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Laboratory and Pilot Investigation on Properties of precast Engineered Cementitious Composites (ECC) Subjected to Carbonation Curing

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Abstract. *The concrete industry is facing an increasing challenge for sustainability. Besides the large embodied carbon, the intensive operational carbon associated with repetitive repair becomes the main hurdle for lifecycle emission reduction. In this study, the feasibility of sequestering CO_2 into durable engineered cementitious composite (ECC) through early-age carbonation curing was investigated. The goal is to demonstrate a simultaneous reduction of the material's embodied carbon (by CO_2 sequestration) and lifecycle emissions (by ECC's superior durability). The material was processed at both lab and pilot scales and was demonstrated on precast pedestrian pavement slabs. Results show that ECC was highly reactive to CO_2 at lab scale, with 26.5% CO_2 uptake by cement mass after 24-hour carbonation. However, the early-age carbonation was subjected to a significant size effect and attained a 4.3% CO_2 uptake for pilot-scale specimens with a low specific surface area. Despite this reduction in carbonation efficiency, the calcite precipitation through carbonation curing was found to densify the fiber/matrix interface and improve the composite ultimate tensile and flexural strength by up to 28.8%. Carbonation curing also enhanced ECC's crack width control, thus mitigating sulfate attack and lowering surface salt scaling on freeze-thaw exposure. It is suggested that producing ECC through carbonation curing is technically viable, and the carbon-sequestered ECC is recommended for small-scale precast components for enhanced durability and sustainability.*

Keywords: *CO_2 sequestration; engineered cementitious composites; carbonation curing; durability*

1 Introduction

Carbonation curing is an emerging technique for sequestering CO_2 in precast Portland cement (PC) products. At the early age, introducing CO_2 into cementitious materials triggers a series of carbonation reactions of calcium silicates and their hydration products. These reactions are driven by CO_2 mineralization and convert gaseous CO_2 into calcium carbonates ($CaCO_3$) which subsequently form an integral structure of the final construction product (Xian et al., 2022). Cement reactions in the presence of CO_2 occur faster than those in a CO_2-free environment, hence the material's early strength development can be expedited (Shao et al., 2015). At the laboratory scale, carbonation curing is normally completed within 24 hours after casting, and the carbonated material continues gaining strength as the subsequent hydration proceeds. The final material is characterized as a hybrid of hydrates and carbonates and exhibits a more refined microstructure attributed to the $CaCO_3$ precipitation (Rostami et al., 2012; Xian et al., 2021).

Engineered Cementitious Composites (ECC) is a class of fiber reinforced cementitious composite featuring high tensile ductility (i.e., two orders of magnitude that of conventional concrete) and intrinsically tight cracks (Li, 2019). The crack width in ECC can be controlled below 100 μm irrespective of the imposed strains, and when loaded in tension, ECC develops multiple fine cracks, which continue carrying load via fiber bridging (Li, 2012). This allows new cracks to initiate elsewhere to accommodate the imposed tensile deformation. By tailoring micromechanical parameters associated with the cementitious matrix, fiber, and fiber/matrix interface, a tensile strain-hardening behavior can be achieved. Owing to the tight crack width, ECC has proved to maintain a low material permeability and desirable durability (Liu et al., 2017), thus lowering the need for repetitive repair and maintenance.

Carbonation curing has been demonstrated on ECC at the laboratory scale, showing a noticeable CO_2 uptake and enhanced composite tensile strengths both immediately after carbonation and at the standard 28 days (Zhang et al., 2021). Carbonation curing reportedly densifies the fiber/matrix interface, thus promoting the microfiber bridging and composite crack width control. By sequestering 30% CO_2 in reference to cement mass, ECC provides a potential opportunity of converting CO_2 into durable construction materials and decarbonizes the built environment in the lifecycle aspect.

While carbonation curing of ECC shows promise in laboratory, the scalability of this process remains an unaccounted domain. The scalability of carbonation curing can be viewed from both environmental and technical points of view, including 1) the consistency of CO_2 sequestration capacity and the net emission reduction efficiency across different length scales, and 2) the acceleration of early strength gain and post-carbonation properties for large-scale products. During the early-age CO_2 exposure, gaseous CO_2 diffuses through the partially evacuated pores to sustain the reaction along depth. As carbonation progresses and material surface becomes heavily carbonated, the built-up of $CaCO_3$ in the porous space tends to hinder the further ingress of CO_2. This diffusion limit was found to forms a layered structure in conventional concrete, comprising of a heavily carbonated surface and a lightly or non-carbonated core. Hence, the efficacy of carbonation curing should be prone to a size effect, and a large product size and a low material porosity tend to dilute the total CO_2 uptake as well as the technical merits associated with carbonation.

It is the interest of this study to investigate whether ECC carbonation curing could be scaled for general precast applications. Slab as a typical precast element is chosen as end-product. ECC's CO_2 uptake and resultant mechanical and durability properties are evaluated. The slabs after carbonation curing are sampled for 4-point bending at standard 28 days. The present study would serve as a reference for the industrial practice of carbonation curing for both ECC/concrete slabs and general precast products.

2 Experimental Program

2.1 Materials and Mix Proportion

The solid ingredients used for ECC included Type I ordinary Portland cement (OPC, Lafarge-Holcim), Class F fly ash (FA, Boral Resources), and F75 grade silica sand (US Silica). Tap water and a polycarboxylate-based water reducer (BASF, Glenium 7920) were used for mixing. Polyvinyl alcohol (PVA) fiber from Nycon (RECS-15) was used to formulate ECC. The PVA fiber is 8 mm in length, 39 μm in diameter, 6% in elongation, 1.3 in specific gravity, 42.8 GPa in Young's modulus, and 1600 MPa in tensile strength. Table 1 lists the ECC mix design.

Table 1. Mix design

Components	Cement	Fly ash	Silica sand	Water	SP	PVA fiber
Mass ratio	1	1.2	0.8	0.58	0.005	2 vol%

2.2 Sample Preparation

The experiment was conducted at two length scales. At the lab scale, ECC was cast into the dogbone-shaped specimens and 50-mm cubic specimens for uniaxial tension (per JSCE recommendation) (JSCE, 2008) and compression (per ASTM C109) (ASTM, 2013), respectively. At the pilot scale, ECC was first cast into full-size slabs (4 in. × 4 in. × 12 ft) for carbonation. The carbonated slabs were then cut into smaller beam specimens for flexural test. The mixing, casting, and curing protocols are specified as follows.

ECC was mixed using 4-liter and 60-liter plenary-type mixers for small- and large-scale mixing, respectively. Table 2 lists the mixing sequence for each. All specimens were cast one-time without separate layers, and the top surface was lightly finished using a trowel. The fresh ECC showed a high flowability and self-consolidating property, and thus no additional compaction was applied during casting. For each specimen type, two parallel groups were prepared for carbonation curing and moisture curing (as control), respectively.

Table 2. ECC mixing sequence

Step	Activity	Mixing time in 4-liter mixer	Mixing time in 60-liter mixer
1	Mix solid ingredients	3 min	5 min
2	Add premixed water and superplasticizer	3 min	10 min
3	Discharge fibers	3 min	15 min

The procedure of carbonation curing was comprised of four steps. First, fresh mixtures were kept in mold after casting for 18 hours. This step is to ensure the material gains sufficient strength for demolding. Second, the specimens were then demolded and subjected to an intentional fan drying at room condition (23 °C and 60±5% RH). The purpose is to partially remove pore water and evacuate interconnected pores to facilitate CO_2 diffusion. Third, specimens were mounted into a pressure vessel for carbonation. As an initial test for scalability, high-purity CO_2 gas (99.8% concentration) was used in this study. The gas pressure was kept at 5 bar during the period of carbonation. Lastly, to enable subsequent long-term hydration, the carbonated specimens were sprayed with water to compensate the water loss during drying and carbonation, and the specimens were cured for additional 26 days before testing.

2.3 Performance Evaluation

The investigation into ECC's mechanical properties included compressive strength, tensile strength and ductility, crack width, and flexural strength. The compression test was conducted using a Forney compression machine, while the tension test was conducted on the dogbone-shaped specimens using an Instron testing machine. The loading speed was 0.5 mm/min, and two LVDT was attached to the center 80 mm section to measure tensile deformation. The flexural test was conducted on beam specimens cut from ECC slabs after carbonation curing. The beams were tested at a loading rate of 0.05 mm/min following ASTM C1609 (ASTM, 2012). The displacement was measured using an OptoTrack system by placing three optical sensors at the midspan center and two ends. An MTS machine with a 100 kN capacity was employed for loading, and the flexural stress is calculated using Eq. 1.

$$\sigma_f = \frac{3PL}{4bd^2} \tag{1}$$

where, σ_f is the flexural stress in MPa, P is the flexural load in N, L is the span length (254 mm), b is the beam width (101.6 mm), and d is the beam depth (101.6 mm).

After flexural test, CO_2 uptake was measured on ECC powder samples drilled at different depths from the same slab specimens. Thermal paralysis was conducted to obtain CO_2 content from the powder sample. To isolate the effect of early-age carbonation, powders from the non-carbonated slab were sampled and tested as control. The CO_2 content in the non-carbonated sample was subtracted to obtain the carbonation-induced CO_2 uptake.

Conventional concrete slabs are commonly subjected to cracking associated with uneven subgrade and chemical attack. In cold regions, concrete is also prone to surface salt scaling and microcracks led by freeze-thaw cyclic action. Hence, the durability criteria for the intended sidewalk application include resistance to sulfate attack and salt scaling. For sulfate resistance, ECC cubes were prepared and subjected to three exposure conditions, i.e., 3% Na_2SO_4 solution, 3% $MgSO_4$ solution, and water. The material compressive properties were evaluated after a 3-year exposure, and the solutions were changed every 3 months to ensure sufficient supply of the detrimental species. The freeze-thaw resistance was measured on 150-mm ECC cubes using a modified RILEM TC 176-IDC CDF/CIF method (RILEM, 2004). The specimens were saw cut to obtain a 70-mm thickness, and the carbonated surface was exposed to 3% NaCl solution and treated in the temperature range of -20-20 °C. The scaled material was collected and weighed.

3 Results and Discussion

3.1 CO_2 Uptake

The results of CO_2 uptake indicate a significant size effect on ECC's carbonation efficiency. Table 3 lists the carbonation depth and average CO_2 uptake for each type of ECC specimens. Compared with dogbones and cubes, the slab specimen exhibited a substantial reduction in CO_2 uptake, recording only 4.3% by cement mass. This can be attributed to the thick specimen depth and low specific surface area, which lead to a relatively larger fraction of the non-carbonated core. The slab specimen also showed a lower carbonation front, with CO_2 content reaching the highest value at the surface 5 mm layer and lowered to less than 0.1% in the core (i.e., depth >20 mm). Besides the limited CO_2 diffusion, the small depth of carbonation can also be associated with a less efficient removal of internal water during pre-conditioning. Therefore, extending the drying process and increasing CO_2 partial pressure seem to be the most effective measure to restore carbonation efficiency at scale.

Table 3. Carbonation depth and CO_2 uptake in different specimen types

Specimen geometry	Specimen thickness (smallest dimension), mm	Specific surface area, m^2/m^3	Depth of carbonation, mm	No. of samples	Average CO_2 uptake, % by cement mass
Dogbone	12.7	175.4	Fully carbonated	18	26.5
Cube	50	120.0	6.5 ± 1.9	18	18.9
Slab	101.6	21.87	4.6 ± 0.6	3	4.3

3.2 Mechanical Properties

The effect of carbonation curing on ECC's mechanical properties was evaluated on 50-mm cubes for compressive strength, dogbone-shaped specimens for tensile properties, and beams cut from slabs for flexural properties. As listed in Table 4, specimens subjected to carbonation curing displayed higher tensile and compressive strengths at both 3 and 28 days compared to the non-carbonated reference. This confirms the accelerated early strength gain and densified microstructure led by carbonation. Additionally, calcite precipitation at the fiber surface was reported to enhance the fiber/matrix interfacial bond, which is accountable for the increased composite tensile strength after carbonation curing.

Table 4. Effect of carbonation curing on ECC's mechanical property

Age, days	Compressive strength, MPa		No. of testing samples
	Carbonation curing	Non-carbonated reference	
3	40.10 ± 2.78	29.80 ± 1.71	15
28	58.19 ± 1.92	57.91 ± 2.06	15
Age, days	Ultimate tensile strength, MPa		No. of samples
	Carbonation curing	Non-carbonated reference	
3	5.35 ± 0.86	5.05 ± 1.17	12
28	5.93 ± 0.36	5.17 ± 0.56	12
Age, days	Tensile strain capacity, %		No. of samples
	Carbonation curing	Non-carbonated reference	
3	3.64 ± 0.93	3.62 ± 1.54	12
28	2.78 ± 0.86	3.46 ± 1.31	12
Age, days	Flexural strength, MPa		No. of samples
	Carbonation curing	Non-carbonated reference	
28	13.33 ± 1.27	10.35 ± 0.62	6

The effect of carbonation curing on the flexural behavior of ECC beams is graphically illustrated in Figure 1. All specimens were found to exhibit flexural strain hardening characteristics, with multiple fine cracking identified on the side and bottom. As shown in Figure 1b, the specimen after carbonation developed a more saturated multiple cracking behavior and a 28.8% higher flexural strength compared to its non-carbonated counterpart. This indicates that carbonation can improve the overall load-carrying capacity of large-sized slabs despite a small carbonation front (approx. 5-10 mm). This enhancement of ECC's structural behavior through carbonation curing may enable a reduction in cement usage for structural components and warrants further investigation.

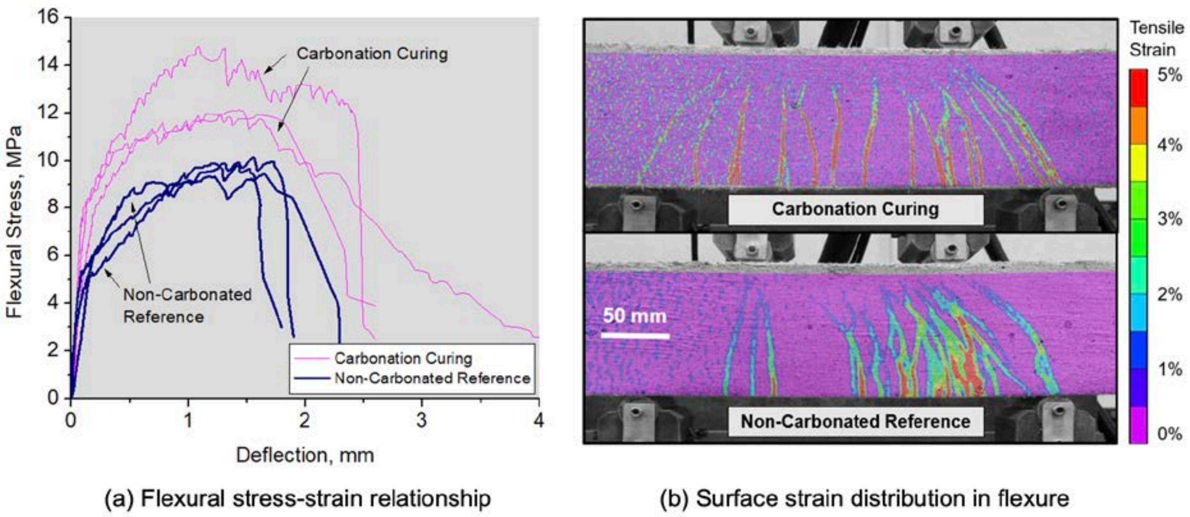
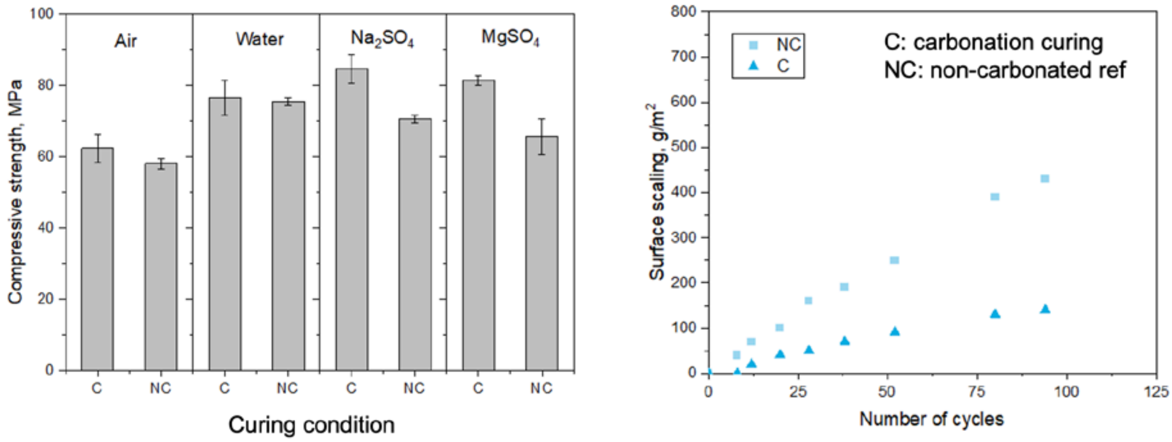


Figure 1. Flexural behavior of ECC beam

3.3 Durability and field deployment

The target application of the carbonated slabs is a pedestrian sidewalk section in contact with soil and exposed to freeze-thaw environment. Hence, the material's long-term strength and surface scaling resistance were examined in corresponding lab conditions before the field deployment. As shown in Figure 2a, the carbonated ECC retained desirable compressive strength in a 3-year period of exposure in sulfate conditions. When stored in air or water, a marginal difference was observed between the specimens subjected to carbonation curing and the non-carbonated reference. Compared to the group exposed to air, carbonated specimens exhibited a 22.8% higher compressive strength when stored in water, indicating that the long-term hydration in supply of water was not impeded by the early carbonation. Additionally, when exposed to sulfate solutions, carbonated specimens displayed a slightly higher strength and exceeded the respective non-carbonated reference. This result confirms the enhancement of sulfate resistance by carbonation curing and can be attributed to the lowered alkalinity and denser microstructure (incl. a stronger bond at the fiber/matrix interface) owing to the early carbonation.



(a) Compressive strength after 3-year exposure (b) Surface scaling exposed to freeze-thaw cycles

Figure 2. Effect of curing condition on ECC's durability on various exposures

ECC has proved to be freeze-thaw resistant due to the microfiber bridging (Şahmaran et al., 2012), and this technical advantage was amplified after the early carbonation. As shown in Figure 2b, the scaled mass was lowered considerably by carbonation curing up to 94 cycles. While the non-carbonated reference displayed noticeable damages on the exposed surface, the carbonated specimen appeared nearly intact throughout the testing period. As the material strength and toughness controls the crack propagation during salt scaling (Valenza & Scherer, 2007), the densified cementitious matrix and fiber/matrix interface led by the calcite precipitation should be most accountable for the enhanced resistance to salt scaling.

Upon the completion of performance testing and evaluation, full-size ECC slabs were prepared and subsequently deployed in the field. Three slabs were made including a non-carbonated reference. The ECC section was surrounded by newly poured traditional concrete pavement to attain a comparative observation for durability. The site is shown in Figure 3, and the long-term performance is being monitored.

Figure 3. Pilot-scale deployment of CO_2-sequestered ECC slabs (photo courtesy: Scott Spooner, City of Ann Arbor Parks and Recreation Services)

4 Conclusions

This study aims to demonstrate the scalability of ECC's carbonation curing and its feasibility for field applications. Pedestrian sidewalk is chosen as an example. The pilot-scale experiment confirms a significant size effect on CO_2 uptake, lowered from 26.5% to 4.3% as the volume specific surface area decreases from 175.4 to 21.87 m^2/m^3. Despite a limited carbonation front, the flexural strength was enhanced by ~28.8%, accompanied by a higher long-term strength in sulfate environment and lower salt scaling on freeze-thaw cyclic exposure. It is concluded that carbonation curing enhances ECC's performance for the intended pavement application and can potentially increase its durability and lower the environmental footprint associated with operation and maintenance. Hence, carbonation curing is feasible and can be adopted for industrial production of non-reinforced precast ECC products for CO_2 sequestration and performance improvement.

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References

ASTM. (2012). C1609 Standard Test Methods for Flexural Performance of Fiber-Reinforced Concrete (Using Beam With Third-Point Loading).

ASTM. (2013). C109 Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens).

JSCE. (2008). Recommendations for Design and Construction of High Performance Fiber Reinforced Cement Composites with Multiple Fine Cracks (HPFRCC). In.

Li, V. C. (2012). Tailoring ECC for special attributes: A review. *International Journal of Concrete Structures and Materials*, *6*(3), 135-144.

Li, V. C. (2019). *Engineered Cementitious Composites (ECC): Bendable Concrete for Sustainable and Resilient Infrastructure*. Springer.

Liu, H., Zhang, Q., Li, V., Su, H., & Gu, C. (2017). Durability study on engineered cementitious composites (ECC) under sulfate and chloride environment. *Construction and Building Materials*, *133*, 171-181.

RILEM. (2004). Final Recommendation of RILEM TC 176-IDC'Internal Damage of Concrete due to frost action'. *Materials and structures, 37*, 754-759.

Rostami, V., Shao, Y., Boyd, A. J., & He, Z. (2012). Microstructure of cement paste subject to early carbonation curing. *Cement and Concrete Research, 42*(1), 186-193.

Şahmaran, M., Özbay, E., Yücel, H. E., Lachemi, M., & Li, V. C. (2012). Frost resistance and microstructure of Engineered Cementitious Composites: Influence of fly ash and micro poly-vinyl-alcohol fiber. *Cement and Concrete Composites, 34*(2), 156-165.

Shao, Y., Zhou, A., & Mahoutian, M. (2015). Pseudo-dynamic carbonation for concrete curing and carbon storage. *International Journal of Materials and Structural Integrity, 9*(1-3), 21-38.

Valenza, J. J., & Scherer, G. W. (2007). A review of salt scaling: II. Mechanisms. *Cement and Concrete Research, 37*(7), 1022-1034.

Xian, X., Zhang, D., Lin, H., & Shao, Y. (2022). Ambient pressure carbonation curing of reinforced concrete for CO2 utilization and corrosion resistance. *Journal of CO2 Utilization*, *56*, 101861.

Xian, X., Zhang, D., & Shao, Y. (2021). Flue gas carbonation curing of cement paste and concrete at ambient pressure. *Journal of Cleaner Production*, 127943.

Zhang, D., Ellis, B. R., Jaworska, B., Hu, W.-H., & Li, V. C. (2021). Carbonation curing for precast Engineered Cementitious Composites. *Construction and Building Materials, 313*, 125502.