SKYONIC CORPORATION
FINAL DOE REPORT
DOE AWARD # DE-FEE0002586 - REVISED

Recovery Act: SkyMine® Beneficial CO_2 Use Project

FINAL REPORT

DOE Award Number:	**DE-FE0002586**
Recipient:	**Skyonic Corporation**
Project Officer:	**Vito Cedro NETL/DOE**
Date Report Issued:	**February 15, 2016**
Reporting Period Start Date:	**January 15, 2010**
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Submitted by:

Jerel Walters

Skyonic Corporation
900 S. Capital of Texas Hwy., Suite 475
Austin, TX 78746

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ABSTRACT:
This Final Report addresses accomplishments achieved during Phase 1 and Phase 2 of the SkyMine® Carbon Mineralization Commercial Scale Pilot Project. The primary objectives of this project were to design, construct, and operate a system to capture CO_2 from a slipstream of flue gas from a commercial coal-fired cement kiln, convert that CO_2 into products having commercial value as an example of Carbon Capture and Beneficial Re-Use, show the economic viability of the CO_2 capture and conversion process, and thereby advance the technology to a level of commercial scale completion with the ability for further proliferation. The project also substantiates market opportunities for the technology by sales of chemicals into existing markets, and has identified opportunities to improve technology performance and reduce costs during the normal course of operations. The primary objectives of Phase 1 of the project were to elaborate proven SkyMine® process chemistry to commercial pilot-scale operation and complete the preliminary design for the pilot plant to be built and operated in Phase 2.

During Phase 2, *Pilot System Engineering Design, Construction and Testing*, supporting research and development was conducted at a field test unit to collect data that aided in detailed designs of the commercial scale pilot unit; final detailed designs were completed; the commercial scale pilot plant is in its final stages of construction; and a test program has been designed and will be implemented to collect data from the facility to evaluate the technical viability of the process for resulting in an overall net reduction in CO_2 emitted into the atmosphere through a process that is run and conducted in a carbon negative manner.

The objective of Phase 2b was to build the commercial scale pilot plant to be operated and tested in Phase 2c. By June 30, 2015, the plant was +99% constructed and completed.

Capitol SkyMine

Inputs: CO_2, Salt, Water, Electricity

Primary Outputs:

- Hydrochloric Acid
- Sodium Bicarbonate
- Sodium Hypochlorite Bleach Solution
- Caustic Soda

Designed to directly capture 75k t/yr of CO_2 & offset a total of 300,000 t/yr

- Sale of outputs to generate profits
- Scrubs SO_x, NO_x and metals
- 1st Commercial plant
- Stand-alone technology

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List of Acronyms and Abbreviations

AHR = AustinHR
CCCG = CCC Group
CAU= Chlor-Alkali Unit
CCU= Carbon Capture Unit
CO_2 = carbon dioxide
CFD = Computational Fluid Dynamics
CPM = critical path method
CPS = CPS Energy
CTA = cellulose triacetate
C_T = total concentration
DBA = Davis Bacon Act
DCAA = Defense Contract Auditing Agency
DCS= Distributed Control System
DOE = Department of Energy
EA = Environmental Assessment
EIV = environmental information volume
EPC = engineering, procurement, and construction
EPCM = engineering, procurement, and construction management
ERT = energy recovery turbine
EVMS = earned value management system
FAT = Factory Acceptance Test
FCC = Food Chemical Codex
FEL = front end loaded engineering
FO = Forward Osmosis
FRP = fiber-reinforced plastic
GFD = gallons/sqft*day
HAZOP = Hazard and Operability Study
HCl = hydrochloric acid
Hg = mercury
IFC = issued for construction
LOI = letter of intent
MCC = Motor Control Center
NEPA = National Environmental Policy Act
NETL = National Energy Technology Laboratory
NDA = non-disclosure agreement
NO_X = nitrogen oxides
P&ID = piping and instrumentation diagram
PFD = process flow diagram
PHA = Process Hazards Analysis
PMP = project management plan
PO = purchase order
ppb = parts per billion
PSSR = Pre-startup Safety Review

PVC = polyvinyl chloride
RO = reverse osmosis
SAWS = San Antonio Water System
SO_x = sulfur oxides
SWPPP = storm water pollution prevention plan (or SWP3, or SWP^3)
TDS = total dissolved solids
TFC = thin-film composite
Ton = short ton, unit of weight equal to 2000 pounds
Tonne = metric ton, unit of mass equal to 1000 kilograms
TPDES = Texas Pollutant Discharge Elimination System
TTUS = Toyo-Thai US
USA = utility services agreement
WBS = work breakdown structure

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1.0 EXECUTIVE SUMMARY

This report summarizes the activities performed and results realized from efforts during the period from January 2010 through June 30, 2015 (Q2FY2015). As of June 30, 2015, about 0.3% of construction remains to be completed. Commissioning is scheduled to be completed by the end of August with production beginning mid-September.

The objectives of this multi-phase project were to implement the SkyMine® technology at a commercial-scale pilot plant that would be designed to capture 75,000 tonnes of CO_2 per year from a slipstream of exhaust gases from a coal fired cement plant kiln. The SkyMine® process is an example of carbon capture and beneficial re-use because it produces products with commercial value. The Project, conducted in two main phases, began on January 15, 2010. Phase 1 consisted of completing a preliminary pilot plant design, conducting a NEPA review, and producing a Project Management Plan. Phase 2 was organized into three subphases. In Phase 2a, the first of the three subphases, Skyonic completed the final detailed design to the point of beginning construction of the commercial-scale pilot plant. In Phase 2b, construction of the Capitol SkyMine plant progressed to >90% completion at the end of December 2014. Phase 2c included the construction, operation and testing of the technology. As of the end of this Project on June 30, 2015, construction was 99.7% completed.

Engineering efforts included modeling and simulation, field tests, and laboratory work which enabled the team to both evaluate and validate the design concepts to be used for large-scale capture and mineralization of CO_2, and purification of products generated by the process. During Phase 1, the SkyMine® field test facility was moved and installed at the host site in San Antonio to further enable staff members to collect sufficient additional data and more fully characterize the emitted flue gas and various process conditions to allow model corroboration and process optimization for scale-up for flue gas conditioning, CO_2 capture efficiency, bicarbonate crystal growth, reaction column recirculation and flow rates, and dewatering. A Life cycle analysis was developed that forecasts the carbon dioxide emissions of the SkyMine® commercial pilot plant, from transportation of incoming raw materials to the shipping of the end product chemicals to the market. It also includes all of the utility requirements. This was submitted as part of the ***Phase 1 Topical Report*** in section 3.6 and updated in Q2 2013. Some changes in the updated analysis include adding the CO2 footprint for comparison of all NaHCO3 production methods and adding a CO2 footprint table for the lowest possible SkyMine energy process. The life cycle analysis shows how the SkyMine® process will be carbon negative, a part of which includes running the chlor-alkali cells at a lower current density which reduces the total power draw required in the process for the production of bicarbonate.

In Phase 2, the collaborative efforts of the Project Team and support entities resulted in the design and construction of a commercial-scale pilot SkyMine® plant that is expected to capture 75,000 tonnes (82,687 tons) of emitted CO_2 on an annual basis which is hosted on the site of Capitol Aggregates' Cement Plant in San Antonio, Texas.

Design changes and delays during construction resulted in the plant not being operational at the end of the DOE's Project timeline. As such, it was not possible to gather data on the performance of the plant to include in this report before the end of the project, but it is anticipated that the completion of construction and the commissioning of the plant will occur in mid-September, and products will begin shipping to customers meeting their intended specs per contracted agreements shortly thereafter.

The final Project area is comprised of approximately 8 acres which includes storage facilities, rail spurs and access roads and is co-located on the 600-acre property of the Capitol Aggregates cement manufacturing plant in San Antonio, Texas.

Below and on the next page are two diagrams that show how the basic SkyMine® process works.

<u>Figure 1.1 – High-Level Process Flow Diagram of SkyMine®</u>

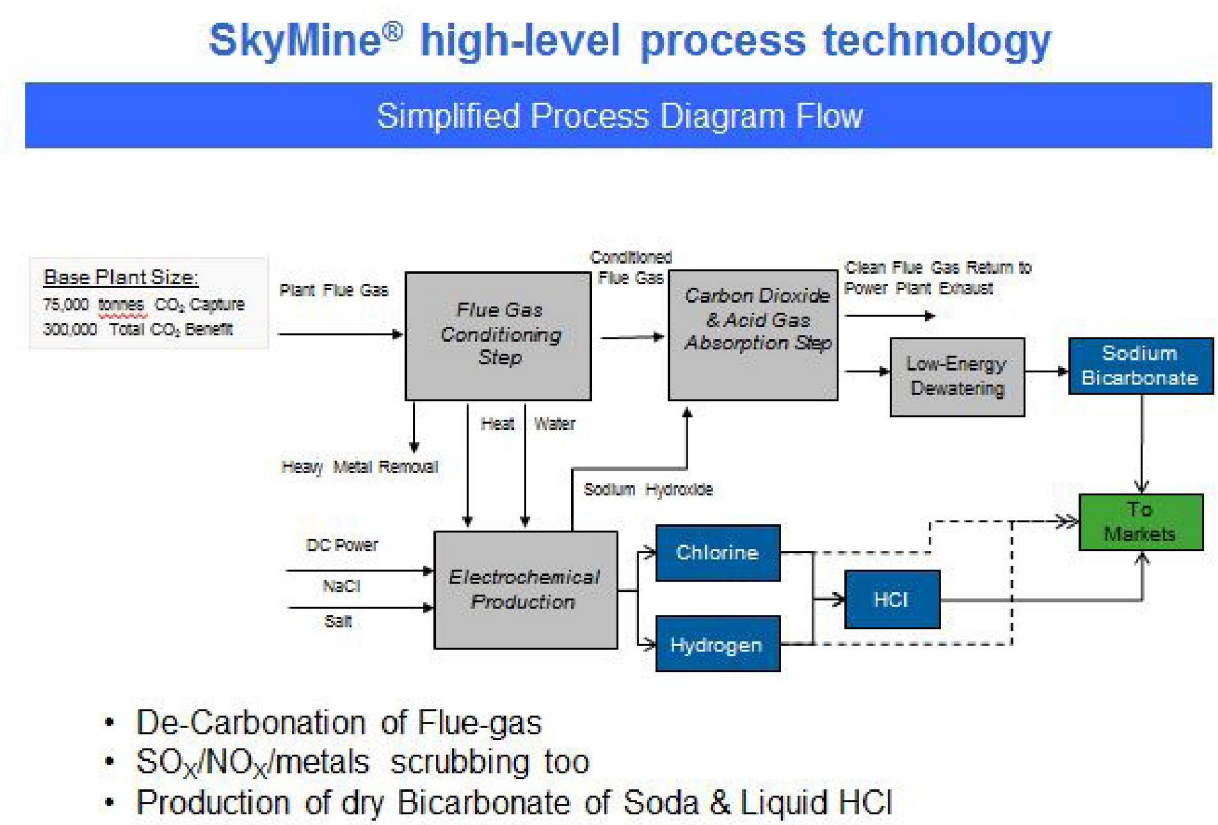
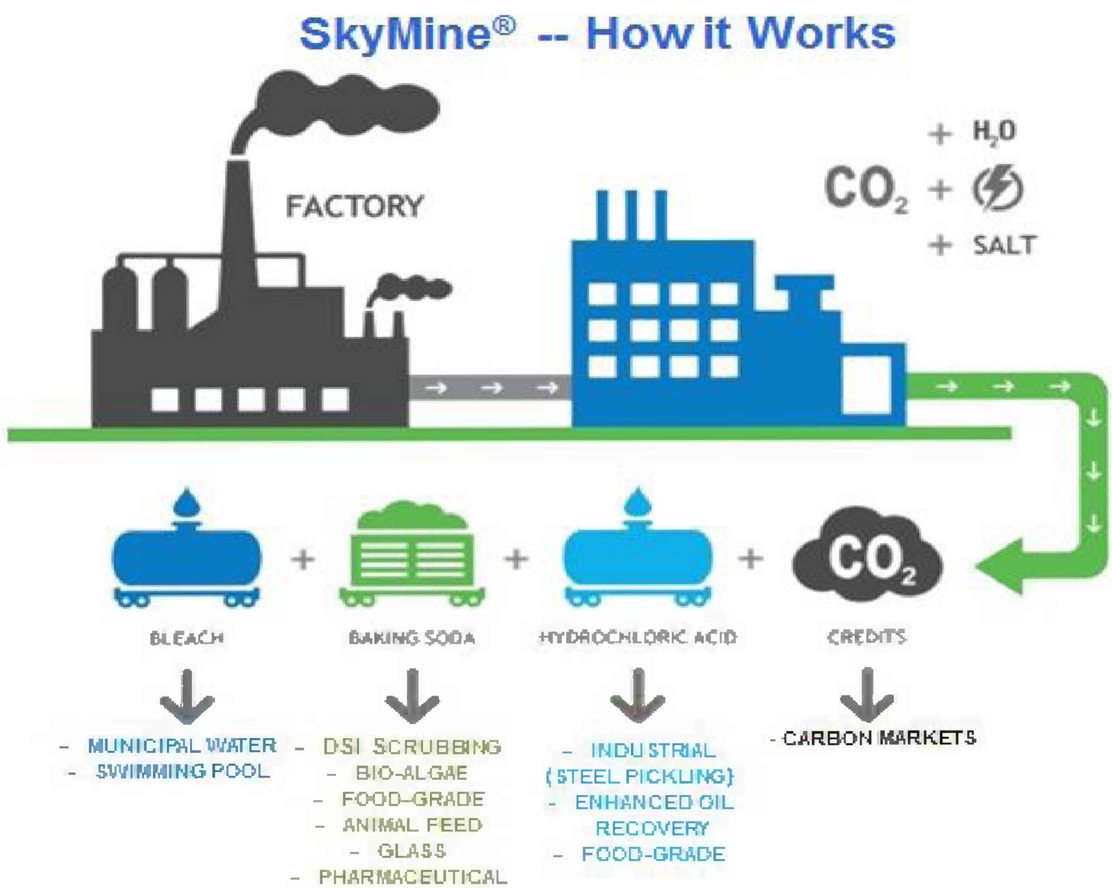


Figure 1.2 – Simplified SkyMine® Process



2.0 PROJECT STATUS

By June 30, 2015, the final quarter of this DOE supported Project, construction progress for Capitol SkyMine was 99.7% leaving approximately 0.3% left to be completed. Much of the final construction activity was focused on the CCU area of the plant.

2.1 HUMAN RESOURCES

Capitol SkyMine is fully staffed with no plans to add additional positions. Positions will be filled if attrition occurs.

2.2 SAFETY

During the total Project which included over 2 years of construction there were only 10 reportable safety incidents; there have been no lost time incidents.

3.0 PROCESS DESIGN

The SkyMine® Process is a unique, patented process designed to remove carbon dioxide from a gaseous waste stream, convert the carbon dioxide to a mineralized carbon product, and produce valuable chemical byproducts. The SkyMine® process, which reacts carbon dioxide from waste flue gas with sodium hydroxide to form sodium bicarbonate, is designed as a multi-column system. In the on-site field test unit, columns were equipped with recirculation pumps, metering pumps, pH sensors, temperature sensors, pressure sensors, sample points (for fluid samples), test points (for gas samples), drip trays (for fluid distribution), flow indicators and manual valves to control recirculation flow. The SkyMine® process is designed to remove over 90 percent of the carbon dioxide from a flue gas stream. This Project is designed to capture ~15% of the flue gas from Capitol Aggregates cement plant. The resulting products produced by the process are of high purity are readily marketable and in demand. Before construction began, Skyonic had multiple off-take agreements in place to sell or distribute these products for use in the oil and gas, animal feed, municipal water treatment and industrial cleaning industries, among others. Because of the purity of the products manufactured, Skyonic expects the products to become certified for use in the food and pharmaceutical industries.

Project engineers worked with multiple vendors to characterize the dewatering and drying processes and determine optimum operating conditions to minimize energy consumption and produce a consistent marketable product. For much of the project Skyonic had pursued the development of proprietary Vibratory Shear Enhancement Processing (VSEP) technology for the removal of excess water generated during the SkyMine process. VSEP is a reverse osmosis (RO) technology that utilizes a vibratory design to greatly reduce membrane fouling of precipitating solids. This technology is designed to recycle the dissolved sodium carbonate and sodium bicarbonate ions to the process instead of losing them to drain and had the advantage of a reduced downtime that is typical of a static RO membrane. Wanting to improve the performance of dewatering, which has an economic impact and improves the environmental efficiency, Skyonic worked with one of their project engineers, Ford Bacon Davis (FBD) to determine an alternative method of water removal. It was determined that using Mechanical Vapor Recompression (MVR) technology lowered the overall energy requirement and met additional goals, some of which were to 1) maintain the low energy approach to water removal, 2) have similar capital and operating costs when compared to VSEP, 3) maintain product quality through the process, 4) eliminate product loss, and 5) further recycle water for use in the process. From this point in the process, Skyonic implemented the MVR technology in their engineering and design.

The SkyMine® process is comprised of nine (9) process- areas identified as Areas 100 - 900:

Area 100 – Flue Gas Preparation and Processing

Flue gas from the cement kiln is the carbon dioxide source for sodium bicarbonate to be produced by the SkyMine® facility. The purpose of the flue gas preparation area is to acquire the required amount of flue gas from the kiln, and bring it to the required pressure and temperature for use in downstream processing. The flue gas is cooled and condensate from the cooling operation is removed from the gas stream and further processed for either use in the process or in preparation for removal from the site as waste. Major equipment in the flue gas preparation area consists of the following equipment:

- Flue Gas Blower
- Flue Gas Cooler
- Knock Out Tank with an associated demister and pump
- Condensate Pump
- Carbon Filters

Area 200 - CO_2 Absorption Columns

Once the flue gas has been cooled, the total flue gas is passed through a pair of CO_2 Absorber Columns operating in parallel. The columns provide a large vapor liquid contact area between flue gas flowing upward through the column and NaOH solution flowing downward. Here the CO_2 in the flue gas reacts to form sodium carbonate:
The sodium carbonate solution is then fed to the bubble columns where it is further reacted with flue gas to yield sodium bicarbonate ($NaHCO_3$).

Major equipment in the CO_2 Absorber Column Area consists of the following equipment:

- CO_2 Absorber Columns
- CO_2 Absorber Pump

Area 300 – Bicarbonate Crystallization, Separation, and Water Recovery

The major processes in Area 300 are sodium bicarbonate crystallization, separation, concentration and water recovery. Bicarbonate crystallization is the continuous process by which conditioned flue gas is compressed and bubbled through a saturated sodium carbonate solution in a bubble column reactor to precipitate bicarbonate crystals. The slurry is separated by a centrifuge into solids, sent to the dryer, and liquids, which are further processed to remove excess water. The system concentrates the remaining carbonate and bicarbonate in solution while recovering water for reuse in the plant. The concentrated solution is recycled to the bubble column reactor for further reaction to allow zero product waste.
Area 300 includes the following major equipment:

- Compressors Flue gas coolers
- Sodium Carbonate cooler
- Knockout pots
- Bubble column reactors
- Decanters

- Membrane Filter package

Area 400 – Sodium Bicarbonate Drying and Rail Loadout

After sodium bicarbonate is produced in the bubble columns and has been dewatered it must be dried and stored for shipment.
The sodium bicarbonate drying and rail loadout system includes the following equipment:

- Dryer Air Blower
- Dryer Air Heater
- Cooling Loop Pump
- Expansion Tank
- Bicarbonate Dryer Package
- Bicarbonate Conveying Package
- Bicarbonate Storage Silos
- Bicarbonate Loadout Package

Area 500 – Salt Unloading and Brine Saturation

Equipment Associated with Salt Unloading and Brine Saturation include the following:

- An Enclosed Salt Unloading Building r
- A Salt Receiving Hopper with a screw conveying system
- A Tubular Drag Chain Conveying System
- A Brine Saturation Tank
- Brine Feed Pumps

AREA 600 – Chlor-Alkali and Bleach Storage

The purpose of the chlor-alkali plant is to provide caustic (NaOH) as a 32 wt% solution for use in carbon capture, scrubbing, and other minor uses. Any caustic required for use within the chlor-alkali plant is stored within the chlor-alkali plant battery limit. Lean Brine is sent from the chlor-alkali plant to the Saturation Tanks in Area 500. Saturated Brine is sent from the Saturation Tanks to the chlor-alkali battery limit.

Sodium hydroxide is generated through electrolysis of sodium chloride brine in a membrane cell. This process is part of a packaged system. The package was purchased as an integrated system, including secondary brine treatment, HCl synthesis, chlorine scrubber, and bleach preparation. The packaging of the system in this fashion provides a well-defined process boundary allowing definable and measurable performance guarantees for the package.

Saturated brine is fed through a number of purification steps to remove hardness and other trace components using flocculation, filtration, and ion exchange steps. Ultrapure saturated brine is fed into the anode side of the electrolysis cell where the chlorine ions are oxidized to chlorine. At the cathode side of the cell, water is reduced to hydrogen gas hence releasing hydroxide ions into solution. The ion exchange membrane installed between the anode and cathode compartment allows the sodium ions to pass to the cathode side where they react with the hydroxide ions to produce sodium hydroxide.

The equipment in this area includes:

- Chlor-Alkali Plant

- Bleach Storage Tanks
- Bleach Loading Package

AREA 700 – HCl Storage

The equipment in this area includes:

- HCl Storage Tanks
- HCl Scrubber
- HCl Rail Loading Package
- HCl Truck Loading Package

AREA 800 – Caustic Storage

The equipment in this area includes:

- Caustic Storage Tanks
- Caustic Storage Pump
- Caustic Dilution Package
- Caustic Unloading Arm

AREA 900 – Utilities

The equipment in this area includes:

- Cooling Tower
- Chiller
- Boiler Package
- Air Compressors, Receivers, Dryer
- Liquid Nitrogen Package
- Fire Protection System
- Waste Water Tank

4. MASS & ENERGY BALANCE

Initial Process Flow Diagrams, P&IDs, and general site and building arrangement plans were issued by FB&D in Phase 1 as part of the project scope package. These PFDs show compositions, temperatures, pressure, mass flow rates, and enthalpies for the major process streams. An overall mass balance shown in Table 4.1 details the incoming raw materials and the products and outgoing waste streams.

The energy balance summary details each process area's power consumption. The projected total plant power consumption at peak performance is shown at the bottom of the Power Requirements table in Table 4.2.

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Table 4.1 – Material Balance (originally submitted as Table 3.1 in ***Final Phase 1 Topical Report**, 2010)*

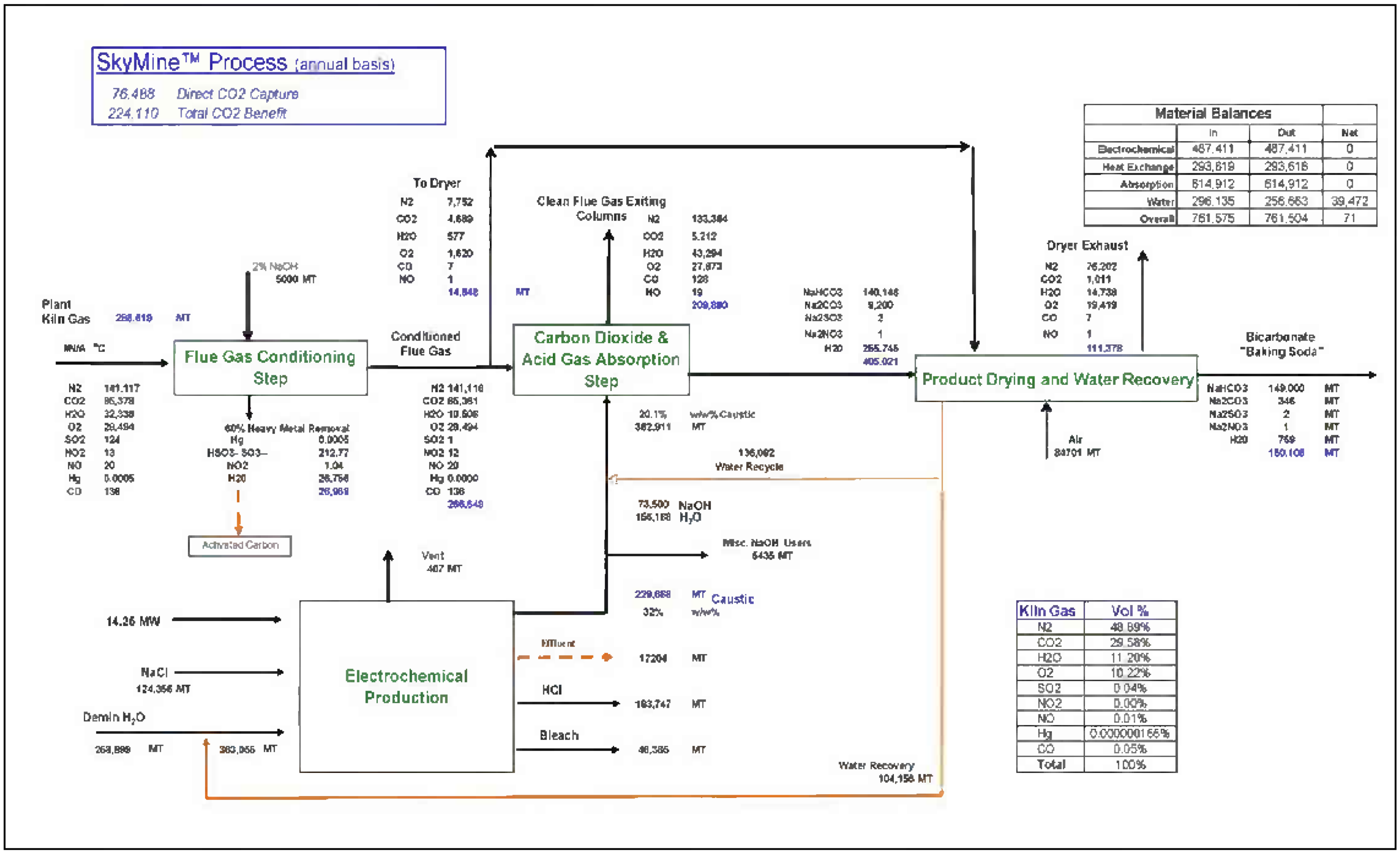

Table 4.2 Material Balance (originally submitted as Figure 3.1 in the ***Final Phase 1 Topical Report***, *2010)*

Area Number	Area Name	Power Required (kW)
100	Flue Gas Preparation and Processing	180.8
200	CO2 Absorption Columns	7.8
300	Bicarbonate Crystallization, Separation, and Water Recovery	958.3
400	Sodium Bicarbonate Drying and Rail Load Out	237.6
500	Salt Unloading and Brine Saturation	138.6
600	Bleach Storage	14.4
600C	Chlor-Alkali Ancillary Equipment	358.6
700	HCl Storage	10.4
800	Caustic Storage	3.9
900	Utilities	788.2
	TOTAL BALANCE OF PLANT POWER REQUIREMENT	2698.6

Area	Power Required (MW)
Balance of Plant	2.70
Electrolysis	18.42
TOTAL	**21.12**

5.0 ENVIRONMENTAL/NEPA PLANNING

Under contract with Skyonic Corporation, qualified environmental consultants helped to develop a comprehensive environmental permitting strategy and assisted in producing the data necessary for DOE to execute its responsibilities under NEPA.

Due to the nature and location of the project and the small amounts of emissions, all permits had to be completed through the permit-by-rule process. All permitting activities were completed prior to construction within the current Project timeline.

Skyonic, together with environmental consultants and legal counsel, reviewed applicable regulations and met with the Texas Commission on Environmental Quality (TCEQ) to determine environmental permit requirements.

6.0 CONSTRUCTION

Skyonic contracted with TTUS to serve as the EPC contractor and to integrate the Ford Bacon & Davis (FBD) design of the Carbon Capture Unit (CCU) with the Chlor-Alkali Unit (CAU) and balance of the SkyMine® pilot plant. CCC Group (CCCG), a U.S. construction firm, under the direction of TTUS, constructed the entire SkyMine® plant. The total installed cost of the project was estimated to be $125 million by TTUS.

The completion of Construction, Subphase 2b of the Project, was designated as the "Mechanical Completion" milestone. To achieve Mechanical Completion, each system and subsystem was verified to be constructed per "Issued For Construction" drawings and documents, and the equipment (or system) has all the mechanical and electrical interconnections to function as designed. The EPC firm, TTUS, with support from their subcontractors, was responsible for meeting the criteria of Mechanical Completion. The equipment for the Project was procured by Toyo Thai Corporation Limited's US Division (TTUS) using competitive bids.

6.1 OPERATOR TRAINING

All operators have been fully trained and were utilized in system reviews to find issues with equipment and system operations. Items were noted and used in the PSSRs of the systems, MCC loop checks, instrument loop checks, field verifications of instruments, and approving punch list corrective actions items.

7.0. CONCLUSIONS

In Phase 1 of the project, the Project Team and support entities completed a design and plan for building their first SkyMine® plant capable of annual capture of 75,000 tonnes (82,687 tons) of emitted CO_2 at a suitable host site in San Antonio, Texas, thereby meeting the primary Phase 1 objectives. While construction was +99% completed, and the process technology testing and final demonstration were starting to be tested during the preliminary commissioning that had occurred by the end of Phase 2.

Secondary Phase 1 objectives, like process optimization, construction planning, and supply chain development, were also met. Advanced process modeling and simulation combined with laboratory experiments, proven principles of basic chemistry and well-known electro-chemical processes, and field validations show that the innovative Capitol SkyMine project will achieve its objectives by capturing and mineralizing CO_2. Significant additional benefit is derived from the SkyMine® process' capability for removing SO_x and NO_x and heavy metals such as Hg from the emitted flue gas.

Analytical results of testing process variables have provided clear optimum values for such items as membrane selection, and pressure and temperature conditions relative to desired flow rates and recovery amounts. A review of some of these results can be found in the published ***Phase 1 Topical Report*** and the ***Decision Point 1 Topical Report.***

The logistics of transport of inputs and outputs, concludes that the SkyMine® process is viable over large geographical bounds without disabling transport impediments, and is not limited (as geo-injection processes are) by geologic storage issues. The Capitol SkyMine Project is expected to capture more than 75,000 tpy of CO_2 from the exhaust gases of a cement kiln. The carbon penalties of this first-plant SkyMine® mineralization process are competitive when compared to other carbon capture technologies. Further improvements in energy penalty range of operation can be achieved by improving SkyMine® internal efficiencies and lowering parasitic loads, which will come about as the project efforts further define the operation of a commercial scale pilot, allowing prioritization of future development efforts.

In matters relevant to the power industry, specifically, the SkyMine® system, in addition to containing its own SO_X/NO_X/Metals scrubbing system, has substantial abilities to interact with its input power provider to draw primarily off-peak energy, improve system carbon intensity, and absorb bursts of renewable power.

The SkyMine® technology, built primarily from proven commercial systems and paired with specifically designed and engineered materials to meet Skyonic's precise technical needs, can be retrofitted to existing plants, and is nearing commercial completion. A large portion of a SkyMine® plant can be constructed from commercially available sub-components, which is valuable in establishing a reference plant design. Each plant will be unique and will go through an adaptive engineering process, but the overall Plant itself is relatively easy to replicate. It is anticipated that it can be potentially down-costed when expanding the size and capacity of carbon capture by utilizing the marginal cost of each additional section or unit. During the normal course of operations it is anticipated that multiple levels of optimization will be achieved. Those improvements will be reflected in future plant designs and could reduce the footprint, layout and materials needed to build of the plant, the energy needed to run the plant, and the operating costs of maintaining the plant. At the time of this report, using the inputs and outputs that are outlined in the Life Cycle Analysis, with the cost of the $125 million capital expenditure at 15%, this project is expected to provide an internal rate of return of approximately 30%. When fully operational, this first of its kind SkyMine® facility will demonstrate the feasibility of the SkyMine® carbon capture technology at commercial scale while producing valuable chemicals that can be sold into the market for profit. This is not only beneficial for the environment, but will also have a positive impact on the economy because of the number of jobs it creates and amount of commerce that it generates.

To read more about the Project and reference some of the lab work and initial experiments that were completed, please refer to the following published reports which are available on the OSTI website:

Phase 1 Topical Report: http://www.osti.gov/scitech/servlets/purl/1027801

Decision Point 1 Topical Report: http://www.osti.gov/scitech/servlets/purl/1238330

Decision Point 2 Topical Report: http://www.osti.gov/scitech/servlets/purl/1176867

Commercial-Scale Pilot Plant Photos

Figure 1 Plant site October 30, 2013

Figure 2 Plant Site January 31, 2014

Figure 3 Plant Site March 30, 2014

Figure 4 Plant Site June 30, 2014

Figure 5 Plant Site September 30, 2014

Figure 6 Plant Site December 31, 2014

Figure 7 Plant Site March 31, 2015

Figure 8 Plant Site June 30, 2015

Figure 9 Night View of Plant

Figure 10 Area 100 Flue Gas Conditioning

Figure 11 Sodium Bicarbonate Bubble Columns

Figure 12 Flue Gas Ducts and HCl Synthesis Units

Figure 13 Carbon Capture Area

Figure 14 -- Sodium Bicarbonate Rail Loading Area