Final Technical Report

HFC Concrete: A Low-Energy, Carbon-Dioxide-Negative Solution for reducing Industrial Greenhouse Gas Emissions

DOE Award Number: DE-EE0003495

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Dr. Larry McCandlish, Principal Investigator
908-315-5901
lmccandlish@solidiatech.com

Solidia Technologies, Inc.
(formerly CCS Materials, Inc.)
11 Colonial Drive, Piscataway, NJ 08854

Dr. Richard Riman, Co-Principal Investigator

Department of Materials Science and Engineering
Rutgers, The State University of New Jersey

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Acknowledgment, Disclaimer and Proprietary Data Notice

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Proprietary Data Notice: There is no proprietary data included in this report.

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List of Acronyms

ASTM	American Society for Testing and Materials
CAGR	Compound Annual Growth Rate
DoD	Department of Defense
DOE	Department of Energy
EERE	Energy Efficiency and Renewable Energy
FEMA	Federal Emergency Management Agency
GHG	Green House Gas
GSA	General Services Administration
HFC	Hydrate Free Cement
LEED	Leadership in Energy and Environmental Design
LTS	Low Temperature Solidification
LW	Light Weight (concrete)
PC	Portland Cement
UBC	Uniform Building Code
UFC	Unified Facilities Criteria

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Executive Summary

Solidia/CCSM received funding for further research and development of its Low Temperature Solidification Process (LTS), which was used to create hydrate-free concrete (HFC). LTS/HFC is a technology/materials platform, which offers wide applicability in the built infrastructure. Most importantly, it provides a means of making concrete without cement.

Cement and concrete production is a major consumer of energy and source of industrial greenhouse gas (GHG) emissions. The primary goal of this project was to develop and commercialize a novel material, Hydrate Free Concrete (HFC), which by replacing traditional concrete and cement, reduces both energy use and GHG emissions in the built infrastructure. Traditional concrete uses Portland Cement (PC) as a binder. PC production involves calcination of limestone at ~1450°C, which releases significant amounts of CO_2 gas to the atmosphere and consumes a large amount of energy due to the high temperature required. In contrast, HFC is a carbonate-based hydrate-free concrete (HFC) that consumes CO_2 gas in its production. HFC is made by reaction of silicate minerals with CO_2 at temperatures below 100°C, more than an order-of-magnitude below the temperature required to make PC. Because of this significant difference in temperature, it is estimated that we will be able to reduce energy use in the cement and concrete industry by up to 30 trillion Btu by 2020. Because of the insulating properties of HFC, we believe we will also be able to significantly reduce energy use in the Building sector, though the extent of this saving is not yet quantified.

This one-year project had three objectives that helped to advance LTS/HFC technology towards commercialization:

a. Optimization of the Low Temperature Solidification (LTS) process to reduce the product cost, develop a manufacturing process at scale, and facilitate entry into the HFC concrete market.

b. Demonstration that HFC has similar or better properties and greater durability than conventional materials for building façade applications since this is a strong entry point for the building market and is easier than larger-scale applications. For many applications the following properties are most important:

- Compressive Strength
- Modulus of Rupture
- Impact Resistance
- H_2O absorption

These properties were used to evaluate the suitability of HFC formulations for building façade applications.

c. Development of a commercialization plan, including development and documentation of market-related information and potential environmental, energy and economic benefits of the technology and product.

Significant results were achieved demonstrating the potential of LTS/HFC to impact the cement/concrete industry:

- Achieved considerable progress in net-shape green body preparation, shortening mixing and forming times from days to minutes.

- Undertook a number of studies to better understand and optimize the LTS reaction.

- Reduced temperature and pressure needed to achieve required properties, from a baseline of 100°C and 20 psig to 80°C and 0 psig (i.e. 14 psia $P = P_{CO2} + P_{H2O}$)

- Evaluated mechanical strength, durability, weathering, staining and adhesion as per ASTM standards, which showed that our materials are not only competitive with but surpass the strength and durability of comparable products in the façade market.

- Carried out an extensive study of the façade market in preparation of commercialization of our first product application.

During the period of this project, relationships with a number of façade manufacturers and distributors, both domestically and abroad, were developed with the intent of licensing LTS technology to various manufacturers to enable more rapid commercialization. These discussions continue and are extremely promising. We hope to have our first licensee during 2012.

This work showed that our technology has significant potential to provide the built infrastructure market with building products that are stronger and more durable than those made from traditional stone and concrete materials, at comparable or lower cost while significantly reducing both embedded energy and building energy use.

It is recommended that further work be undertaken to develop additional applications and products outside of facades, including large pre-cast concrete structures.

1.0 Introduction

Solidia/CCSM received funding for further research and development of its Low Temperature Solidification Process (LTS), which is used to create hydrate-free concrete (HFC). LTS/HFC is a technology/materials platform that offers wide applicability in the built infrastructure. Most importantly, it provides a means of making concrete without Portland cement.

Cement and concrete production is a major consumer of energy and source of industrial greenhouse gas (GHG) emissions. The primary goal of this project was to develop and commercialize a novel material, HFC, which by replacing traditional concrete and cement, reduces both energy use and GHG emissions in the built infrastructure.

Traditional concrete uses Portland Cement (PC) as a binder. PC production involves calcination of limestone at ~1450°C, which releases significant amounts of CO_2 gas to the atmosphere and consumes a large amount of energy due to the high temperature required.

In contrast, HFC is a carbonate-based hydrate-free concrete (HFC) that consumes CO_2 gas in its production. HFC is made by reaction of silicate minerals with CO_2 at temperatures below 100°C, more than an order-of-magnitude below the temperature required to make PC. Because of this significant difference in temperature, it is estimated that we will be able to reduce energy use in the cement and concrete industry by up to 30 trillion Btu by 2020. Because of the insulating properties of HFC, we believe we will also be able to significantly reduce energy use in the Building sector, though the extent of this saving is not yet quantified.

It is estimated that production of a tonne of PC-based concrete requires about 6.2 million Btu of energy and produces over 1 tonne of CO_2 emissions (Choate, 2003). These can be reduced to 1.9 million Btu and 0.025 tonnes of CO_2 emissions per tonne of HFC (with overall CO_2-negativity possible by increasing carbonation yield). In this way, by replacing PC-based concrete with HFC in infrastructure we can reduce energy use in concrete production by 70%, and reduce CO_2 emissions by 98%; thus the potential to reduce the impact of building materials on global warming and climate change is highly significant.

Low Temperature Solidification (LTS) is a breakthrough technology that enables the densification of inorganic materials via a hydrothermal process. The resulting product exhibits excellent control of chemistry and microstructure, to provide durability and mechanical performance that exceeds that of concrete or natural stone.

The technology can be used in a wide range of applications including façade panels, interior tiles, roof tiles, countertops, and pre-cast concrete. Replacing traditional building materials and concrete in these applications will result in significant reduction in both energy consumption and CO_2 emissions.

2.0 Background

2.1 Process Description (Chemistry)

Concrete is the world's most frequently used construction material and second only to water as the most consumed substance on Earth, in large measure because it is the least expensive, widely available building material.

The manufacture of Portland Cement (PC), an essential ingredient in modern concrete, emits about 1.2 tonnes of CO_2 for each tonne of cement that is produced (Choate, 2003).

PC results from the calcination of limestone (calcium carbonate) and silica (from clay) according to the reaction (simplified):

$$5CaCO_3 + 2SiO_2 \Rightarrow 3CaO \bullet SiO_2 + 2CaO \bullet SiO_2 + 5CO_2$$

The production of 1 tonne of cement directly generates 0.54 tonnes of chemical-CO_2 and requires the combustion of fuel to yield an additional 0.66 tonnes of fuel-CO_2 (Choate, 2003). Almost all of the PC that is manufactured goes into concrete (Mehta, 2006).

In the United States, the cement industry ranked 9th among industrial users of energy, consuming 494 trillion Btus in 2002 (Schipper, 2005). The industry ranked 2nd in energy consumption per dollar of value added, at 95.5 KBtu, behind the petroleum refining industry (ICF International, 2007).

Solidia Technologies is working to eliminate the need for PC in concrete by replacing it with a low-temperature solidification (LTS) processed ceramic binding material that is produced at less than 100°C, called Hydrate-Free Concrete (HFC). Forming HFC at this lower temperature produces about 0.2 tonnes of combustion-based CO_2 per tonne of binder as part of its formation.

HFC *consumes* at least 0.17 tonnes of reaction-based CO_2 per tonne of binder as part of its forming reaction:

$$(Ca,Mg)SiO_3 + CO_2 \Rightarrow (Ca,Mg)CO_3 + SiO_2$$

Energy savings and CO_2 emissions reductions can be seen in Table 1 below.

Table 1: Energy and CO_2 Comparisons for Portland Cement and Hydrate Free Concretes

	PC Concrete	Hydrate Free Concrete	% Reduction
Energy Used per tonne	6.24 x 10^6 Btu	1.88 x 10^6 Btu	70%
CO_2 Emitted per tonne	1.2 tonnes	0.03 tonnes	98%

Thus, in the long-term, use of HFC in lieu of cement in pre-cast concrete products will significantly reduce both energy use and CO_2 emissions in this sector.

The long-term objective of Solidia is to create a low energy-consuming, low CO_2 emitting process that can replace cement in a wide range of applications, including concrete. In order to accomplish this goal, our process must be refined to balance reaction conditions and raw material formulation to increase the size of products that can be made and to scale-up the manufacturing process to produce product in large quantities.

In making these products a low-temperature solidification (LTS) process is used to remove porosity in a permeable, net-shape structure that is formed, for example, by casting or extrusion of a slurry. This molded preform structure comprises a reactive matrix that consists of particles that can release calcium ions and other filler particles that are not reactive.

In the illustration below the white circles represent this reactive matrix. The void space between the matrix particles is a network of connected pores so that the structure is pervious to gas and liquid phases.

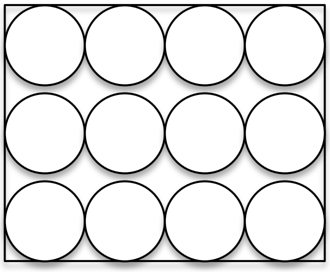

Figure 1. Schematic representation of reactive matrix (circles) and void space

The porous reactive matrix is infiltrated with a reactive fluid that is shown in the illustration below as the blue region.

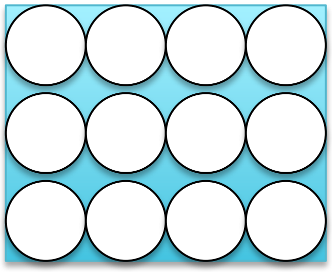

Figure 2. Reactive fluid infiltration of void space

The reactive fluid dissolves part of the matrix structure, releasing calcium ions and supplying carbonate ions. The ions react to fill the pore structure with cementitious material, in this case calcium carbonate. The reactive fluid is excluded from the shrinking pores as the solidification reaction proceeds. In the illustration below, the precipitated cementitious material formed by the LTS reaction is shown in gray. Note that the net shape of the preform does not change much even though material has been added to the structure, because the volume of the precipitated material is balanced by the original pore volume plus the volume created by dissolution of reactive matrix material.

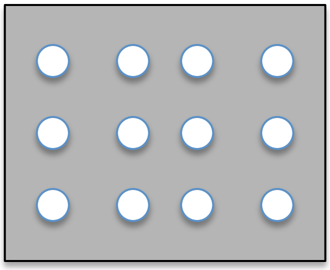

Figure 3. Filling of former void space by new carbonate material

In this way a high-density ceramic material is created without using a high temperature kiln.

Another way of understanding this solidification process is to realize that it is analogous to the lithification process by which nature creates limestone or completely fills the void space inside a geode. The main difference is that nature requires millions and millions of years and perhaps higher temperatures while the LTS process requires only hours and temperatures below 100°C.

2.2 Process Description (Physical and Chemical Operations)

The figure below is a process flow diagram:

Figure 4. Sequence of processes for making building materials from minerals and CO_2 gas

The mixing, molding and finishing operations are straightforward adaptations of processes currently used in making concrete and/or synthetic stone materials.

The specifics of formulation, drying and reacting are unique to HFC and the LTS reaction, because composition and control of porosity are essential to achieving green-body characteristics that lead to an acceptable LTS reaction rate and acceptable mechanical properties of the final product compositions. Thus research and development is focused on understanding and improving green-body formulation, drying and reacting. Process improvement is measured in relation to cost and performance of the product. Generally, cost and performance are positively correlated: low cost = low performance; high cost = high performance. It is known that HFC, with high performance properties along with low energy and small GHG footprints, is possible. HFC's price/performance ratio, and hence its competitive advantage is yet to be determined, and of course this will vary from one market to another.

2.3 Technology Status at Beginning of Project

The first HFC objects ever produced were 1" long x 1" diameter die pressed cylinders and then 4" x 3" x ½" die-pressed tiles. These were dimensionally stable and exceptionally strong in compression, over 100 MPa. This was the technology status at the time that we wrote our research proposal for the project. By the time that the DOE award was made we were attempting to cast 8" x 8" x 1" and 48" x 20" x ½ " tiles using vibratory casting techniques. We were mostly unable to dry these larger castings without the appearance of cracks. We had no way of reacting the 48" x 20" x ½ " tiles.

In making these early objects, compositions were mixed in a small, slow planetary mixer. After adding water, the resulting water/powder mixes were homogenized by jar rolling. The majority of the early research was on pure Wollastonite or 60% Wollastonite/40% calcite compositions.

The LTS reaction was carried out in small Parr autoclaves at temperatures above 100 C. CO_2 was introduced into the Parr vessels as weighed amounts of dry-ice pellets. The Parr reactors also contained liquid water at the reaction temperature so that P_{total} had two components $P_{CO2} + P_{H2O}$. We could measure P_{total} . Since P_{H2O} was the equilibrium vapor pressure of water at the reaction temperature (a known quantity), we could calculate P_{CO2}.

We were also developing modified commercial sterilizers, which were similar to any ordinary pressure cooker that you might find in a kitchen, as LTS reactors. These were designed for operation at temperatures below 100 C. The sterilizers were large enough to accommodate 8” x 8” x 1” tiles.

2.4 Tasks and Milestones

This project was organized around four tasks to accomplish our commercial and engineering objectives:

Task 1: Potential Benefits Assessment

Task 2: Develop Product Strategy for First Market Entry

Task 3: Process Optimization Studies

Task 4: Product Evaluation and Acceptance of Building Facades

Three milestones were set as a measure of success in meeting our objectives:

Milestone 1: Show a 20% temperature and a pressure reduction in the LTS process relative to the baseline of 100 °C and 20 psi (i.e. 80°C & 16 psi)

Milestone 2: Complete a competitive market analysis for building facades.

Milestone 3: Produce HFC façade panels measuring five feet long by 2 feet wide by 0.5 inch thick.

3.0 Results and Discussion

Our low-energy manufacturing process for making HFC can be divided into three major operations:

1) Forming raw materials (powdered minerals) into a preliminary shape (panel, block, shingle, tile, slab, etc.) suitable for reacting with CO_2. This relatively fragile shape is called a green body.

2) LTS reacting the green body with CO_2 to develop robust useful properties;

3) Finishing the reacted shape to make a commercial product for deployment in the built infrastructure.

A major task of our research program has been the optimization of this process with a view toward making larger and larger objects (scalability) while at the same time decreasing energy consumption (increasing efficiency). Both of these goals are obtainable by lowering the reaction temperature and pressure.

3.1 Forming Operations

During this project we formulated powder mixtures for producing green bodies, mixed the powders according to formulation, and developed procedures for casting the mixtures in a variety of mold types.

While a variety of formulations were studied. The baseline powder mixture comprised silicate and carbonate minerals along with a small amount of water. Initially, we used a conventional "contractor" cement mixer to blend the minerals and followed this by rolling the mixed material in 50 gallon drums to further improve the homogeneity of the mix.

This two-step mixing procedure was sufficient for making material for our initial studies, but was too labor intensive, too slow and used too much space to be commercially viable.

Using a commercial high-intensity mixer, which eliminated the need for drum rolling and cut mixture preparation time from days to minutes, solved the scalability problem for the mixing part of the process. The mixing process we developed is scalable to commercial production.

High-intensity mixing produces a granular material, which becomes a viscous fluid upon vibration. This unusual behavior permits us to use vibratory casting to mold a wide variety of green body shapes.

The as-cast green bodies are rather fragile but their mechanical strength can be improved by drying, which helps avoid damage due to handling. The avoidance of cracking and curling due to shrinkage is a challenge in drying green bodies.

Consequently, the drying kinetics of full-size (5’ x 2’ x 0.5”) green-body panels at fixed relative humidity and temperature as well as smaller panels at greater thicknesses was studied. To accomplish this a weighing platform using Sartorius load cells was designed, built and installed in a Tenney Environmental Test Chamber. We also installed two Vaisala relative humidity sensors so that we could monitor the relative humidity difference between air near the surface of the drying panel and the relative humidity of the conditioned air within the chamber.

A computer program to collect temperature, relative humidity and weight loss data and to display the data in real time (Figures 5 & 6) was written. The drying rate, as it changes during the process, was calculated from the weight loss curve. The well-known problem of calculating the instantaneous derivative of a noisy digital data stream was overcome by applying a second-degree Savitzky-Golay (Savitzky, 1964) first derivative filter to weight loss data collected at one-minute intervals.   A filter length spanning one hour of data calculates the instantaneous drying rate at any given time by taking into account weight loss behavior looking backward in time for 30 minutes and forward in time for 30 minutes. The program stores data in an Excel spreadsheet for archiving and post-run analyses.

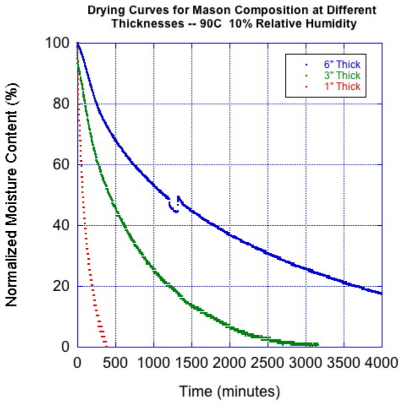

Figure 5. Drying curves for a 2’ x 2’ panel of a single composition at different thicknesses

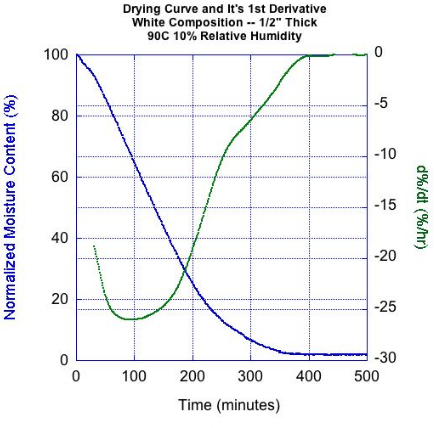

Figure 6. Drying and drying-rate curves for 5’ x 2’ x 1/2” panel

As can be seen in Figure 5, thicker panels require significantly longer drying times at the same drying temperature and relative humidity.

Going from a 1” thick panel to a 6” thick panel increases the drying time by more than an order-of-magnitude. The small glitch in the drying curve for the 6” thick panel shows the upset and recovery from a short power outage.

Figure 6 shows that a full-size façade panel can be dried at 90 C and 10% relative humidity in about 400 minutes. The drying-rate curve computed from the drying curve reveals several different stages of drying. The fastest drying rate occurs at about 100 minutes, which is at the end of the transient-on response as the panel finishes heating-up from room temperature to 90 C. During the heat-up time the panel is shrinking and free water expelled from the shrinking pores is evaporated. A second stage of drying occurs between 100 minutes and 270 minutes where liquid water within the pores is transported to the surface and evaporates. A third drying stage is observed between 270 minutes and 390 minutes where more strongly bound water is removed from the pore surfaces at a slower rate. After 400 minutes the panel is at equilibrium with the drying atmosphere and is essentially fully dried.

3.2 LTS Reaction

Green body shapes are hardened by LTS reaction in an autoclave (Figure 7).

Figure 7. LTS Autoclave

The need for an autoclave, which is an expensive pressure containment vessel, in the LTS process limits the size of objects that can be made. The scalability of the process to economically make objects larger than about 10’ by 30’ depends on being able to eliminate the autoclave from the process, which is facilitated by lower temperature and lower pressure operating conditions. More specifically, elimination of the autoclave depends on if the reaction pressure can be lowered to about 1 atmosphere of CO_2(g).

Effect of Temperature and Pressure on Extent-of-Reaction and Compressive Strength

We designed a series of experiments to quantitatively determine the temperature and pressure effects on extent-of-reaction and compressive strength of HFC made by the LTS reaction.

In this study we examined a series of one-inch-diameter by one-inch-long HFC cylinders. This small size was chosen as a matter of convenience.

The study comprised a replicated 2^2-factorial design. The temperature variable was studied at two levels, 50 C (the T_L condition) and 90 C (the T_H condition); the pressure variable was studied at 14 psig (the P_L condition) and 20 psig (the P_H condition). The variables were set at four combinations of conditions in this design (T_LP_L, T_HP_L, T_LP_H, and T_HP_H). All reaction trials were terminated after 20 hours. The first three combinations of reaction conditions were replicated five times, the last combination three times, for a total of 18 trials. Responses are summarized in Table 2.

Table 2. Extent-of-Reaction and Compressive Strength Responses to LTS Reaction at Combinations of two levels each of Temperature and Pressure

Reaction Condition	Extent-of-Reaction (%)	Compressive Strength (Mpa)	Replicates
T_LP_L	39(7.3)*	111(14)*	5
T_HP_L	49(1.1)	187(17)	5
T_LP_H	36(4.1)	133(25)	5
T_HP_H	52(1.4)	171(16)	3

*Estimated standard deviations, based on replicate experiments at each condition, are shown in parentheses after the average response for each condition.

Yates's algorithm (Box, 1978) was applied to the average responses tabulated in Table 2 to extract the main effects (T effect & P effect) and the two-factor interaction (T x P interaction effect) for each response. The estimated standard deviations computed from the replicates were pooled to calculate an estimated standard deviation for the calculated effects. Our results are shown in Table 3.

Table 3. Temperature Effect, Pressure Effect, and (Temperature x Pressure) Interaction

Effect	Response	Magnitude-of-Effect
T	Extent-of-Reaction	13(4)* %
P	Extent-of-Reaction	0(4) %
T x P	Extent-of-Reaction	3(4) %
T	Compressive Strength	57(9) Mpa
P	Compressive Strength	3(9) Mpa
T x P	Compressive Strength	-19(9) Mpa

*Estimated standard deviations are shown in parentheses.

The results shown in Table 3 can be interpreted as follows:

Extent-of-Reaction

Temperature has a significant effect on extent-of-reaction at 20 hours:

> Increasing the temperature by 40 C increases the extent-of-reaction at 20 hours by 13%, independent of the reaction pressure.

Also, based on a t-test (scaled by the estimated standard deviation determined from the replicates), the observed apparent (T x P) interaction is indistinguishable from noise.

Figure 8 shows these results as a response surface. The response surface is the least squares plane (LSP) fit to the data, which is shown as a gradient-shaded plane surface in the figure. The top surface of the frame is at 100% reaction; the bottom surface at 0% reaction.

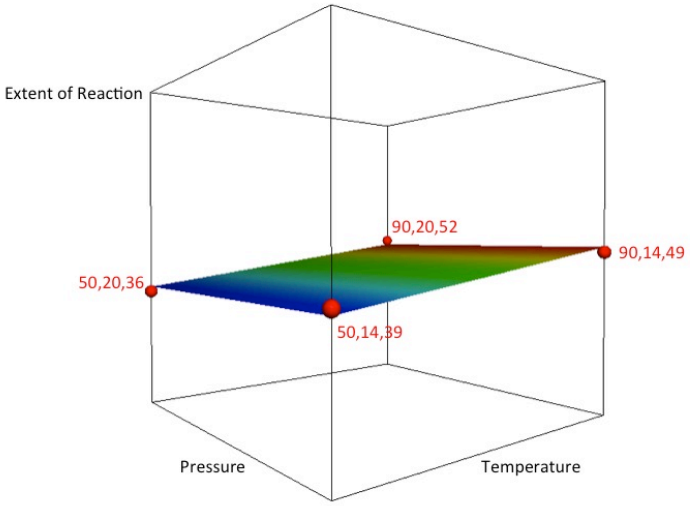

Figure 8. Extent-of-Reaction Response Surface

Compressive Strength

Temperature has a significant effect on compressive strength:

> Increasing the reaction temperature from 50 °C to 90 °C increases the compressive strength, after 20 hours of reaction by an average of 57 Mpa (8,267 psi).

The apparent small pressure effect is indistinguishable from noise based on a t-test (scaled by the estimated standard deviation determined from the replicates).

Unlike the extent-of-reaction case, the t-test puts the apparent (T x P) interaction effect just above the noise floor for the compressive strength case (just outside two standard deviations). The safe tentative interpretation is that in the case of compressive strength, we cannot consider the effect of T or P alone. We must consider the effects of temperature and pressure jointly:

At 90 °C, increasing P decreases compressive strength.
At 50 °C, increasing P increases compressive strength.

At 14 psi, increasing T increases compressive strength.
At 20 psi, increasing T increases compressive strength.

This is better understood from a response surface point-of-view (Figure 9).

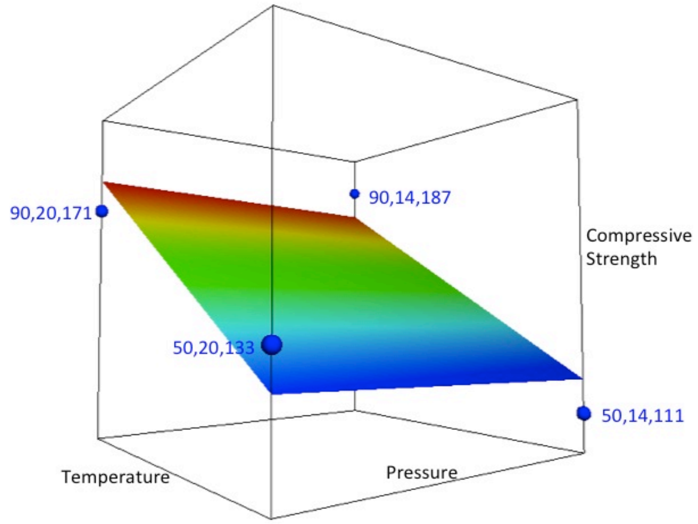

Figure 9. Compressive Strength Response Surface

Figure 9 shows the compressive strength response at the four combinations of reaction conditions that were studied. It also shows the LSP fit to the data. This plane is the true response surface if there is no T x P interaction. If the apparent T x P interaction is real then the true response surface has a twist that is hinted at by the distribution of responses about the LSP. The top surface of the frame is at 200 MPa compressive strength; the bottom surface is at 100 MPa compressive strength.

In our experience, for objects as small as 1” x 1” cylinders to objects as large as 5’ x 2’ x ½ “ panels, most material properties are improved by greater extent-of-reaction. We conclude that it is most likely that we will be able to scale the LTS process because the extent-of-reaction is independent of pressure.

We need to be careful of two-factor interactions for some properties, however, as is seen for the case of compressive strength, where at 90 C decreasing P may raise compressive strength but at 50 C decreasing P may lower compressive strength. Fortunately, in this case the apparent two-factor interaction is small.

LTS Activation Energy

We observed the weight gained, over a 20-hour reaction period, by three identical 1-inch high by 1-inch diameter cylinders. Three sets of reaction conditions were examined: 50 °C at 20 psig, 75 °C at 20 psig, and 90 °C at 20 psig. The reaction rates (0.65 %/hr, 0.85 %/hr and 0.95 %/hr, respectively for the three reaction conditions) were used to calculate the activation energy for the LTS process.

These data can be used to extract the activation energy for the reaction from the Arrhenius equation (Connors, 1990), which is best regarded as an empirical relation:

$$r = A \exp(-E_a/RT)$$

where r is the rate, A is a constant that is independent of temperature and E_a is the Arrhenius activation energy. R is the universal gas constant and T the absolute temperature. Taking the natural logarithm of both sides of the equation gives

$$\ln r = -E_a/RT + \ln A$$

Plotting ln r against 1/T should yield a straight line whose slope is $-E_a/R$.

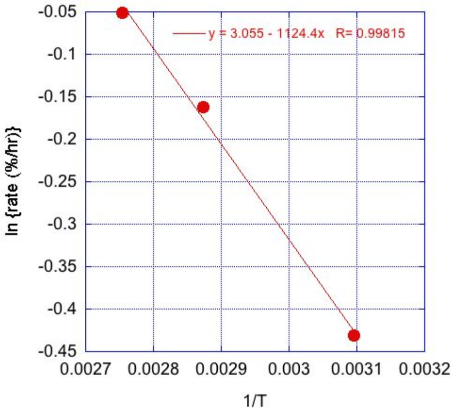

Figure 10. Arrhenius plot for LTS reaction

The slope of the straight line yields the activation energy of 2.2 Kcal/mole (8.3 kJ/mole) for the process, which suggests that the reaction is mass transfer limited by diffusion.

Experiments Supporting the Feasibility of Eliminating the Need for an Autoclave

Experiments were carried out at both Rutgers and Solidia, to reduce reaction pressure and temperature while still maintaining excellent mechanical properties in the product material.

Experiments at Rutgers on small pressed samples of 100% $CaSiCO_3$ suggests that the reaction temperature can be reduced from 90°C to 50°C at a cost of increased reaction time to reach a given level of conversion to $CaCO_2$ + SiO_2. For samples reacted at 50°C we obtain a compressive strength of 85 Mpa (12,300 psi). CO_2 pressure was reduced from 20 psig to about 5 psig without much effect on extent of conversion (approximately constant at 47%). The most important conclusion from the Rutgers work is that CO_2 pressure does not have a strong effect on reaction rate. The Rutgers results are consistent with results from the factorial design reported above.

Following the Rutgers finding on the effect of CO_2 pressure, experiments at Solidia shifted to pushing the envelope of useful reaction conditions into a region where we can eliminate the need for an autoclave. We also focused more on compositions typical of the practical mixture designs. To eliminate the need for an autoclave we need to work at or very near to ambient pressure.

LTS reaction in a temperature gradient:

The following experiment demonstrates the feasibility of carrying out the LTS reaction on practical mixtures at CO_2 pressures near 14.7 psia (i.e. 0 psig):

We cast a test block (8” long x 8” wide x 7.25” thick) whose composition was 23.6 weight % $CaSiO_3$, 43.8 % dolomitic sand, and 32.6 % Nycor 100 aggregate. The test block was weighed dry (16 Kg) and then soaked in water. The test block was placed in pure CO_2(g) in a glove box at atmospheric pressure. The CO_2(g) pressure was maintained at between 14.75 psia and 15.0 psia by a Photohelic pressure controller. The test block was heated by a planar array of three infrared quartz lamps (each lamp 300-watt,12”-long). The array was placed 2” above the block. The temperature at the top of the block was maintained at about 215°C. In this way a near-linear temperature gradient was established across the thickness direction of the block (Figure 11). The temperature measured at the bottom of the block was 70°C, which is lower than we currently use in our autoclave.

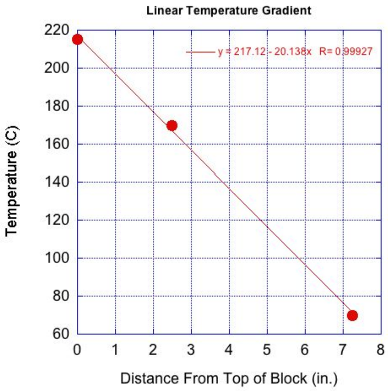

Figure 11. Temperature Gradient From Top to Bottom of Block

Under these conditions the LTS reaction proceeded spontaneously. We expected the LTS reaction rate to vary over the thickness of the test block since the temperature of the block varied from top to bottom. The LTS reaction was interrupted at successive 24-hour intervals to measure the weight gained due to CO_2 uptake in the interval so that the cumulative average extent-of-reaction could be calculated. It was necessary to dry the test block before it was weighed. The block had to be re-wetted with distilled water each time the extent-of-reaction was determined because water is required for the LTS reaction to proceed.

Figure 12 shows the cumulative average extent-of-reaction as a function of accumulated reaction time.

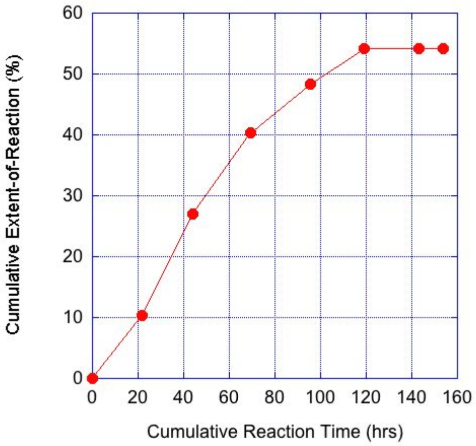

Figure 12. Cumulative Extent-of-Reaction as a Function of Time at a CO_2 Pressure of One Atmosphere

The cumulative average extent-of-reaction increased from interval to interval. It saturated at about 54%.

The reaction rate (and thus the extent-of-reaction) varied across the thickness of the test block due to the temperature gradient. The relative variation was quantified by measuring the transit time of an ultrasonic pulse over a distance of 150 mm using a pulse generator and detector. The speed of sound in the block at different heights was then calculated. The speed of sound in a solid material depends on its density. Density, in this case, is related to extent-of-reaction. Higher speed of sound corresponds to greater extent-of-reaction.

Figure 13 shows speed of sound as a function of distance from the top face of the test block (i.e. along the direction of the temperature gradient).

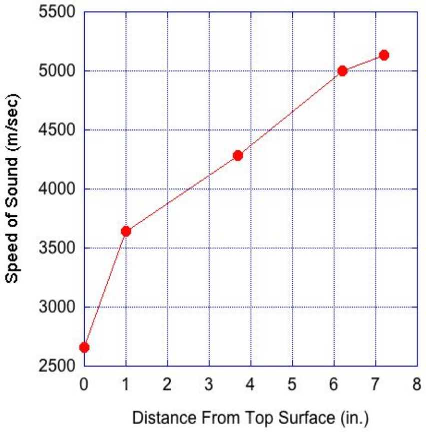

Figure 13. Speed of Sound in Test Block as a Function of Height

We know from our experience that panels having the best mechanical properties (e.g. compressive strength > 95 Mpa = 13,778 PSI) exhibit transit times in the 25-30 μs range (speeds = 5000 to 6000 m/s), which correspond to extents-of-reaction above 70%. Panels with transit times in the 30-40 μs range (speeds = 3750 to 5000 m/s) have excellent to very good mechanical properties (e.g. compressive strengths > 55 Mpa = 7,977 PSI), with extents-of-reaction in the 50-70% range. Unreacted panels exhibit transit times longer than 60 μs (speeds < 2500 m/s). Many concrete applications have compressive strengths in the 20.7-27.6 Mpa = 3000-4000 PSI range.

Figure 13 shows that measured speeds of sound were between from about 3750 m/s to above 5000 m/s over most of the block, which implies that the compressive strength ranges from about 55 Mpa (~8000 psi) to about 95 Mpa (~13,800 psi) depending on the reaction temperature, with the best properties emerging at the lowest temperature.

The experiment also gives us new information on how temperature affects the extent-of-reaction:

The speed of sound increased from top to bottom of the test block. Thus the extent-of-reaction at the top of the block was lowest and at the bottom of the block highest, i.e. extent-of-reaction is negatively correlated with temperature. This finding seems to contradict the result obtained above in the experimental design where we looked at the effect of temperature on extent-of-reaction. We found, in that case, that extent-of-reaction was positively correlated with an increase in temperature.

The results of the two experiments can be explained and made consistent by recognizing that the temperature domains for the experiments are different (50 - 90 °C in the former case and 70 – 215 °C in the present case) and have only a small overlap.

The conversion of CO_2 to bicarbonate ion, HCO_3^-, and then to carbonate ion, CO_3^{2-} requires water according to the equilibrium reaction:

$$CO_2(g) + H_2O(l) = H_2CO_3(a) = HCO_3^-(a) + H^+(a) = CO_3^{2-}(a) + 2H^+(a).$$

The LTS reaction would stop if the temperature were high enough to "dry out" the block. Thus, the high temperatures in the top half of the block could have caused water to evaporate preferentially from the top region, which could slow the reaction rate at the top relative to the rate at the bottom.

Furthermore, another factor is likely to be involved in the reduced extent-of-reaction at the top of the block: CO_2 solubility in water is lower at high temperatures. The solubility is also lower at low pressures, but we have found that increasing CO_2 pressure generally does not increase the reaction rate at constant temperature. This suggests that there is a critical CO_2 concentration that maintains an adequate carbonic acid concentration and thus bicarbonate concentration in solution to feed the turnover of CO_2 to CO_3^{2-}. At one atmosphere of CO_2 and high temperature, the dissolved CO_2 concentration could drop below the critical concentration, slowing the reaction. In this case the reaction rate could be limited by the formation rate of carbonic acid from CO_2 and H_2O, or by the rate of carbonic acid dissociation.

It follows from the two experiments that there will be an optimum temperature that maximizes overall reaction rate since the rate slows at high temperature due to insufficient water or insufficient HCO_3^-, and at low temperature due to insufficient energy to overcome the activation energy barrier for the reaction or diffusion.

This study establishes feasibility that the LTS reaction can be used to make carbonate-bonded concrete having excellent mechanical properties by reaction at 1 atmosphere CO_2 pressure and at a *temperature of 70 C (with no autoclave needed) in reasonable time (5 days).*

3.3 Finishing Operations

Finish processing comprises those steps that are necessary to bring the reacted shape to its final commercial shape and finish. Relevant finishing operations involve cutting to size; edge shaping; and grinding, polishing and sealing the surface.

Commercial processes and equipment for finishing natural stone products are readily available in the market. Such equipment, with little or no modification, is suitable for finishing HFC materials.

4.0 Product Evaluation and Acceptance of Building Facades

4.1 Mechanical Performance Evaluations

Compressive strength (ASTM C170) and Flexural Strength (ASTM C880) were measured at Solidia. Depending on mixture formulation and reaction parameters we saw compressive strengths in the range of 50 to 170 MPa (8,000 to 25,000 psi), and modulus of rupture (flexural strength) in the range: 10 to 20 MPa (1,000 to 3,000 psi). All properties were measured by the indicated tests on samples taken from 5’ by 2’ façade panels that were made from calcium silicate, dolomitic sand and precipitated calcium carbonate.

Table 4. Mechanical Properties

Test	Property	Total number of Samples	Mean	Std. Dev.	Max.	Min.
ASTM C67 ASTM C170	Compressive Strength	302	114 MPa	22 MPa	173 MPa	52 MPa
ASTM C99	Modulus of Rupture	100	16 MPa	2 MPa	22 MPa	11 MPa

4.2 Durability Evaluations

Water absorption, tensile strength and impact resistance were also measured at Solidia on samples taken from 5’ by 2’ façade panels or on full panels. None of these panels or samples was resin-treated. The results are shown in Table 5.

Table 5. Properties that Correlate with Durability

Test	Property	Total Number of Samples	Mean	Std. Dev.	Max.	Min.
C67 C97	Water Absorption (5 hr boil)	315	4.0 %	0.6 %	5.7 %	2.2 %*
Anchor Pull	Tensile Strength	46	3.2 kN	0.8 kN	4.5 kN	1.7 kN
Test	**Property**	**Total Number of Samples**	**Passing @ 0.76 lb-ft**	**Passing @ 1.29 lb-ft**	**Passing @ 2.02 lb-ft**	**Passing @ 3.13 lb-ft**
TAS 201-94 ISO 7892	Impact Resistance	100	100%	96%	66%	45%

* Panels sealed with epoxy resin absorb less than 1% water by weight and are therefore more durable than untreated panels, however at increased cost.

4.3 Accelerated Weathering

In the salt spray test *(ASTM B117)* a 5% NaCl solution is aerosolized in a salt fog chamber at 35C. 8” x 4” test samples, cut from façade panels, were exposed to the salt fog for 500 hours. The test is pass/fail according to whether there is visible damage, cracking, or significant weight change.

In the freeze-thaw test *(ASTM C67)* a 4” x 8” test sample is cut from a 5’ x 2’ façade panel. The test sample is cut to yield a pair of 4” x 4” pieces. One of the 4” x 4” pieces is maintained at ambient conditions while the other one is subjected to 50 cycles of freeze/thaw consisting of 20 hours of freezing at < -9°C and 4 hours of thawing in a recirculating water bath that is maintained at 25°C. The test is performed using deionized water (for normal freeze/thaw) or pH 4 sulfuric acid solution (for acid freeze/thaw). The test is carried out until damage is visible or 50 cycles are complete without visible damage.

Table 6. Accelerated Weathering Tests

Test	Property	Samples	Hours or Cycles	Number Passed
ASTM B117	Salt Spray	10	500 hours	10
ASTM 67	Freeze/Thaw	4	50 cycles	4
ASTM 67	Acid Freeze/Thaw*	4	50 cycles	4

*Acid freeze/thaw uses pH 4 sulfuric acid instead of pH 7 water and is a more severe test.

4.4 Staining and Adhesion

Various grouts, adhesives and caulking were used to place arrays of 1’ x 1’ tiles on a test wall using various mortars to determine suitability of mortar for adhesion. As expected some mortars are better than others. We have had no problems with staining, bleeding or loss of adhesion.

Table 7. Products Tested for Staining and Adhesive Strength

Product	Manufacturer	Function
White Universal Adhesive	Laticrete International, Inc.	Adhesion to substrate
Megabond 1 Part Adhesive	Laticrete International, Inc.	Adhesion to substrate
Granirapid 2 Part Adhesive	Mapei Corporation	Adhesion to substrate
Sanded Grout 1500	Laticrete International, Inc.	Fill joints
Grout Enhancer	Laticrete International, Inc.	Fungicide & Mildew protection

5.0 Benefits Assessment

To calculate Btu savings for concrete production, energy use per tonne of $CaSiO_3$ used is compared to energy use per tonne of PC used. 8.3 tonne of concrete is assumed to contain 1 tonne of PC or 1 tonne of carbonated $CaSiO_3$. The difference in energy use for the two cases is calculated to be 70%. Table 8, below, outlines the basic calculation and is followed by a line-by-line accounting of the assumptions that go into the table. This calculation is conservative. The extent-of-reaction for $CaSiO_3$ was assumed to be only 45 wt% and heat losses for the drying oven and autoclave were not minimized.

On this model, replacement of 25% of the US concrete market by HFC instead of traditional concrete would result in approximately 18% savings of energy.

This calculation does not include additional projected energy use savings from the reduction in material required for each application because of increased strength of HFC as compared to PC concrete.

Table 8. Energy and CO_2 per Tonne of Cement Associated with U.S. Cement Manufacturing and Concrete Production from U.S. Cement: PC vs. HFC

		PC		HFC	
Component	**Process Operation**	**10^3 BTU/tonne cement**	**Kg CO_2 /tonne cement**	**10^3 BTU/tonne cement**	**Kg CO_2[6] /tonne cement**
Cement raw materials	Quarry+Beneficiation	42.9[2]	4.0	59.9[2]	5.6
Concrete raw materials	Quarry+Beneficiation	160.7	14.4	160.7[3]	14.4
Cement	Raw Grinding	93.9	16.9	341.6[4]	61.5
Cement	Kiln Fuels	4617.1	432.7		
Cement	Kiln Reaction		543.8		
Cement	Finish Milling	270.6	48.6		
Concrete	Mixing	353.7	63.6	353.7[3]	63.6
Concrete	Transport	697.3	51.0	697.3[3]	51.0
Concrete	Hydration or Carbonation	-336.3[1]	0.0	262.6[5]	-170.5[7]
Concrete	**Total**	6236.3	1175	1875.5	25.4
	% of PC	100%	100%	30%	2%
	% change on using HFC			**-70%**	**-98%**

Energy and CO_2 data on Portland cement are cited from(Choate (2003).

[1] Average heat of hydration of Portland Cement (type I, II and III). Assume Portland cement is hydrated for 7 days and released to the environment.

[2] Average $CaSiO_3$ ore concentration is 50% (Penner (2004). Thus, HFC quarrying and beneficiation is take as 2X that of limestone in Choate Table A.5

[3] PC concrete and HFC concrete energy and CO_2 costs assumed to be the same.

[4] Assumes grinding $CaSiO_3$ to -400 mesh at 91 kWh/short ton = 100 kWh/tonne (Penner 2004).

[5] Incorporates energy due to drying (3 h) and carbonation (12 h) steps . Assumes released heat is 100% recovered. [5]Incorporates CO_2 emission from drying (3 h) and carbonation (12 h) processes.

[6] Same as PC case but scaled in proportion to relative energy usage.
[7] Assumes 45 wt% $CaSiO_3$ is converted to $CaCO_3$.

Methodology for Determining Energy Consumption during $CaSiO_3$ Carbonation (Table 8):

Total energy consumed during the LTS is mainly composed of three processes: a) Quarrying and crushing, b) Grinding, and c) LTS reaction.

Assumptions:

a) LTS casting process is similar to cement casting process. Heat gain or loss is not considered.
b) Energy consumed during quarrying and crushing of different minerals are similar.
c) Heat released from $CaSiO_3$ carbonation is used in maintaining autoclave temperature and heat released in Portland cement hydration is lost to the environment.
d) No heat loss during the transfer of the dried samples from the drying oven to the autoclave.
e) Dimension of the drying oven is same as that of the autoclave.

Footnote numbers in the following calculations are keyed to the footnote numbers shown in selected cells in Table 8.

Hereafter, the details for the calculations given in the Table 1 in the main text is

explained as follows.

[1] Heat generated by hydration of PC (average of Type I, II and III cements) is -354.8 kJ/kg X 1000 Kg/tonne X 0.9478 btu/kJ = 336279 BTU/tonne after 7 days.

[2] Energy consumed on quarrying and crushing process: There is 44 % weight loss during calcination ($CaCO_{3(s)} = CaO_{(s)} + CO_{2(g)}$) of Portland Cement (PC). If PC consists of approximately 65 wt% CaO and other constituents (SiO_2, Al_2O_3, Fe_2O_3, etc), to produce 1 metric tons of PC the total amount of $CaCO_3$ (1.16 tonne) and other constituents (0.35 tonne) required will be 1.51 metric tons, which corresponds to 42.9 BTU energy expenditure (Table A.6 in Choate (2003)).

Compared to PC manufacturing, $CaSiO_3$ mineral is used directly in the carbonation reaction without a kiln processing. Assuming 50 wt% $CaSiO_3$ in wollastonite ore (Penner (2004)). Two metric tons of ore are used to produce 1 metric ton of $CaSiO_3$, which corresponds to 59.9 BTU of energy (twice the energy in Table A.5 in Choate (2003) assuming mining cost for wollastonite and limestone are the same).

[4] Raw material grinding: The energy consumed on grinding pure $CaSiO_3$ to -400 mesh is 91 kWh/short ton, Penner (2004), which is 100.1 kWh/mt X 3412.1416 BTU/kWh = 341555 kWh/mt.

[5]For simplicity, we calculate the energy consumption on drying and reacting 1 mt (1000 kg) of $CaSiO_3$. To get the energy consumption for concrete we add in energy required to heat aggregate and sand 90 °C during the carbonation reaction.

If the green density of a cast tile is 2.2 kg/liter and dimensions of the tiles are 0.91 m (3 feet, width) × 1.52 m (5 feet, length)× 0.0254 m (1 inch, thickness), then 13 tiles can be casted from 1 mt of $CaSiO_3$. These 13 tiles will be vertically stacked at a spacing of 0.0254 m (1 inch) and placed into the drying oven/ autoclave. If the oven/autoclave is 60% full, the dimensions of the oven/autoclave will be 1.8 m (6.0 feet, width) ×1.1 m (3.6 feet, height) × 0.8 m (2.5 feet, depth). Therefore, the volume of the drying oven/autoclave is about 1.5 m^3 (52 ft^3). A stainless steel cart is required to hold the tiles. The weight of the cart is assumed to be 0.2 mt (200 kg).

The LTS process contains three steps:

Step 1: Casting of tiles: Energy consumed in this process is not considered (main assumption (a)).

Step 2: Drying of cast tiles: Placing of wet tiles (including ~15 wt% water, i.e. 85% solid loading) on the cart at ambient temperature, 23°C (296 K), and then transferring the cart to the drying oven at 90°C (363 K) and holding for three hours.

Energy consumption during drying in the oven operating at 90 ^oC (363 K)*:*

Using $Q = mc_p\Delta T$, where, Q = heat lost or gained (kJ), m = mass of object heated/cooled (kg), c_p = specific heat capacity (kJ/(kg-K)), and ΔT = change in temperature (K):

Heat 1 mt of casted tiles (c_p = 0.75 kJ/(kgK) (calcium silicate):

Q1 = 1000 kg X 0.75 kJ/(kgK) X (363 -296) K = 50250 kJ = 13.96 kWh

Heat 15 wt% of water in 1 mt of the tiles (c_p = 4.19 kJ/(kgK) for water:

Q2 = 1000 kg X 15% X 4.19 kJ/(kgK) X (363 -296) K = 42110 kJ
= 11.70 kWh

Heat cart (c_p = 0.50 kJ/(kgK) (stainless steel 304):

Q3 = 200 kg X 0.50 kJ/(kgK) X (363 -296) K = 6700 kJ = 1.86 kWh

Heat loss of the drying oven: In our plant, natural gas 6HP steam boiler is used. The boiler capacity is ~72 kW and its efficiency is 80%. If heat loss of the oven is controlled to be ~3%, the total heat loss (Q4) after 3 hours of drying will be Q4 = 72 kW X 80% X 3% X 3 h = 5.18 kWh

Thus, total energy consumed in drying Step 2 is:

Q1 + Q2 + Q3 + Q4 = (13.96 + 11.70 + 1.86 + 5.18) kWh = 32.70 kWh

Step 3: Carbonation reaction: - Transferring the dried tiles into the autoclave and heating the autoclave to 90 ºC (363 K) for 19 h.

Energy consumption during LTS reaction operating at 90 °C (363 K)*:*

Using Q = $mc_p\Delta T$, where, Q = heat lost or gained (kJ), m = mass of object heated/cooled (kg), c_p = specific heat capacity (kJ/(kg-K)), and ΔT = change in temperature (K):

Heat water in the autoclave (c_p = 4.19 kJ/(kgK)

Q5 = 11.8 liter X 1 kg/liter X 4.19 kJ/(kgK) X (363 -296) K = 3312.6 kJ
= 0.92 kWh

(Note that water used in 500 cubic feet of reactor in our plant is 30 gallon (maximum), therefore the water required for the designed autoclave above (52 cubic feet) is 3.12 gallon, i.e., 11.8 liter.)

Heat cart (c_p = 0.50 kJ/(kgK) (stainless steel 304):

Q6 = 200 kg X 0.50 kJ/(kgK) X (363 -296) K = 6700 kJ = 1.86 kWh

Heat loss from the autoclave: Similar to the heat loss from the drying oven, the total heat loss after 19 hours of the reaction will be

Q7 = 72 kW X 80% X 3% X 19 h = 32.83 kWh

Thus, total energy consumed on Step 3 is:

Q5+Q6+Q7 = (0.92 + 1.86 + 32.83) kWh = 35.61 kWh.

Total energy consumption for the carbonation process is the summation of step 1, step 2 and step 3, i.e., 68.31 kWh/mt (32.70 + 35.61 = 68.31 kWh/mt).

$CaSiO_3$ carbonation is an exothermic reaction releasing 87 kJ/mol of heat. If the carbonation conversion for $CaSiO_3$ is 45 wt%, the total heat released from 1 mt of $CaSiO_3$ carbonation will be -93.75 kWh.

If we assume the released heat is 100% recovered, since it contributes to maintaining the temperature of the autoclave, then the **net energy consumption for carbonation of 1 mt of $CaSiO_3$ in the LTS process is**

-28.77 kWh ((68.31 - 93.75) kWh = -25.44 kWh, exothermic).

For the case of concrete, coarse and fine aggregates must also be heated to the temperature of the carbonation reaction. We assume the concrete is 12% binder and 88% aggregate. 1 tonne of cement would make 8.3 tonnes of concrete of which 7.33 tonnes would be aggregate.

Assuming the heat capacity of the aggregate to be 0.75 kJ/Kg-K, the heat required to heat the aggregate from ambient temperature to 90 °C is

Q8 = 7333 kg X 0.75 kJ/(kg-K) X (363 -296) K = 368483 kJ
= 102.36 kWh

Therefore, the net energy needed to make 8.3 tonnes of HFC concrete, which uses 1 tonne of $CaSiO_3$, is

-25.44 kWh + 102.36 kWh = 76.92 kWh = 262461 BTU

[6]Energy consumption vs. CO_2 emission:

For the LTS process, we calculate the CO_2 emission for a process step to be the emission in the PC case scaled by the energy ratio for the two cases. For example, for the Raw Grinding operation the CO_2 emitted in the case of HFC is 16.9 X (341.6 / 93.9) = 61.5 Kg CO_2 per tonne of cement.
The total emitted CO_2 for all processing steps except the carbonation reaction is then 195.9 kg CO_2 per tonne of cement

[7]If 1 tonne of $CaSiO_3$ is completely carbonated, 378.9 kg of CO2 is sequestered as $CaCO_3$. If 45 wt% is carbonated then 170.5 kg of CO_2 is sequestered.

Thus, the net CO_2 emission for concrete, per tonne of $CaSiO_3$ consumed is

195.9 kg – 170.5 kg = 25.4 kg

6.0 Commercialization

Solidia Technologies has pioneered a breakthrough ceramics technology based on a process called Low Temperature Solidification (LTS) enabling a suite of cost competitive and sustainable building and construction products. In order to maintain capital efficiency and significantly increase the speed by which the technology is dispersed, the company has decided to license production of end applications to existing manufacturers.

While the potential scope for Solidia technology within the building and construction segments is quite large and expected to be upwards of $150B worldwide[1], Solidia has identified the US cladding market (addressable market of $1.45B) as its first target market due to its performance fit with our technology and the stage of technical development achieved. High-end cladding also is priced in a range in which we can be cost competitive ($7-$15 per square foot) and offer margins that can support sharing of revenue with a licensor (15-40%).

The cladding market is fragmented and geographically dispersed, providing a challenge for large scale licensing. The company will target large-scale producers as its primary licensees in an effort to gain the largest market share in the shortest period of time. Large-scale producers also have been found to have a greater capacity to absorb new technology and adapt to new capability.

We are currently in discussion with a number of potential licensees, though no formal agreements have been signed.

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[1] Total addressable US building and construction market for Solidia technology is ~ $50 B; world-wide market is estimated as 3X US market

7.0 Accomplishments

We made a number of significant accomplishments during the course of this project:

During the period of this project, we agreed to partner with a global equipment manufacturer named Simec. Simec has a long history of designing and manufacturing equipment for the stone industry. They will work with us to develop a fully-automated line which we will provide to potential licensees as a turn-key manufacturing process along with the “recipe” for creating our materials.

We also developed relationships with a number of façade manufacturers and distributors both domestically and abroad with the intent of licensing the technology to various manufacturers to enable more rapid commercialization. These discussions continue and are extremely promising. We hope to have our first licensee during 2012.

The company closed a second round of private investment and secured $27M from KPCB, Bright Capital (the venture capital arm of Ru-Com, a large Russian construction conglomerate), BASF and BP.

We also recently launched our company website: www.solidiatech.com

8.0 Conclusions

Hydrate Free Concrete (HFC) is an extremely strong and durable advanced material that significantly reduces energy use in the cement and concrete industry and consumes CO_2 in the production process. In this project, we were able to significantly enhance the production process and optimize the reaction conditions to enable progress toward developing a scalable and commercial ready manufacturing process. We were also able to assess and compare a wide range of mechanical properties, showing that our materials are stronger and more durable than traditional stone and concrete building products. We made significant progress toward understanding the façade market, which we plan to use as our first point of market entry. We developed relationships with strategic partners that will enable rapid commercialization.

From the research undertaken we found that:

- An existing commercial mixer can be configured to develop the highly sensitive mix of powders required for our process
- Temperature has a significant effect on extent-of-reaction; the extent-of-reaction in turn is highly correlated with compressive strength of the material
- The LTS reaction appears to be mass-transfer-limited by diffusion
- Extent-of-reaction is not strongly dependent on pressure
- Through experimentation with process and composition, we showed a 20% temperature and pressure reduction in the LTS process relative to the baseline of 100 °C and 20 psi to 80°C & 16 psi
- It is possible to create a 7” block of hydrate-free-concrete at ambient temperature and atmospheric pressure
- Our materials exhibit comparable or superior mechanical and durability properties as compared to traditional stone and concrete

9.0 Recommendations

There are a number of areas in which further research is highly recommended.

The process of creating hydrate-free-concrete (HFC) can be viewed as a technology platform from which a very wide range of applications can be derived. This study focused on the production of façade panels. Further work should be undertaken to understand required mechanical and durability properties for other building materials applications such as roof tiles, floor tiles, interior wall panels, pavers, etc.

In addition, because the process of production is undertaken at such low temperatures and pressures, sensors and electronics can easily be incorporated directly into the material during production. This should be further investigated and processes to ensure successful integration of electronic equipment into the material should be developed.

In order to make large pre-cast panels while reducing production cost, it will be necessary to move away from using an autoclave as a reactor. Further study of methods to carryout the reaction at ambient pressure and further reduced temperature should be undertaken.

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