

Performance of CO2-reducing cement based on calcium silicates

Sada Sahu^{1,a}, Richard C. Meininger^{2,b}

¹Solidia Technologies, Piscataway, United States ²Federal Highway Adminstration, Turner-Fairbank Highway Research Center, McLean, United States

> ^assahu@solidiatech.com ^brichard.meinininger@dot.gov

ABSTRACT

A new type of cement is made from raw materials that are typically used to make ordinary Portland cement (OPC) clinker. The kiln feed raw meal chemistry is adjusted to produce a clinker rich in low-lime calcium silicate phases such as CS and C3S2 instead of the high-lime phases C3S and C2S typical of OPC. Production of such clinker requires 30% less limestone compared to OPC clinker and sintering temperatures can be reduced to as low as 1200°C compared to 1450°C for OPC. Manufacturing of this cement reduces the CO2 emissions by 30% than OPC. Addition of gypsum is not needed during the grinding of this clinker to produce the cement. This cement does not harden by hydration; rather, it hardens by carbonation. Its main carbonation products are CaCO3 and SiO2. During the carbonation process this cement can sequester up to 300 kg per ton of cement.

The mechanical properties of concrete made using this cement were tested using ASTM C39 (compressive strength), C78 (flexure strength), C469 (splitting tensile strength), and C496 (modulus of elasticity); durability performance following ASTM C666 (freezing and thawing resistance), C227 (ASR) and C1012 (sulfate resistance) are reported in this paper.

This cement is a patented cement of Solidia Technologies and is commercially available as Solidia Cement [™].

Keywords: CO2-reduction, carbonation, CaCO3, CO2-emission, CS, durability



1. INTRODUCTION

Portland cement production is a very energy intensive process and is a significant contributor to global greenhouse gas (GHG) emissions. World cement production has reached 4.65Gt in 2016 (Cembureau.eu) and is estimated to contribute about 8% of total anthropogenic CO₂ emissions (carbonbrief.org). Cement industry is the 2nd largest industrial greenhouse gas (GHG) emitter. During the UNFCC, COP-21 meeting in Paris (The Paris Agreement) the member nations agreed to keep the global temperature rise within 2°C of the preindustrial era by the end of 21st century (UNFCC.int). Following the International Energy Agency's guidelines to meet this 2 Degree Celsius Scenario (2DS), the World Business Council for Sustainable Development's (WBCSD) Cement Sustainability Initiative (CSI) group has developed a Global Technology Roadmap called "Low-Carbon Transition in Cement Industry" (wbcsdcement.org). This roadmap has set a target to reduce the CO₂ emissions from 2.2 Gt emitted in 2014 to 1.7 Gt by 2050. This must be accomplished despite a predicted 12 to 23% growth in worldwide cement production. To achieve this goal, a radical approach to cement manufacturing, use and alternative CO₂-funtional cement chemistry must be undertaken.

1.1 Portland Cement and CO₂ Emissions

Portland cement clinker is produced by burning a mixture of calcareous and siliceous raw materials in a rotary kiln at very high temperature. In this process, calcareous material, such as limestone, and siliceous materials, such as sand, clay, or similarly composed materials, are ground and sintered at a temperature of ~1450°C in a rotary kiln. The clinker nodules produced by the sintering process are then ground and combined with ~5% gypsum to produce finished cement. Portland cement production emits CO_2 by two direct mechanisms:

- The primary source of CO₂ emission is the decomposition of CaCO₃ to produce CaO(s) and CO₂(g).
- Heat necessary to achieve the high sintering temperature in the kiln is supplied through the burning of fossil fuels.

 CO_2 -emissions are also indirectly created through the use of electricity. Mining, crushing and grinding of raw materials to create the raw meal feed to the kiln followed by final grinding of the cement clinker contribute to this category. Emissions from used electricity can vary widely depending on the source of the electricity and are relatively small, about 10% of the total CO_2 emissions. Due to this, the CO_2 emissions from the use of electricity are omitted in the calculation here.

Portland cement clinker typically contains up to 70% CaO by weight. The calcination of limestone used to achieve this proportion of CaO releases ~540 kg of CO₂ gas per ton of clinker (Barcelo L., *et al*, 2013). The CO₂ emitted from combustion within the kiln can vary depending on the type and efficiency of the kiln as well as the fuel source. A high efficiency kiln with a five stage preheater and precalciner has an efficiency of 58% which results in a CO₂ emission of ~ 270 kg CO₂ per ton of clinker. An older wet-process kiln with an efficiency of 26% can have a CO₂ emission from combustion process of Portland clinker production contributes an associated specific CO₂ emission of 810 kg to 1,146 kg per ton of clinker produced.

1.2 Carbonatable Calcium Silicate Cement

A new type of cement, carbonatable calcium silicate cement (CCSC), has a chemistry and functionality that allows it to have a significantly reduced CO₂ footprint compared to Portland cement. In CCSC, low-lime calcium silicate phases such as wollastonite/pseudowollastonite (CaO·SiO₂, CS) and rankinite (3CaO·2SiO₂, C₃S₂) are preferred to the high-lime alite (3CaO·SiO₂, C₃S), belite (2CaO·SiO₂, C₂S) tricalcium aluminate (3CaO·Al₂O₃, C₃A), and tetracalcium aluminoferrite (4CaO·Al₂O₃·Fe₂O₃, C₄AF) phases of Portland cement. Gypsum addition is not required during the grinding process of the clinker.

1.3 Carbonatable Calcium Silicate Cement CO2 Emissions

As the CCSC contains low-lime containing calcium silicate phases, the lime content can be reduced from 70% to 45%, which translates to a reduction of CO_2 emissions from 540 to 370 kg per ton of



clinker during the calcination process. As this clinker is sintered at ~1250°C as opposed to ~1450°C for OPC the fuel consumption is reduced by approximately 30%. This translates to approximately 30% reduction in CO_2 emissions from the fossil fuel combustion process in the kiln. The CO_2 emissions as calculated by reduction of $CaCO_3$ in the raw mix and an estimated 30% fuel savings are shown in Table 1. The details of the CO_2 saving opportunities are also discussed in previous publications (DeCristofaro N., Sahu S., 2014, Atakan V., *et al*, 2014, DeCristofaro N., et al, 2017).

CO ₂ Emission source	Per ton of OPC clinker	Per ton of CCSC clinker	Reduction, %
Calcination	540 kg	375 kg	30
Combustion	270 kg	190 kg	30
Total CO ₂ Emission	810 kg	565 kg	30

Table 1. Summary of CO₂ emissions as reported for Portland cement clinker (OPC) compared to the predicted CO₂ emissions for a carbonatable calcium silicate cement clinker (CCSC).

2. EXPERIMENTAL

2.1 Synthesis of Carbonatable Calcium Silicate Cement

CCSC was produced using a rotary cement kiln. Limestone and sand typical of ordinary Portland cement production were ground to 82% passing 200 mesh. The ground raw material was processed in an industrial rotary kiln with a 4-stage preheater and precalciner. The burning zone temperature was maintained at ~1260°C.

2.2 Finish Milling

The produced clinker was ground using a vertical roller mill (VRM) to a Blaine fineness of approximately 500 m²/kg. The particle size of the ground cement was measured by laser diffraction in water suspension (Malvern Mastersizer 2000, Malvern, UK).

2.3 Chemical and Phase Analysis

The clinker was sampled and analyzed for elemental composition by X-ray fluorescence (XRF) and phase composition by X-ray diffraction (XRD). Clinker samples were made into fused glass beads for XRF analysis and measured using a Panalytical Axios WDS spectrometer (Alemo, NL). Data was collected using a Panalytical Cubix³ diffractomer (Almeno, NL) with 1600W Cu K_a radiation from 5° - 65° 2Θ, 0.017°/s, 60s per step. Amorphous content was determined by comparison of the collected pattern to a crystalline rutile standard. The reported silica was the sum of quartz, cristobalite and trydimite.

2.4 Concrete, Mortar Formulation and Curing

Air-entrained concrete with 75 mm maximum-size coarse aggregate was produced using the mixture proportions provided in Table 2. The water/cement ratio (w/c) was maintained at 0.39. Concrete cylinders of 100mm x 200mm, prisms of 75mm x 100mm x 400mm, 100mm x 100mm x 350mm and 150mm x 150mm x 525mm were cast. The concrete samples were demolded after precuring in ambient condition for ~6h and further cured in 100% CO_2 environment for 72h at elevated temperatures (< 100°C). The cured samples were tested for compressive strength according to ASTM C39, split tensile strength according to ASTM C496, flexural strength according to ASTM C78, and elastic modulus according to ASTM C469. Freeze-thaw resitance tests on prisms of 75mm x 100mm x 400mm were carried out according to ASTM C666, procedure-A.

Mortar bars, 25mm x 25mm x 300mm with ASTM C490 gage studs for length change measurement, were prepared following the procedure described in ASTM C227 for alkali-silica reaction testing. The mixture proportions of materials used are provided in Table 3. These mortar mixes were produced using the same admixtures as concrete mixes only without air entering admixture. To comply with the gradation requirement, a small percentage of the concrete sand was used in addition to the reactive fused silica sand. The reference OPC used in the study had a total alkali content of Na₂Oeq = 0.96%. The mortar bars were precured in the mold for ~5h before demolding. The demolded mortar bars were further cured in 100% CO₂ atmosphere for 40h. The alkali-aggregate reaction tests were carried out



according to the test procedure described in ASTM C227. The length change measurements were made weekly for the first four weeks and subsequently after every four weeks.

Ingredients	Contents (kg/m3)
Solidia Cement	350.0
Concrete Sand (FM = 2.35)	821.0
Coarse Aggregate 3/8" (9.5 mm)	414.0
Coarse Aggregate 3/4" (19 mm)	737.0
Water	136.0
High-Range Water Reducer (Glenium 7500)	5 ml/kg of cement
Air Entraining Admixture (MB AE-90)	3 ml/kg of cement
Set-Retarding Admixture (Admixture-13)	5 ml/kg of cement

Table 2. Mixture proportions of air-entrained CCSC concrete.

Table 3. Mixture proportions of mortar for ASR testing.

Ingredients	Contents (g)
Solidia Cement	600
Fused Silica Sand	1317
Concrete Sand	333
Water	175
High-Range Water Reducer (Glenium 7500)	5 ml/kg of cement
Set-Retarding Admixture (Admixture-13)	5 ml/kg of cement

Mortar bars, 25mm x 25mm x 300mm with ASTM C490 gage studs for length change measurement, were also prepared following the procedure described in ASTM C1012. The mixture proportion of materials used to prepare the mortar is provided in Table 4. However, in this mortar mix, 3ml/kg of high-range water reducer was used. The mortar bars were precured in the mold for ~5h before demolding. The demolded mortar bars were further cured in 100% CO₂ atmosphere for 40h. The test specimens were exposed to 352 moles of sodium sulfate solution (50g/L). Sulfate expansion tests were carried out according to the test procedure described in ASTM C1012. The length change measurements were made weekly for the first four weeks and subsequently after every four weeks.

Ingredients	Contents (g)
Solidia Cement	500
ASTM Standard Sand	1375
Water	175
High-Range Water Reducer	3 ml/kg of cement

Table 4. Mixture proportions of mortar for sulfate resistance testing.

2.5 Air-Void Analysis

Air-void content and distribution in hardened concrete was measured by an automated air-void analyzer, RapidAir457. This procedure is consistent with ASTM C457 Procedure C.



2.6 Scanning Electron Microscopy (SEM)

A small cut portion of a concrete cylinder was dried, epoxy impregnated and cured. After curing, one of the surfaces was polished to ¼ micron finish. The polished surface was sputter coated with carbon and examined under a SEM in backscattered mode. A Tescan, Mira 3 Field Emission SEM at 20 keV was used to acquire the images.

3. RESULTS AND DISCUSSION

3.1 Chemical Composition of the Clinker

The average elemental composition of the CCSC clinker as measured by XRF and expressed as oxides is shown in Table 5.

Oxides	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	Na₂O	K ₂ O	SO ₃
(Wt. %)	46.6	47.9	2.6	0.8	0.8	0.4	0.7	0.2

Table 5. The average composition of CCSC clinker.

3.2 Phase Composition of the Clinker

The average phase composition of the CCSC clinker as determined by quantitative X-ray diffraction with Reitveld refinement is shown in Table 6.

Table 6. The average phase composition of CCSC clinker as measured by X-ray diffraction.

Phases	Formula	Concentration (Wt. %)
Pseudowollastonite	CaSiO₃	51.0
Wollastonite	CaSiO₃	0.2
Rankinite	Ca ₃ Si ₂ O ₇	13.1
Belite	Ca ₂ SiO ₄	2.7
Amorphous		24.4
Melilite	(Ca,Na,K)2(Al,Mg,Fe ²⁺)[(Al,Si)SiO7]	5.9
Brownmillerite	Ca ₂ (Fe,Al) ₂ O ₅	0.6
Silica	SiO ₂	1.9
Lime	CaO	0.4

3.3 Particle size of the Solidia Cement

The statistics of the particle size distribution is provided in Table 7.

Table 7. Particle size distribution statistics

d 10	d 50	d 90
1.52 µm	14.56 µm	44.30 μm

3.4 Carbonation Reactions During Concrete Curing

The calcium silicate phases present in the Solidia Cement react with CO_2 as described in equations 1-3.

$$CaSiO_{3}(s) + CO_{2}(g) \xrightarrow{H_{2}O} CaCO_{3}(s) + SiO_{2}(s)$$
(1)



$$Ca_{3}Si_{2}O_{7}(s) + 3CO_{2}(g) \xrightarrow{H_{2}O} 3CaCO_{3}(s) + 2SiO_{2}(s)$$
(2)

$$Ca_2SiO_4(s) + CO_2(g) \xrightarrow{H_2O} 2CaCO_3(s) + SiO_2(s)$$
(3)

Additionally, the amorphous component of the cement can carbonate to some extent, depending on its calcium content and bulk chemistry. The formation of $CaCO_3(s)$ and $SiO_2(s)$ is associated with a net solid volume increase (due to the incorporation of CO_2). This reaction is what gives CCSC the ability to generate strength, similarly to the solid volume increase produced by hydration in Portland cements (due to the incorporation of water). Details of the carbonation process of this cement are provided in other publications (Sahu S., et al, 2015).

Microstructure of cured concrete was examined by scanning electron microscopy (SEM) in backscattered electron (BSE) imaging mode. An SEM-BSE image of the hardened CCSC concrete is provided in Figure 1. The brightest particle in the image is ferrite phase. The second brightest particles are reactive calcium silicate phases and are generally surrounded by a dark rim of calcium depleted amorphous silica. In some instances the cement particle is completely reacted leaving the imprint of original cement particle rich in silica. The space between cement particles is filled with CaCO₃ particles of intermediate brightness. The dark area is the porosity left after the carbonation process. The carbonation of CCSC produces a densified material through the direct precipitation of CaCO₃ in the pores, where it acts to bind together the components of the concrete.



Figure 1. FESEM-BSE image showing the microstructure of the carbonated paste area.



For effective CO_2 curing of test specimens and concrete products, an enclosure is required with a CO_2 -rich gas environment, along with control of temperature and relative humidity. The curing process is counter diffusion between moisture and CO_2 . The cross section of concrete elements thus produced is limited by the depth that the CO_2 can travel internally from a free surface exposed to a sufficient supply of gaseous CO_2 . This limitation mostly comes from the type and efficiency of curing equipment deployed. In the 1st phase this technology has been used to cure thinner concrete bodies. The process has been proven in making concrete masonry units, bricks, tiles, pavers, and other smaller precast concrete elements.

3.5 Mechanical properties of the concrete

The results of mechanical properties of concrete are provided in Table 8. The results show that the performance of this carbonated CCSC concrete is comparable to that of OPC concrete of similar mix design.

Property	ASTM Test	Results
Compressive strength, psi	C39	9,145
Split tensile strength, psi	C496	931
Flexural strength, psi	C78	783
Modulus of elasticity, psi	C469	7,192,400
Poisson's ratio	C469	0.17

Table 8	. Test	results	of	mechanical	pro	perties	of	CCSC concrete.	
		results	U 1	meenamoar	piv	perties	U 1		

3.6 Freeze-thaw resistance of the concrete

Corner 4

Average

5.91

6.52

Freeze-thaw durability is a crucial property of concrete for cold weather applications and is influenced by a wide variety of factors such as aggregate type, water content, and mix composition. In Portland cement concrete, air-entraining admixtures are used to significantly increase service life in cold weather. The effectiveness of air entrainment in CCSC concrete is therefore an important component of freeze-thaw resistance. The air-void content and distribution of this particular mix is provided in Table 9. The air-void content and spacing factor of this concrete are within the range to provide good protection from freeze-thaw damage.

specimen.									
TFHRC ID	Corner	Air Content (%)	Specific Surface (in ⁻¹)	Spacing Factor (in.)	Void Freq. Vds/in. (in ⁻¹)	Average Void Length (in.)			
	Corner 1	7.04	660.6	0.0056	11.63	0.0061			
	Corner 2	6.9	663.4	0.0057	11.45	0.006			
15219	Corner 3	6.24	619.9	0.0067	9.67	0.0065			

848.8

698.18

0.0051

0.0058

12.54

11.32

0.0047

0.0058

Table 9. Air-void parameters of CCSC-concrete as determined by ASTM C457, Procedure C, with four replicates, each starting from a different corner of the prepared polished concrete specimen

Assessment of CCSC freeze-thaw durability was performed as specified in Procedure A (freezing in water and thawing in water) of ASTM C666: Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing. This method evaluates freeze-thaw resistance of concrete as a function of percentage relative dynamic modulus of elasticity (RDME) with relation to the number of freeze-thaw cycles. The results are plotted in Figure 2. ASTM C666 requires testing up to 300 cycles. If the RDME values of a concrete specimen drop below 60% it is considered to have failed. As there was



not a significant drop in RDME values after 300 cycles, the test was continued until 540 cycles. After 540 cycles the RDME values dropped to only 96%. The mass change over this period is plotted in Figure 3. It shows the specimens gained a small amount of mass over the testing period. This is most likely due to saturation of the nano-pores present in the silica gel over time (Villiani C., et al, 2014). The specimens showed very minor scaling at the end of testing. These results demonstrate that carbonated concrete has good freeze-thaw durability when the appropriate amount and distribution of entrained air-voids are present in the system.



Figure 2. Change in RDME values of CSC-concrete prisms at various freeze-thaw cycles.





3.7 ASR resistance of the concrete

Expansion and cracking, leading to loss of strength, elasticity, and durability, can result from chemical reactions involving hydroxyl ions and alkali ions in Portland cement (or from other sources) with certain siliceous constituents of aggregates in OPC concrete. The types of silica that are susceptible to alkalisilica reaction (ASR) are quartz with sufficiently strained or microcrystalline, tridymite, cristobalite, and glass, which can occur in various rock formations such as opals, flints, cherts, etc. (Diamond S., 1975). This can render the aggregates unusable or potentially detrimental to the performance of OPC concrete. Therefore, ASR resistance in the presence of reactive aggregates is an important factor in concrete durability.



To evaluate the ASR resistance of CCSC concrete, highly reactive fused silica sand was used in this study. Results of length change measurements with time are provided in Figure 4 using ASTM C227 mortar bars. The results show OPC mortar samples expanded beyond the threshold limit of 0.1% within the first three weeks. After about 12 weeks, the expansion rate was significantly reduced. The CCSC mortar showed slight expansion in the initial two weeks but did not expand further for the duration of testing.

These results show improved ASR resistance of CCSC in comparison to OPC used in this study. Therefore, some of the reactive aggregates not usable in OPC concrete could potentially be used in CCSC concrete.



Figure 4. Length change of mortar bars made with ASR aggregate (fused silica) using ASTM C227 procedures with both CCSC and OPC mixtures.

3.8 Sulfate resistance of the cement

Upon exposure to sulfate bearing soil or water, the paste of OPC concrete interacts with sulfate ions to form gypsum and ettringite leading to expansion and cracking within hardened concrete. Recent studies have also reported formation of thaumasite in colder regions. Thaumasite can cause severe damage to concrete through deterioration of C-S-H, the primary binding phase within the cement paste. As a result, hydraulic cements, especially those high in C_3A content, are susceptible to structural cracking, loss of strength, stiffness reduction, or disintegration in the presence of sulfates (Müllauer, W., et al, 2013, Tian, B. & Cohen M. D., 2000).

Sulfate resistance of CCSC was evaluated by measuring the length change of mortar bars upon exposure to sodium sulfate solution. Results of length change with time are provided in Figure 5. In this test, OPC mortar samples expanded beyond the threshold limit of 0.1% in the first sixteen weeks of exposure and continued to expand significantly. After about ~24 weeks the mortar bars expanded by 0.5% and bowed. No further measurements were obtained for OPC mortar bars, as accurate expansion measurements were unattainable. In contrast, the CCSC mortar bars showed negligible expansion throughout the testing period.

In comparison to the reference OPC specimen, the CCSC specimen exhibited significantly improved sulfate resistance. Therefore, concrete made with this type of cement may be suitable for use in aggressive sulfate environments.





Figure 5. Length change of mortar bars during exposure to sodium Sulfate solution.

4. CONCLUSIONS

- The production of CCSC typically emits about 30% less CO₂ than the production of Portland cement, and the total energy consumption is also about 30% less. Concretes made by carbonating CCSC can achieve a reduction in carbon footprint by 50-70% compared to conventional OPC-based concretes. This is achieved a) by reducing the CO₂ emitted during cement production from 810 kg per ton of OPC clinker to 565 kg per ton of CCSC clinker and b) by consuming up to 300 kg of CO₂ per ton of this cement during the CO₂-curing. The carbonation of the cement produces calcite (CaCO₃) and amorphous silica (SiO₂).
- The mechanical properties of CCSC-concrete are equivalent or better than OPC-based concrete with a shorter curing period.
- CCSC concrete passes freeze-thaw testing in fresh water as per ASTM C666 procedure A (Relative dynamic modulus of elasticity >90% after 540 freeze-thaw cycles).
- CCSC concrete exhibits negligible mass loss due to scaling during the freeze-thaw testing. Rather, the specimens have gained a small weight due to saturation of nano-pores.
- CCSC mortar bars made with fused silica sand show minimum expansion. CCSC has an excellent resistance to ASR.
- CCSC mortar bars exposed to sodium sulfate solution show minimum expansion. CCSC has an excellent resistance to sulfate attack.

Acknowledgements

Part of this work was performed under a cooperative agreement between Solidia Technologies and Federal Highway Administration (FHWA) under the agreement: DTFH6115H00020. Prior to that agreement preliminary evaluations were conducted at the FHWA-TFHRC using a CRADA (Cooperative Research And Development Agreement.) FHWA neither endorses nor approves this product and the FHWA name and logo are not to be used in the marketing of the product. The purpose of the cooperative agreements was to evaluate this new proprietary technology with respect to potential applications in highway construction and repair.



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