate produced in the pilot demonstration plant from CO_2 in flue gas has successfully been used for a wide range of applications including as a partial cement substitute, precipitated calcium carbonate, aerated concrete, and fiber cement boards. Fiber cement boards were selected as the demonstration product for the pilot demonstration project based upon considerations of technology advantages over current products, overall product market size, technical feasibility and product differentiating factors. Calera's prototype fiber cement products for both backer board and sheathing applications have passed the interior use fiber cement standard, ASTM C 1288, and exterior use fiber cement standard, ASTM C 1186. The prototype fiber cement boards achieved the development target of 1,400psi equilibrium flexural strength and 1,000 psi saturated flexural strength at a density of 0.97 g/cm^3. Other mechanical properties such as nail head pull-through significantly exceeded the specification requirements. The produced boards were shown to pass the specific requirements for all durability performance including mold resistance, surface burning, moisture movement, warm water resistance, water tightness, and freeze thaw resistance.

The Calera Project, funded by the ARRA, demonstrates a first-of-a-kind process that captures CO_2 from power plant and industrial flue gas and converts it into reactive carbonate minerals that can be made into marketable building materials. In particular, the program accomplishments were as follows:

- Designed and built an integrated CO_2 conversion to material products pilot demonstration plant that can capture 1 ton per day of CO_2 and convert it into full size fiber cement boards (4ft by 8ft) while co-producing useful commodity chemicals at 40% lower energy than current state of the art technologies.
- The project has already had a substantial effect on the local economy, creating over 30 jobs during construction, and more than 12 new permanent full-time jobs.
- The manufacture of the cement products elsewhere normally produces 0.9 tonnes of CO_2 per tonne while the Calera product sequesters a net 0.170 tonnes of CO_2 per tonne of product.
- Construction of the plant is 100 % complete, pilot plant management, operating and maintenance personnel have been hired, and training of pilot plant operating personnel has been completed. The pilot demonstration plant is currently in full operation.
- The Calera technology was designed to retrofit existing coal-burning facilities, but also has potential applications for heavy industry, including cement, glass, steel and natural gas power.
- During the course of the program to date, CO_2 from both natural gas and coal fired flue gas has been captured and converted into approximately 65 tonnes of calcium carbonate, representing roughly 30 tonnes of sequestered CO_2.
- The project successfully demonstrated the use of the calcium carbonate with sequestered CO_2 in a public works projects in Santa Cruz, as well as multiple commercial building projects in Northern California. http://www.santacruzsentinel.com/general-news/20101203/green-cement-manufacturer-to-test-product-in-santa-cruz, http://sccrtc.org/wp-content/uploads/2010/12/2013-08-29-

aux-faq.pdf, http://www.sccrtc.org/projects/streets-highways/highway-1-aux-lanes/

- The project successfully developed and scaled to a pilot demonstration plant a new process that uses waste oxides such as cement kiln dust, high calcium flyash and carbide lime (byproduct from acetylene manufacturing from coal) from industrial processes to cost effectively capture CO_2 from flue gas and convert it to cementitious calcium carbonate that sequesters CO_2 and has beneficial use. Calera has subsequently scaled the carbide lime processing technology to the same scale as the pilot demonstration plant and has successfully tested in the pilot demonstration unit.
- Construction of Calera's cement fiber board pilot plant was completed as part of the ARRA program. This plant has the ability to continuously produce commercial scale boards (4 ft x 8 ft) from Calera's novel calcium carbonate binder system as the only binder material. To date, over 300 boards (9,600 sq. ft.) have been produced which have met ASTM standards. Calera intends to place these boards into demonstration projects throughout 2015 to gain market acceptance. To learn more on Calera's AIRock™ boards, check out www.airock.com.
- In addition the program has demonstrated the use of the calcium carbonate material with sequestered CO_2 in a wide array of applications such as fillers for paper and plastics, architectural applications, and building materials.
- Through utilizing Calera's chemicals technology as opposed to the traditional chlor-alkali and direct chlorination technologies, the United States could avoid 5MM tonnes per year of CO_2 emitted into the atmosphere through Calera's lower energy and CO_2 route.

The Calera beneficial use technology is now fully ready for large-scale demonstration in preparation for commercial deployment.

2. DESCRIPTION OF INTEGRATED CO_2 TO MATERIAL PRODUCTS DEMONSTRATION PLANT

2.1 Overview of Subsystems in the Integrated Plant

In Phase 2a, the CCMP plant was been designed, in Phase 2b, the CCMP plant was constructed and in Phase 2c the CCMP plant was commissioned and tested. The high level subsystems that are included into the CCMP plant are shown in the Figure 2-1. The CCMP plant has been constructed to allow testing on various feedstock inputs and to generate either dry reactive carbonate cement or full commercial size fiber cement boards. The manufactured product generated is full, commercial size, fiber cement boards that are generated with no additional cement other than the reactive carbonate cements produced in the CCMP pilot demonstration plant. In addition, the CCMP plant was built to absorb and use of CO_2 from flue gas generated at the adjacent Dynegy Power Station in Moss Landing, California or from simulated coal flue gas using a boiler simulator facility.

The subsystems that have been constructed and tested as part of this program include the following:

Mineralization Subsystem. The first subsystem is the Mineralization via Aqueous Precipitation (MAP). In this subsystem the alkalinity is used to capture CO_2 from the flue gas and then the aqueous carbonated material is contacted with a high calcium brine to produce reactive calcium carbonate (vaterite) slurry. The slurry is then dewatered and the wet solid is dried to yield dry reactive carbonate powder. This reactive vaterite carbonate is a self-cementing material when mixed with water and certain additives where it recrystallizes to a calcium carbonate (aragonite) cement. The components included in the mineralization subsystem are an absorber, precipitator reactor, lamella clarifier, filter press and a circulating bed dryer. Several upgrades of this system where carried out in the program to allow continuous operation and increased output. This subsystem has been extensively tested in the program including numerous production test runs, which has demonstrated the ability to continuously produce carbonate cements in large volumes. More details on this subsystem are provided in section 2.2 and performance-testing results are provided in section 3.1.

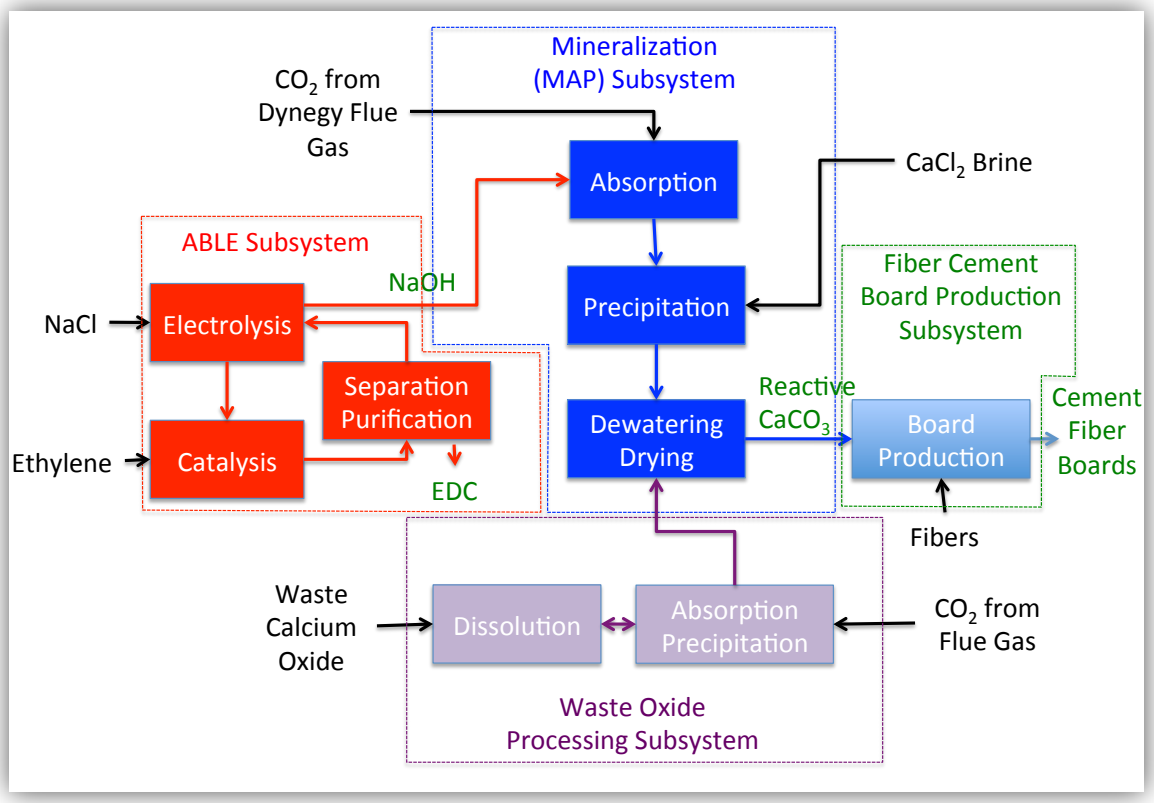


Figure 2-1 The subsystems in the CO_2 Conversion to Material Products (CCMP) pilot demonstration plant

ABLE Subsystem. The alkalinity needed to capture CO_2 can be produced using electrochemistry in a process called Alkalinity Based on Low Energy (ABLE). This subsystem produces alkalinity in the form of sodium hydroxide (NaOH) using electrochemical splitting of salt (NaCl). The ABLE process also produces a useful chemical byproduct, ethylene dichloride (EDC) through the use of a chlorine shuttle reaction using copper chloride to transfer chlorine atoms where they are subsequently reacted with ethylene in a catalysis reactor. Ethylene dichloride is a high volume commodity chemical used in the production of PVC plastics. The depleted copper chloride is recycled back to the electrochemical cell where it is regenerated. The components built in the construction phase include all of those necessary for continuous and integrated pilot demonstration plant operation: brine production and treatment, electrochemical cell, catalysis reactor, distillation and separation components for EDC, and caustic flow and storage. These components have been successfully commissioned and full operational testing has been completed in this program. More details on this subsystem are provided in section 2.4 and commissioning and performance test results are provided in section 3-3.

Waste Oxide Processing Subsystem. In addition to the use of manufactured alkalinity using ABLE, the CMPP pilot demonstration plant includes the option to use waste calcium oxide sources such as carbide lime as the source of both alkalinity and calcium. In this subsystem, the raw waste is dissolved using ammonia chloride and impurities are filtered out. The dissolved

calcium oxide is then used to absorb CO_2 and form the reactive calcium carbonate vaterite slurry in an absorber reactor. The slurry is then dewatered and dried with the same equipment as in the MAP subsystem. The components included in this subsystem include the waste lime storage tank, dissolution reactor, impurity filter, absorber reactor and ammonia chloride storage and recycle tanks. Several upgrades to this subsystem were carried out in the program including the addition of the impurity filtration component and integration with the MAP subsystem for continuous operation. Several commissioning, optimization, performance assessment tests and production runs were carried out in the program. More details on this subsystem are provided in section 2-3 and the performance test results are provided in section 3-2.

Fiber Cement Board Production Subsystem. The main product generated in this demonstration program is fiber cement board. This subsystem is a complete fiber cement board production line that uses the carbonate cement along with cellulosic fibers as input and produces commercial size 4-foot by 8-foot boards. The components included in this subsystem include mixing tanks, cellulose preparation, feeding and dosing systems, sheeting machine, sheet cutoffs, pre-cure oven, autoclave and drying oven. The production line is fully operational and has been used in the program to generate hundreds of boards used in use demonstrations. More details on this subsystem are provided in section 2-5 and the results of the production runs and fiber boards acceptance tests are provided in section 4.

A schematic and layout of the entire pilot demonstration unit is provided in Figure 2-2. This drawing shows the location of the 36-inch diameter flue gas pipe that is connected to the Dynegy Power Plant stack that is across the road from the Calera CCMP pilot demonstration plant. Whenever flue gas containing CO_2 is needed for operation of the CCMP plant, Calera calls the Dynegy control room, who then opens their side of a double block and bleed valve located on one of two stacks (depending on which unit is operating). Calera then opens its valve downstream of the Dynegy valve to allow flue gas to enter the flue gas pipe and flow via an ID fan into the CCMP plant. The flue gas can be directed to either the mineralization subsystem absorber or to the waste oxide absorber reactor depending upon the input mode of operation. Also shown in the figure is the boiler simulator furnace where different fuels including pulverized coals, can be burned to generate flue gas for testing.

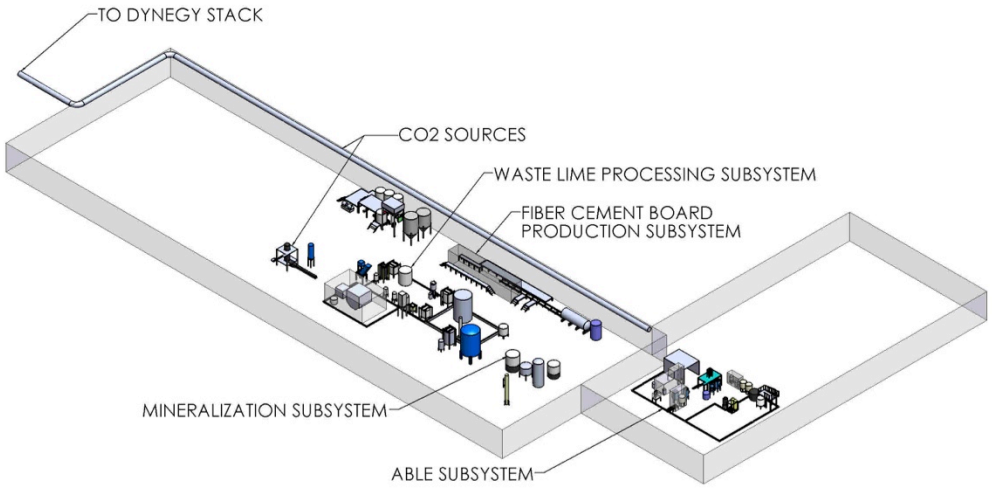


Figure 2-2 Schematic layout of the as-built integrated CCMP pilot demonstration plant

2.2 Mineralization via Aqueous Precipitation (MAP) Subsystem

In the MAP process, CO_2 is reacted with calcium and alkalinity to produce a calcium carbonate product that contains 44 wt% of CO_2 that would have been otherwise emitted to the atmosphere. Using concentrated brines containing calcium and alkalinity, precipitation of solid calcium carbonate is carried out, producing vaterite, a metastable form of calcium carbonate. The process involves carbonation of NaOH in a packed tower absorber using flue gas from the Dynegy power plant. Concentrated naturally occurring $CaCl_2$ solution is diluted with municipal water and doped with additives. During production, the calcium and carbonate brines are added to a stirred tank reactor. Through level control of the tank, the mean residence time is maintained to control particle size. Slurry is continuously pumped from the tank into a lamella-settling unit for solids concentration. The concentrated calcium carbonate slurry is fed into a filter press and subsequently dried via hot gas fluidization. A process flow diagram can be seen in Figure 2-3 and the physical layout is shown in Figure 2-4. Mid-run analysis consists of: filtrate analysis via titration and particle morphology observation via Scanning Electron Microscopy (SEM). Post run analysis includes X-ray Diffraction (XRD) determination of polymorphs, particle size analysis via light scattering, vaterite to aragonite conversion rate, and compressive strength of 2”x2” cured paste cubes.

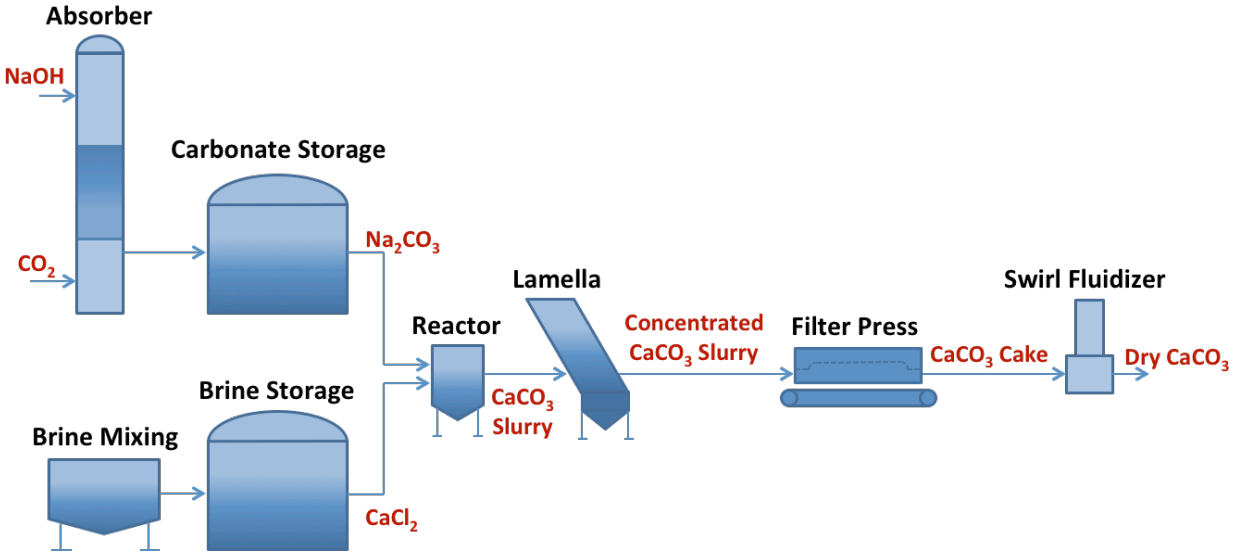


Figure 2-3 MAP process flow diagram

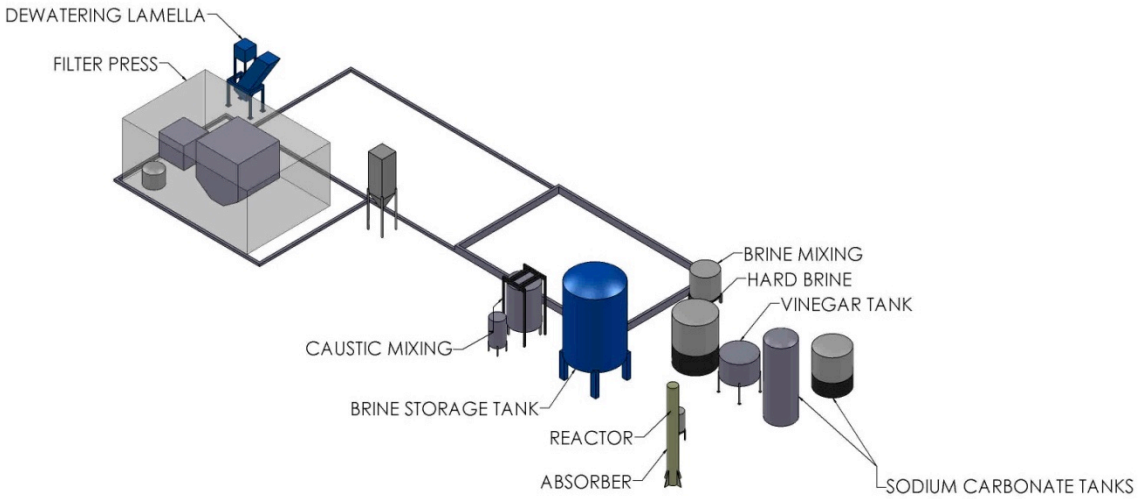


Figure 2-4 Physical layout of the MAP subsystem in the CCMP pilot demonstration plant

In the course of the program, the MAP pilot plant has produced approximately 65 tonnes of calcium carbonate (vaterite) cement used both as partial cement substitute (replaced portion of portland cement in traditional concrete mixes) and as the self-cementing material used in concrete applications and for fiber cement boards as well as for PCC applications.

2.3 Waste Oxide Processing Subsystem

As an alternative source of alkalinity and calcium, a subsystem was developed that can process waste calcium oxide/hydroxide sources such as carbide lime residue and steel slags. The subsystem allows the waste calcium oxide sources to be used to capture and convert CO_2 to the vaterite form of calcium carbonate. The physical layout of the subsystem is shown in Figure 2-5

with pictures of the units shown in Figure 2-6. In this subsystem, waste calcium oxide sources are dissolved with ammonium chloride (NH_4Cl) and insoluble impurities are separated and removed. The purified dissolved calcium hydroxide slurry is then sent to an absorber reactor where CO_2 flue gas is introduced under specific controlled conditions that allow the generation of reactive calcium carbonate. The reactive calcium carbonate slurry is then moved to the dewatering and drying equipment shared with the MAP plant. The liquid reagent containing ammonium chloride is recycled back to the dissolution tank for reuse.

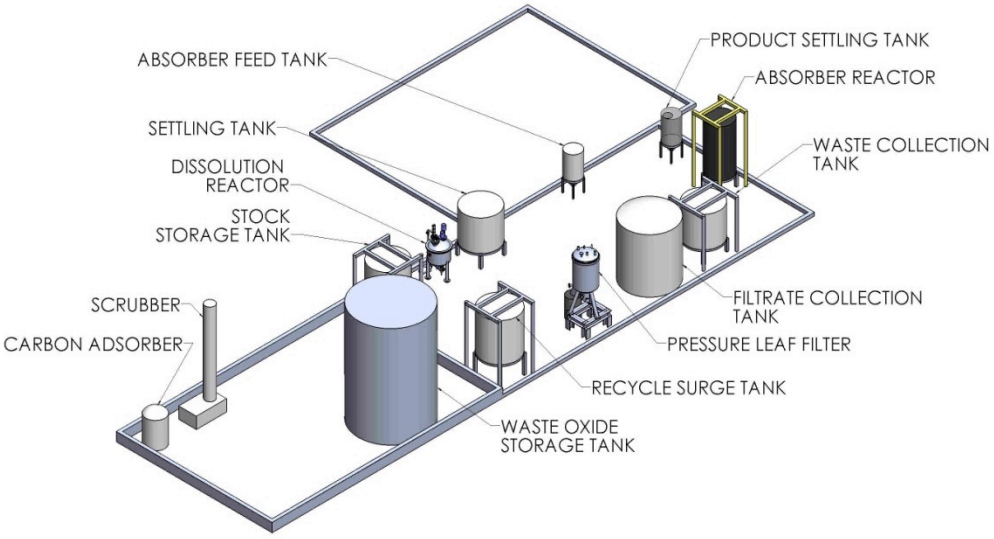


Figure 2-5 Physical layout of waste oxide processing subsystem

Figure 2-6 Picture of the waste oxide processing subsystem in the Moss Landing pilot demonstration plant

A simplified block flow diagram of the waste oxide processing subsystem is provided in Figure 2-7 for the example of carbide lime residue. The subsystems is made up of the following major components:

Waste Oxide Storage Tank. Waste oxide/hydroxides can be received in the plant either as a dried powder or water slurry. Since waste hydroxides such as carbide lime residue are produced as slurry that is the general mode of acceptance. The CCMP pilot demonstration has a large (7,500 gal, 33,000 L) stirred tank as the receiving and storage tank for slurries. Shipments are brought into the plant in tanker trucks and directly transferred to the storage tank.

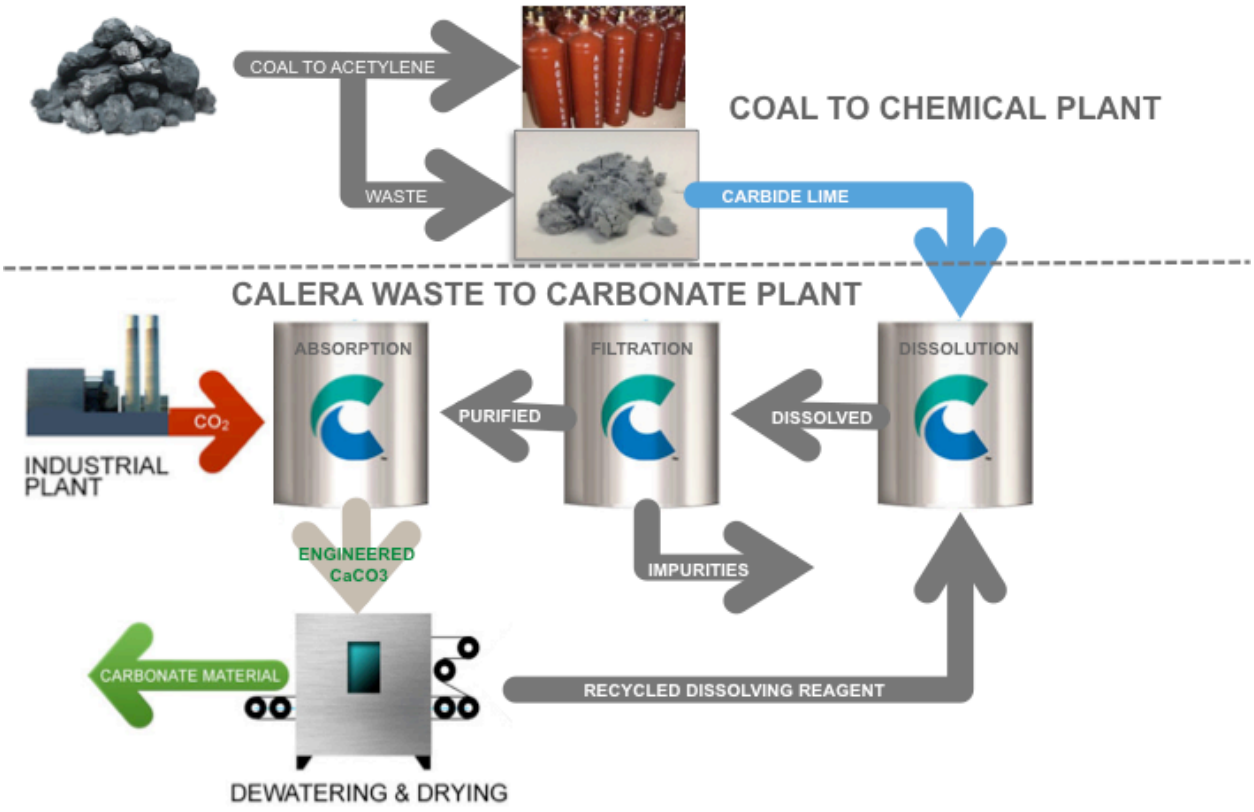


Figure 2-7 Simplified Block Flow Diagram (BFD) of the waste oxide processing subsystem in the Moss Landing pilot demonstration plant

Dissolution Reactor. The slurry is transferred at a prescribed rate into a dissolution reactor where it is mixed with an ammonium chloride solution. The ammonium chloride dissolves the calcium hydroxide while leaving impurities such as silica, carbon, calcite and other insoluble materials as solids. The dissolution reactor is a well mixed, gas sealed 90 gal (340 L) cone bottom tank.

Filtration. The resulting mixture is then transferred to a settling tank and the top portion is transferred to a 250 Gal/100 psig centrifugal discharge filter shown in Figure 2-8. The centrifugal discharge filter type consists mainly of a pressure vessel with a hollow center shaft around which series of round filter elements are vertically stacked at specific, but variable spacing. The filter stack, consisting of both the hollow shaft and the elements, is installed in the vessel, so that it can freely rotate. To clean the filter, the whole stack is spun by means of a drive system. The hollow shaft, which serves as a filtrate discharge manifold, is connected to an external drive motor permitting the removal of cake by centrifugal action. The centrifugal discharge filter removes the insoluble impurities from the dissolved waste oxide. The clear solution is then directed to a feed tank for further use.

Figure 2-8 Centrifugal discharge filter used to remove impurities from dissolved waste oxide

Absorption Reactor. The absorber reactor, shown in Figure 2-9, is a well-mixed reactor with the flue gas introduced through a sparging gas diffusing system at the bottom of the reactor. In the absorber reactor, the purified dissolved waste oxide is mixed under tightly controlled conditions with CO_2 containing flue gas. Under these conditions, reactive calcium carbonate will form as a precipitate. The resulting product slurry is then directed to the same dewatering and drying components as described above in the MAP subsystem. Once the solid product is removed from the slurry, the resulting water still containing the ammonium chloride reagent is recycled back to the dissolution reactor and reused in the process.

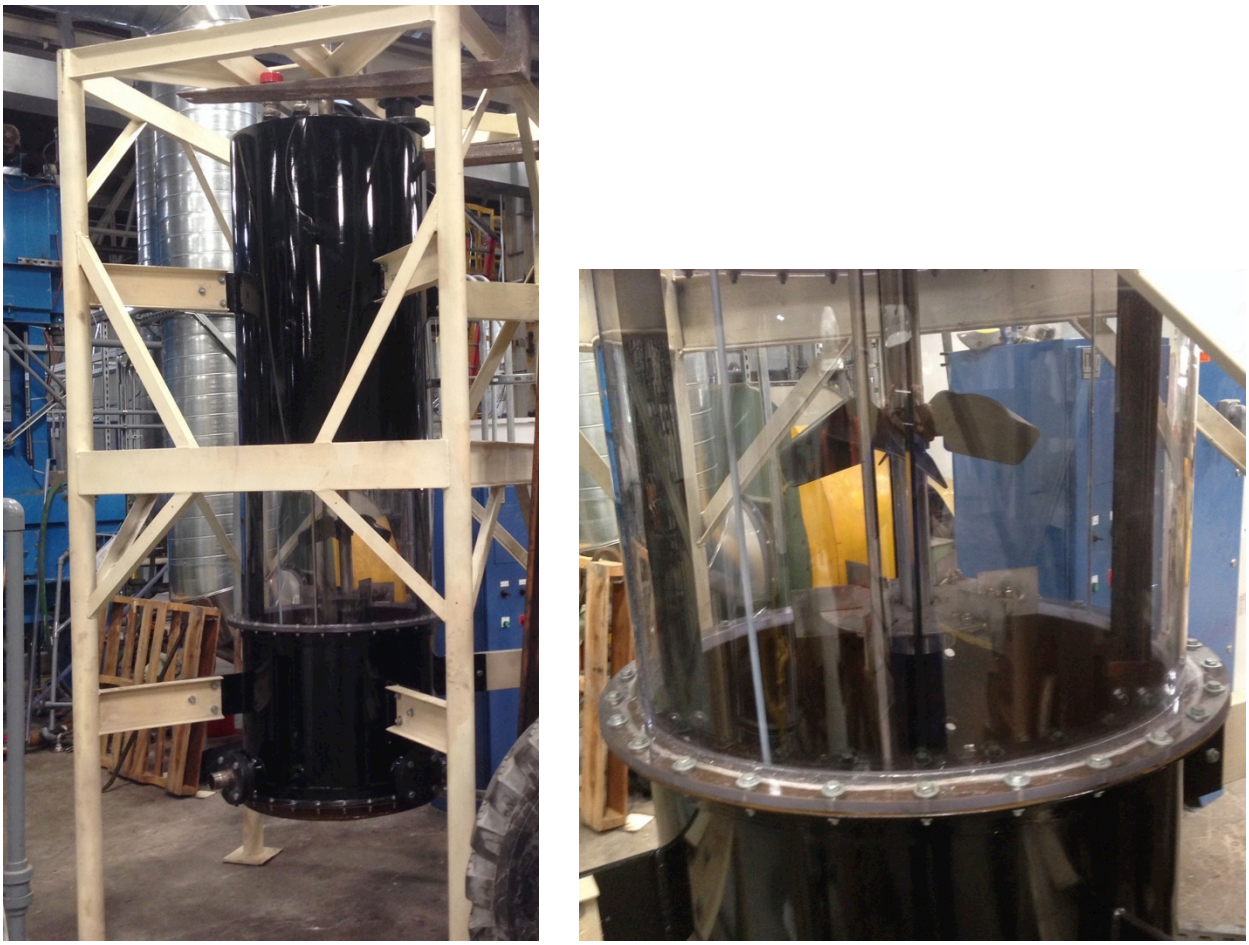


Figure 2-9 Absorber reactor (left); detail of clear reactor section (right)

Subsystem Integration and Control. The waste oxide plant is a fully integrated and instrumented continuous subsystem. The components, feed rates, and condition set points are automatically monitored and controlled using Programmable Logic Controllers (PLC) tied to Human Machine Interface (HMI) using touch screen displays. All data from test runs are stored electronically using PI System operational historian software.

Air Pollution Control. The subsystem is equipped with an air pollution control device to ensure low air emissions of any residual sulfur or particulate matter from contaminates in the waste oxide and any ammonia stripped from the liquids in tanks. The Air Pollution control equipment includes both an acid (H_2SO_4) scrubber and a carbon bed.

2.4 ABLE E-CHEM & CAT REACTOR SUBSYSTEM

2.4.1 ABLE Pilot Plant As-Built Design

The physical layout of the ABLE plant is shown in Figure 2-10. The electrochemistry subunit consists of three electrolyte feed systems located adjacent to the mezzanine which houses the E-Chem cell rack. The E-Chem cell is assembled in a cell assembly room also adjacent to the

mezzanine. The catalysis subunit is contained inside a Plexiglas splash room. The copper mixing station is positioned between the subunits and serves as a hold-up volume between E-Chem and catalysis. The actual plant floor is shown in Figure 2-11 where the catalysis and adsorption systems are oriented in the front of the picture.

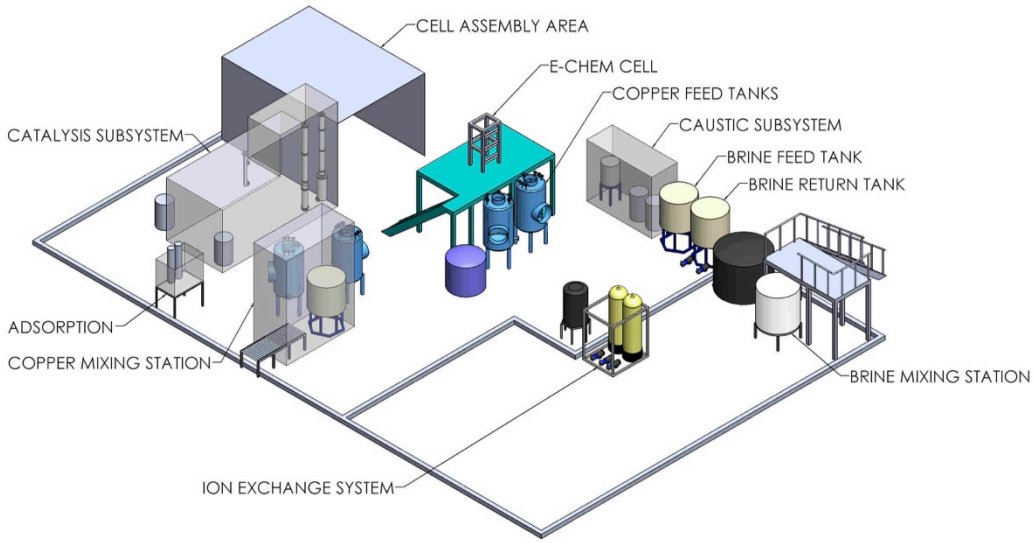


Figure 2-10 Schematic layout of the as-built ABLE pilot demonstration plant

Figure 2-11 Actual layout of the ABLE pilot demonstration plant

2.4.2 ABLE Process Flow Diagram

Figure 2-12 below represents a process flow diagram (PFD) for the ABLE pilot plant. Copper monochloride is oxidized to copper dichloride in the electrochemical cell (U4 in Figure 2-13) with simultaneous production of 30 wt% sodium hydroxide and hydrogen gas. Copper dichloride is then reduced back to copper monochloride in the catalysis reactor (U8) by reaction with dissolved ethylene to produce ethylene dichloride (EDC). This process requires the copper chloride stream to be shuttled between the E-chem and catalysis subsystems during integrated operation (stream A25). The majority of the EDC product is separated from the copper chloride stream in a distillation system downstream of the reactor (U9). Water and EDC are then separated via liquid-liquid separation (U10). If needed, small concentrations of residual organics from the catalysis subunit are removed in the adsorption unit (U11) before transfer to the E-chem subunit.

The single cell and catalysis reactor subsystems were operated independently in a batch stand-alone mode throughout initial commissioning and performance optimization. Once the commissioning phase was completed the plant moved forward to the testing phase with the goal of conducting long term integrated test campaigns. The as-built design of each of these subunits is described in the sections below.

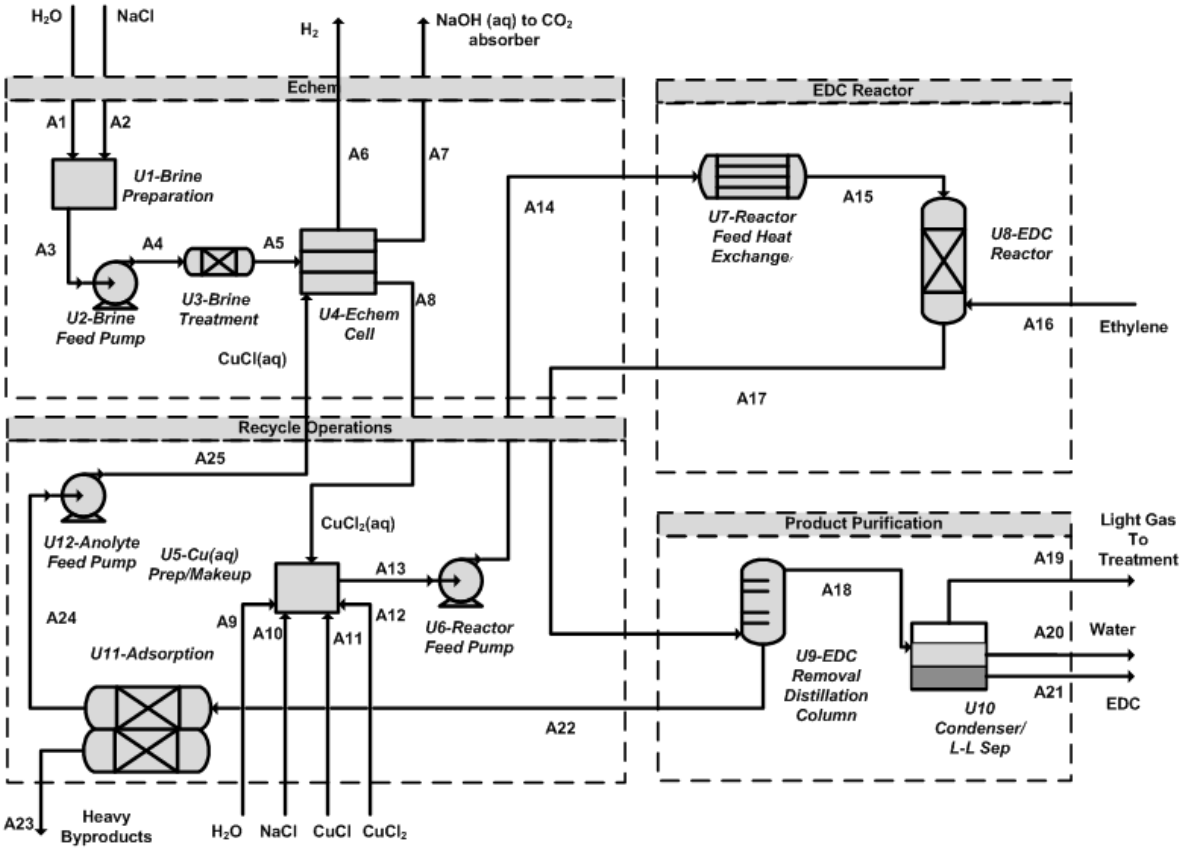


Figure 2-12 ABLE process flow diagram

2.4.2 Electrochemistry Subsystem Design

The electrochemistry subsystem consists of three electrolyte feed systems: caustic, brine, and copper chloride. All electrolytes are pumped to the cell rack which is elevated above the feed tanks on a mezzanine structure. The cell compartment outlets then gravity drain back to the feed and product tanks allowing the cell to be operated near atmospheric pressure. The electrolyte feed systems were upgraded for 24/7 operation to avoid flow disruptions to the electrochemical cell which would result in system shut-down and unwanted interruption in long term data collection.

Brine: The brine feed system provides purified brine to the cell within Chlor-alkali specifications via an ion exchange unit. Figure 2-13 below shows the ion exchange unit which has been designed to remove membrane-poisoning ions that are inherent in the solid sodium chloride feedstock. The depleted brine effluent is collected separately for return to the ion exchange system where it is re-concentrated and purified. An inline heat exchanger brings the brine from room temperature to process temperature (90°C) en route to the brine compartment. Brine compartment pressure is controlled by an adjustable head leg at the compartment outlet and is important for affecting proper flow as well as membrane transport properties.

All piping sections that are maintained at cell temperature in the brine system were constructed with PFA tubing and fittings to meet material compatibility standards. These streams are heated and cooled in-line before and after the cell. Heat exchangers were built in-house with PFA tubing and/or Hastelloy for the brine system. Initial commissioning and performance optimization was completed with single peristaltic pumps. These were replaced with centrifugal pumps in parallel for all electrolytes as shown in Figure 2-14 below. These systems were designed to automatically start the redundant pump in case of a pump failure. This will decrease downtime by decreasing the frequency of unwanted shut-downs. Flow control valves are installed downstream of the recirculation loop for all electrolyte feed tanks to accomplish a controlled flow rate to the cell in addition to continuous tank recirculation.

Figure 2-13 Brine preparation tanks and ion exchange unit

Figure 2-14 Centrifugal pumps installed in parallel for all electrolytes

Caustic: The caustic system is designed to provide and collect 33 wt% NaOH to the cathode system from the surge tank where level increases as caustic is produced. The caustic surge tank and product totes are shown inside the splash room in Figure 2-15 below. Concentrated caustic is then diluted en route to the cell with DI water feed via a peristaltic pump. The dilution flow rate is controlled by the inlet density (used it with the temperature to calculate caustic concentration) and it is measured by the inlet Coriolis flow meter. The inlet and outlet Coriolis flow meters provide a continuous measure of Cation Exchange Membrane (CEM) efficiency which is confirmed by sample titrations at cell inlet and outlet. Similar to the brine system, feed is heated from room temperature to process temperatures via an inline heat exchanger. Hydrogen is separated from the product before return to product totes in a liquid/gas separator.

All piping sections that are maintained at cell temperature in the caustic system were constructed with PFA tubing and fittings to meet material compatibility standards. The inlet stream is heated inline with a Inconel 600 heat exchanger.

Figure 2-15 Caustic feed tank and product drums

Copper: The copper feed system is comprised of two tanks shown in Figure 2-16 below—a copper E-chem recirculation tank and a E-chem copper(I) make-up tank which are designed to maintain well-mixed copper solutions at 90°C. The copper(I) make-up tank receives copper(I) rich solution from the catalysis subsystem. This copper(I) rich solution is pumped into the copper E-chem recirculation tank at a flow rate that matches the Catalysis reactor feed rate. The E-chem Recirculation tank is maintained at the anode inlet composition in steady state operation. The copper(II) rich solution from the outlet of the electrochemical cell goes back to the catalysis unit and the E-chem recirculation tank. A control valve determines the ratio between the flow going back to catalysis unit and E-chem in order to keep the E-chem recirculation tank at constant level.

Integrating the E-Chem and catalysis plants requires pumping the copper chloride stream with some residual organics between the E-Chem and catalysis units. All low pressure copper piping is of butt welded PVDF construction for material compatibility purposes. All copper piping is equipped with self-regulating heat strips and insulation to prevent cold spots and precipitation causing pipe clogs.

The mass balance around the anode compartment is accomplished by flow meters and sample ports at anode inlet and outlet, that provides mass flow rate and composition of the copper stream as well as indicate any integrated process upsets.

Figure 2-16 CuClx feed tanks and circulation loops

2.4.3 Catalysis Plant Design

The catalysis system is comprised of three subunits: the catalyst feed unit, the reactor and the EDC recovery unit. All subunits have been automated by programming all instrumentation through a central PLC. The PLC is used to control the desired process flows, conditions, and safety interlocks by means of safe and remote operation. The control schemes and safety systems for each sub-system are described below.

Catalyst feed unit: The catalyst hold-up system is designed to maintain the copper (II)-rich anode effluent at 90^0C in a sealed environment. Level switches provide a safety interlock to prevent overfilling and overheating. Parallel centrifugal pumps are employed to re-circulate the solution continuously in the feed tank and maintain a well-mixed catalyst solution. Progressive cavity pumps bring the copper up to reactor pressure at a controlled flow rate. The Coriolis flow meter and sample valve provide an accurate measure of mass flow and composition into the reactor for mass balance purposes. The copper stream is subsequently heated to reactor temperature via an inline oil heat exchanger.

Catalyst reactor: The reactor is shown in Figure 2-17. The reactor is maintained under constant ethylene pressure by a pressure control valve on the ethylene inlet stream and a flow control valve on the gas outlet stream which is used to purge inert gases from the system. A flow meter and Quadrapole Mass Spectrometer (QMS) sample port on the purge stream closes the mass balance on the gas side of the reactor. Over-pressurization is prevented with an automated solenoid valve on an emergency vent line from the headspace as well as a rupture disk routed to a separate discharge receiver tank. Constant temperature is maintained with self-regulating heat

strips and insulation to compensate for heat loss from the headspace. Over heating is prevented by redundant high temperature cut-outs on the utility side and process side of the heat exchanger. Liquid level is regulated by a level control valve and differential pressure transmitters placed across the vapor phase of the packed bed and the liquid outlet. The copper(I) rich catalyst and organic product is transferred under pressure from the reactor to the distillation unit through an inline flow meter which provides a liquid mass balance and a means to identify process upsets.

The Catalysis system required materials of construction that would stand up to greater pressures and temperatures while maintaining corrosion resistance. The design point for this equipment was 175°C at 350 psig.

Figure 2-17 Picture of top of catalysis reactor

EDC Recovery: The separations system drops the pressure to flash organic products and some water into the vapor phase. The flash is conducted by regulating pressure to the desired set point using pressure control valves on the vapor outlet stream. The distillation unit pictured in Figure 2-18, below, removes EDC remaining in the copper stream after the flash. Heat duty to the distillation unit's reboiler is controlled via hot oil flow rate to optimize EDC and H_2O split fraction. The copper residence time in the reboiler can also be adjusted to decrease the amount of chlorinated products leaving the reactor in the copper chloride stream. Water and organics from the distillation vapor phase are collected in the subsequent condenser and separated in a liquid-liquid separator. The water stream is returned to the top of the distillation column to maintain total copper concentration. Flow meters and sample valves on the copper return line downstream of the separations unit provide the liquid split fraction. EDC is directed to a product drum equipped with a scale and sampling valve for mass balance and purity assessment. The catalyst solution with a low concentration of organics can be directed to either the E-Chem Make Up tank or through the adsorption system described below before returning to the E-Chem subsystem.

Organic products in the copper effluent are quantified by offline liquid-liquid extraction followed by GC-MS analysis and provide a measure of reaction rate and selectivity. Flow meters and a QMS enable quantification of the gas vent stream composition which allows a calculation of ethylene consumption (inlet minus outlet mass flow rate) to provide a second calculation of reaction rate. Copper concentrations are quantified by titration and provide a third means of assessing reaction rate.

All vent streams from the reactor and separations units are analyzed via QMS. Downstream of the selector valve, vent streams are sent to absorber barrels consisting of carbon beds to remove organic products and catalyst beds to oxidize remaining ethylene.

Figure 2-18 Distillation column used for separation of product from copper reagent

Adsorption: Figure 2-19 below shows a schematic of the organic adsorption unit obtained from Chemionex Inc. This system was tested but later work with an improved distillation column indicated that it is not necessary for the integrated design. The system is comprised of two adsorption columns that are operated in a lead/lag configuration where the organic contaminated copper solution passes through both columns in series during the load step. Automated valves are programmed to cycle automatically through a series of operating cycles including water void, raffinate, feed void, and strip cycles for each column. The system is started up in the water void

stage to push residual water out of both columns with the organic contaminated copper feed stream. The raffinate stage is then initiated which redirects the clean copper effluent to the E-chem copper enrichment tank. This stage continues until low levels of organics are present in the effluent of the lead column. The feed void stage is then initiated to remove the remaining bed volume of copper from the lead bed. These cycles are continued until all contaminated solution has been processed.

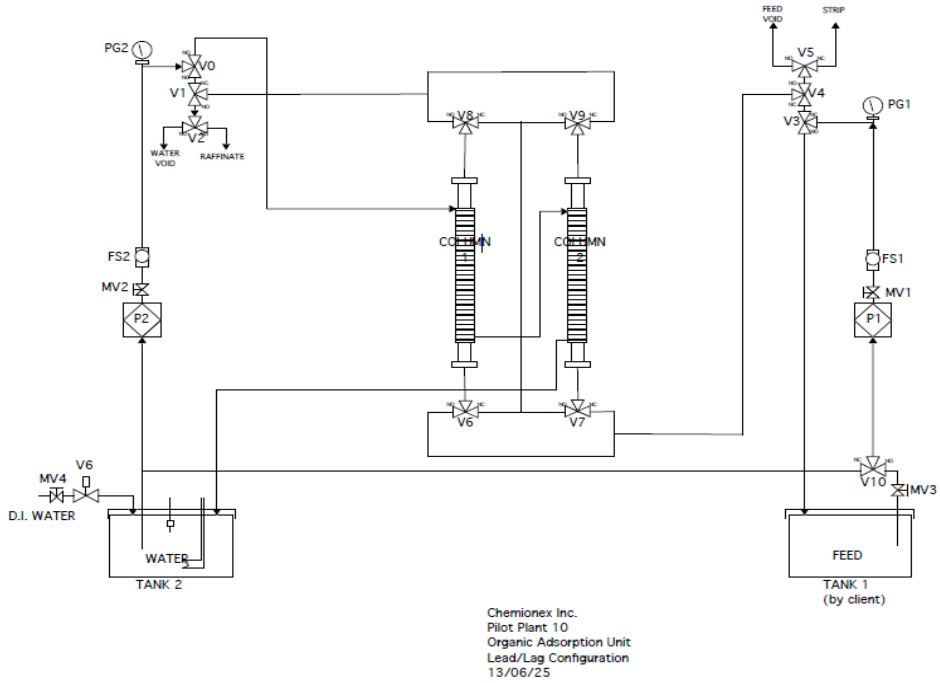


Figure 2-19 Adsorption system schematic

PLC: All of the upgrades implemented to enable 24/7 operation required additional inputs and outputs to the PLC. A more robust 5000 series PLC model was desired to replace the previous 500 series PLC as well as additional HMI screens to facilitate ease of operation. To accomplish these things a new PLC cabinet was installed. Figure 2-20 below shows the new cabinet as installed on the plant floor and completed installation. All instruments and equipment were removed from the previous PLC and re-wired into the new locations. To commission the upgraded plant, each input and output were tested individually to verify correct mapping between the plant for and the PLC. Instrument and equipment programming was re-written to suit the updated software model. Alarms have been programmed to alert operators of deviations in temperature, pressure, flow, etc. on all streams with interlocks to return the system to a safe state.

Figure 2-20 Programmable Logic Controller (PLC) and instrumentation cabinet for the ABLE subsystem

2.5 Carbonate Production Subsystem

As discussed above, the selected application for the carbonate products was commercial size fiber cement boards. The carbonate cement produced from CO_2 in either the Mineralization or the Waste Calcium Oxide Processing subsystem is utilized to form fiber cement sheets and cut into 4ft by 8ft (or alternatively 3ft by 5ft) fiber cement boards. This section discusses the fiber cement sheet production process that was constructed in Phase 2b. The capacity of this unit is matched to the production of carbonate cement production plant of 2 tonnes per day. The plant has the capability to go to higher production rates as well. The section also includes a physical layout and pictures of the pilot demonstration plant at the Calera Moss Landing plant.

The physical layout of Fiber Cement Board Production Subsystem is shown in Figure 2-21. The subsystem is directly adjacent to the carbonate production facilities to allow pneumatic transfer of carbonate cement powder from the Mineralization and Waste Calcium Oxide processing subsystems to a feed silo. The subsystem is organized into a line starting at the feed silo and progressing down the line to cellulose preparation tanks, dosing and mixing tanks through to the sheeting machine and cut off jets then to stacking, curing, autoclaving and drying of the boards. The actual plant floor is shown in Figure 2-22.

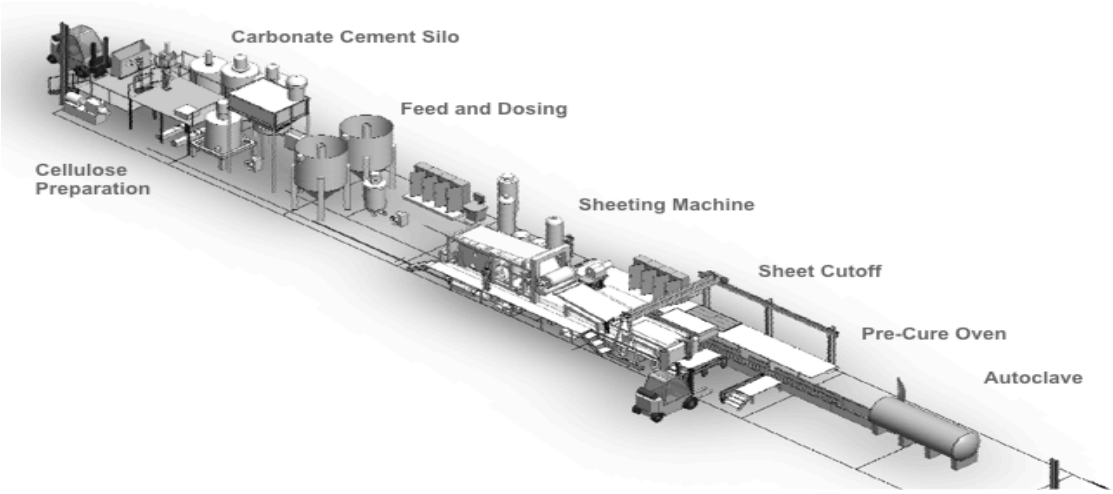


Figure 2-21 Physical layout of Fiber Cement Board Production Subsystem

Figure 2-22 Panoramic view of Fiber Cement Board Production Subsystem

In Figure 2-23 is shown the process flow diagram for Fiber Cement Board Subsystem including the various components that are used to convert cellulose, water and reactive carbonate into finished boards. The following is a description of the as-built machinery and equipment included in this subsystem.

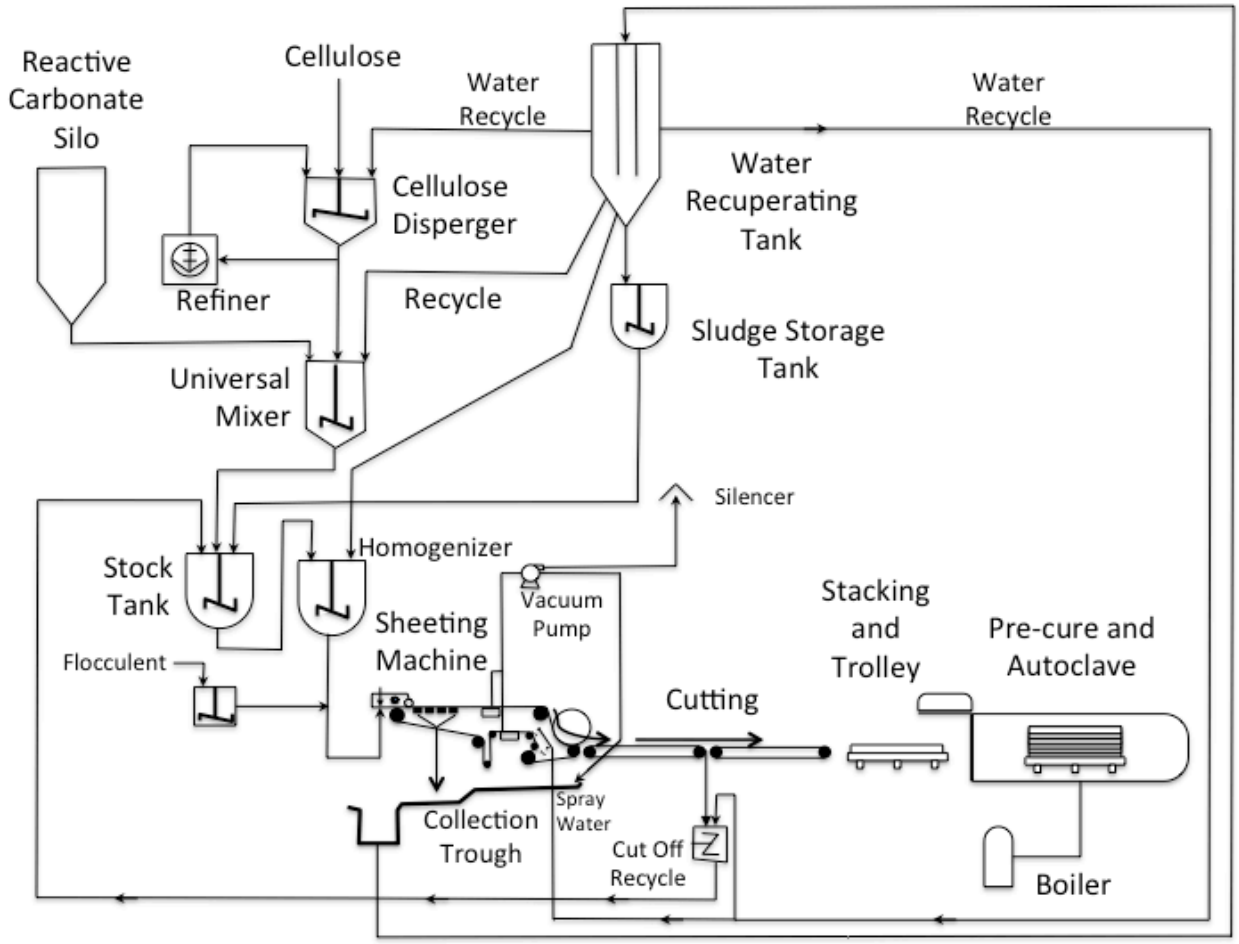


Figure 2-23 Process Flow Diagram for Fiber Cement Board Production Subsystem

2.5.1 Material Preparation

Cellulose Refiner

Cellulose fibers, in bleached and unbleached form, act as reinforcement for the fiber cement sheet. Calera fiber cement boards use cellulose as the only fiber. The cellulose arrives in bales and is fed manually to a cellulose disperger. In Figure 2-24 is shown the cellulose disperger that is equipped with an agitator and is used to initially mix the cellulose with water. Then the slurry is directed to a refiner is shown in Figure 2-25 to open up the fibers. After slushing with water the fibers are well refined (fibrillation) and then stored in a 5000-liter cellulose slurry tank as shown in Figure 2-26.

Figure 2-24 Cellulose Disperger used in the Fiber Cement Board Production Subsystem

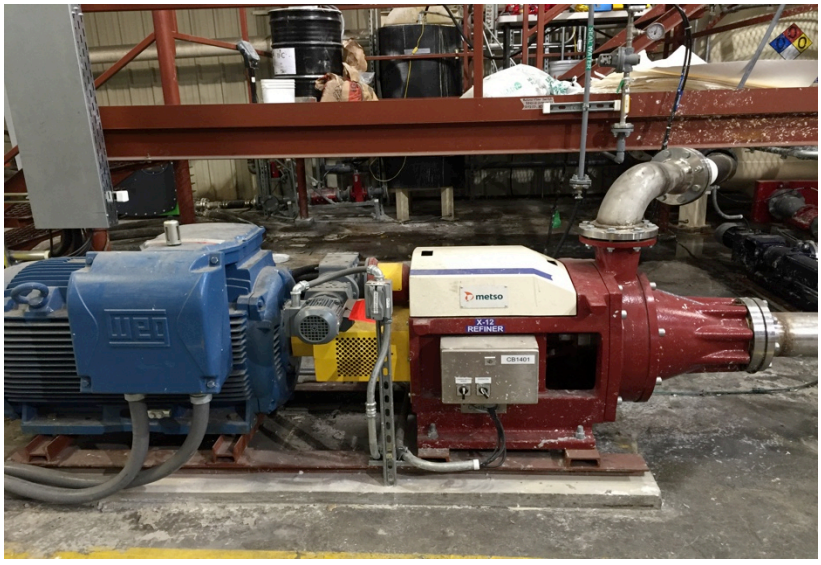


Figure 2-25 Cellulose Refiner used in the Fiber Cement Board Production Subsystem

Figure 2-26 Cellulose Slurry Tank used in the Fiber Cement Board Production Subsystem

Carbonate Silo

Calera carbonate cement produced in the Mineralization or Waste Oxide Processing Subsystems is the only cement used in the board manufacturing. The carbonate cement is pneumatically transferred into separate silos from the Mineralization or Waste Oxide Processing Subsystems for subsequent storage and use is shown in Figure 2-27. The capacity of the silo is 1850 Gal (8,000 L) or roughly 6.5 tonnes of carbonate cement.

Figure 2-27 Carbonate silo used in the Fiber Cement Board Production Subsystem

Universal Mixer and Homogenizer

In the universal mixer shown in Figure 2-28, the reactive carbonate cement, refined cellulose slurry and recycled water are mixed using an agitated tank. The carbonate cement is fed using a horizontal screw conveyor. The refined cellulose slurry is fed to the mixer using a progressive cavity slurry pump. Any additives needed to accelerate or retard setting times for the particular formulation are also added to this mixer. Additives are individually dosed using dosing screws or manually into the batchers. The dosing is down through a fully automatic control system that weighs all materials to high accuracy according to a self-learning system. The fully mixed material is then fed to a 1,360 Gal (6,000 L) stock tank and then to a homogenizer mixer tank for final adjustments of water content and flocculants as needed for the specific mix design. The homogenizer, shown in Figure 2-29, thoroughly mixes fresh slurry from the stock tank, recycled rejects and cut-off material with process water to reach the required slurry density for the sheet production.

Figure 2-28 Universal mixer used to mix carbonate cement, refined cellulose slurry, recycled water and additives in preparation for feeding to sheeting machine

Figure 2-29 Homogenizer Tank used for final adjustments of water content, required flocculants, mixed fresh slurry, recycled rejects and cut-off material in preparation for target slurry density for the sheet production

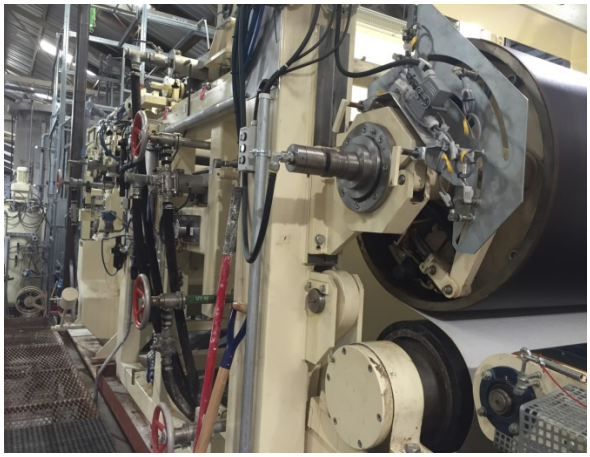
Water Recycle

The total weight of water is important in the mix formula to allow formation of sheets and to provide proper water for setting and final weight of boards. Based on the required density, the computer calculates the water already contained in the slurries and adjusts the water batcher to the required total water content. As far as possible, process water is recycled in the process. A collection trough under the sheeting machines is used to collect slurries from over runs and spills, water spray used to clean the felt and vacuumed cleaning material. These slurries are pumped to two water recuperation tanks for settling. The concentrated slurries from the recuperating tanks are then reintroduced into the feed systems either at the universal mixer or the final homogenizer. The recycled separated water from the recuperating tanks is used to reslurry cutoffs solids, sprays to clean the felt belts in the sheeting machine and water for the cellulose disperger.

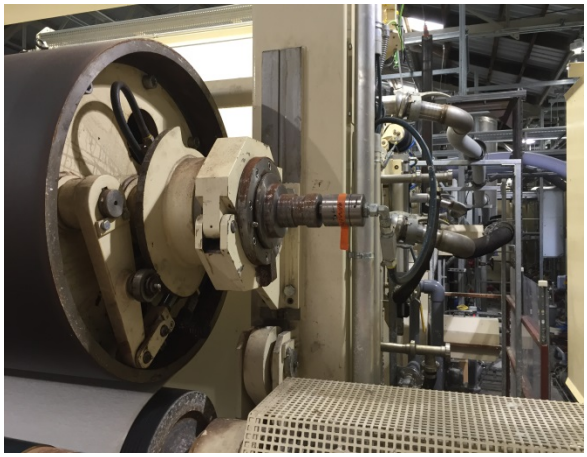
2.5.2 Sheet Production

The sheeting machine is the main equipment used for making the fiber cement boards. Figure 2-30 shows a set of pictures of the Calera sheeting machine. The heart of the sheet production line is the Hatschek machine, which consists of a vat in which a cylindrical sieve rotates in contact with dilute water based slurry of fibers and Calera reactive carbonate material as the cementitious agent to form a filtering film. The sieve cylinder is mounted on an axle and driven by a continuous felt wrapped around the top of the sieve by a couch roller. The felt is threaded around a drive roller and a tail roller. The drive roller is pushed into hard contact with an accumulating forming roller. The top pictures in Figure 2-30 show the accumulating forming roller from the front and back of the machine. The bottom pictures in Figure 2-30 show the felt rollers and vat.

Sheeting Machine (front)



Sheeting Machine (back)



Sheeting Machine (front)

Sheeting Machine (back)

Figure 2-30 Fiber cement board sheeting machine used in the CCMP pilot demonstration plant

Sheets are formed on the Hatschek machine as follows.

Adhering the fiber-carbonate slurry to the continuous felt: As the clean sieve is pulled under the slurry in the vat, water from the slurry runs through the sieve depositing a soft porous film of fibers and carbonate on the surface of the sieve. The sieve cylinder on the Calera sheeting machine is shown in Figure 2-31 fitted into the vat. Special frequency controlled pumps feed the individual sieve cylinder tubs of the sheeting machines. The rotating sieve cylinders collect a thin layer of solid materials while most of the excess water passes through the wire mesh of the sieve. The thin layer emerges from the slurry and is further dewatered and compressed by the couch rollers as it is transferred to the felt.

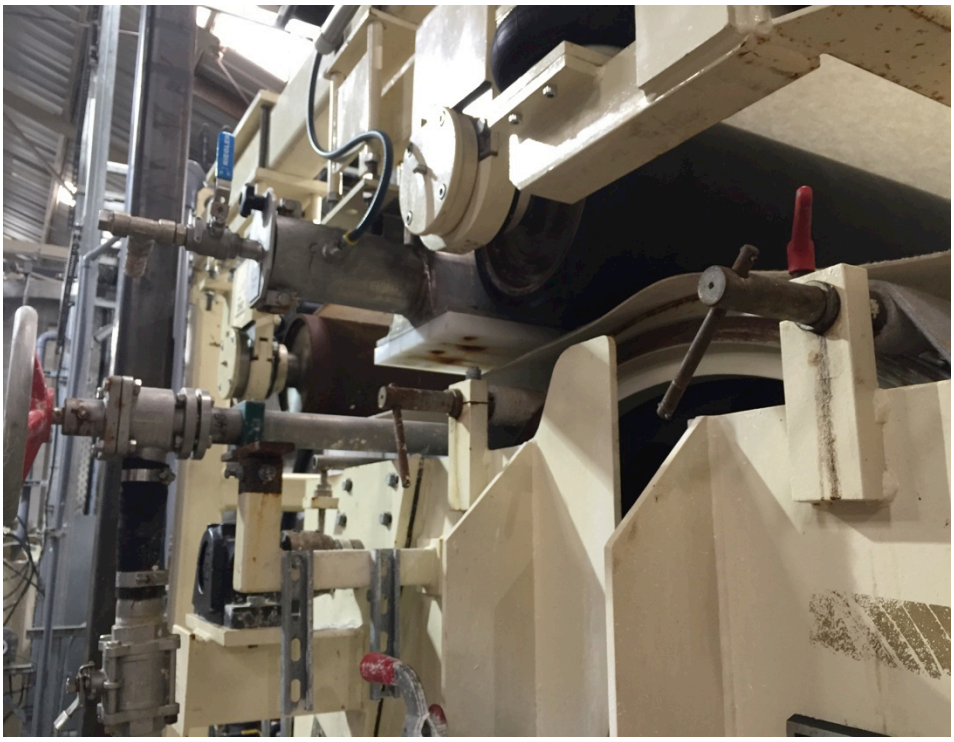


Figure 2-31 Sieve cylinder fitted into the carbonate cellulose fiber vat of the Calera sheeting machine

The sieve carrying the film exiting the vat is brought into contact with the felt stretched tightly across the sieve. This removes much of the water from the film by forcing it back through the film. The solid film floats on this layer of water and is transferred to the felt partly in response to the effect of removal of water and partly because the felt has a greater affinity for the film than the sieve. The thin layers of all sieve cylinders are collected on the felt which runs with speed of 50 – 75 m/min over dewatering vacuum boxes to the accumulating forming roller.

Forming the Sheet: The film is carried on the felt to a accumulating forming roll is shown in Figure 2-32 to which it is transferred by further removal of water at high pressure. The circumference of the accumulating forming roller determines the sheet length i.e., 8 foot in the Calera sheeting machine. A sufficient number of films are wrapped on the forming roll to form a sheet of the desired thickness, the stack of films is then removed from the roller and laid out flat to form the sheet. The action of dewatering successive films in contact with each other under pressure is sufficient to bind the films together to form a contiguous solid sheet. The forming roller accumulates layer after layer until the programmed number revolutions and the required sheet thickness is reached, and the automatic cut-off mechanism cuts through the layers. The sheet thickness is measured by high precision laser control. The sheet drops onto the sheet receiver conveyor.

Sheet Thickness Control: Advanced automatic sheet thickness control system ensures minimum thickness variations. This saves material and reduces rejects that in turn will increase plant output and economy of manufacturing. The measured sheet thickness in relation to the pre-set

number of forming roller revolutions is used to control the slurry density in the homogenizer. Intelligent control system automatically adjusts the correct proportion of fresh- and returns slurry as well as water to provide the correct slurry density into the sheeting machine.

Figure 2-32 Accumulating forming roller on the Calera carbonate to fiber cement sheeting machine

2.5.3 Board Completion

The sheets formed in the machine are referred to as green sheet. The board completion involves cutting the green sheet into board lengths. The boards are then moved via conveyors and stacked on top of each other with separators. The stacks are then pre-cured, autoclaved and dried.

Cutting: The raw sheet is cut into various lengths utilizing water jet cutting commonly used for autoclave fiber cement sheets. Cutting of the green sheet can either be the final cut, or a pre-cut, which is followed by dry finishing after curing. A high-pressure (400 bar, 5800 psi) pump is used to pump the water into the cutting jets.

Stacking: The cut green boards are then pulled off of the conveyor using a stacking device shown in Figure 2-33. The lifting devices uses a vacuum box to grab the board and then the unit uses pneumatic drives on the carriage to transfer the board to a autoclave trolley.

Figure 2-33 Green board stacking device on the Calera fiber cement board production plant

Sheets Pre-Curing, Curing And Transport: The stacked green sheets on the stacking cart are then moved by trolley into the autoclave unit is shown in Figure 2-34. The Calera equipment has the ability for automatic transport systems for the autoclave trolleys, pre-curing trolleys and autoclave pallets. After closing the door, the autoclave is set under vacuum. The steam hardening process starts by first feeding steam and maintaining the autoclave temperature at 80 °C for pre-curing. After a defined pre-curing time, the sheets are hard enough for further high pressure curing. Steam is future introduced to the autoclave chamber and gradually increasing the pressure up to 2 bars over pressure equivalent to 130-140 °C.

The whole autoclave cycle is controlled by a computerized autoclave control operating with special valves is shown in Figure 2-35. Pressure and temperature are kept for several hours for full property development. At the end of the autoclave cycle the pressure is gradually reduced to ambient pressure. Condensate accumulates due to temperature differences especially at the beginning of the autoclave cycle. Automatic autoclave drainage system is used to drain the condensate water, which is recycled back in the process as make up water.

After autoclaving the door opens and the autoclave trolleys are one by one pulled out for unloading.

Figure 2-34 Calera carbonate fiber cement sheets stacked on the stacking cart moved by trolley into the pre-curing unit

Figure 2-35 Autoclave system used in the fiber cement board subsystem

Steam Boiler Plant: The steam for the autoclave unit is supplied via a previously used 50 HP vertical packaged steam boiler fired with propane is shown in Figure 2-36.

Figure 2-36 Steam boiler system supplied steam for autoclave unit fired with propane

Destacking And Packing: After the sheets have been cured (autoclaved), sheets are separated and stacked and packed onto wooden pallets is shown in Figure 2-37. The destacker runs with an electro mechanical drive in banana mode provides fastest possible cycle times of less than 10 sec/sheet. Specially designed suction boxes allow sorting of two or more shorter sheets produced out of the longer sheet or efficient sheet separation even if sheets stick after autoclaving.

Figure 2-37 Fiber cement sheets stacked and packed onto wooden pallet after autoclaved

3.0 OPERATIONAL PERFORMANCE TESTING OF THE SUBSYSTEM AND INTEGRATED DEMONSTRATION PLANT

3.1 MAP and Seed Production

Map Production

To date, approximately 30 tonnes of material produced by the MAP subsystem has been consumed by the fiber cement board subsystem. The MAP pilot plant continues to be used as a source of material for commissioning of the fiberboard plant as well as production of fiber cement boards to be used in demonstration projects.

The results of a typical MAP campaign can be seen in Figure 3-1. A production campaign was conducted to test the plants ability to continuously produce material as needed for commissioning of the fiber cement board plant. This particular campaign produced 5 tonnes of calcium carbonate (vaterite) and took place over 11 plant runs.

Table 3-1 Summary of initial self-cement production for fiber cement board commissioning

Batch	Production Date	Total Recorded Yield (lbs)	PSA mean (μm)	7-day Compressive strength (psi)	$[CaCl_2]$ (M)	$[Na_2CO_3]$ (M)	%Aragonite @ 7 days
PNRN603	10/3/13	1078	14	4900	0.371	1.17	59
PNRN604	10/4/13	309	16	3900	0.371	1.17	61
PNRN605	10/7/13	712	17	5000	0.376	1.22	62
PNRN606	10/8/13	465	15	5400	0.376	1.22	66
PNRN607	10/9/13	1142	15	4700	0.361	1.25	56
PNRN608	10/14/13	988	17	3800	0.352	1.30	76
PNRN609	10/15/13	823	18	4300	0.352	1.30	60
PNRN611	10/16/13	1725	13	4500	0.409	1.46	77
PNRN614	10/21/13	2112	15	5900	0.382	1.47	85
PNRN616	10/23/13	438	14	4000	0.383	1.52	58
PNRN617	10/25/13	1228	14	4800	0.378	1.52	64

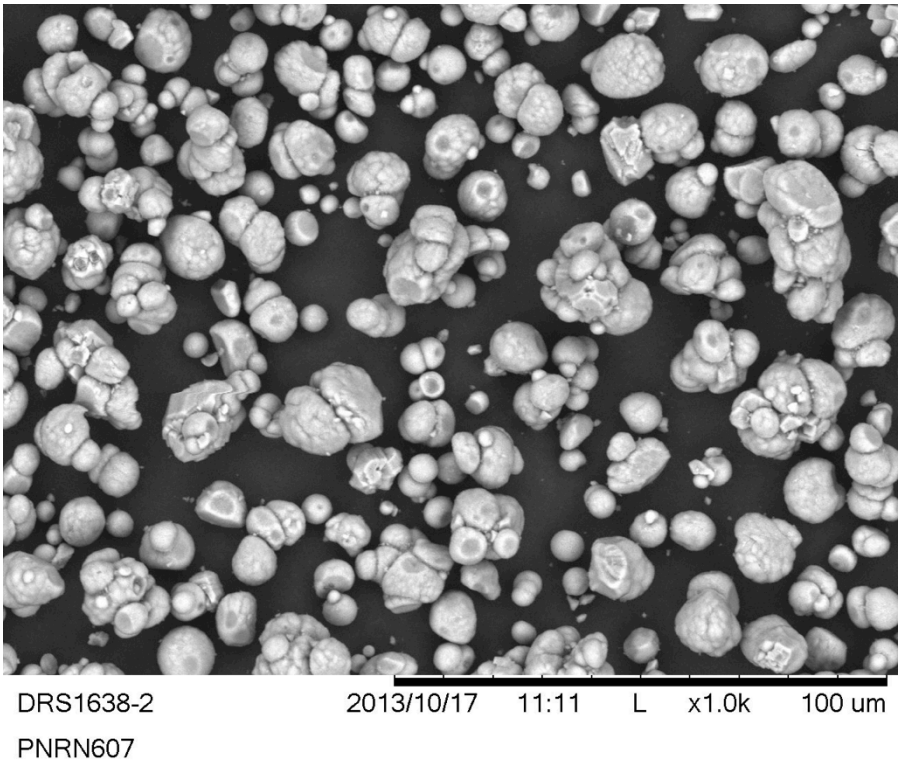


Figure 3-1 SEM image of metastable vaterite produced via the MAP process

Seed Production

Because the presence of chloride salts in pre-cast building materials compromises durability, there exists a driver to replace chloride salt solutions as the principal activator of the vaterite transformation into aragonite. A viable alternative to salt activation is the introduction of sub-micron aragonite particles into the self-cement vaterite matrix.

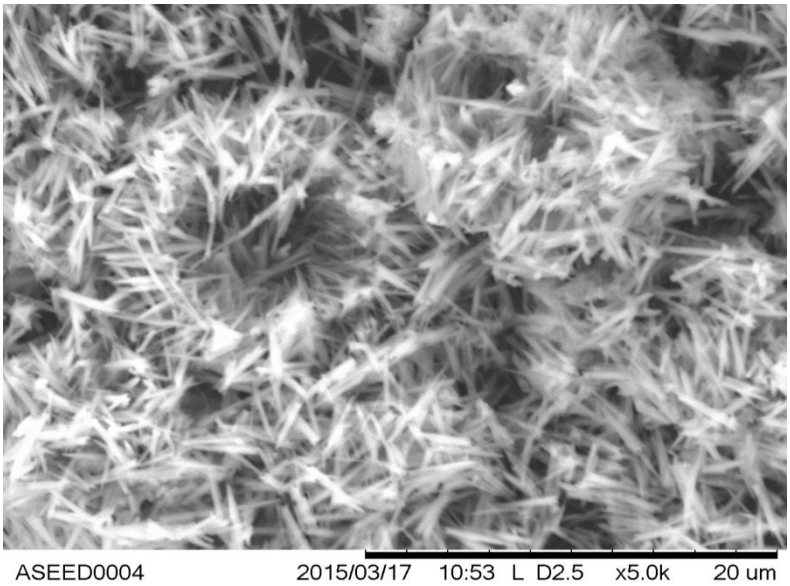


Figure 3-2 High-magnification SEM image of aragonite seed material

In Figure 3-2 is shown the characteristics of the aragonite seeds which have been successfully used in fiber cement board production

3.2 Waste Oxide Processing Subsystem Test

Initial testing of the waste oxide processing subsystem was performed by circulating air and water through all process units. All sections were systematically tested to evaluate and fix leaks, PLC logic and operability, and the function of alarms, emergency interlocks, instruments, pumps, mixers and ventilation. Most of the needed fixes or modifications were simple, however modifications to the insoluble impurity removal subsystem required a more involved approach.

3.2.1 Calcium Oxide Process Commissioning

Insoluble Impurity Removal
Laboratory studies had indicated that it was preferable to remove the insoluble impurities (introduced with the carbide lime) from the process to achieve the desired product. The initial plant design specified a two-stage process, beginning with a continuous sedimentation tank, followed by a drum filter, to remove any residual solids in the supernate. During initial plant runs, it became apparent that this design was not adequate, as insoluble impurities were still able to bypass the separation equipment. The size and geometry of the sedimentation tank resulted in decreasing solids removal effectiveness, over the duration of the runs. The atmospheric operating pressure of the drum filter resulted in filtration rates were so low, that the majority of the flow was bypassed around the filter, including the insoluble impurities. Propagation of these impurities through the system resulted in poor product quality. Alternate methods were researched to improve the separation of the insoluble impurities.

Disk Stack Centrifuge
Results from bench scale centrifuge testing suggested that industrial centrifuges might be a viable option for the separation of insoluble impurities from the process fluid. To evaluate this technology in an industrially representative setting, a pilot-scale disc stack centrifuge was rented from Alfa Laval (Figure 3-3), and integrated into the pilot system. While the test unit did seem to remove solids from the process fluid, eventually the solid accumulation in the unit resulted in clogging, ultimately causing the unit to fail. It was determined that the sticky nature of the insoluble impurities impeded the ability of the sludge to flow out of the unit, making it inherently incompatible with this type of system.

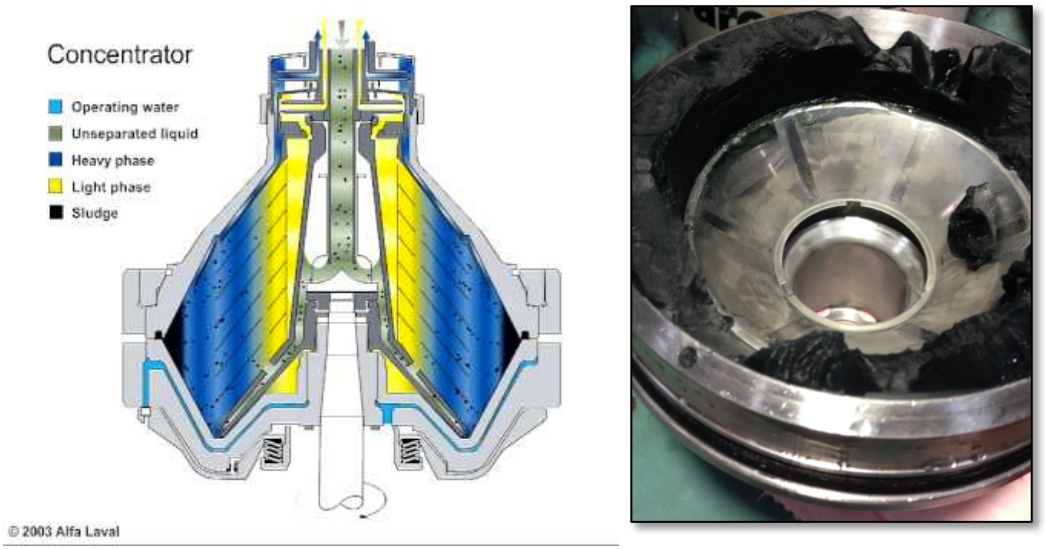
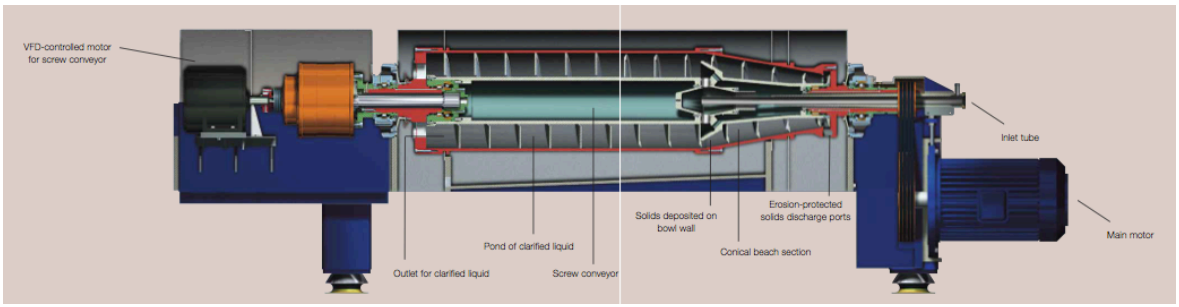


Figure 3-3 Illustration of design and operating principle of a disc stack centrifuge (left). Solids buildup and clogging in test unit (right).

Decanter Centrifuge

Since the centrifugal mechanism was demonstrated to be promising, it was decided to evaluate a centrifuge with a different solids clearing mechanism. A decanter centrifuge (Figure 3-4) was chosen, as the solids are mechanically removed by a screw feeder, which does not require that the concentrated solids be free flowing. During testing, no mechanical failures occurred, however the acceptable solid-liquid separation performance was only achieved when running at ~15% of the unit's maximum rated capacity. The addition of a flocculant to the feed slurry resulted in slightly better performance of ~20% capacity, suggesting that an optimized flocculant program could achieve even higher performance. Optimal flocculant selection and dosing concentrations are currently being investigated.



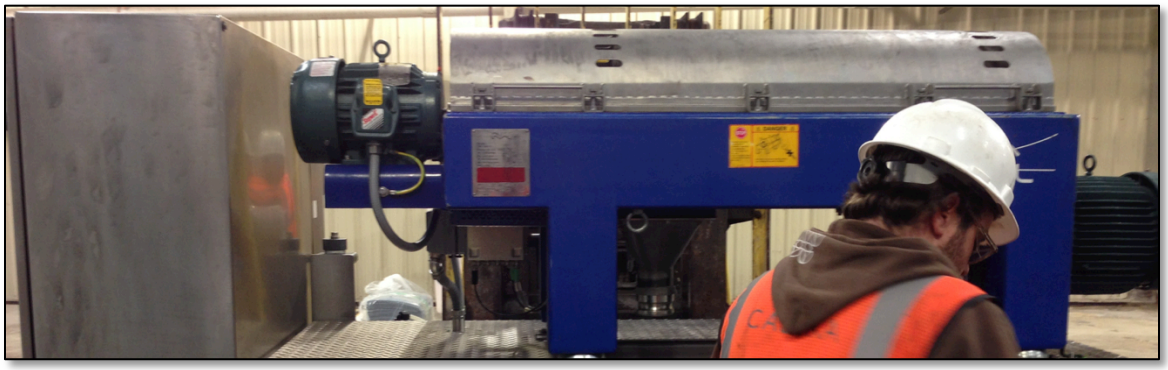


Figure 3-4 Decanter centrifuge illustration (top) and rental unit being installed (bottom)

Leaf Filter

To delineate the overall process development from the insoluble impurity separation development, a filter unit was purchased and installed as a low-risk interim solution. Filtration had been previously evaluated using several lab-scale pressure filter units, including candle filters and leaf filters. The separation was completely effective, however the filtration rates were extremely low, due to primarily to the ultra-fine nature of the impurities. Due to the low filtration rates and linear dependence on filtration surface area, it is not believed that filtration will economically scale at commercial plants, however at the pilot scale the economics are nearly identical to centrifugation.

Flue Gas Source

For the first shakedown runs, propane was chosen as a fuel for flue gas generation. In later runs, several types of coal of varying sulfur content were used, to more closely represent operation at a coal-based power plant. In Figure 3-5 is shown the boiler simulator facility used to burn coal and typical inlet and outlet concentrations of CO_2 and SO_2. Gas concentrations (particularly CO_2 and SO_2) in and out of the reactor were monitored using dual continuous emissions monitoring systems (CEMS), to allow determination of pollutant removal efficiency.

Figure 3-5 Boiler simulator for generating flue gas from coal or propane

Shakedown Production Runs

Initial operating conditions for the pilot system were chosen to mimic the laboratory conditions as closely as possible. Key process parameters to be tested are mixing, temperature, reagent concentrations, coal sulfur content, gas and liquid flow rates and their respective residence time distributions. Early runs were characterized by a consistent decay in the product vaterite purity over time but subsequent optimization of the process parameters have resulted in conditions that allow for the steady state production of the calcium carbonate vaterite polymorph. This progress is illustrated in Figure 3-6.

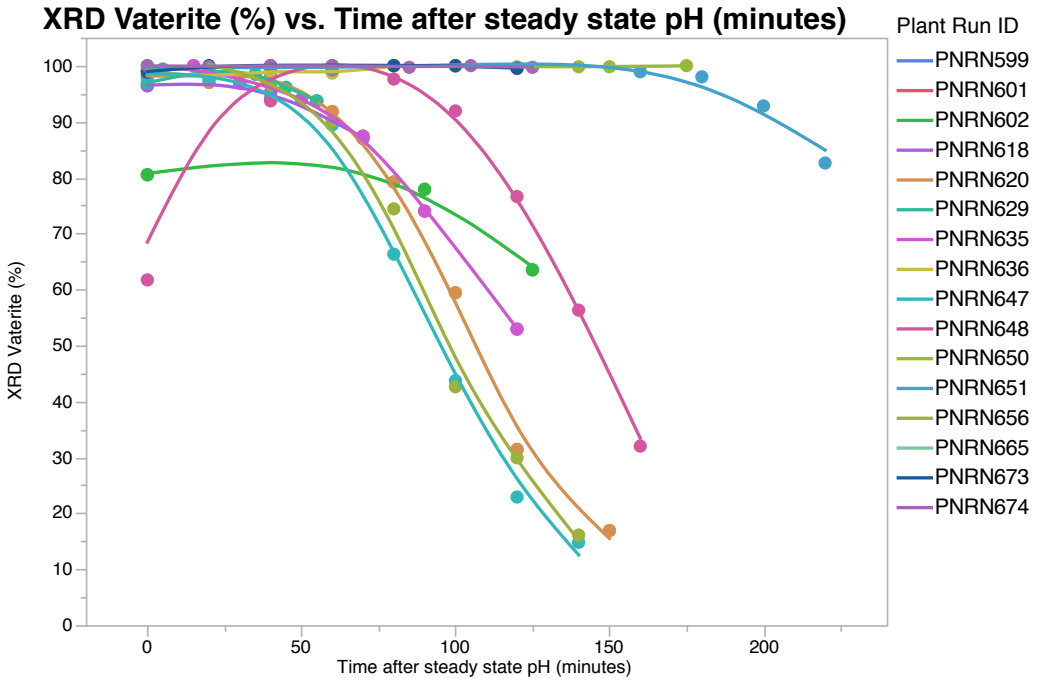


Figure 3-6 Illustration of run-to-run improvements in sustained product vaterite purity, as determined by x-ray diffraction (XRD)

3.2.2 Carbide Lime Test Results

To date, 38 full test runs have been completed using the waste oxide processing subsystem. Key performance indicators for the runs were the CO_2 absorption rate of the process and the purity of the vaterite product, which determines the quality of the cement. Continued operation of the subsystem led to optimization of process parameters, which allows the plant to reliably produce a product which is >98% pure vaterite. The run data are summarized below in Table 3-2.

Table 3-2 Subsystem run data

Date	Plant Run ID	Fuel Type	Average Inlet [CO2] (vol%)	Average Outlet [CO2] (vol%)	Average Abs [CO2] (vol%)	XRD Vaterite (wt%) Average	PSA Mean (µm) Average
10/31/13	PNRN618	Propane				67	14.5
11/7/13	PNRN620	Propane				62	
11/26/13	PNRN629	Propane				93	
12/11/13	PNRN635	Propane				57	
	PNRN636	Coal				94	
1/30/14	PNRN647	Propane				62	8.5
2/5/14	PNRN648	Propane				44	11.5
2/7/14	PNRN650	Coal				98	11
2/11/14	PNRN651	Mixed				85	8.8

	DRS10010	Propane				40	
2/28/14	PNRN656	Propane				57	
3/18/14	PNRN665	Coal					
4/9/14	PNRN673	Coal				95	12.2
4/18/14	PNRN674	Coal				96	14.5
4/24/14	PNRN676	Propane				99	10.3
4/29/14	PNRN677	Coal				98	7.5
5/6/14	PNRN680	Coal	13.80%			99	3.1
5/13/14	PNRN684	Coal				99	4.2
6/3/14	PNRN691	Propane					
6/11/14	PNRN695	Propane	11.22%	6.48%	45%	98	4.2
6/12/14	PNRN696	Propane	4.70%	2.63%	45%	96	6.7
6/19/14	PNRN700	Propane	10.85%	7.87%	30%	89	8
6/30/14	PNRN701	Propane	10.93%	7.22%	37%	99	12
4/28/15	PNRN977	Propane	11.80%	9.08%	25%	99	13.7
5/21/15	PNRN1004	Coal	12.67%	8.56%	35%	99	

3.2.3 Gas and Coal Flue Gas Absorption and Carbonation

The waste oxide processing subsystem was tested utilizing flue gas generated from the combustion of propane, as well as the combustion of several types of coals. The primary variable of interest with the different coals was the sulfur content as summarized in Table 3-3. The elemental sulfur content of the coal determines the amount of sulfur dioxide gas ($SO_{2(g)}$) generated during combustion. In most cases the flue gas of all of the fuels was controlled to approximately 12% CO_2, by controlling the amount of combustion air, to normalized absorption conditions to be representative of typical coal combustion gas concentrations.

Table 3-3 Coals used for pilot plant testing runs

Mine	Region	Type	Total Sulfur Content (wt%)	Theoretical [SO_2] at 3% O_2 Combustion (ppm)	Pilot Runs Used In
Caballo Rojo	Powder River Basin	Subbituminous	0.54	335	PNRN665
Rawhide	Powder River Basin	Subbituminous	0.70	587	PNRN673 PNRN674 PNRN1004
(Utah)	Southwest	Bituminous	0.98	968	PNRN636 PNRN677 PNRN680 PNRN684

In Figure 3-7 is provided examples of continuous emissions data during the burning of Rawhide coal. The alkaline conditions and relatively long gas residence times of subsystem's absorption/precipitation reactor, make it very similar to the conditions typical hydrated lime or

caustic-based flue gas desulfurization (FGD) units, with respect to it's ability to capture SO_2. SO_2 capture rates were >99% as determined by CEMS monitoring.

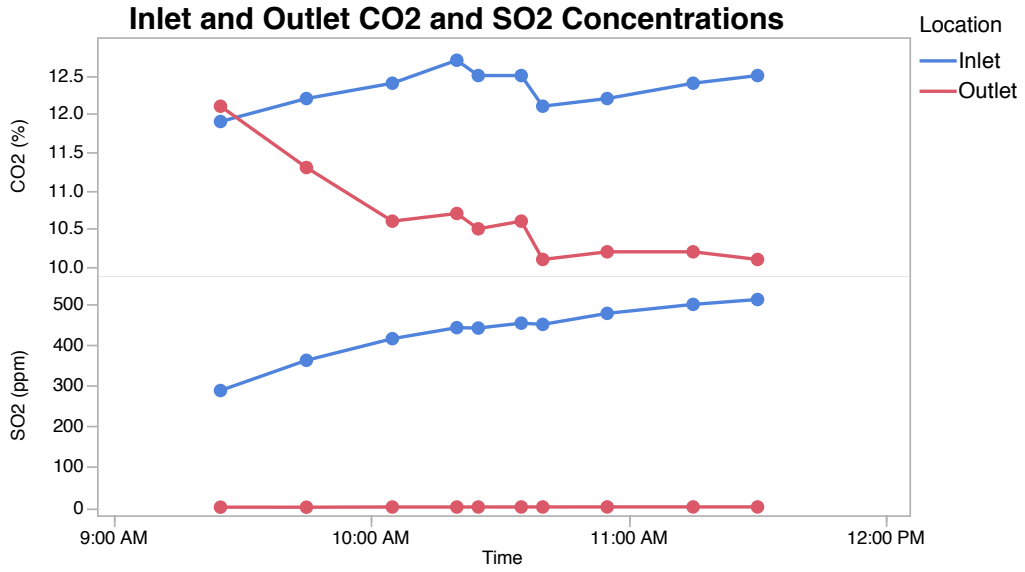


Figure 3-7 Example of CEMS data demonstrating CO_2 and SO_2 capture (Rawhide Mine, Powder River Basin coal)

The inlet and outlet CO_2 emissions from the absorber are shown in Figures 3-8 and 3-9 for two different runs using coal flue gas. The CO_2 absorption percentage ranged between 25-45%, and was found to be primarily a function of:

- Operating pH of the absorber (positive)
- Operating temperature of the absorber (positive)
- Mixing rate in the absorber (positive)
- Liquid/slurry fill height of the absorber (positive)
- Flue gas flow rate (negative)
- Initial CO_2 concentration of flue gas (positive)

While certain absorption parameters were found to increase the percentage of CO_2 absorbed from the flue gas, the parameters were constrained by the necessity to make the desired vaterite cement and process units of practical size. For example, while the flue gas flow rate could be decreased to increase the percentage of CO_2 absorbed, the absolute mass of CO_2 absorbed would decrease, necessitating larger process units to achieve the same overall absorption and production throughput.

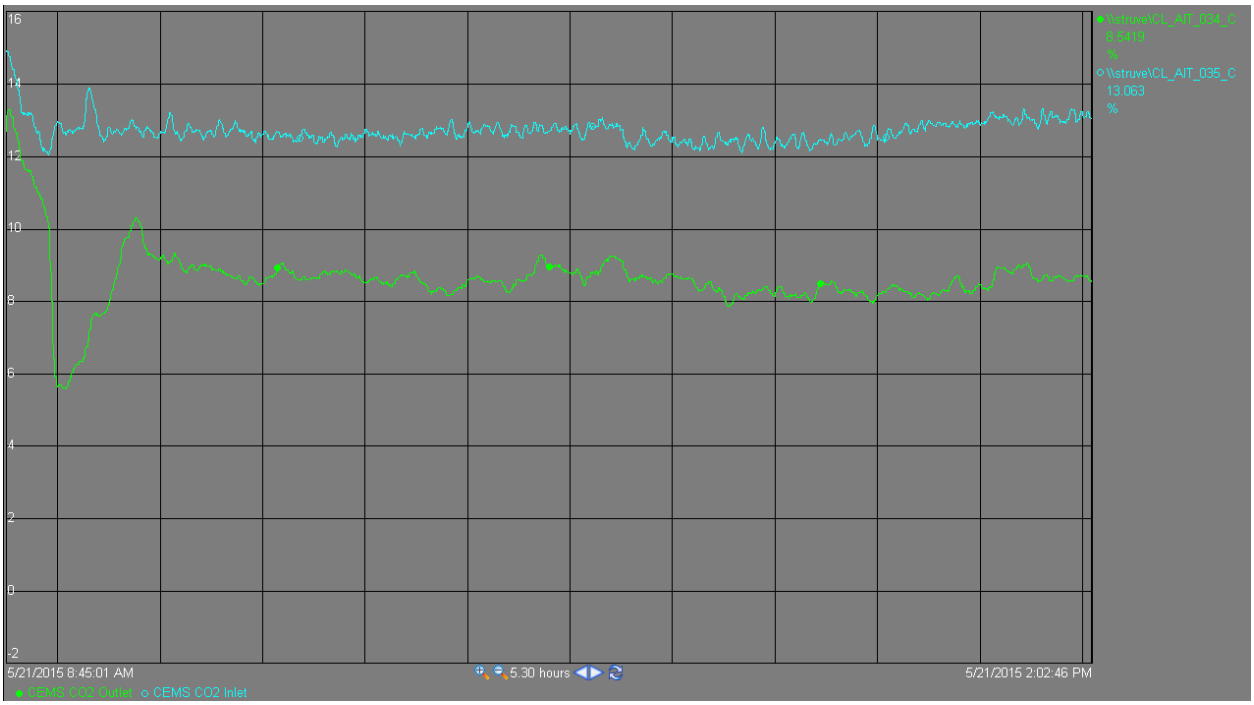


Figure 3-8 High-resolution CO_2 log data from CEMS, showing inlet CO_2 (blue) and outlet CO_2 (green) from coal-based run, PNRN1004

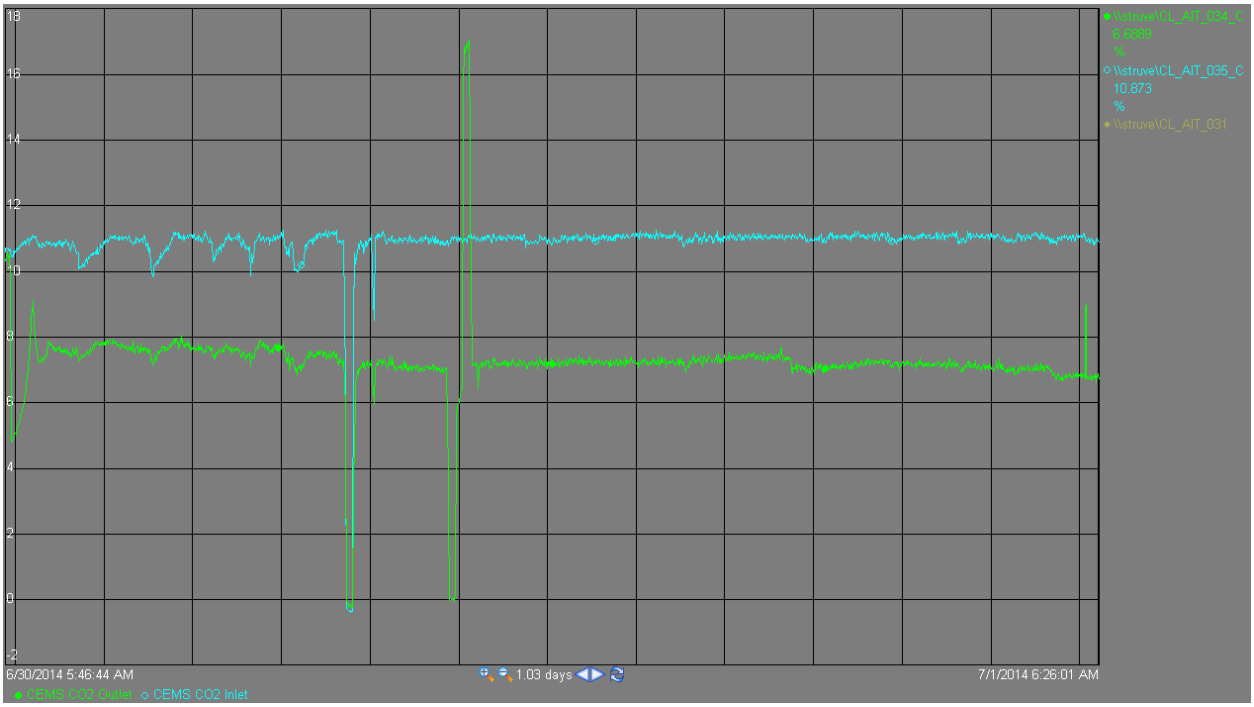


Figure 3-9 CO_2 absorption data collected during 24-hour testing, PNRN701

3.2.4 Conclusions

Calera has successfully demonstrated the technical industrial scale feasibility of recycling waste oxide materials to capture CO_2 and SO_2 pollutant gases, while also producing a valuable and novel cement product.

While all of the pilot scale testing was done using carbide lime, lab testing has shown that the demonstrated process may be applied to other waste metal oxides, including various steel slags, with changes to only the initial oxide dissolution step. The versatility of the process creates a much more geographically broad range of applicability.

3.3 **ABLE Subsystem**

3.3.1 **Electrochemical Cell Performance Tests**

The key performance index for the electrochemical subsystem is the operational cell voltage at the target current density. Figure 3-10 highlights the main voltage milestones achieved throughout commissioning subphase 2b. The electrochemical subsystem currently operates within the targeted voltage range required for continuous operation. The cell improvements throughout this phase included electrode re-design and construction, membrane optimization, and more uniform flow and pressure distribution within each compartment.

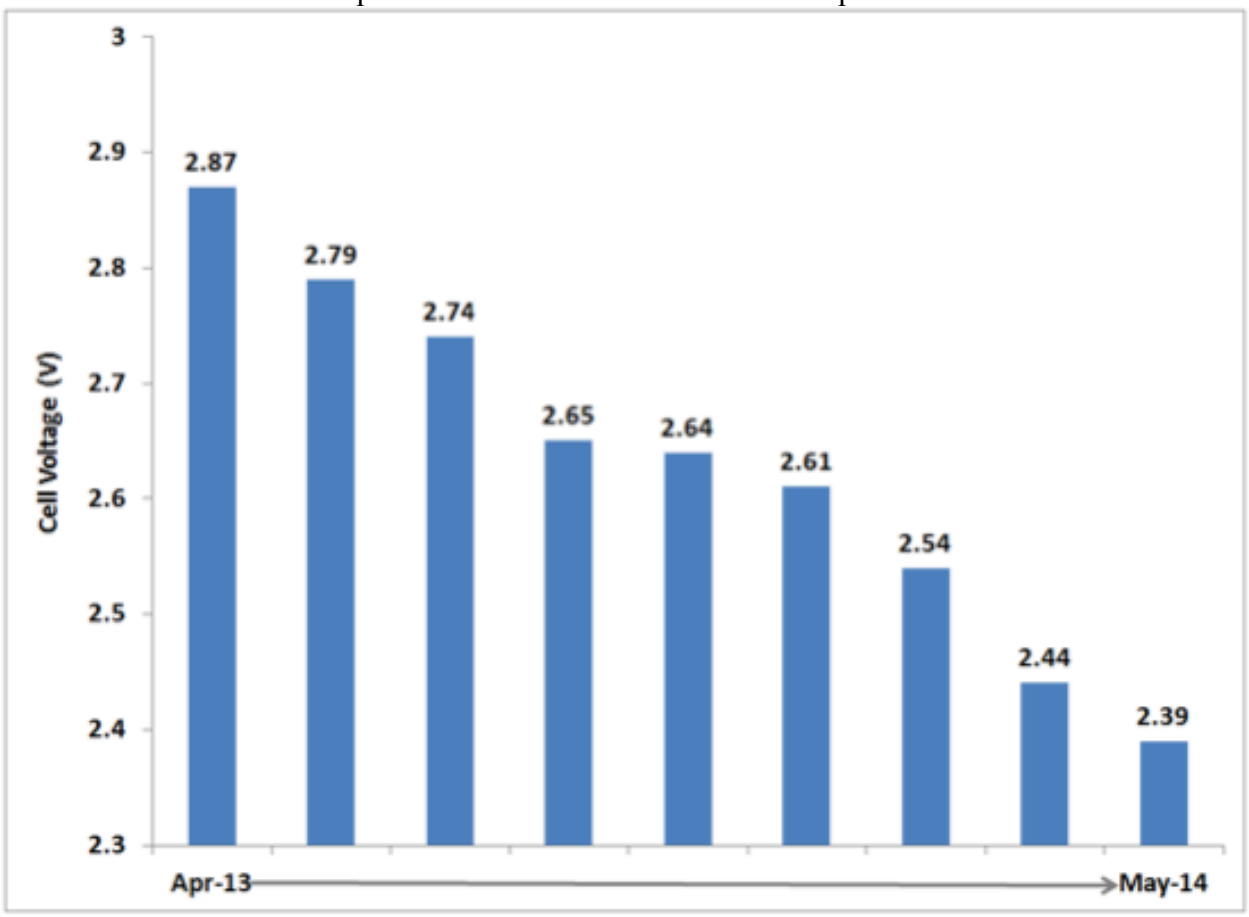


Figure 3-10 Progression of cell voltage at for the electrochemical subsystem

Currently the electrochemical subsystem consumes around 1650 kwh of electricity for one ton of caustic soda production, that is 20~25% lower energy consumption than state of the art chlor-alkali technology. If one-third of the chlor-alkali plants in US retrofit with Calera's ABLE technology, the energy saving would be enough to power the entire San Francisco area. That is why the electrochemical subsystem is currently focused on further modification of the cell for ease of manufacture, robustness and cost saving for Demo scale plant.

A novel detection system for copper monochloride has also been developed in the electrochemical subsystem. This method utilizes ultramicroelectrodes (UMEs) and electrochemical principles, which enables fast and online measurement of copper monochloride. The system electrochemically oxidizes copper monochloride to copper dichloride. With designed system control and data analysis algorithm, the oxidation current can be translated to copper monochloride concentration as shown in Figure 3-11.

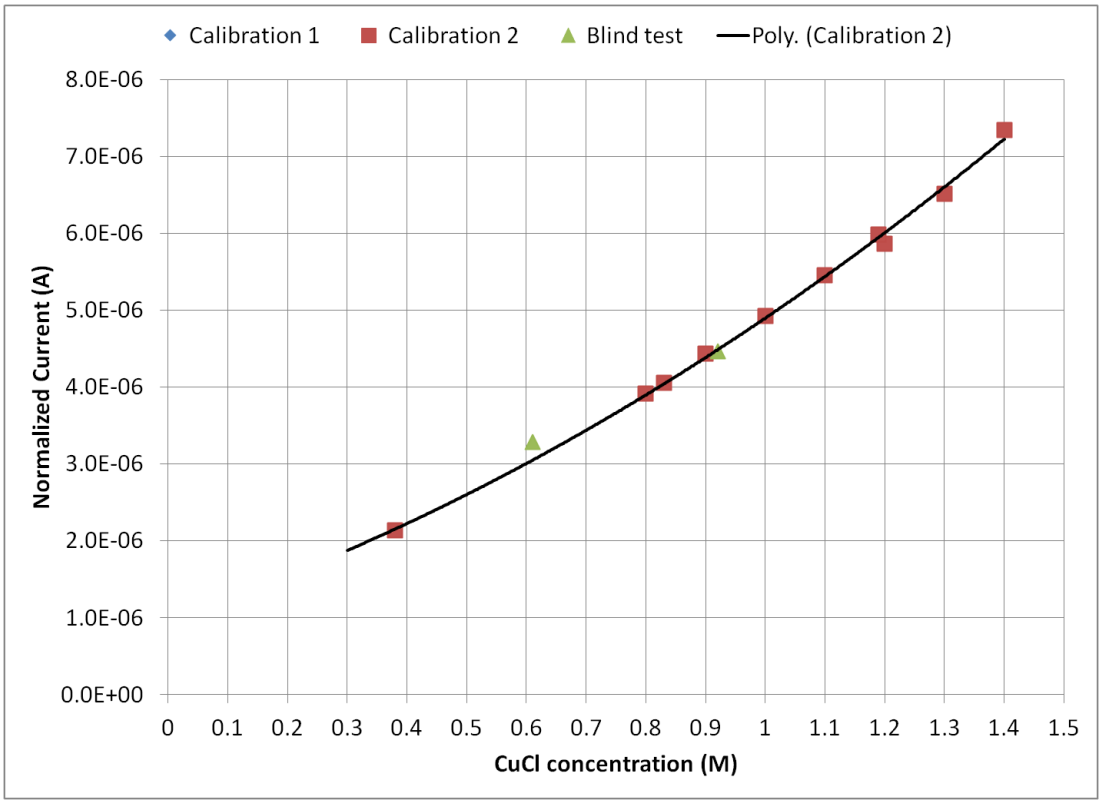


Figure 3-11 Calibration and measurements of copper monochloride using UME

3.3.2 **Catalysis Performance Tests**

As described above, copper monochloride is required for the anodic reaction in the E-chem sub-unit where it is oxidized copper dichloride. In order to sustain low energy alkalinity production long term, this copper monochloride in the anolyte feed stream must be regenerated. The catalysis test campaign was designed with the primary goal of proving that ethylene oxidation coupled with production of ethylene dichloride (EDC) is an economical solution to complete this copper cycle challenge. A few of the key economic drivers are catalysis performance criteria

including target copper conversion rates (EDC formation rates) and percent recovery of EDC versus byproducts (EDC degradation rate). Design parameters influencing these criteria were investigated at pilot scale in order to obtain EDC formation and degradation rate models. These studies are described below.

EDC formation rate:

Gaseous ethylene must come in contact with aqueous copper chloride in order for the reaction to occur. It follows that gas/liquid interfacial area can be an influential parameter in reaction rate. Reactor design and configuration can thus have a large impact on EDC formation rate. Multiple reactor types and aspect ratios were tested at the pilot scale.

Space time yield (STY) is a measure of EDC formation rate per reactor volume and a useful parameter for scale-up purposes as it directly influences capital costs. Both a continuously stirred tank reactor (CSTR) configuration as well as a modified trickle bed reactor design have been tested at the pilot plant scale.

Trickle bed reactor designs are sensitive to liquid flow rates as well as packing selection. Copper electrolyte flow rate showed a large impact on STY in the trickle bed reactors tested at pilot (when operated in absence of flooding conditions), likely due to an effect of increasing gas/liquid interfacial area. Figure 3-12 below shows this trend in a 3" diameter reactor, where STY increases roughly linearly with flow rate.

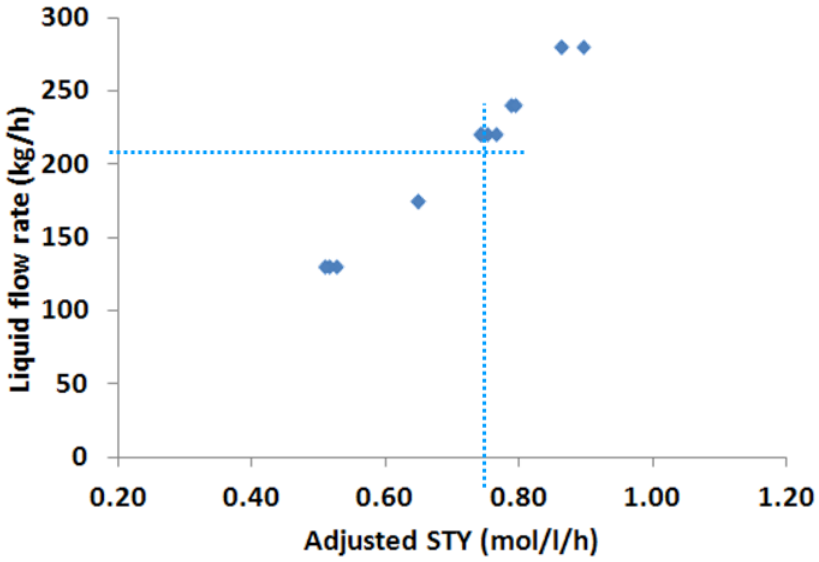


Figure 3-12 Impact of copper flow rate on STY (3" dia. trickle bed reactor)

However, varying reactor diameter at a constant liquid flow rate also influences gas/liquid contact area, as shown in Figure 3-13 below. Superficial liquid velocity is a translatable parameter for scaling reactor capacity across various aspect ratios. The data in Figure 3-14 collected for the 3" reactor was used to tie corresponding flow rate requirements to larger reactor diameters. As shown in Figure 3-15 below, a 4" reactor diameter was chosen based on the

maximum pump capacity procured for pilot demonstration scale. The reactor height required to match target sodium hydroxide production rates in integrated operation was subsequently determined based on these assumptions. As shown in Figure 3-15 below, a 20' reactor column was selected to meet copper cycle regeneration needs for integrated testing described herein.

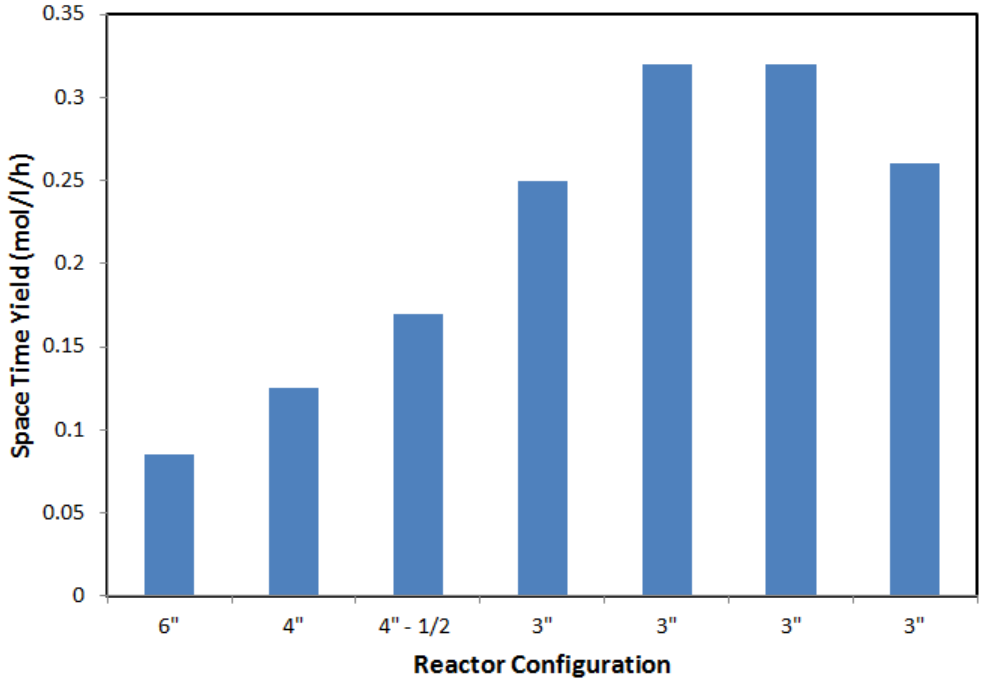


Figure 3-13 Decreasing reactor diameter resulted in higher STY at constant liquid flow rate

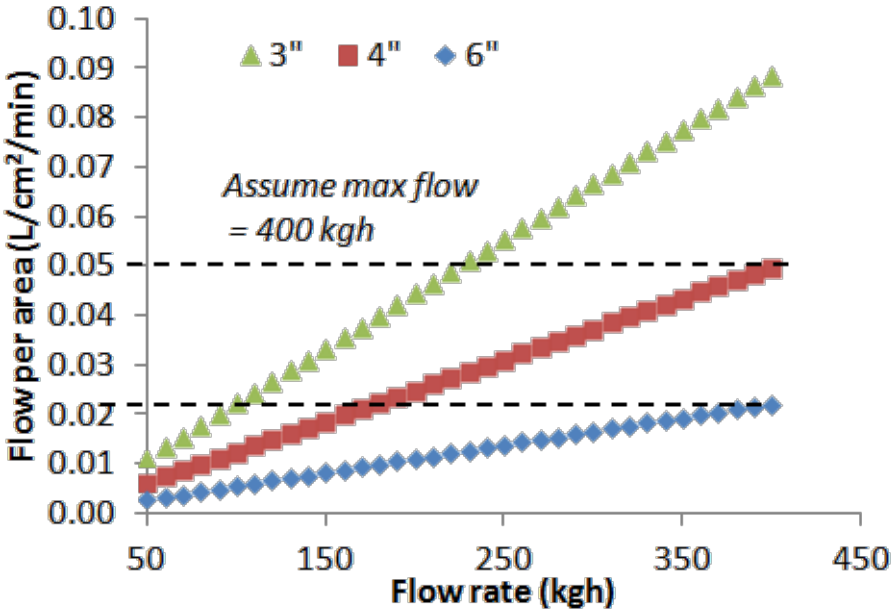


Figure 3-14 Selected 4" reactor based on tie point with 3" reactor data at 0.05 l/cm^2/min

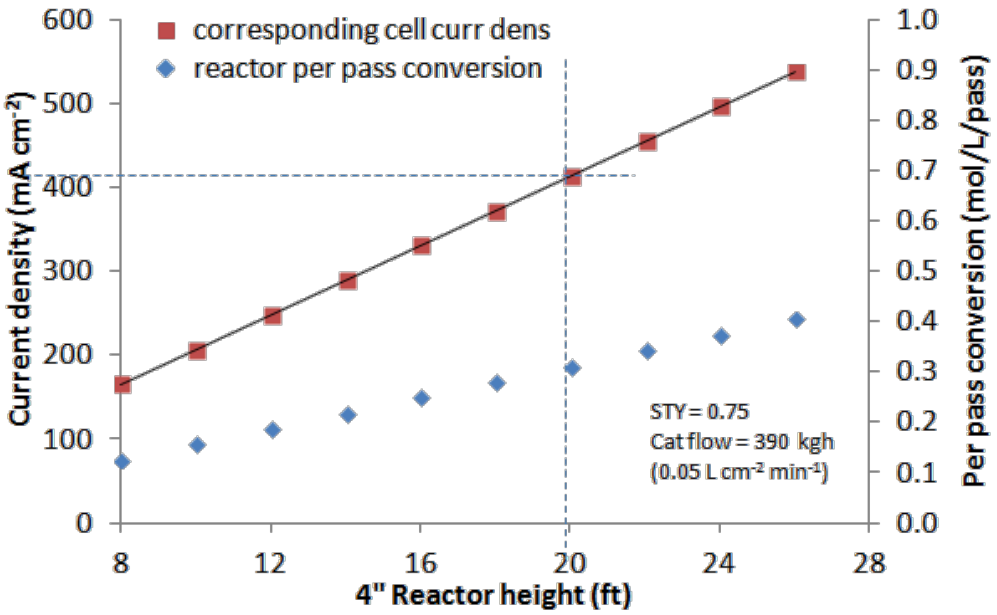


Figure 3-15 Reactor scaling to meet sodium hydroxide production rates

Packing material is a critical component of reactor design due to its impact on gas/liquid interfacial area. Packing materials were tested in a cold flow column over a range of flow rates and qualitatively assessed for flow regime—film flow vs. filament flow. Subsequent testing in the pilot scale reactor correlated well with the cold flow results. Pilot reactor results shown in Figure 3-16 below shows that Raschig rings provided a favorable gas/liquid contact area in comparison to glass and ceramic ball packing. Pre-wetting the cold flow column showed an observable improvement in liquid distribution. This flooding effect was not observed in the pilot reactor.

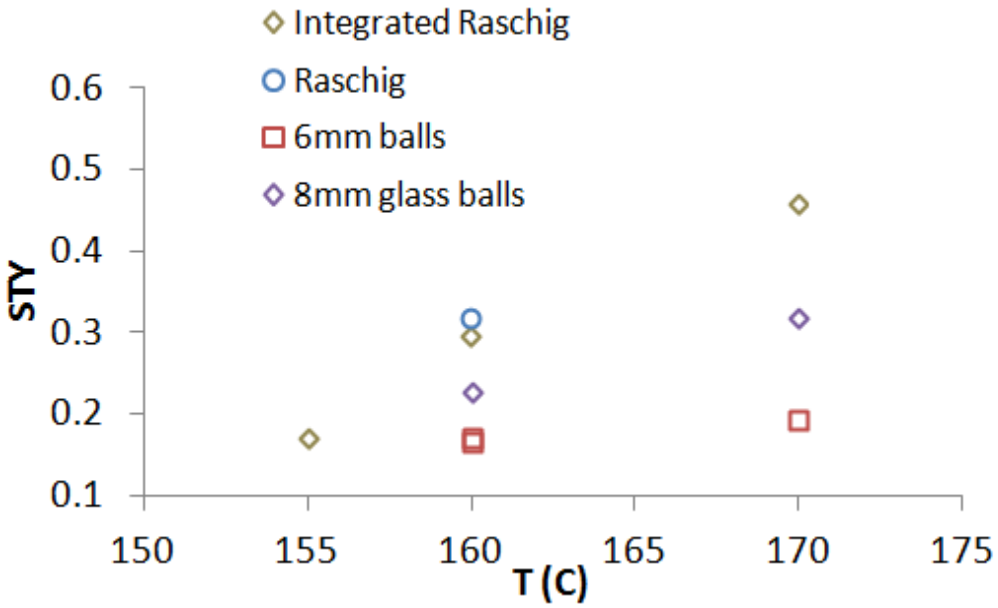


Figure 3-16 Type and size of reactor packing impacts reaction rates

The partial pressure of ethylene in the vapor phase is directly proportional to the concentration of ethylene dissolved in the aqueous phase. It is the dissolved ethylene molecules that can bind and react with copper chloride molecules. As expected, ethylene partial pressure was shown to have a positive correlation with STY in lab studies presented previously. Figure 3-17 below shows that this trend translates similarly to pilot scale performance.

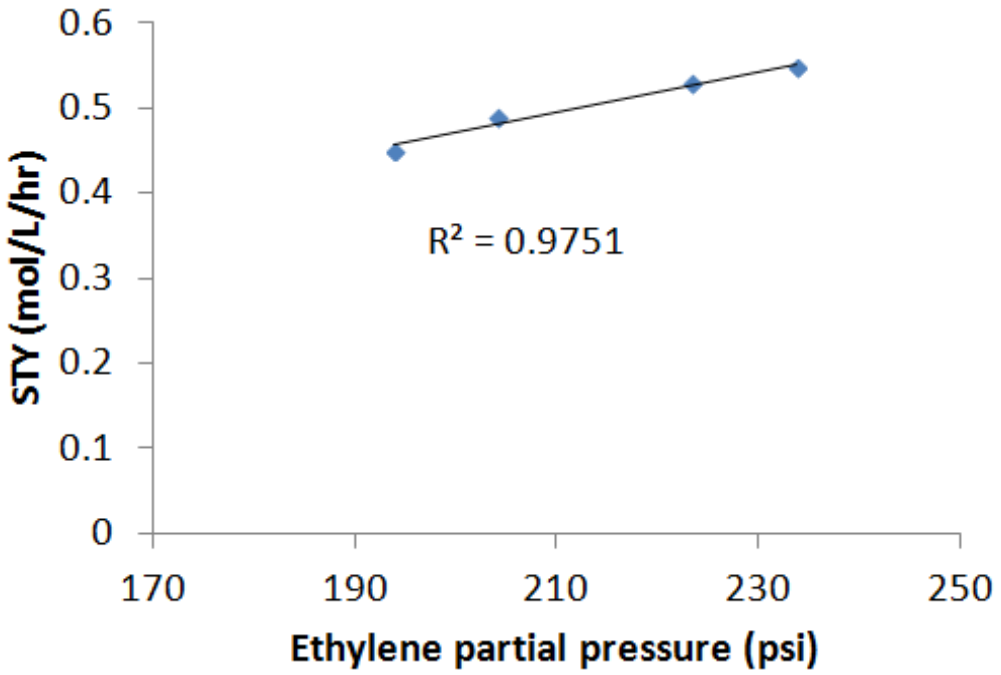


Figure 3-17 Linear relationship of STY with ethylene partial pressure is reproducible at pilot

The concentration of copper chloride molecules in the aqueous phase is also a driver for ethylene reaction rate. It has been reported from lab studies that increasing copper dichloride concentration has shown to increase STY. Density is a convenient measurement and can consistently indicate increases or decreases in copper chloride content. Density can be altered easily within a catalysis run via water removal in the distillation column and subsequent condensers. Several pilot experiments were conducted with varying copper density throughout the duration of the run while monitoring impact on STY. Figure 3-18 below shows that increasing density (i.e. copper chloride concentration) positively impacts STY, in agreement with lab data.

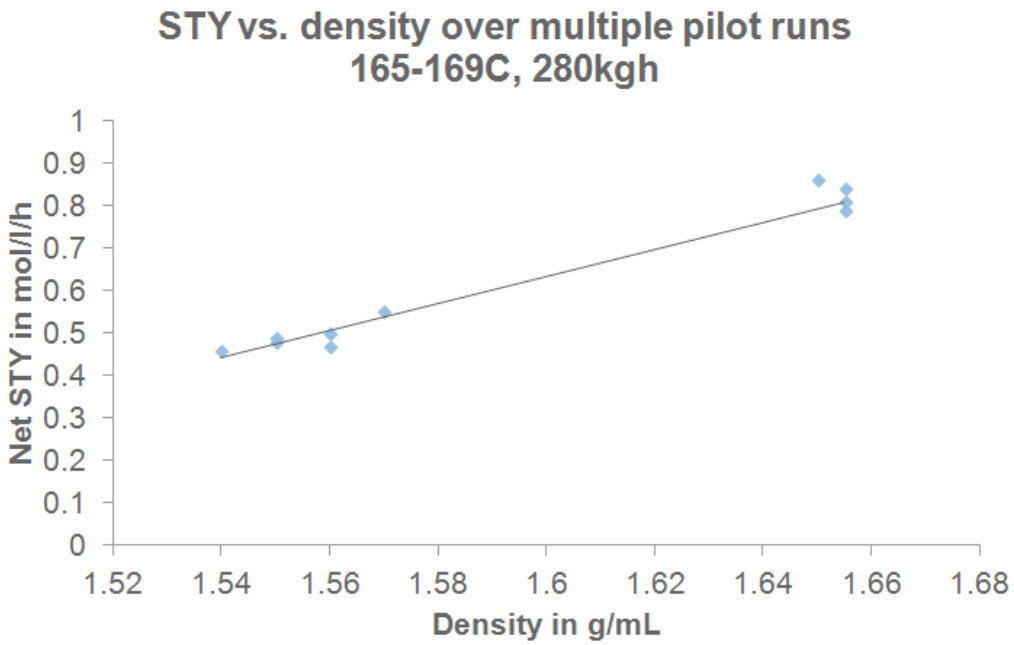


Figure 3-18 STY increases with increasing copper chloride concentration (correlated as density)

Lab data suggested that STY is sensitive to reactor temperature. Figure 3-19 below shows that pilot scale experiments agree with this trend. The activation energy for EDC formation was determined by conducting experiments with constant conditions and varying only temperature.

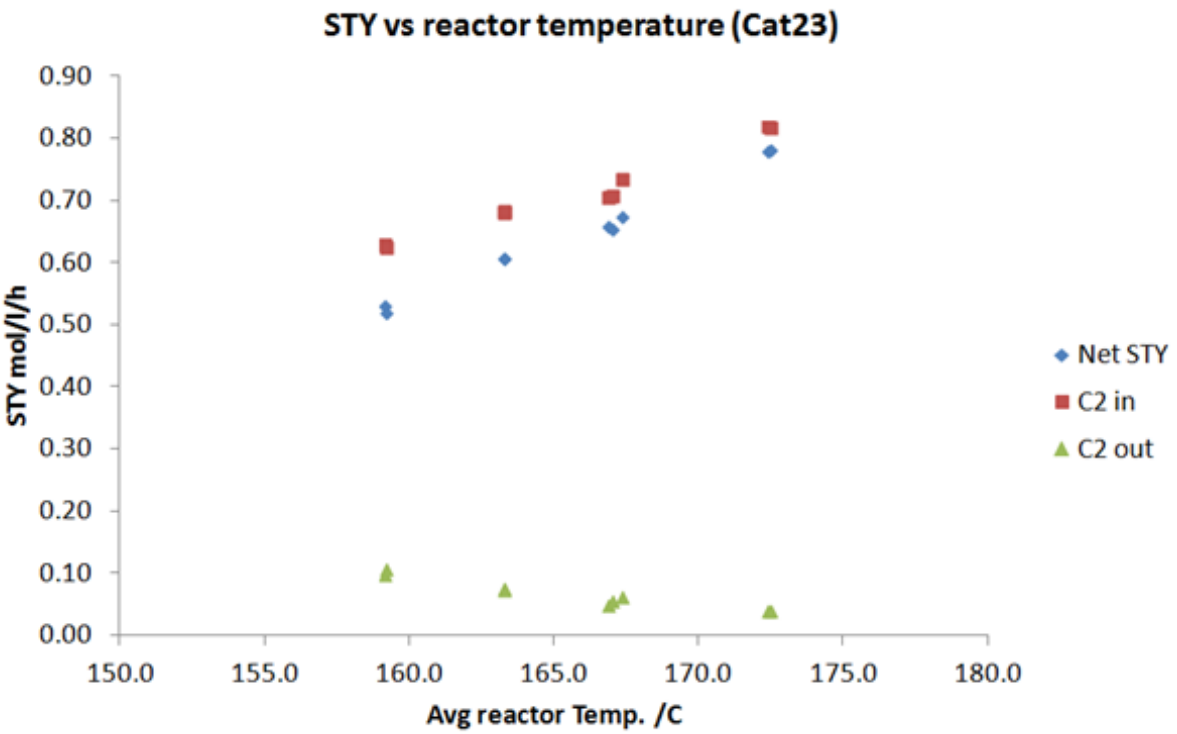


Figure 3-19 EDC formation rate temperature dependency consistent with lab data

EDC selectivity:

EDC selectivity is a measure of the moles of EDC produced per moles of ethylene consumed, where the dividend is attributed to byproduct formation. This is an important performance criterion because it has direct implications on operating costs, mostly due to high price of ethylene gas. In order to determine quantifiable impacts of various parameters on selectivity the system mass balance must be closed. EDC doping tests were conducted as a first step in the selectivity investigation to demonstrate mass balance closure around the reactor and separations units. These mass balance experiments were carried out at relatively low temperatures (130ºC) where EDC degradation rate is known to be low. EDC dosing was measured via a Coriolis flow meter. EDC recovered from the separations units was measured as a sum of mass of pure EDC collected and EDC collected in aqueous condensate. Aqueous condensate EDC concentrations were verified by liquid:liquid extraction and subsequent quantification via gas chromatography coupled with a flame ionization detector. A mass balance closure of 97% was achieved consistently based on EDC recovery alone.

Lab studies have shown that hydrolysis of EDC to chloroethanol occurs under reactor conditions. The extent of conversion is dependent upon copper composition, reactor residence time, and temperature. Since water is required for hydrolysis of EDC to chloroethanol, less free water available in solution should favor the EDC side of the equilibrium. Copper dichloride and total chloride concentrations have an inverse relationship with free water concentration. Copper dichloride and total chloride concentration has been shown to impact selectivity of ethylene conversion to EDC vs. chloroethanol in lab scale experiments. Copper dichloride variations were carried out at the pilot to confirm this correlation.

Longer copper residence times in the reactor promotes further degradation to chloroethanol and other byproducts. Packing material is a critical component of reactor design due to its impact on dynamic liquid holdup volume and therefore residence time. Packing materials were tested in a cold flow column under similar flow conditions to simulate residence time and dynamic liquid holdup for various reactor aspect ratios and packing materials. Figure 3-20 below shows these results for several packing types in the cold flow column in comparison to repeated tests conducted in the pilot reactor itself. Results of the cold flow column and pilot data are in close agreement. Little difference was found in testing water vs. copper electrolyte thus validating results collected in the cold flow column with water. Structured packing has yet to be tested at pilot scale, but cold flow testing suggests it may provide room for further improvement of selectivity.

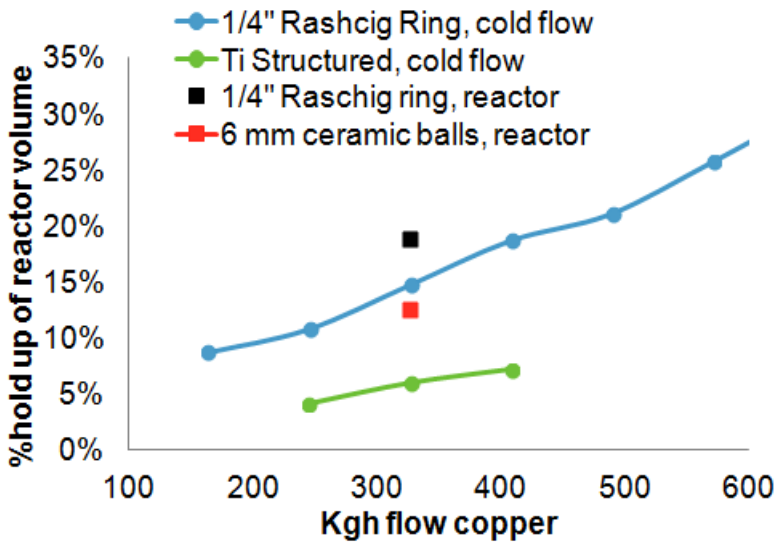


Figure 3-20 Cold flow testing shows further opportunity for selectivity improvement by moving toward structured packing with lower liquid holdup

Dynamic liquid hold-up volumes quantified using the cold flow column were then used to design a set of EDC doping experiments at various known residence times with the aim of understanding the impact of residence time in the pilot plant. This information is critical for guiding design for scale-up. Figure 3-21 below shows that EDC recovery in these doping tests decreases linearly with increasing residence time.

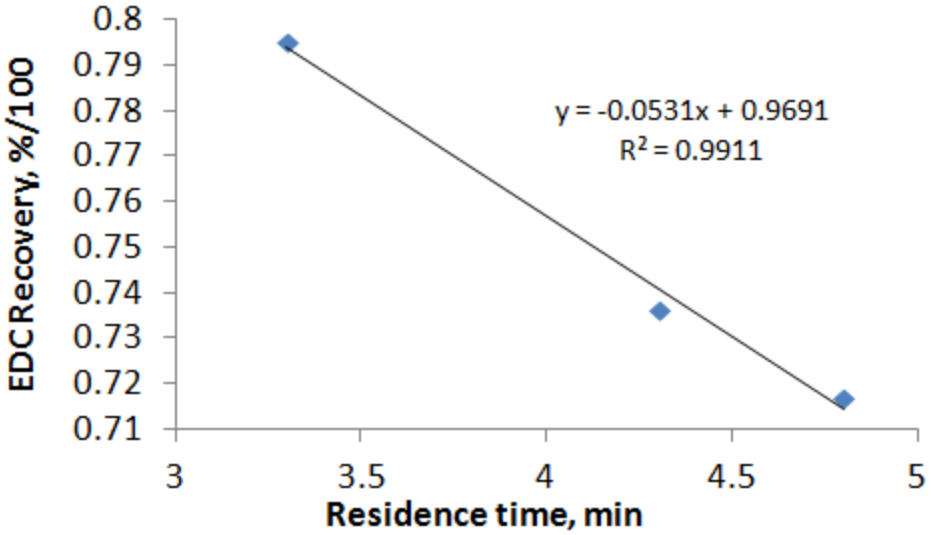


Figure 3-21 EDC degradation increases linearly with residence time

3.3.3 **Integrated Operation**

The primary objective of integrated testing was to demonstrate sustainable production of sodium hydroxide by regenerating copper monochloride in the catalysis sub-unit within target performance requirements. These performance requirements include NaOH production rates, copper cycle efficiency, cell voltage, EDC selectivity, product purity, and component lifetime.

The target sodium hydroxide production rate dictates the copper conversion and therefore EDC formation rate needed to maintain steady state within the ABLE system. For integrated operation it is critical that the rate of copper reduction in catalysis matches the rate of copper oxidation in the E-chem cell. Mass balance closure discussed above is critical to maintaining tight process control. As presented above, catalysis production rate and cell voltage are both dependent upon the copper chloride electrolyte composition. Integrated tests were conducted to find a range of copper compositions which could sustain operation of both systems within performance targets. Copper monochloride to copper dichloride ratios were varied during the run by altering the ratio of cell current to ethylene oxidation during an integrated run. Total copper chloride concentrations were investigated by removing net water from the system in the distillation unit during an integrated run. A resulting composition was found which allowed operation of both systems within performance targets. Figure 3-22 below shows catalysis copper conversion rate meeting the target sodium hydroxide production rate during integrated operation.

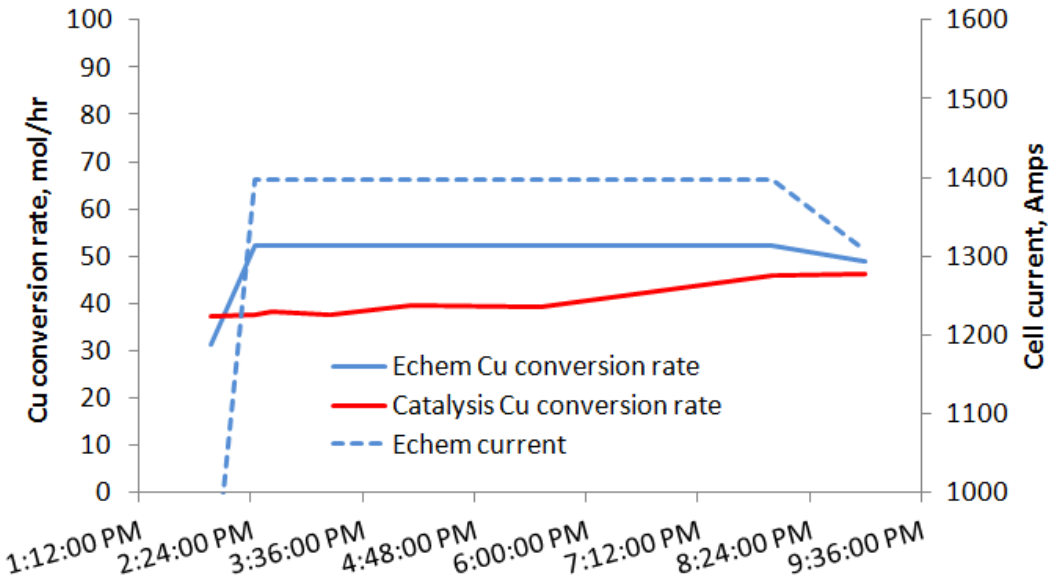


Figure 3-22 Example of copper conversion rate for ABLE subsystem integrated operation

Integrated runs provide a solid platform for testing copper cycle efficiency over longer time scales. Copper cycle efficiency was verified by sampling and analyzing both E-chem and catalysis feed streams during integrated operation. Copper analytics are accomplished with a combination of ICP and other methods developed in-house, including titrations and electrochemical methods.

Sodium hydroxide concentration and purity must meet certain requirements to be suitable for CO_2 capture and/or as a separate value-adding product. EDC produced must meet purity requirements for downstream industrial applications. Sodium hydroxide concentration was measured continuously by Coriolis flow meter where the weight percent of sodium hydroxide can be determined by measured temperature and density. Sodium hydroxide concentration was also measured routinely by acid/base titration. Sodium hydroxide purity was confirmed by colorimetry. EDC purity was measured through routine sampling by gas chromatography in combination with a flame ionization detector (FID). All integrated runs conducted at pilot demonstration scale have exceeded these requirements.

Extended integrated testing at pilot demonstration scale also provided a framework for learning about materials of construction and component lifetimes. The E-chem components of most relevance are the anion exchange membranes. Membranes of various thicknesses and compositions were tested in integrated operation in order to aid in selection for larger demonstration scale. Those anion exchange membranes which were found to develop pinholes or bulk brine flux were eliminated from use. Integrated test campaigns also allowed for optimization of cell design including types of sealing surfaces, mechanical pressure impacts in the cell compartment stack, and electrode stability. On the catalysis side where the copper stream is maintained at higher temperature and pressure, integrated runs provided a solid basis for learning which materials of construction are or are not compatible. An assortment of corrosion resistant coatings for plant instrumentation, valves, and piping were explored in order

to determine which compositions and methods of application are acceptable for ABLE catalysis applications. Metal components constructed from multiple grades of titanium and stainless steel were put to use in various locations throughout the reactor, distillation, and separations units in order to map out potential service conditions for these materials. Over 1 year of integrated testing all materials of construction used in techno-economic analysis assumptions were validated for scale-up operations.

3.3.4 **Readiness for Large Demonstration Scale Testing of ABLE**

The ABLE process is now ready for more extensive demonstration scale evaluation. Through extensive pilot scale testing, we were able to identify and demonstrate the necessary copper reagent compositions that yield required performance of catalysis and E-chem in a recycle system. Final reactor design criteria have been established for scale-up to large demonstration phase. The ability to meet fundamental criteria used in the techno-economic analysis has been supported through the testing phase of the program.

4.0 MATERIAL PRODUCT QUALITY TESTING RESULTS

4.1 Alternative Uses Of Calera Reactive Carbonates

4.1.1 Partial Cement Substitute

14 tonnes of partial cement substitute (PCS) was produced in Calera's MAP Pilot Plant. PCS was produced with a particular particle size and used as a partial cement substitute to replace a portion of portland cement in traditional concrete mixes. 10% NaOH was pre-carbonated using flue gas from the Dynegy natural gas power plant and stored until use. Concentrated $CaCl_2$ solution was diluted with municipal water one day before PCS production. All PCS produced is tested for mineralogy and particle distribution, and in a standard ASTM mortar mix for compressive strength after 24 hours, according to ASTM C 109. The PCS produced was supplied to two external projects: 1) Khosla Ventures Office Building and 2) Santa Cruz Sidewalk as described in DOE Technical Report Phase 2a Design, Section 3.

4.1.2 Precipitated Calcium Carbonate

Calera has demonstrated pilot-scale production of a prototype, 2-3 micron precipitated calcium carbonate (PCC) product suitable to be used as filler in rubber and plastics applications. A combination of internal and external material characterization is being performed to compare the pilot materials to the Chinese HG/T 2226 specification for PCC fillers for rubber and plastics applications (Table 4-1). Calera's material has met or exceeded almost all of the specified criteria, with the exception of whiteness, with a value of 91 versus the specification of 92. The lower whiteness is potentially resulted from process contamination and impurities and is currently being optimized.

Table 4-1 Calera Pilot PCC versus Chinese standard

	Calera Pilot PCC	HG/T 226-2010 Standard
CaCO3 (wt%) ≥	99	97-98
Whiteness	91	92-95
Particle Size Median (μm)	3.0	(not specified)
Particle Size Standard Deviation (μm)	1.5	(not specified)
Fe (ppm) ≤	630	500-800
Mn (ppm) ≤	0	50-80
Pb (ppm) ≤	0	10
Cr (ppm) ≤	2	5
Hg (ppm) ≤	0	2
Cd (ppm) ≤	0	2
As (ppm) ≤	0	3

4.1.3 Aerated Concrete

Calera has attempted to diversify its product portfolio by expanding and developing aerated concrete formulations. Preliminary lab-scale results show that Calera's aerated concrete formulations could meet the aerated concrete standard, ASTM C 1693. However, further optimization on strength is required to further validate this line of products as the lab prototypes are about 20-30% weaker in compressive strength at the same density compared to commercial aerated concrete products.

Table 4-2 Aerated concrete ASTM C 1693 testing results

	Mid-Density	High-Density	ASTM C 1693 AAC-2	ASTM C 1693 AAC-4
Density (kg/m^3)	500	650	350-550	450-650
Porosity (vol.%)	73	78	N/A	N/A
Thermal Conductivity (W/m/K)	0.17	0.22	N/A	N/A
Compressive strength (MPa)	2	4	≥2	≥4

4.2 Selection of Fiber Cement Board Products

Approximately 12 months was spent researching the existing board products markets in the US, and other countries. This included understanding existing technologies, market size, building standards, contractor needs, competition, pricing, raw material availability, etc. Calera's goal is to produce a new disruptive cementitious board product targeted initially at the North American market. Initial products selection was based on market size (>$200M/yr), price such that Calera's product can have a price advantage at commercial scale, a "green" advantage over all others, and exceed other products in key differentiation areas such as aesthetics, weight, durability, safety, and handling. Based on these criteria, Calera decided to focus on developing fiber cement board products for tile backer and floor/roof underlayment (backer board), and exterior sheathing applications (sheathing).

4.3 Fiber Cement Board Formulation

A wide range of fiber cement formulations were evaluated on Fiber Cement Board Production Subsystem with varying carbonate cement, cellulose, and additives contents with the objectives to optimize density, mechanical, and durability properties. Calera's prototype fiber cement products for both backer board and sheathing applications is composed of 89.5% carbonate cement, 10% cellulose fiber, 0.2% anti-fungal additive, and 0.3% water repellant additive.

4.4 Performance Measures for Fiber Cement Boards

Fiber cement boards require high flexural strength to support handling, installation, and application specific usage. After every fiber cement board production run, 1 out of every 10 boards is randomly selected for evaluation of equilibrium density and flexural strength, and water saturated flexural strength in order to evaluate the effect of changing process and formulation parameters. For both backer board and sheathing applications, the fiber cement boards were targeted to achieve an average equilibrium flexural strength of 1400 psi and water

saturated flexural strength of 1000 psi with an equilibrium density of <1 g/cm3, which is at least 20% lighter than common commercial fiber cement board products. Once the density and flexural strength are determined satisfactory, additional fiber cement boards from the same production run are submitted for internal specification testing according to the interior use fiber cement standard, ASTM C 1288, and exterior use fiber cement standard, ASTM C 1186 to cover additional mechanical and durability performance measures.

4.5 Fiber Cement Board Testing Results

Calera's prototype fiber cement products for both backer board and sheathing applications have passed majority of the interior use fiber cement standard, ASTM C 1288, and exterior use fiber cement standard, ASTM C 1186. In Table 4-3 is shown the testing results of Calera's prototype fiber cement products according to the specifications. The prototype fiber cement boards achieved the development target of 1400 psi equilibrium flexural strength and 1000 psi saturated flexural strength at a density of 0.97 g/cm^3. Other mechanical properties such as nail head pull through significantly exceeded the specification requirements. Durability performance including mold resistance, surface burning, moisture movement, warm water resistance, water tightness, and freeze thaw resistance all passed the specification requirements. For the remaining properties, heat rain resistance is a long-term durability test that is in progress. Shear bond strength passed for both standard mortar and latex modified mortar but failed with organic adhesive as the adhesive is deemed not compatible with the carbonate cement. Recommendation of only using standard and latex modified mortar for attaching tile has been included in the prototype products installation guideline.

Table 4-3 Fiber cement prototype products ASTM C 1288 and C 1186 testing results

	ASTM C1288 ASTM C1186 (US)	Prototype Fiber Cement Board Product
Density (g/cm3)	-	0.98 Grade L (China)
Dry Flexural Strength (MPa; ASTM C947)	≥4/10/16 (Grade I/II/III)	10 Grade II (US) Grade III (China)
Wet Flexural Strength (MPa; ASTM C947)	≥4/7/13 (Grade I/II/III)	6.5 Grade I (US) Grade II (China)
Nail Head Pull Through (kgf; ASTM D1037)	≥40	≥100
Mold Resistance (rating; ASTM G21)	≤1	0
Moisture Movement (ASTM D1037)	≤0.07%	≤0.03%
Surface Burning, flam spread/ smoke development (index; ASTM E84)	0 flame spread ≤5 smoke (Class A)	0 flame spread ≤5 smoke (Class A)
Warm Water Resistance (ASTM C1185)	≥80% control flexural strength	≥90% control flexural strength
Shear Bond Strength (kPa; ANSI A118.1)	≥350	≥700 Except organic adhesive
Water Tightness (ASTM C1185)	No water drops on underside	No water drops on underside
Freeze Thaw Resistance (ASTM C666)	≥50 cycles ≥80% control flexural strength	≥50 cycles ≥90% control flexural strength

4.6 DEMONSTRATION PROJECTS

In order to facilitate the use of Calera's prototype fiber cement products in demonstration projects, several supporting documents were drafted such as datasheet, safety datasheet, and installation guide. Figure 4-1, 4-2, and 4-3 are shown examples of these supporting documents. Currently, the production is being ramped up at Fiber Cement Board Production Subsystem to produce 100 passed ASTM C 1288 and C 1186 prototype fiber cement boards for use in external demonstration projects. Figure 4-4 is shown an internal demonstration project where Calera's prototype fiber cement board was installed on a 20" by 12" wood frame at the Moss Landing Pilot Plant.

AIRock™
TILE BACKERBOARD

TECHNICAL DATA SHEET

Lowest CO_2 Footprint | Lowest Energy Footprint | Environmentally Friendly | Easier Installation | Light Weight | Mold Resistant | Water Resistant

DESCRIPTION

AIRock Tile BackerBoard is a next generation fiber-cement board that can replace conventional tile backer products. AIRock Tile BackerBoard is 20% lighter than conventional products while maintaining equivalent performance properties, making it effortless to handle and install. AIRock Tile BackerBoard can be scored and snapped with a utility knife or cut with shears or saw, and can be installed with standard corrosion-resistant screws. Unlike conventional tile backer products, AIRock contains no silica (silica may cause health issues), no fiberglass (which can cause skin irritation), is made from 90%+ recycled content, and has a 120% lower CO_2 and 65% energy footprint.

PHYSICAL PROPERTIES

Properties	AIRock Tile BackerBoard		
Dimensions	32" x 48" x 1/2"	36" x 60" x 1/2"	48" x 96" x 1/2"
Dimensions	32" x 48" x 5/8"	36" x 60" x 5/8"	48" x 96" x 5/8"
1/2" Weight	27 lbs	38 lbs	80 lbs
Density	60 lbs/ft		

MAY CONTRIBUTE UP TO 10 LEED POINTS

U.S. GREEN BUILDING COUNCIL LEED USGBC

SPECIFICATIONS

	Properties	Test Method	Requirements	Test Results
Mechanical Properties	Dimensional Tolerances	ASTM C 1185	Length ±0.25 in Width ±0.25 in Thickness ±0.05 in Squareness ≤1/32 in/ft Edge Straightness ≤1/32 in/ft	Pass
	Flexural Strength	ASTM C 1185	>1450 psi (Grade 3)	1750 psi
	Nail Head Pull Through	ASTM D 1037	>90 lbs	Pass
	Nail Pull Resistance	ASTM C 473	>80 lbs	Pass
	Shear Bonding	ANSI A118	>50 psi	Pass
Durability Performance	Water Tightness	ASTM C 1185	No water drops on underside	Pass
	Moisture Movement	ASTM C 1185	≤0.07%	≤0.01%
	Warm Water Resistance	ASTM C 1185	≥80% control flexural strength	100%
	Mold Resistance	ASTM G 21	≤1	0
	Surface Burning	ASTM E 84	0 flame spread 0 smoke	0 flame spread 0 smoke (Class A)

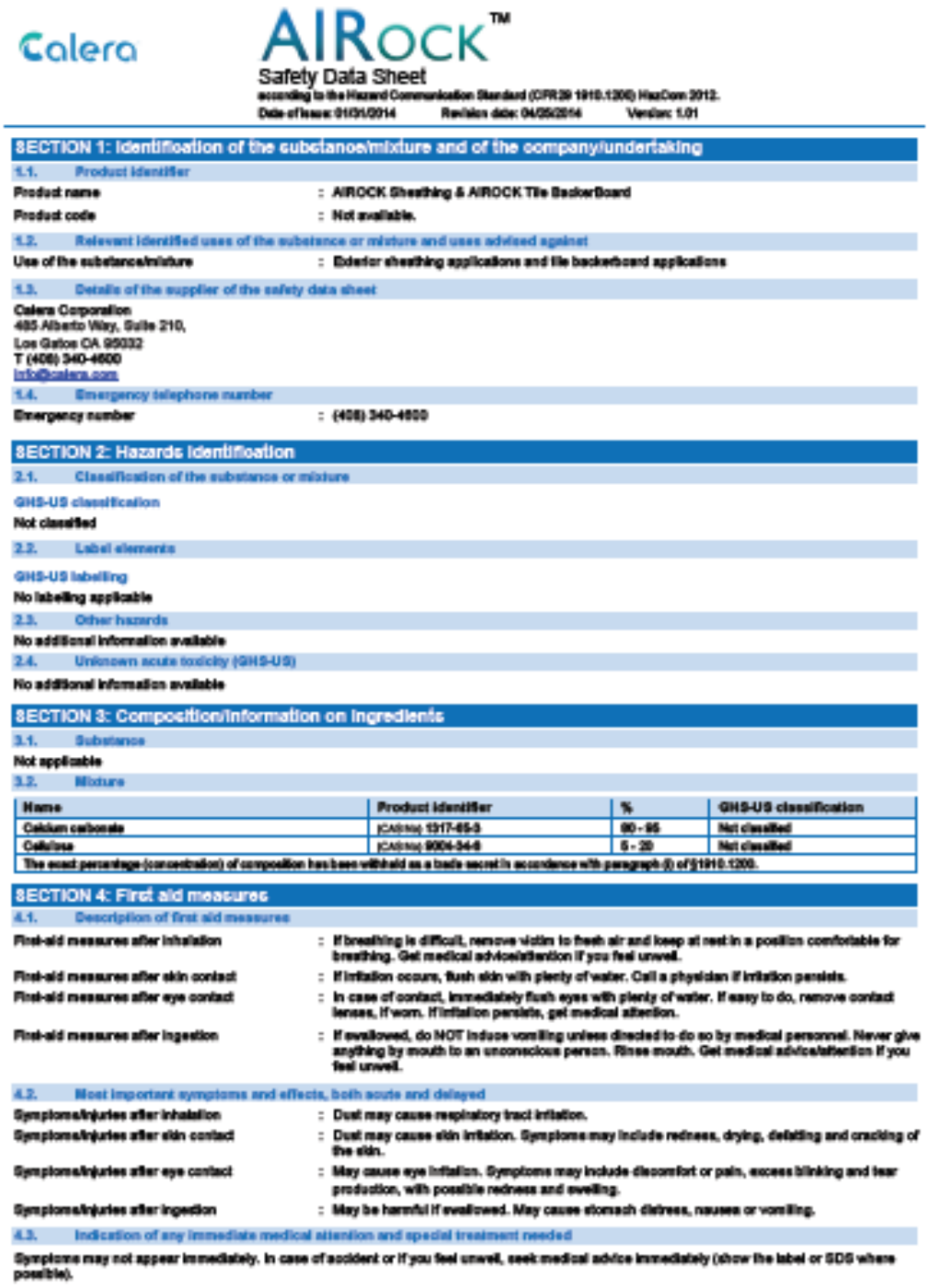
Certification: AIRock is externally validated to meet the U.S. standard for tile backerboards - ASTM C1288 and TCNA.
Composition: AIRock is composed of calcium carbonate, cellulose and selected additives.
Safety: AIRock contains no harmful asbestos, silica, glass fiber, or formaldehyde.

AIRock Tile BackerBoard is intended as an internal substrate for tiling in residential and commercial properties. It is a water resistant board and can be used in wet areas in both new construction and renovation.
See Warranty for warranty limitations. US & international patents issued & pending.
Installation: Refer to installation guide for details of proper installation.

Calera

485 Alberto Way, Suite 210, Los Gatos CA 95032

Figure 4-1 Draft datasheet for Calera's prototype fiber cement products



Calera

AIROCK™

Safety Data Sheet

according to the Hazard Communication Standard (CFR29 1910.1200) HazCom 2012.

Date of issue: 01/31/2014 Revision date: 04/25/2014 Version: 1.01

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1. Product identifier

Product name : AIROCK Sheathing & AIROCK Tile BackerBoard

Product code : Not available.

1.2. Relevant identified uses of the substance or mixture and uses advised against

Use of the substance/mixture : Exterior sheathing applications and tile backerboard applications

1.3. Details of the supplier of the safety data sheet

Calera Corporation
485 Alberto Way, Suite 210,
Los Gatos CA 95032
T (408) 340-4600
info@calera.com

1.4. Emergency telephone number

Emergency number : (408) 340-4600

SECTION 2: Hazards identification

2.1. Classification of the substance or mixture

GHS-US classification

Not classified

2.2. Label elements

GHS-US labelling

No labelling applicable

2.3. Other hazards

No additional information available

2.4. Unknown acute toxicity (GHS-US)

No additional information available

SECTION 3: Composition/information on ingredients

3.1. Substance

Not applicable

3.2. Mixture

Name	Product identifier	%	GHS-US classification
Calcium carbonate	(CAS No) 1317-65-3	80 - 95	Not classified
Cellulose	(CAS No) 9004-34-6	5 - 20	Not classified

The exact percentage (concentration) of composition has been withheld as a trade secret in accordance with paragraph (i) of §1910.1200.

SECTION 4: First aid measures

4.1. Description of first aid measures

First-aid measures after inhalation : If breathing is difficult, remove victim to fresh air and keep at rest in a position comfortable for breathing. Get medical advice/attention if you feel unwell.

First-aid measures after skin contact : If irritation occurs, flush skin with plenty of water. Call a physician if irritation persists.

First-aid measures after eye contact : In case of contact, immediately flush eyes with plenty of water. If easy to do, remove contact lenses, if worn. If irritation persists, get medical attention.

First-aid measures after ingestion : If swallowed, do NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Rinse mouth. Get medical advice/attention if you feel unwell.

4.2. Most important symptoms and effects, both acute and delayed

Symptoms/injuries after inhalation : Dust may cause respiratory tract irritation.

Symptoms/injuries after skin contact : Dust may cause skin irritation. Symptoms may include redness, drying, defatting and cracking of the skin.

Symptoms/injuries after eye contact : May cause eye irritation. Symptoms may include discomfort or pain, excess blinking and tear production, with possible redness and swelling.

Symptoms/injuries after ingestion : May be harmful if swallowed. May cause stomach distress, nausea or vomiting.

4.3. Indication of any immediate medical attention and special treatment needed

Symptoms may not appear immediately. In case of accident or if you feel unwell, seek medical advice immediately (show the label or SDS where possible).

Figure 4-2 Draft safety datasheet for Calera's prototype fiber cement products

AIROCK
TILE BACKERBOARD

AIRock Backerboard Installation Guide

Materials Required

- 1 1/4" (minimum) long wafer head exterior grade or fiber cement backerboard screws
- Polymer or latex modified thinset mortar
- 2" alkali-resistant glass fiber joint tape
- 1/4" square notched trowel
- Screw gun or drill
- Utility knife, carbide-tipped scoring blade, or electric shears
- Straight edge
- Hammer

Cutting AIRock backerboard

- Always cut backerboard on a flat stable surface.
- To cut straight lines across the entire length or width of the board, use a straight edge as a guide and score the face of the backerboard using a utility knife or carbide tipped scoring blade. Next, snap the backerboard upwards along the scored line.
- To cut out a hole, score along the perimeter of the hole and then knock out the undesired material using a hammer. Alternatively a hole saw can be used at low rpm to cut holes into AIRock backerboard. When using power tools to cut AIRock minimize dust generation and wear appropriate personal protective equipment.
- To cut a 90° section out of a board, score along the long cut and use electric shears along the short cut. After the making the short cut with shears, snap along the scored line.

Floor Installation

- Receiving surface: Sub-floor should be a minimum of 5/8" exterior grade plywood or 23/32 OSB (Exposure 1 classification or better) conforming to local building codes. Joists should be at a maximum 24" o.c. The sub-floor assembly should have a maximum deflection of L/360 if receiving tile and L/720 if receiving natural stone under any loading scenario. Check that the subfloor does not pull up from the joists and that all joints are level. The sub-floor should be clean of any debris or oil.
- Determine AIRock backerboard layout ensuring that the backerboards run perpendicular to the subfloor layout and backerboard joints are not within 12" of a subfloor joint. Leave a 1/8" gap between adjoining backerboards and between backerboards and vertical surfaces such as walls or cabinets.
- Apply a supporting bed of thinset mortar with a ¼" square notched trowel according to manufacturer's recommendations.
- Screw AIRock backerboard to sub-floor. Screws should be placed between 3/8" and 3/4" from the edge of the board but not within 2" of the corner of the board. Screws should be placed every 8" in a grid pattern. Sink screws flush with the top of the backerboard.

Figure 4-3 Draft installation guide for Calera's prototype fiber cement products

Figure 4-4 Internal demonstration project of the use of Calera's prototype fiber cement products

5.0 DESIGN OF A LARGER-SCALE ABLE PLANT

5.1 Demonstration Scale ABLE Plant

Calera intends to build a new demonstration scale unit that will demonstrate Calera proprietary ABLE process at an existing Chlor-Alkali and direct chlorination plant site. The Calera ABLE plant will include Calera's ABLE core technology, control system and the connections to brine (NaCl), caustic (NaOH), product (EDC) & the utilities that will be supplied by the existing plant.

The ABLE demonstration unit will include a 10-cell Electrolyzer stack with 56x the active area of the pilot plant and will produce 3.5 ton/day EDC and 2.75 ton/day NaOH (dry basis). The unit will: demonstrate 1 year of continuous fully integrated operation of all the ABLE subsystems; prove out the scalability and long-term viability of the process technology; generate operational data to assist in plant design of a commercially scaled-up facility.

The layout of the ABLE demonstration plant is shown in Figure 5-1. Calera intends to have a US fabricator build process modules that will be transported and erected at an existing Chlor-Alkali plant site. The E-Chem and Catalysis subunits and the storage tanks will each be constructed in a separate module. The Catalysis subunit, which is very tall, will be constructed of a few sub-modules to allow transportation to the site.

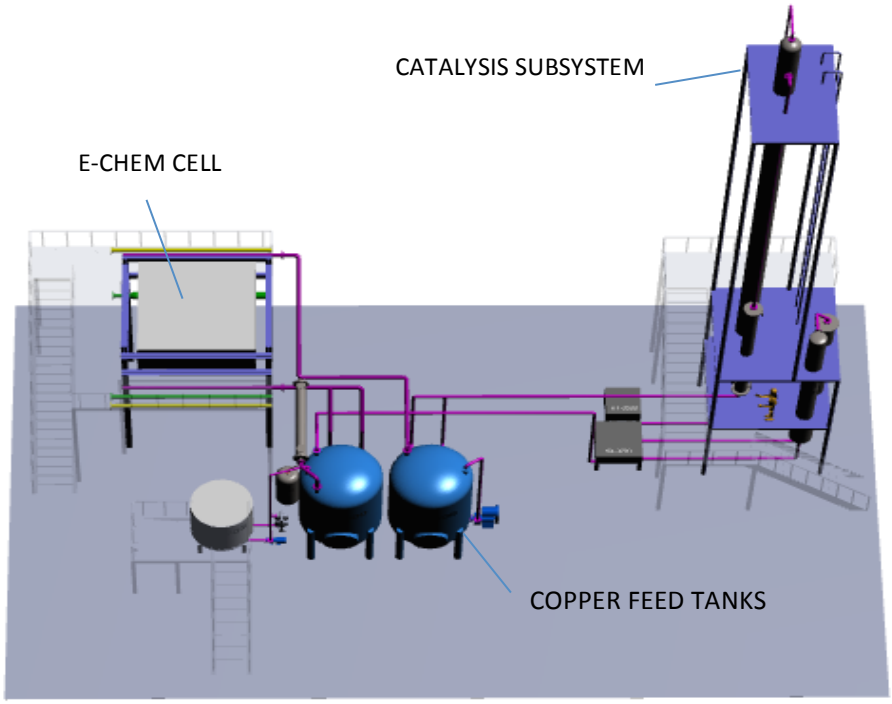


Figure 5-1 Schematic 3D layout of the ABLE demonstration plant

5.1.1 ABLE Process Flow Diagram

Figure 5-2 below gives an overview of the ABLE process. Figures 5-3 and 5-4 below represents a process flow diagram (PFD) for the ABLE Demo plant. The ABLE demonstration unit will use a proprietary low energy process to produce sodium hydroxide and ethylene dichloride from sodium chloride, water and ethylene. The unit utilizes an electrochemical cell to split NaCl to make NaOH and hydrogen in the cathode compartment and convert cuprous chloride (CuCl) to cupric chloride ($CuCl_2$) in the anode compartment. NaCl, NaOH and CuCl are all in aqueous solutions. $CuCl_2$ acts as a shuttle carrying the chloride to the catalysis area where the chloride is reacted with ethylene to make EDC and reduce the $CuCl_2$ to CuCl. The EDC is distilled from the copper chloride solution which is recycled back to EChem area.

The design of the demonstration unit is based on more than 1 year of testing at the pilot scale. All subunits have been automated by programming all instrumentation through a central PLC. The PLC is used to control the desired process flows, conditions, and safety interlocks by means of safe and remote operation. The operation and control schemes for each sub-system in the ABLE demonstration plant are described below.

Figure 5-2 ABLE process PFD, page 1

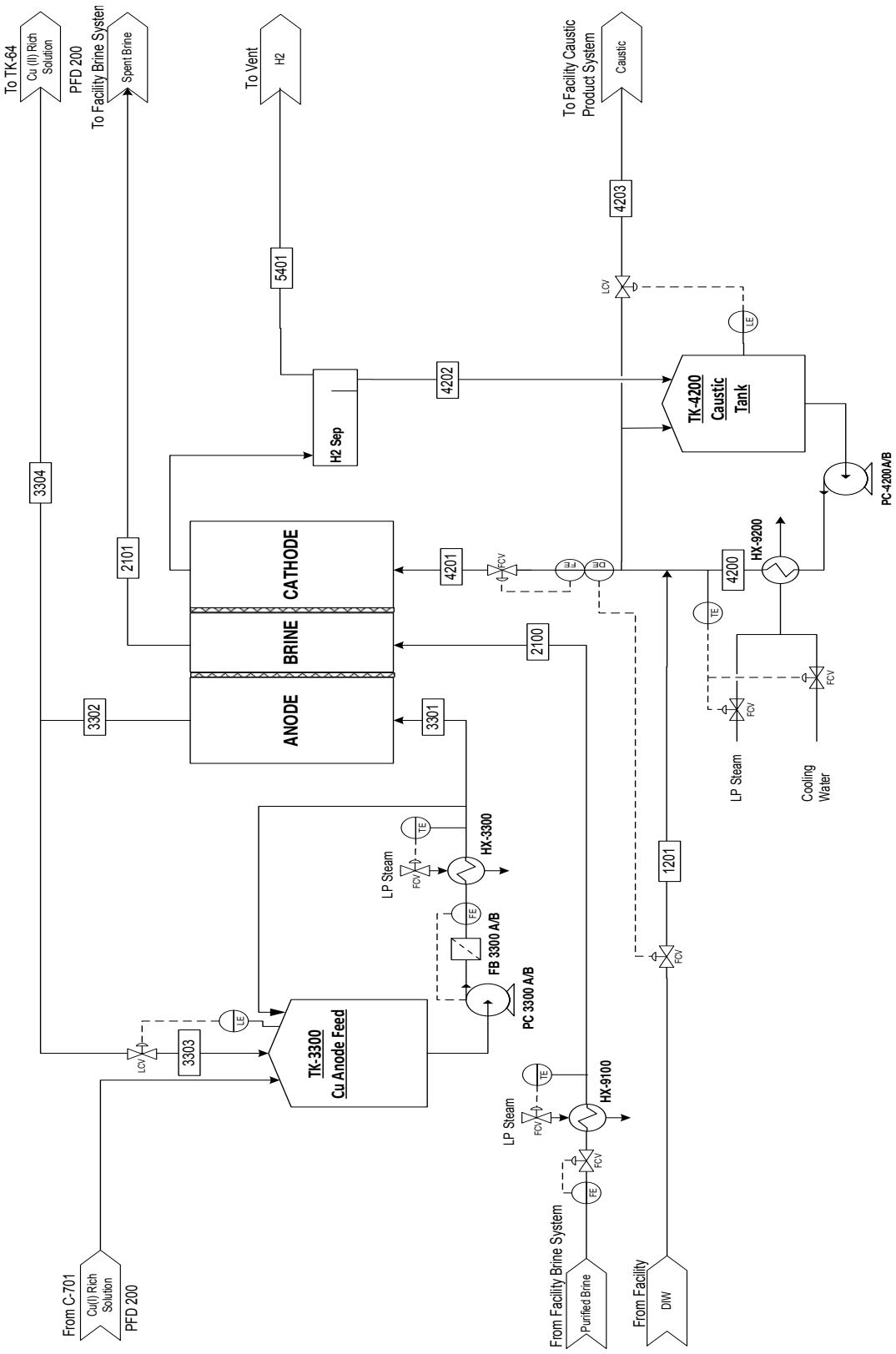
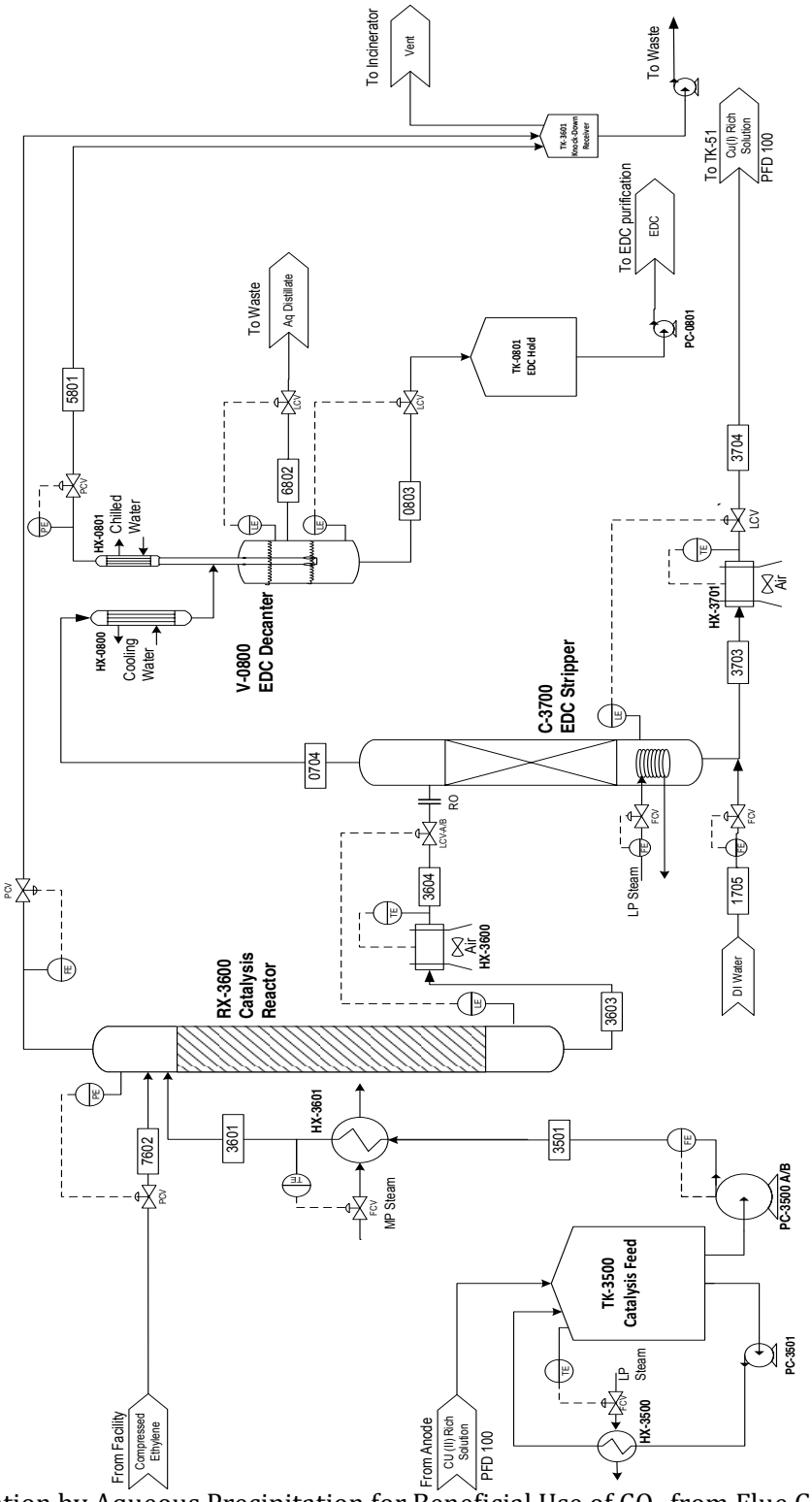


Figure 5-3 ABLE process PFD, page 2



5.1.2 Electrochemistry Subsystem Design

The Electrolyzer is a bipolar stack consisting of ten cells, with each cell having an active area of ~3 m^2. Each cell is composed of three compartment, Brine, Caustic (Cathode) and Copper Chloride (Anode). Each of the compartments is fed from a feed header connected individually to the ten cells. The product of each compartment exits to a product header. The sample valves and most of the instrumentation are located on the headers. By-pass lines with automated valves are installed to allow bypassing the three compartments during startup/shut down. The Electrolyzer is located on an elevated structure and the product headers gravity drain to their respective tanks which allows the cell to operate at atmospheric pressure.

Brine: Purified brine is supplied as a slip stream from the existing Chlor-Alkali plant pumping system. The brine is heated to Calera's Electrolyzer operating temperature using a steam fed heat exchanger. Inlet flow rate is controlled with a control valve. Inlet and outlet pH and pressure are measured to detect any abnormalities in operation.

Caustic (Cathode): The caustic system is designed to provide and collect 32 wt% NaOH to the cathode system from the surge tank where level increases as caustic is produced. Concentrated caustic is pumped, using redundant centrifugal pumps, from the surge tank and then diluted en route to the cell. Dilution is done with DI feed via a control valve, where dilution flow rate is controlled by the feed density measured by a coriolis flow meter. The outlet flow rate and density is also measured by a coriolis flow meter; these Coriolis flow meters also provide a continuous measure of the cathode exchange membrane (CEM) efficiency which is confirmed by sample titrations at cell inlet and outlet.

Caustic feed is heated/cooled by a steam/cooling water fed heat exchanger. During start-up the caustic is heated and during operation, where some heat is generated in the Electrolyzer, the caustic is cooled. Hydrogen produced the cathode is separated in a liquid/gas separator before the caustic returns to the surge tank.

Copper Chloride (Anode): Copper aqueous solution is pumped, using redundant centrifugal pumps, from the copper feed tank to the cathode. Feed temperature is controlled by a steam fed heat exchanger and flow rate is controlled by a control valve. About a 1/3 of the anode outlet is sent to Catalysis, and 2/3 is recirculated back to the tank. This ratio is maintained by controlling to a constant level in the copper feed tank and setting the copper feed rate to E-Chem to 3x the copper feed rate out of the catalysis feed tank. Mixing in the tank is improved by recirculation of some of the E-chem feed back to the tank. The improved mixing gives a constant copper composition in the E-chem feed.

The mass balance around the anode compartment is accomplished by flow meters and sample ports at anode inlet and outlet to provide mass flow rate and composition of the copper stream as well as indicate any process upsets.

5.1.3 Catalysis Plant Design

The catalysis system is comprised of three subunits: a catalyst feed unit, reactor, and s product recovery/distillation unit. The Catalysis system operates at much higher temperatures than the E-Chem system. At a commercial plant, most of the heating energy will be supplied by heat exchanging the reactor and distillation outlet streams with the reactor feed stream. Sizing those recuperating heat exchangers is fairly straight forward and their operation does not need to be proven in the demo scale. In order to save capital cost and shorten start up times, the ABLE demonstration plant does not include heat recovery and all heating is done by steam.

Catalyst feed unit: The catalyst copper feed tank provides hold-up time to buffer between the E-chem system and the Catalysis system. The tank is maintained at 90^0C by recirculation loop heated by a steam fed heat exchanger. Copper solution from the tank is fed at high pressure to the reactor using redundant multi-stage centrifugal pumps. Flow rate is controlled by pump VFD and reactor feed temperature is controlled by a steam fed heat exchanger.

Catalyst reactor: A trickle bed reactor is used to react ethylene gas with $CuCl_2$ in the liquid copper solution to make EDC and CuCl. The reactor is maintained under constant ethylene pressure by a pressure control valve on the gas inlet stream and a flow control valve on the gas outlet stream which is used to purge inert gases from the system. A rupture disk/relief valve combination protects the reactor from overpressure. A liquid level is kept at the bottom of the reactor to seal the ethylene gas in the reactor. The level is regulated by a level control valve and differential pressure transmitters placed across the vapor phase of the packed bed and the liquid outlet. The copper (I)-rich catalyst and organic product is cooled with an air cooled heat exchanger to distillation operating temperature in order to simulate heat integrated operation. The outlet flow rate is measured to provide a liquid mass balance and a means to identify process upsets.

The Catalysis system requires materials of construction that would stand up to greater pressures and temperatures while maintaining corrosion resistance. The design point for this equipment is 400 psig at 185°C.

Distillation: The distillation column recovers EDC from the reactor product to produce a bottom stream of a copper solution free of EDC and an overhead two phase stream. The overhead stream is condensed, cooled and separated to two liquid phases in a separation vessel. The aqueous phase contains small amount of EDC and reaction by-products and is sent to waste or recycled back to column. The organic phase contains 99wt%+ EDC and is sent to the EDC purification train of the exiting Chlor-Alkali plant.

The distillation column includes a packed bed that is located under the feed nozzle and a Kettle reboiler. As the copper solution flows down the bed, steam rising from the reboiler remove/strip the EDC from the copper. The amount of steam generated in the reboiler is controlled by a steam control valve feeding the tube side of the kettle. The level in the reboiler is measured using a differential pressure transmitter and controlled with a level control valve. The bottoms stream leaving the reboiler is cooled to 90°C in an air fed heat exchanger and sent to the E-Chem feed tank.

5.2 Design of a Full Scale ABLE-CMAP Plant

This section describes a combined ABLE-CMAP process for the production of EDC and $CaCO_3$ cement fiber board. In this process, the ABLE process generates EDC, NaOH, and H_2 from NaCl and ethylene. The NaOH is then used in the CMAP process to capture CO_2, followed by formation of a $CaCO_3$ cement slurry and formation of cement fiber board. A conceptual design is provided in this section, including a block flow diagram, design basis, and mass/energy balance. These analyses are used in section 7.2 as a starting point for the techno-economic analysis of this combined process.

5.2.1 Process Description

An overview of the proposed process is presented in Figure 5-5 at the block flow diagram level.

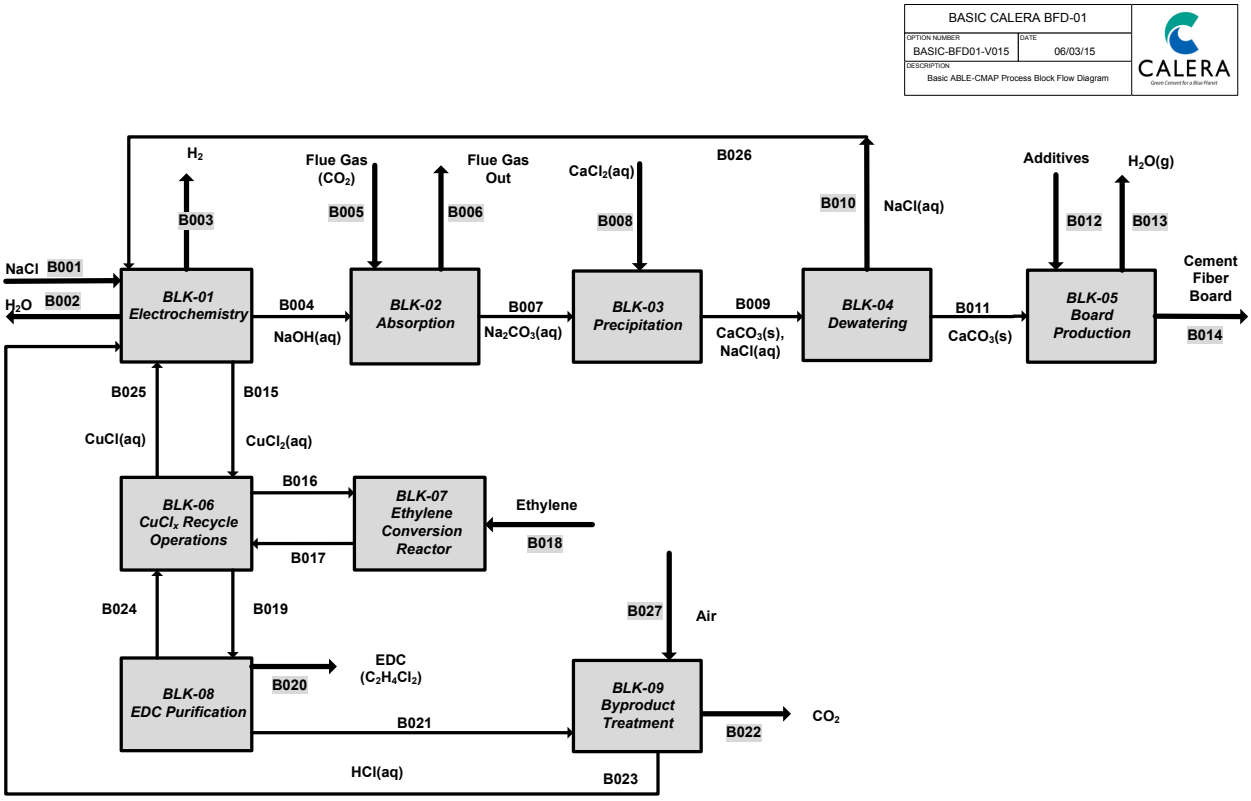


Figure 5-4 Calera Process Block Flow Diagram

The overall chemistry of the process in Figure 5-5 can be (ignoring for the moment byproducts and additives) in the simplest terms summarized as:

$$CaCl_2 + CO_2 + H_2O + C_2H_4 \rightarrow CaCO_3 + C_2H_4Cl_2 + H_2 \qquad (5.2.1\text{-}1)$$

From an overall perspective, the process thus takes calcium chloride, carbon dioxide, ethylene, and water and produces EDC ($C_2H_4Cl_2$), hydrogen, and calcium carbonate (in the form of a fiber cement board product). The overall reaction 5.2.1-1 can be expanded as 5.2.1-2:

$CaCl_2 + CO_2 + H_2O + C_2H_4 + additives \rightarrow CaCO_3$-Fiber Board + $C_2H_4Cl_2 + H_2$ (5.2.1-2)

A description of the proposed process may begin with the electrochemistry process (BLK-01), in which sodium chloride and water are reacted in an electrochemical process with recycled CuCl(aq) to produce $CuCl_2$(aq), sodium hydroxide (NaOH), and hydrogen via reaction 5.2.1-3:

$2\ NaCl(aq) + 2\ H_2O + 2\ CuCl(aq) \rightarrow 2\ NaOH(aq) + H_2(g) + 2\ CuCl_2(aq)$ (5.2.1-3)

The NaOH(aq) product formed at the cathode of the electrochemical process is fed to an absorber (BLK-02) in which the NaOH is contacted with flue gas, resulting in the capture of carbon dioxide and the formation of sodium carbonate (Na_2CO_3) via reaction 5.2.1-4:

$2\ NaOH(aq) + CO_2(g) \rightarrow Na_2CO_3(aq) + H_2O(l)$ (5.2.1-4)

In the precipitator (BLK-03), the sodium carbonate formed in BLK-02 is combined with calcium chloride ($CaCl_2$), resulting in the formation of a cementitious calcium carbonate ($CaCO_3$) via 5.2.1-5:

$Na_2CO_3(aq) + CaCl_2(aq) \rightarrow CaCO_3\ (s) + 2\ NaCl(aq)$ (5.2.1-5)

Following the precipitator (BLK-03), the cementitious calcium carbonate ($CaCO_3$) is dewatered (BLK-04), with the dissolved sodium chloride recycled as a feedstock for the electrochemical process (BLK-01).

After dewatering, the calcium carbonate is combined with various additives and processed to produce a cement fiber board (BLK-05).

Shifting back to the electrochemical unit (BLK-01), the product of the anode containing an aqueous stream rich in $CuCl_2$ passes through the recycle operations (BLK-06), in which, the stream is heated through heat exchange and pumped to the ethylene conversion reactor (BLK-07). In the reactor, ethylene is reacted with the $CuCl_2$-rich stream to produce EDC and CuCl(aq) via 5.2.1-6:

$2\ CuCl_2(aq) + C_2H_4(g) \rightarrow C_2H_4Cl_2(l) + 2\ CuCl(aq)$ (5.2.1-6)

In addition to the formation of the desired product (EDC), a number of byproducts may also be formed in BLK-07 including 2-chloroethanol (CE), cloroacetaldehyde (MCA), dichoroacetaldehyde (DCA), and trichloroacetaldehyde (TCA). The chemical reactions for the production of these byproducts are shown in 5.2.1-7, -8, -9, and -10.

$2\ CuCl_2(aq) + C_2H_4(g) + H_2O(l) \rightarrow C_2H_4Cl(OH)(l) + 2\ CuCl(aq) + HCl(aq)$ (5.2.1-7)

$4\ CuCl_2(aq) + C_2H_4(g) + H_2O(l) \rightarrow C_2H_3ClO(l)$ [MCA]+ $4\ CuCl(aq) + 3\ HCl(aq)$ (5.2.1-8)

$6\ CuCl_2(aq) + C_2H_4(g) + H_2O(l) \rightarrow C_2H_2Cl_2O(l)$ [DCA]+ $6\ CuCl(aq) + 4\ HCl(aq)$ (5.2.1-9)

$8\ CuCl_2(aq) + C_2H_4(g) + H_2O(l) \rightarrow C_2HCl_3O(l)\ [TCA] + 8\ CuCl(aq) + 5\ HCl(aq)$ (5.2.1-10)

After the reactor (BLK-07), the liquid stream is fed to BLK-06 (recycle operations) in which organics are removed from the aqueous stream. The CuCl-rich aqueous stream is returned to the electrochemical unit (BLK-01) and the organic product and byproducts are fed to the purification system (BLK-08). In the purification system, the EDC product is purified to meet the required specification and byproducts are separated and fed to a treatment unit (BLK-09). As in the currently used oxychlorination process for the production of EDC, BLK-09 will incinerate byproducts, resulting in the production of a small amount of CO_2 as well as an aqueous stream containing HCl. The HCl(aq) will be recycled to the electrochemical unit where the chlorine can be utilized for the production of EDC, although without a corresponding unit of NaOH. In locations with a high demand for relatively concentrated HCl, the HCl from the treatment process (BLK-09) may be sold separately; however, the base case conservatively assumes that the HCl is recycled.

5.2.2 Process Basis

The scale of operation is an important variable for estimating process economics. With two principle products, EDC and cement fiber board, one must propose a process basis that is consistent with the market sizes for both products. Starting with EDC, note that a typical new plant size is typically consistent with an associated chlor-alkali capacity of 360,000 tonne Cl_2/yr capacity (capacity defined at 100% plant utilization/capacity). The resulting EDC capacity of a typical new plant is, thus, 400,000 tonne EDC/yr (also defined at 100% plant utilization).

With a typical plant capacity factor of 88.5%, the actual plant output would be 354,000 tonne EDC/yr. Using the stoichiometry of the process, this output corresponds to 358,000 tonne/yr of $CaCO_3$ in the cement fiber board. This output is compatible with the size of the cement board product markets and further justification and support can be found in section 7.1.

5.2.3 Process Performance Assumptions

The experimental program for the validation of process performance assumptions is discussed in the preceding sections of this document with additional documentation in previous reports to DOE.

Table 5-1 Summary of process performance assumptions

Operation	Parameter	Value
1-Electrochemistry	Cell Voltage	2.3 V
1-Electrochemistry	Current Density	300 mA/cm^2
2-Absorption	CO_2 capture	80%
3-Precipitation	Na_2CO_3 conversion	>99.8%
3-Precipitation	Residence Time	5 min
4-Dewatering	Solids %	30 wt%
5-Board Production – Product Properties	Compatible with Cement Fiber Board Specifications	Compatibility verified
5-Board Production-Process Performance	Fuel Demand	2 MMBTU/tonne of Cement Fiber Board
6-CuClx Recycle Operations – Adsorption and VLE, LLE	Residence Time in Adsorption	<2 min
7-Ethylene Conversion Reactor	Space-time-yield	0.85 mol EDC/liter/h
7-Ethylene Conversion Reactor	EDC selectivity projected to pilot scale reactor	97.2%

5.2.4 Mass and Energy Balance

An overall mass and energy balance for the 400,000 tonne EDC plant is given in Table 5-2 with inputs, outputs, and utilities expressed in four different bases (per 1 tonne each of EDC, CO_2, $CaCO_3$, and fiber cement board).

Table 5-2 Overall mass and energy balance for the process depicted in figure 5-5

Basis	per 1 tonne EDC	per 1 tonne CO2	per 1 tonne CaCO3	per 1 tonne FCB
Material Inputs				
tonne NaCl	1.244	2.845	1.180	0.885
tonne C2H4	0.293	0.671	0.278	0.209
tonne CO2 (net)	0.437	1.000	0.415	0.311
tonne CaCl2	1.210	2.769	1.148	0.861
tonne Additives	0.351	0.804	0.333	0.250
Material Outputs				
tonne H2	0.021	0.049	0.020	0.015
tonne CaCO3	1.054	2.411	1.000	0.750
tonne Cement Fiber Board	1.405	3.215	1.333	1.000
tonne EDC	1.000	2.288	0.949	0.712
Energy Inputs				
kWh electricity	1375.561	3147.420	1305.191	978.893
tonne 50 psig steam	0.080	0.183	0.076	0.057
tonne 200 psig steam	0.237	0.542	0.225	0.169
GJ Fuel	2.110	4.828	2.002	1.502
m3 cooling water	90.710	207.553	86.069	64.552

Table 5-3 provides additional detail on the utilities required by each process block.

Table 5-3 Additional details on the utilities breakdown by process block

			Steam			
Block		Electricity	50 psig	200 psig	Fuel	Cooling Water
Num	Name	kWh	tonne	tonne	GJ	m3
BLK-01	Electrochemistry	1,027				10.0
BLK-02	Absorption	49				4.5
BLK-03	Precipitation	4				
BLK-04	Dewatering	21				
BLK-05	Board Production	14	0.03		2.11	
BLK-06	CuClx Recycle Operations	73				11.8
BLK-07	Ethylene Conversion Reactor	56				
BLK-08	EDC Purification	123	0.05	0.237		59.9
BLK-09	Byproduct Treatment	8				4.5
TOTAL		1,376	0.08	0.237	2.11	90.7

Table 5-4 gives a full material balance for the process on the following pages.

Table 5-4 Detailed material balance

		B001	B002	B003	B004	B005	B006	B007
Mass Flow	TONNE/ HR	67.810	200.000	1.169	203.020	250.000	271.797	181.224
Volume Flow	L/MIN	522	3336	288279	3068	3775990	4966720	2445
Mass Density	GM/CC	2.163	0.999	0.000	1.103	0.001	0.001	1.235
Pressure_PSIA	PSIA	14.696	14.696	14.696	14.696	14.696	14.696	14.696
Pressure_ATM	ATM	1.000	1.000	1.000	1.000	1.000	1.000	1.000
Temperature	C	15.000	15.000	90.000	90.000	48.889	69.223	69.223
Component Mass Fraction								
H2O		0.0000	1.0000	0.0000	0.7714	0.0400	0.2109	0.6607
CO2		0.0000	0.0000	0.0000	0.0000	0.1600	0.0532	0.0000
N2		0.0000	0.0000	0.0000	0.0000	0.7800	0.7174	0.0000
O2		0.0000	0.0000	0.0000	0.0000	0.0200	0.0184	0.0000
C2H4		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
EDC		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
CE		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
MCA		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
DCA		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
TCA		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
HCL		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H3O+		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
NAOH		0.0000	0.0000	0.0000	0.2286	0.0000	0.0000	0.0001
CUCL		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
CUCL2		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
CACO3		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
CACL2		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.3392
NACL		1.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
CL-		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H2		0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000
ADDITIVES		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

		B008	B009	B010	B011	B012	B013	B014
Mass Flow	TONNE/ HR	966.000	1147.224	986.088	161.136	2.001	33.315	129.82
Volume Flow	L/MIN	15515	18020	16562	1457	13	1070170	112625
Mass Density	GM/CC	1.038	1.061	0.992	1.843	2.648	0.001	0.019
Pressure_PSIA	PSIA	14.696	14.696	14.696	14.696	14.696	14.696	14.696
Pressure_ATM	ATM	1.000	1.000	1.000	1.000	1.000	1.000	1.000
Temperature	C	15.000	40.000	40.000	40.000	15.000	150.000	4100.16
Component Mass Fraction								
H2O		0.9317	0.8889	1.0000	0.2088	0.0000	1.0000	0.0026
CO2		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
N2		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
O2		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
C2H4		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
EDC		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
CE		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
MCA		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
DCA		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
TCA		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
HCL		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H3O+		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
NAOH		0.0000	0.0000	0.0000	0.0001	0.0000	0.0000	0.0001
CUCL		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
CUCL2		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
CACO3		0.0000	0.0501	0.0000	0.3567	0.0000	0.0000	0.4427
CACL2		0.0683	0.0020	0.0000	0.0141	0.0000	0.0000	0.0175
NA2CO3		0.0000	0.0005	0.0000	0.0038	0.0000	0.0000	0.0047
NACL		0.0000	0.0585	0.0000	0.4165	0.0000	0.0000	0.5170
CL-		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H2		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
ADDITIVES		0.0000	0.0000	0.0000	0.0000	1.0000	0.0000	0.0154

		B015	B016	B017	B018	B019	B020	B021
Mass Flow	TONNE/HR	2063.620	2063.620	2079.620	16.000	2079.620	54.531	2.751
Volume Flow	L/MIN	22705	23624	24539	8889	23524	798	44
Mass Density	GM/CC	1.515	1.456	1.412	0.030	1.473	1.139	1.053
Pressure_PSIA	PSIA	14.696	220.439	220.439	314.696	220.439	220.439	220.439
Pressure_ATM	ATM	1.000	15.000	15.000	21.414	15.000	15.000	15.000
Temperature	C	90.000	150.000	150.000	15.000	90.000	90.000	90.000
Component Mass Fraction								
H2O		0.4955	0.4955	0.4912	0.0000	0.4912	0.0000	0.3712
CO2		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
N2		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
O2		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
C2H4		0.0000	0.0000	0.0000	1.0000	0.0000	0.0000	0.0291
EDC		0.0000	0.0000	0.0262	0.0000	0.0262	1.0000	0.0198
CE		0.0000	0.0000	0.0003	0.0000	0.0003	0.0000	0.2325
MCA		0.0000	0.0000	0.0001	0.0000	0.0001	0.0000	0.0486
DCA		0.0000	0.0000	0.0002	0.0000	0.0002	0.0000	0.1165
TCA		0.0000	0.0000	0.0002	0.0000	0.0002	0.0000	0.1824
HCL		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
H3O+		0.0000	0.0000	0.0004	0.0000	0.0004	0.0000	0.0000
NAOH		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
CUCL		0.0655	0.0655	0.1207	0.0000	0.1207	0.0000	0.0000
CUCL2		0.3179	0.3179	0.2398	0.0000	0.2398	0.0000	0.0000
CACO3		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
CACL2		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
NACL		0.1211	0.1211	0.1202	0.0000	0.1202	0.0000	0.0000
CL-		0.0000	0.0000	0.0007	0.0000	0.0007	0.0000	0.0000
H2		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
ADDITIVES		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

		B023	B024	B025	B027	B028
Mass Flow	TONNE/HR	1.994	2012.123	2024.332	8.000	8.757
Volume Flow	L/MIN	10804	22506	22724	109408	148925
Mass Density	GM/CC	0.003	1.490	1.485	0.001	0.001
Pressure_PSIA	PSIA	14.696	220.439	14.696	14.696	14.696
Pressure_ATM	ATM	1.000	15.000	1.000	1.000	1.000
Temperature	C	90.000	90.000	90.354	15.000	90.000
Component Mass Fraction						
H2O		0.4168	0.5021	0.5044	0.0200	0.0602
CO2		0.0000	0.0000	0.0000	0.0000	0.1939
N2		0.0000	0.0000	0.0000	0.7900	0.7217
O2		0.0000	0.0000	0.0000	0.1900	0.0243
C2H4		0.0000	0.0000	0.0000	0.0000	0.0000
EDC		0.0000	0.0000	0.0000	0.0000	0.0000
CE		0.0000	0.0000	0.0000	0.0000	0.0000
MCA		0.0000	0.0000	0.0000	0.0000	0.0000
DCA		0.0000	0.0000	0.0000	0.0000	0.0000
TCA		0.0000	0.0000	0.0000	0.0000	0.0000
HCL		0.2925	0.0000	0.0000	0.0000	0.0000
H3O+		0.1015	0.0004	0.0006	0.0000	0.0000
NAOH		0.0000	0.0000	0.0000	0.0000	0.0000
CUCL		0.0000	0.1247	0.1240	0.0000	0.0000
CUCL2		0.0000	0.2478	0.2463	0.0000	0.0000
CACO3		0.0000	0.0000	0.0000	0.0000	0.0000
CACL2		0.0000	0.0000	0.0000	0.0000	0.0000
NA2CO3		0.0000	0.0000	0.0000	0.0000	0.0000
NACL		0.0000	0.1242	0.1235	0.0000	0.0000
CL-		0.1892	0.0007	0.0012	0.0000	0.0000
H2		0.0000	0.0000	0.0000	0.0000	0.0000
ADDITIVES		0.0000	0.0000	0.0000	0.0000	0.0000

6. DESIGN OF A FULL-SCALE WASTE OXIDE TO FIBER CEMENT BOARD PRODUCTION FACILITY

Work has begun to design a commercial scale waste oxide processing plant capable of producing 50,000 tonnes of dry $CaCO_3$ per year. The block flow diagram, shown in Figure 6-1, shows a simplified version of the commercial process. The scale up to a commercial size plant requires some process changes from the pilot waste oxide processing plant detailed in section 2.3. The changes are to the waste oxide feed into the dissolution reactor and the solid impurities removal equipment. The waste oxide will be fed into the reactor as a dry solid instead of slurry in order to minimize the dilution of the re-circulated ammonium chloride stream. Feeding the waste oxide into the dissolution reactor as slurry is simple and effective in the pilot plant. However, any water that comes in with the waste oxide needs to exit the system otherwise the water is carried into the recirculation loop and dilutes the NH_4Cl. In order to reduce capital expenditures a centrifuge will be used to remove the solid impurities instead of a pressure leaf filter. A decanter centrifuge is capable to continuous operation unlike a pressure leaf filter which needs to be operated in batch mode in order to dry and remove the solids. Pressure leaf filters also have physical limits on how large they can be built. A plant of this size would require multiple units to achieve an adequate filtration rate.

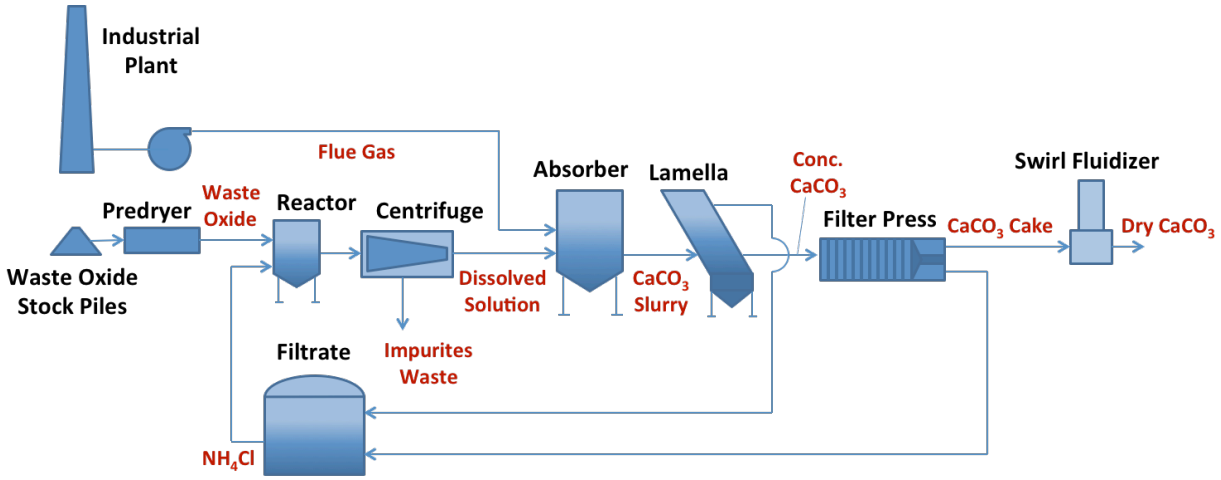


Figure 6-1 Waste Oxide to $CaCO_3$ subsystem Block Flow Diagram (BFD)

Table 6-1 Waste oxide to $CaCO_3$ subsystem mass balance

PROPERTIES	UNITS	WASTE OXIDE TO REACTOR	NH_4CL TO REACTOR	SOLUTION TO CENTRIFUGE	IMPURITES TO DISPOSAL	SOLUTION TO ABSORBER	FLUE GAS TO ABSORBER	$CaCO_3$ SLURRY TO LAMELLA	CONC. $CaCO_3$ TO FILTER	$CaCO_3$ CAKE TO SW. FLUID.	CaCO3 PRODUCT
TOTAL DESIGN FLOW	LBS/HR (CFM)	14,068	235,376	249,444	1,876	247,568	(83,998)	250,000	83,333	15,625	12,626
WATER (SOLUTION)	LBS/HR	2,814	208,848	(248,318)	750	(247,568)	0	210,733	62,850	3,125	126
$Ca(OH)_2$	LBS/HR	11,254	0	0	0	0	0	0	0	0	0
NH_4Cl	LBS/HR	0	26,528	0	0	0	0	26,767	7,983	0	0
$CaCl_2$	LBS/HR	0	0	13,883	0	13883	0	0	0	0	0
$CaCO_3$	LBS/HR	0	0	0	0	0	0	12,500	12,500	12,500	12,500
CO2	LBS/HR	0	0	0	0	0	5,496*	0	0	0	0
OTHER	LBS/HR	0	0	1,125	1,125	0	0	0	0	0	0
WT % SOLIDS	%	80	N/A	0.45	60	0	N/A	5	15	80	99
TOTAL VOLUME FLOW	GPM (CFM)	N/A	448	475	N/A	471	(83,998)	476	151	N/A	N/A
FLUID PROPERTIES											
TEMPERATURE	zF	X	X	X	X	X	X	X	X	X	X
PRESSURE	PSIG	X	X	X	X	X	X	X	X	X	X
SPECIFIC GRAVITY @ TEMP		X	1.050	1.050	X	1.050	1.014	1.050	1.100	X	X

The dry $CaCO_3$ product from the Waste Oxide Processing Plant will be used as the binder in the Fiber Cement Board process detailed in Figure 6-2. The $CaCO_3$ will be mixed with refined cellulose slurry in the universal mixer. From there the slurry will be diluted in a homogenizer tank and fed to a Hatschek sheeting machine to produce fiber cement boards. After the boards are formed on the sheeting machine they will be stacked and moved into an autoclave for curing.

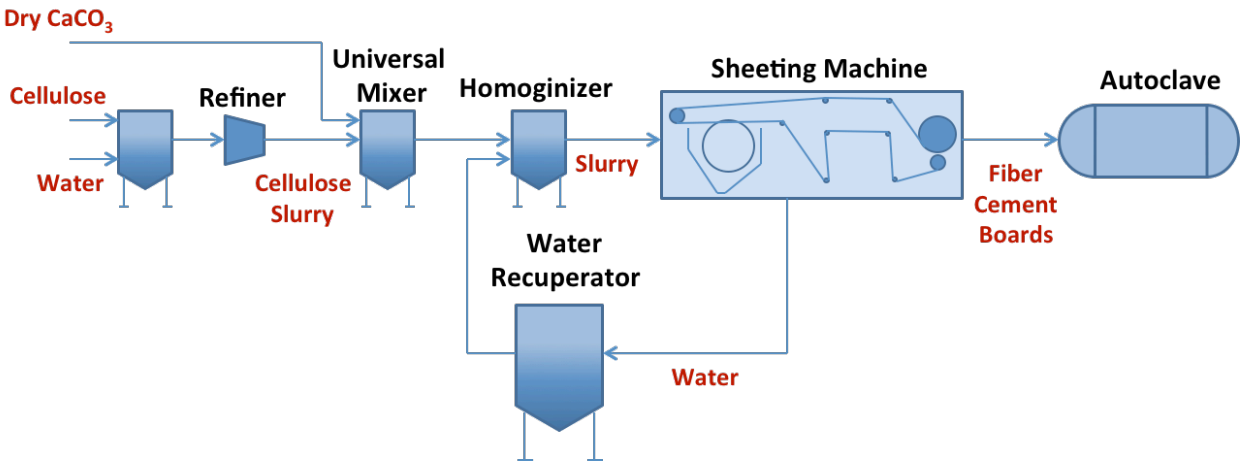


Figure 6-2 Fiber cement board subsystem Block Flow Diagram (BFD)

7.0 TECHNO-ECONOMIC ANALYSIS AND CO_2 LIFECYCLE ANALYSIS

7.1 Carbide Lime to Fiber Cement Boards

7.1.1 Availability of Raw Materials

Carbide lime – $Ca(OH)_2$ – is a by-product essentially associated with the production of acetylene – C_2H_2 – from calcium carbide – CaC_2. Calcium carbide is itself produced from metallurgical coke and limestone in an electric arc furnace.

In the first half of the 20th century, coal-based acetylene was the most common starting material for organic synthesis. Production reached a peak in the 1960s and has steadily declined since then. This evolution was due to the substitution of acetylene by olefins such as ethylene and propylene, driven by lower production cost, lower environmental impact and greater safety. Since, the chemicals industry has fully transitioned in Western countries such as the US and Germany. Memories of past industrial activities may be seen in these countries in the form of waste ponds of carbide lime slurry. Indeed, by-product carbide lime used to be landfilled. Some ponds are estimated to contain several million metric tons of waste materials.

Only China offers a different industrial landscape. The coal-based acetylene route still dominates the domestic chemicals industry, not only because of the lack of natural gas and oil, but also the abundance of low-cost coal. Polyvinyl chloride (PVC), polyvinyl acetate (PVA), calcium cyanamide and 1,4-butanediol (1,4-BDO) are largely produced from calcium carbide. As an example, more than 80% of Chinese PVC was produced from coal-based acetylene in 2013.

Today, approximately 20 million metric tons of carbide lime is generated in China, mostly in coal-rich provinces such as Inner Mongolia, Xinjiang, Ningxia, Shanxi and Sichuan. Regulators are progressively forcing emitters to recycle carbide lime, because of the environmental issues associated with disposal. The most common solution is partial substitution of limestone for cement production. Cement performance is usually negatively impacted, but direct carbon dioxide emissions are lowered.

7.1.2 Market Analysis for Fiber Cement Boards

The cement produced by the Calera process can replace existing inorganic binders, such as gypsum, magnesium oxide, calcium silicate and portland cement in a variety of board building products.

These products are defined by their two-dimensional shape (small thickness with respect to the two other dimensions) and are ubiquitous worldwide for a diversity of exterior and interior applications: roofing, siding, shingle, soffit, trim, fascia, sheathing, decking, ceiling, interior cladding, wall partitioning, tile backer, and underlayment. Various material types can be used in these applications (e.g., wood, mineral, polymer), though inorganic binders are either dominating, or increasingly more common. By volume, the main applications for inorganic binders are wall partitioning, external cladding, roofing, tile backer, and underlayment.

Because of the brittle nature of cementitious binders, reinforcement is required for board products. The most common methods are paper facing (e.g., plasterboard or gypsum drywall),

fiberglass mat or mesh facing (e.g., gypsum sheathing and cement boards) and core-reinforcement with fibers (e.g., gypsum fiberboard and fiber cement). In the later category, cellulosic and synthetic fibers are generally employed today. These fibers have largely replaced asbestos fibers.

7.1.2.1 The US Market

Fiber cement products were introduced in the US market in the early 1990s. Since, they have gained considerable ground. Demand for fiber cement was estimated around 2.2 million metric tons in 2012 and was valued at $1.2 billion. The residential segment represented about three-quarters of the US demand, essentially in the repair and remodeling sub-segment.

The main application today is siding, which accounted for nearly 70% of total demand in 2012. In this category, fiber cement products offer lower maintenance and greater performance than wood siding, lower cost than brick, and greater aesthetic advantages. The second major market is tile backer, which accounted for about 25% of total demand in 2012. Fiber cement tile backer products show greater moisture resistance, lower cost and are easier to handle than wood and gypsum alternatives. Fiber cement products are gaining market shares in each application.

The industry is highly concentrated and two companies control approximately 90% of the US supply. US demand is forecast to rise 8.5% annually and reach 3.4 million metric tons in 2017. Market size would then reach $2.2 billion.

7.1.2.1 Global Market

Fiber cement products are popular building materials in the world. 2014 global demand for fiber cement products is estimated around 26-31 million metric tons. The main applications are roofing and siding and the Asia/Pacific region represents about 50% of global demand. Global demand is forecast to reach 32-38 million metric tons in 2019.

The industry has been facing a major restructuration associated with the transition from asbestos fibers to cellulosic and synthetic fibers. This transition started decades ago, but is still happening in some countries today.

In China, the production of fiber cement products is estimated around 8-12 million metric tons today, with asbestos fiber-reinforced cement products accounting for 70-80% of total production. The industry is highly fragmented with small, specialty players. Asbestos-free cement products are considered as new building materials and have shown net market share growth in recent years. Chinese and foreign companies have both entered this new market.

Fiber cement products are classified in the building panel product category in China, which represents a $15 billion market. It is forecast to rise 20% annually and reach a $40 billion market size in 2017.

7.1.3 Process Economics

7.1.3.1 Techno-Economic Analysis Boundaries and Scenario Presentation

The manufacturing process can be divided in two sequential steps: (1) calcium carbonate cement manufacturing and (2) fiber cement boards manufacturing from the calcium carbonate cement. The second step is almost identical to the traditional board process utilizing portland cement. As a consequence, capital expenditures and operating expenditures for the Calera fiber cement boards are expected to be equivalent to traditional fiber cement boards, except for cement economics. Therefore, this section only details the economics of manufacturing the calcium carbonate cement.

The techno-economic analysis is based on a China scenario where the $CaCO_3$ cement plant is co-located to the CO_2 emitting source. Flue gas is supplied for free. Other inputs – including carbide lime – are supplied by road transportation. They are all purchased at standard market prices, except carbide lime that is supplied for free. Carbide lime transportation cost is included in the analysis and corresponds to the shipment of carbide lime by truck or train on a short distance. This scenario is highly probable as carbide lime and carbon dioxide emissions are often co-located in China.

Techno-economic calculations are based on a size of 500,000 metric tons per year (calcium carbonate cement basis) with a capacity factor of 85%. This plant is referred as 'commercial plant'. Economic impact of operating parameters such as raw materials cost, labor cost, maintenance cost, general and administrative expense, and depreciation expense are included. This analysis is a cost model and does not include revenues from either sales or carbon credits. Moreover access to capital is assumed free. The study indeed does not include financial expenses associated with debt financing for instance. Hence, capital expenditures are close to an overnight cost.

7.1.3.2 Capital Expenditures Estimation

Equipment costs were obtained from Chinese collaborators for a plant producing 50,000 metric tons per year of calcium carbonate cement, referred here as 'demo plant'. An approximation of the equipment cost for the larger commercial plant is determined by using the classical following exponential scaling expression:

$$Equipment\ Cost_{Commercial\ Plant} = Equipment\ Cost_{Demo\ Plant} \times \left(\frac{Commercial\ Plant\ Scale}{Demo\ plant\ Scale}\right)^{0.65}$$

Capital expenditures are then estimated from equipment cost using typical Lang multiplication (scaling) factors for a solids-fluid processing plant. Total capital cost is estimated around $59-68 million.

7.1.3.3 Mass and Energy Balance

The process requires carbon dioxide (CO_2), carbide lime, energy and minor inputs, such as ammonium chloride, ammonia and flocculants. The mass and energy balance was obtained from the ASPEN model and is presented in Table 7-1. The waste sludge by-product originates from the impurity filtration step and may be further valorized. In the analysis, a conservative scenario is considered, where the sludge is treated at a cost.

Table 7-1 High-level mass and energy balance normalized to 1,000 kg of $CaCO_3$ cement

INPUTS	
Carbon Dioxide	440 kg
Carbide Lime (dry)	863 kg
Ammonium Chloride	3.4 kg
Ammonia	3.5 kg
Flocculants	0.1 kg
Electricity	182 kWh
Fuel	3,210 MJ
OUTPUTS	
Calcium Carbonate Cement	1,000 kg
Waste Sludge	151 kg

7.1.3.4 Operating Expenditures Estimation

Assumptions for the key economic parameters are summarized in Table 7-2. It was estimated that approximately 30 full-time equivalent units of labor are required for the demo plant. Labor for the commercial plant is estimated with the following exponential scaling expression:

$$Labor_{Commercial\ Plant} = Labor_{Demo\ Plant} \times \left(\frac{Commercial\ Plant\ Scale}{Demo\ plant\ Scale}\right)^{0.40}$$

Table 7-2 Main assumptions for economic calculations

		COMMENTS
Carbon Dioxide Price	$0/metric ton	
Carbide Lime Cost of Transportation	$5/metric ton	Local Supply
Ammonium Chloride Price	$250/metric ton	
Ammonia Price	$520/metric ton	
Flocculants Price	$2,200/metric ton	
Electricity Price	$0.104/kWh	
Fuel Price (Coal)	$3.4GJ	Bituminous Coal
Cost of Waste Sludge Disposal	$8/metric ton	Light Treatment Cost
	VALUE	**COMMENTS**
Labor Requirements	76	Full-Time Equivalent (FTE)
Labor Cost (annual)	$15,000	Per FTE
Maintenance Cost	4.0%	Of total direct plant cost
G&A Cost	3.0%	Of total direct plant cost
Depreciation – Service Year	12 years	
Depreciation – Residual Value	0%	

The economic summary is summarized in Table 7-3.

Table 7-3 Summary of operating expenditures normalized to 1 metric ton of $CaCO_3$ cement

Normalized to 1 metric ton of CaCO3		
Cost of Goods Sold		
Raw Materials and Energy	$	32.5
Labor	$	2.3
Maintenance	$	4.0
	$	38.8
Operating Expenses		
General & Administrative	$	3.0
Amortization and Depreciation	$	11.3
	$	14.4
Operating Expenditures	**$**	**53.2**

7.1.4 Life Cycle Story

The calcium carbonate cement manufacturing process captures, converts and stores CO_2 flue gas emissions as mineral $CaCO_3$. Stoichiometrically, the cement is composed of 44 wt% carbon dioxide, which corresponds of 440 kg of CO_2 captured per metric ton of cement.

As mentioned in section 7.1.3, the process requires both electrical energy for mechanical processes and thermal energy for the drying and decomposition steps. Producing 1 metric ton of calcium carbonate cement requires approximately 182 kWh of electricity and 3,210 MJ of fuel. It is not reasonable to expect that renewable energies (e.g., solar, biomass) would be solely used to run the process. Therefore, CO_2 emissions would occur during the manufacturing process. These emissions depend on fuel type, plant location and plant efficiency. The worst-case scenario in China corresponds to a plant running on coal without recovering waste heat energy from a co-located industrial facility. More optimistic scenarios may either consider a lower carbon intensive fuel source (e.g., natural gas), or some waste heat energy supply.

The overall carbon footprint of the process is shown in Figure 7-1 for different scenarios. Calculations are based on emission factors indicated in Table 7-4.

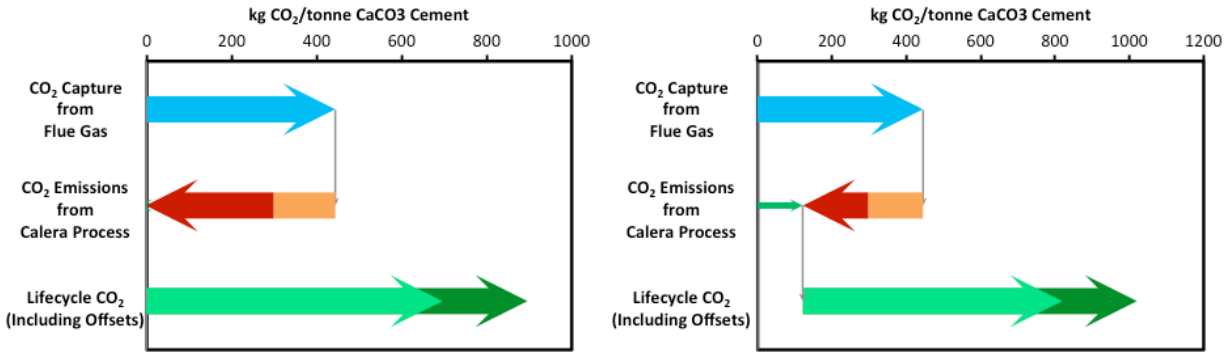


Figure 7-1 Life cycle story of $CaCO_3$ cement manufacture in China for coal (left) and natural gas (right). Breakdown of emissions: electricity (orange color) and fuel (red color). The small green arrow shows net capture CO_2.

Table 7-4 Emission factor assumptions

	EMISSION FACTORS
Fuel Emission Factor (Coal)	340 kg CO_2 per MWh
Fuel Emission Factor (Natural Gas)	200 kg CO_2 per MWh
Electricity Emission Factor, China	800 kg CO_2 per MWh
Offsets, Low (based on cement)	700 kg CO_2 per metric ton
Offsets, High (based on cement)	900 kg CO_2 per metric ton

In the worst-case scenario presented, the calcium carbonate cement is approximately carbon neutral, indicating that as much carbon dioxide is captured than is emitted during the manufacturing process. In a more optimistic scenario, the $CaCO_3$ cement is carbon negative, indicating that more carbon dioxide is captured than is emitted during the manufacturing process. The process results in the net capture of approximately 120 kg of carbon dioxide per metric ton of $CaCO_3$ cement.

The calcium carbonate cement also benefits from CO_2 offsets i.e., avoided carbon dioxide emissions by displacing for instance portland cement in construction materials.

7.2 Techno-Economic Analysis of the ABLE-MAP Process

This section provides a techno-economic analysis of the ABLE-MAP process for the production of EDC and $CaCO_3$ cement fiber board. The starting point for this process is the design details provided in section 5.2.1.

7.2.1 Process Description

The block flow diagram of Figure 5-5 is reproduced below as Figure 7-2 to provide a depiction of the process being analyzed.

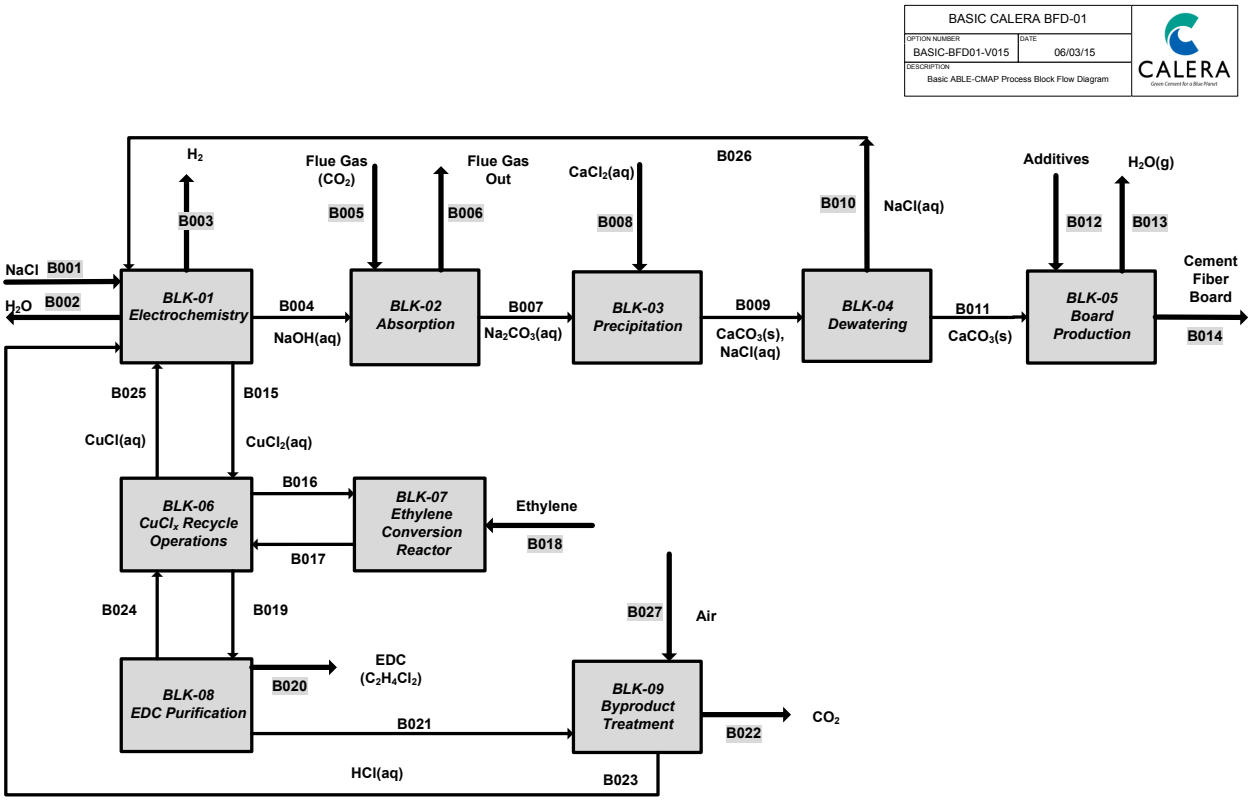


Figure 7-2 Calera process Block Flow Diagram (BFD)

7.2.2 Process Basis

The scale of operation is an important variable for estimating process economics. With two principle products, EDC and cement fiber board, one must propose a process basis that is consistent with the market sizes for both products. Starting with EDC, note that a typical new plant size is typically consistent with an associated chlor-alkali capacity of 360,000 tonne Cl_2/yr capacity (capacity defined at 100% plant utilization/capacity). The resulting EDC capacity of a typical new plant is, thus, 400,000 tonne EDC/yr (also defined at 100% plant utilization).

With a typical plant capacity factor of 88.5%, the actual plant output would be 354,000 tonne EDC/yr. Using the stoichiometry of the process, this output corresponds to 358,000 tonne/yr of $CaCO_3$ in the cement fiber board. This output is compatible with the size of the cement board product markets and further justification and support can be found in section 7.1.

7.2.3 Mass and Energy Balance

The overall mass and energy balance for the 400,000 tonne EDC plant is given in Table 7.2.3-1 with inputs, outputs, and utilities expressed in four different bases (per 1 tonne each of EDC, CO_2, $CaCO_3$, and fiber cement board).

Table 7-5 Overall mass and energy balance for the process depicted in figure 7-2

Basis	per 1 tonne EDC	per 1 tonne CO2	per 1 tonne CaCO3	per 1 tonne FCB
Material Inputs				
tonne NaCl	1.244	2.845	1.180	0.885
tonne C2H4	0.293	0.671	0.278	0.209
tonne CO2 (net)	0.437	1.000	0.415	0.311
tonne CaCl2	1.210	2.769	1.148	0.861
tonne Additives	0.351	0.804	0.333	0.250
Material Outputs				
tonne H2	0.021	0.049	0.020	0.015
tonne CaCO3	1.054	2.411	1.000	0.750
tonne Cement Fiber Board	1.405	3.215	1.333	1.000
tonne EDC	1.000	2.288	0.949	0.712
Energy Inputs				
kWh electricity	1375.561	3147.420	1305.191	978.893
tonne 50 psig steam	0.080	0.183	0.076	0.057
tonne 200 psig steam	0.237	0.542	0.225	0.169
GJ Fuel	2.110	4.828	2.002	1.502
m3 cooling water	90.710	207.553	86.069	64.552

7.2.4 Process Economics

A capital cost estimate for a 400,000 tonne EDC/yr plant was developed using ASPEN Process Economic Analyzer in conjunction with conceptual benchmarking versus similar existing processes: 1) membrane chlor-alkali, 2) EDC production via direct chlorination, and 3) fiber cement board production with ordinary Portland cement. The results of the bottoms-up costing are summarized in Table 7-3.

We note that the capital cost estimate for the integrated Calera process is conceptually very similar to a combination of the three processes listed above on the same basis. Roughly speaking, the Calera electrochemical system saves some capital associated with chlorine avoidance while the Calera EDC production and purification units has elevated capital costs owing to new units associated with circulating metal salt solutions. The capital associated with the Calera fiber cement board plant is expected to be quite similar to existing technologies owing to similarities in process steps and conditions.

Table 7-6 Estimated total invested capital for a 400,000 tonne EDC/yr Plant (US Gulf Coast, Q1-2015 basis)

Block		
Num	Name	Total Invested Capital
BLK-01	Electrochemistry	244,000,000
BLK-02	Absorption	31,000,000
BLK-03	Precipitation	18,300,000
BLK-04	Dewatering	32,000,000
BLK-05	Board Production	71,800,000
BLK-06	CuClx Recycle Operations	36,300,000
BLK-07	Ethylene Conversion Reactor	44,000,000
BLK-08	EDC Purification	43,700,000
BLK-09	Byproduct Treatment	6,700,000
TOTAL		527,800,000

Assumptions relevant to the timing of capital expenditures as well as maintenance and refurbishment expenses can be found in Table 7-4. The initial capital investment is spread over a three year period with a 25%/50%/25% split. In addition to an annual maintenance expense of 3.5% of total invested capital that is typical of chemical processes, additional refurbishment costs are incurred at 4 and 8 year intervals for membrane and cell room refurbishment, respectively. These component lifetimes are typical of the similar components employed in chlor-alkali processes.

Table 7-7 Maintenance and capital timing assumptions

Initial CAPEX Investment Timing	
% of initial in year -2	25%
% of initial in year -1	50%
% of initial in year 0	25%
Annual Maintenance (% of Total Invested CAPEX)	3.50%
Membrane Refurbishment	
Start in Year 4	
Repeat every 4 years	
% of Initial Echem CAPEX	5%
Cell Refurbishment	
Start in Year 8	
Repeat every 8 years	
% of Initial Echem CAPEX	10%

Assumptions related to feedstock, product, and utility pricing are tabulated in Table 7-5. Most critically, we note that the base case conservatively assumes no credit for carbon capture or avoidance. Thus, in a scenario where carbon credits are available, additional financial incentive will exist for the deployment of the technology should product markets prove less robust than expected or should the technology be deemed more costly as the scale-up process progresses.

Table 7-8 Feedstock, product, and utility pricing assumptions

CO2 price ($/tonne)	$	-
Natural Gas ($/MMBTU)	$	5
Electricity ($/MWh)	$	55
NaCl ($/tonne)	$	15
Ethylene ($/tonne)	$	701
CaCl2 ($/tonne)	$	44
Weighted avg all-in-labor ($/FTE/yr)	$	70,000
NaOH ($/tonne)	$	300
Hydrogen ($/tonne)	$	482
EDC($/tonne)	$	450
Fiber Cement Board ($/tonne)	$	532

Because of the variable nature of the refurbishment expenses listed in Table 7-5, the process economics are best summarized in a cash flow statement encompassing the construction period as well as 20 years of operation. Such a cash flow statement is displayed in Table 7-6.

Table 7-9 Cash Flow Statement for Proposed Process

		CAPEX ($MM)				OPEX ($MM)						Revenue ($MM)					CASH FLOW ($MM)
						Feedstock			OPEX (ex-feedstock)								
Year		Echem	EDC	CaCO3	TOTAL	C2H4	NaCl	CaCl2	Echem	EDC	CaCO3	TOTAL	EDC	H2	CaCO3	CO2	TOTAL	
-3		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
-2		61.0	32.7	38.2	*131.9*	-	-	-	-	-	-	-	-	-	-	-	-	*(131.9)*
-1		122.0	65.4	76.5	*263.8*	-	-	-	-	-	-	-	-	-	-	-	-	*(263.8)*
0		61.0	32.7	38.2	*131.9*	-	-	-	-	-	-	-	-	-	-	-	-	*(131.9)*
1		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
2		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
3		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
4		12.2	-	-	*12.2*	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	203.7
5		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
6		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
7		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
8		36.6	-	-	*36.6*	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	*179.3*
9		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
10		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
11		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
12		12.2	-	-	*12.2*	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	*203.7*
13		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
14		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
15		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
16		36.6	-	-	*36.6*	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	*179.3*
17		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
18		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
19		-	-	-	-	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	215.9
20		12.2	-	-	*12.2*	81.3	0.5	15.9	35.7	16.9	23.4	*173.7*	180.0	3.8	205.8	-	389.6	*203.7*
Tot		353.8	130.7	153.0	*637.5*	1,627	9	318	713	339	467	*3,473*	3,600	75	4,116	-	7,791	*3,680*
%		55%	21%	24%	*100%*	47%	0%	9%	21%	10%	13%	*100%*	46.2%	1.0%	52.8%	0.0%	100.0%	

The key metrics from the cash flow statement are summarized in Table 7-7, leading to the following observations and conclusions:

1. The projected $528MM capital investments generates $210MM in earnings before interest, taxes, depreciation, and amortization (EBITDA); a healthy 40% ratio
2. An internal rate of return of over 30% is calculated; a level promising for a project at this stage of development and under the assumptions employed.
3. With respect to the assumptions employed, we again note that no credit was taken for carbon dioxide captured or avoided (the latter of which would likely manifest itself in relative cost savings and not a direct cash credit).

Table 7-10 Key financial metrics from cash flow statement

Initial CAPEX	$ 528 MM
EBITDA (AVG)	$ 210 MM
EBITDA/CAPEX	40%
IRR:	30.3%
NPV (@10%)	$831 MM

7.2.5 CO_2 Lifecycle Analysis

Based on the mass and energy balances balance shown in previous sections, a lifecycle analysis is shown graphically below for a the production of EDC and a fiber cement board, displacing an Ordinary Portland Cement product with a footprint of 1.0 tonne of CO_2/tonne FCB. The critical assumptions are the use of US grid electricity with carbon intensity of 0.5 tonne CO_2/MWh and natural gas as the fuel source with footprint of 0.0507 tonne CO_2/GJ.. Additional detail on key assumptions follows the overview.

In Figure 7-3, the top green bar represents gross CO_2 capture. Included here as a penalty is the carbon dioxide emitted from incineration of organic byproducts. Subtracted (moving left) from the green bar, in red, are emissions from the Calera Fiber Cement Process. A credit is then taken in blue for the emissions avoided by displacing traditional Fiber Cement Board. The large value for this component is a function of the use of carbon-intensive ordinary portland cement in the competing technology along with substantial energy for curing and drying. Subtracting Calera EDC emissions (energy in red, ethylene feedstock in gray) is followed by the bottom bar, which adds a credit for avoidance of traditional EDC manufacture.

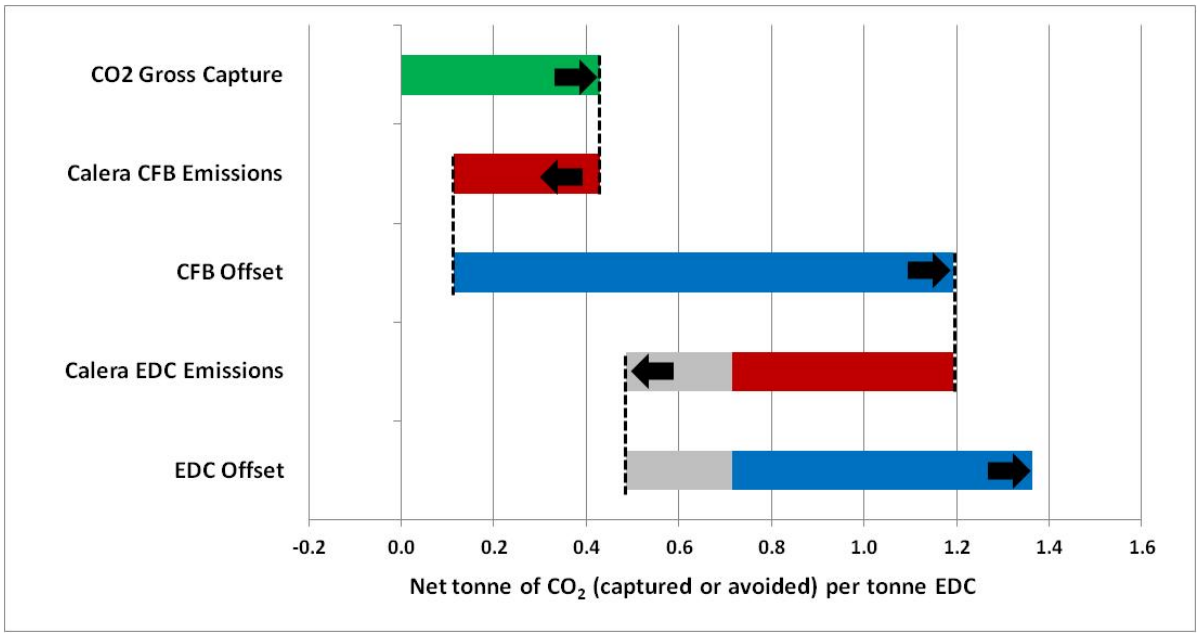


Figure 7-3 Lifecycle CO_2 analysis for Calera EDC + fiber cement board process

Detail behind the carbon intensity of EDC production by traditional means is summarized in Table 7-8.

Table 7-11 Carbon intensity of EDC production (via IEA)

	Electricity (MWh)	Heat MMBTU		tonne CO2
1 tonne Ethylene	0.06	12.19		0.677
1 tonne EDC (ex feedstock)	0.06	4.21		0.256
1 tonne Cl2	1.37	0.27		0.701
0.2845 tonne C2H4	0.39	0.08		0.199
0.72 tonne Cl2	0.99	0.19		0.505
1 tonne EDC process only	0.20	12.80		0.144
+ 0.72 tonne Cl2	0.99	0.19		0.505
1 tonne EDC (ex-C2H4)	1.19	12.99		0.648
+ 0.2845 tonne C2H4	0.39	0.08		0.199
1 tonne EDC (total)	1.58	13.07		0.847

The global potential of the Calera process as depicted is dependent on the market penetration of the EDC market. To generate some simple and approximate order-of-magnitude estimates, we use a static market size estimate for 2016 of 50 MM tonne EDC/yr along with various penetration scenarios to generate estimates in Table 7-9. For example, with a market penetration of 10%, 2.2MM tonne of CO_2 is captured with a total lifecycle impact of 6.8 MM tonne CO_2 captured and avoided per year through the avoidance of carbon intensive competing processes.

Table 7-12 Simplified analysis of potential CO_2 impact vs. EDC market penetration. (Note that this analysis simplistically does not account for market growth, and the conservatism of the lack of market growth may be balanced with a more aggressive assumption for market penetration)

			Annual Carbon Dioxide Impact (MM tonne)			
	Tonne CO2		Versus % Capture of 50MM tonne EDC Market			
	Per Tonne of EDC		1%	5%	10%	20%
Gross CO2 Capture	0.43		0.2	1.1	2.2	4.3
Lifecycle CO2 Captured and Avoided	1.36		0.7	3.4	6.8	13.6

7.2.6 Market Analysis for Ethylene Dichloride (EDC)

EDC is used almost exclusively in the manufacture of vinyl chloride monomer (VCM), which is itself used almost solely in the polymerization manufacture of poly vinyl chloride (PVC). Some key market observations developed in conjunction with external consultantss include:

- EDC is made principally by the direct chlorination or oxychlorination of ethylene
- Most EDC plants are integrated with VCM plants; the VCM process generates considerable quantities of hydrogen chloride (HCl), which is then recycled in the oxychlorination process to generate more EDC
- VCM can also be made by the hydrochlorination of acetylene; this process is not used much in the world except for China where integrated coal chemical sites are under development
- Most producers are balanced, such that direct chlorination capacity is roughly equal to oxychlorination
- The three major exporters of EDC are the United States (mainly to the Republic of Korea and Japan), Germany and Belgium

As depicted in Figure 7-4, external consultants project global EDC demand to grow at about 3% per year in the coming years, projecting a total global market size in excess of 50 million tonnes per year by 2016. Related analysis shown in Figure 7-5, projects North American EDC pricing in the $450 to $500 range by 2016 with higher pricing possible for exports to China.

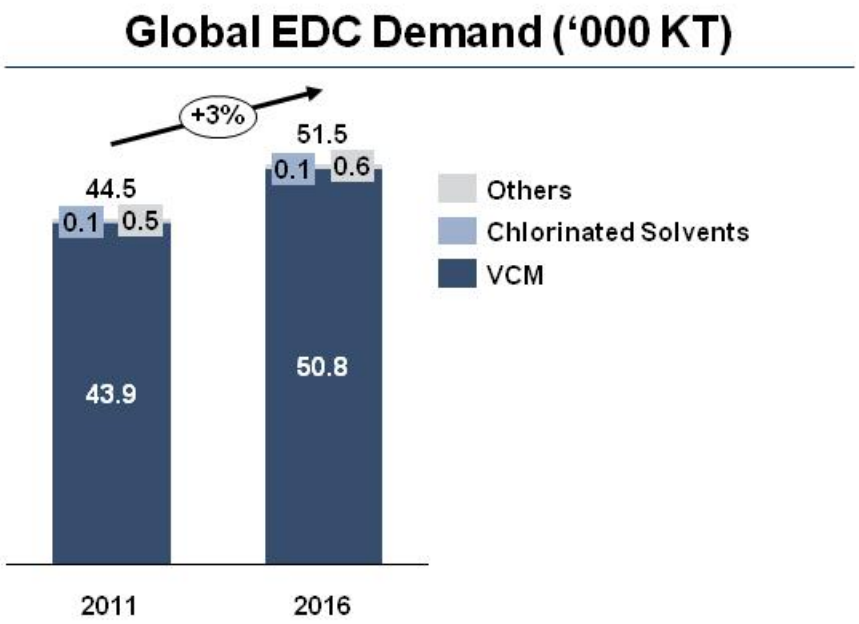


Figure 7-4 Global EDC Demand (projected by external consulting firm)
(KT = 1000 metric tons)

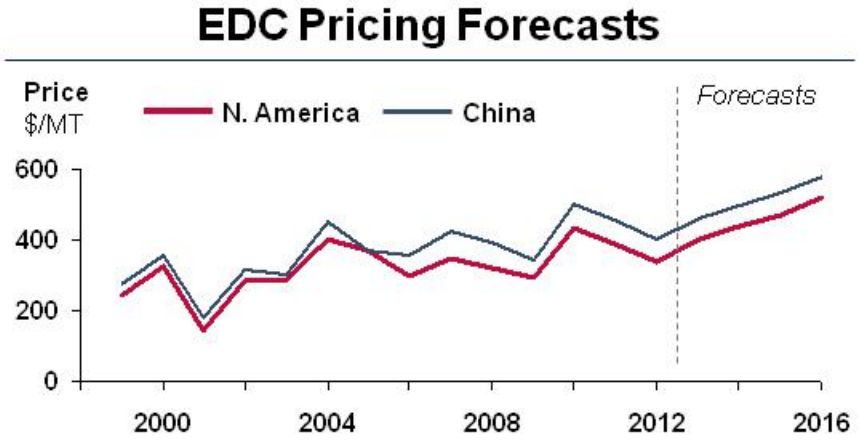


Figure 7-5 EDC Pricing (projected by external consulting firm)

7.2.7 Conclusions for Techno-Economic Analysis and CO_2 Lifecycle Analysis

In summary, the main conclusions of the techno-economic and lifecycle analyses are:

1. Process economics are projected for a 400,000 tonne EDC/yr plant (consistent with current plant scale) and are found to yield favorable projected returns on capital even without carbon credits.
2. The Calera process will have a gross capture of 0.43 tonne CO_2 for every tonne of EDC produced and an even larger impact of 1.36 tonne CO_2 on a lifecycle basis. The larger lifecycle value results from the avoidance of the carbon-intensive intermediates (chlorine and Ordinary Portland Cement) that are used in existing EDC processes and fiber cement board products.
3. If the Calera process were to penetrate 10% of an EDC market that is estimated (2016) to be 50MM tonne/yr, the annual carbon capture would be 2.2MM tonne of CO_2 with a total annual lifecycle impact of 6.8 MM tonne CO_2 captured and avoided.