



# Early carbonation curing of concrete masonry units with Portland limestone cement



Hilal El-Hassan<sup>a</sup>, Yixin Shao<sup>b,\*</sup>

<sup>a</sup> Department of Civil Engineering, American University in Dubai, P.O. Box 28282, Dubai, United Arab Emirates

<sup>b</sup> Department of Civil Engineering and Applied Mechanics, McGill University, 817 Sherbrooke Street West, Montreal, Quebec H3A 2K6, Canada

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## ABSTRACT

The effect of carbonation curing on the mechanical properties and microstructure of concrete masonry units (CMU) with Portland limestone cement (PLC) as binder was examined. Slab samples, representing the web of a CMU, were initially cured at 25 °C and 50% relative humidity for durations up to 18 h. Carbonation was then carried out for 4 h in a chamber at a pressure of 0.1 MPa. Based on Portland limestone cement content, CO<sub>2</sub> uptake of PLC concrete after 18 h of initial curing reached 18%. Carbonated and hydrated concretes showed comparable compressive strength at both early and late ages due to the 18-h initial curing. Carbonation reaction converted early hydration products to a crystalline microstructure and subsequent hydration transformed amorphous carbonates into more crystalline calcite. Portland limestone cement could replace Ordinary Portland Cement (OPC) in making equivalent CMUs which have shown similar carbon sequestration potential.

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## 1. Introduction

Portland limestone cement (PLC) is the product of intergrinding Portland cement clinker with limestone and calcium sulfate. When added to cement, limestone may affect hydration, carbonation and compressive strength of concrete. Currently, major cement producers are marketing and investing in PLC with up to 20% cement replacement by limestone. Such a replacement could reduce the carbon footprint associated with cement production by 20%. Early research believed that fine limestone particles acted as nucleation sites, thus increasing the rate of hydration of the calcium silicates at early age and possibly improving the distribution of hydrates [1,2]. Ramachandran had reported that the hydration rate of C<sub>3</sub>S was also increased with an increase in the fineness and amount of CaCO<sub>3</sub> [3]. The formation of hydration products as ettringite had also been accelerated in the presence of CaCO<sub>3</sub>, while C–A–H incorporated significant amount of calcium carbonates into its structure and formed calcium silicocarbonated hydrates [3]. Additionally, CaCO<sub>3</sub> will react chemically with aluminate phases to form carboaluminate phases [4]. Finally, because limestone is softer than clinker, when interground, it will achieve a finer particle size, thus refining particle packing and improving particle size distribution [5].

With similar major components as Ordinary Portland Cement (OPC), PLC concrete can be carbonated to sequester CO<sub>2</sub> to further reduce the carbon footprint from cement production. This early-age carbonation is the reaction between calcium silicates or early hydration products with carbon dioxide producing a hybrid binder structure of calcium silicate hydrates and calcium carbonates [6]. It is different from weathering carbonation. Weathering carbonation of limestone cement concrete was studied by Parrott and Tsvivilis et al. [7,8]. Results showed that the amount of limestone present in the cement affected the degree of weathering carbonation. When more than 19% of Portland cement clinker was replaced with limestone, the microstructure analysis showed that the depth of weathering carbonation of concrete increased during 18-month drying (at 20 °C and 60% relative humidity) [7]. However, with less than 15% replacement, weathering carbonation degree was not intensified [7]. The effect of limestone addition of up to 35% on the durability of concrete was apparent in other research [8]. Within 15% addition, the limestone additive could reduce water permeability and sorptivity of the concrete due to improved cement particle size distribution [8].

The physical performance and carbonation behavior of Ordinary Portland Cement (OPC) concrete was investigated in separate work [9,10]. To promote high degree of carbonation, the procedure necessitated the introduction of initial air curing prior to carbon exposure. Carbonation curing was then performed by injecting CO<sub>2</sub> gas (99% purity) into a sealed chamber. Of the examined curing

\* Corresponding author.

E-mail address: [yixin.shao@mcgill.ca](mailto:yixin.shao@mcgill.ca) (Y. Shao).

schemes, the optimum combination appeared to be a procedure of air curing of 18 h, followed by carbonation curing of 4 h, and immediate surface water spray for water loss compensation. On the other hand, the sealed hydration (0a) with no water loss during entire curing served as the reference.

The purpose of this paper is to investigate if PLC can be used to replace OPC in making equivalent concrete masonry units (CMU). The effect of carbonation process parameters on the microstructure and characteristics of the reaction products of PLC concrete was examined. A static carbonation setup developed in carbonation of OPC concrete will be used to perform carbonation curing of PLC concrete [10]. The reaction products of the concretes are characterized utilizing X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Thermogravimetry/Differential Thermogravimetry (TG/DTG). A unique method was developed to determine the cement content in concrete powder to allow a quantitative TG analysis for concrete [9]. The produced PLC concrete blocks may serve as a potential carbon sink whilst using lesser Portland cement in the production.

**2. Experimental investigations**

**2.1. PLC concrete sample preparation and curing**

To simulate a typical web or face shell of a 200-mm PLC concrete masonry unit, rectangular concrete samples 127 mm long, 76 mm wide, and 38 mm thick were cast. PLC used in this research is a commercial product manufactured and marketed by Holcim Canada. About 13–15% limestone is interground with OPC clinker. The chemical compositions of both PLC and OPC are reported in Table 1. The CO<sub>2</sub> content determined by infrared carbon analyzer in as-received PLC is about 7.0%, which is equivalent to a limestone content of 15.9%. The cement is considered as high limestone cement and is therefore labeled as Portland limestone cement. To maintain comparable early compressive strength to OPC, PLC is ground finer. The Blaine fineness of PLC is 500 m<sup>2</sup>/kg in comparison to 390 m<sup>2</sup>/kg in general use OPC. The as-received lightweight expanded slag aggregates had a water content of 5% by mass at saturated surface dry (SSD). The sieve analysis of the same aggregate blend was performed in previous work; the well graded aggregates ranged in size between 0.2 and 6 mm [10]. The concrete mixture proportion shown in Table 2 is based on a commercial CMU mix design. The water-to-cement ratio (w/c) was 0.71 taking into account the mixing water and the water in the aggregates (5%). The raw materials were mixed in a pan mixer and samples were compact formed using a vibrating hammer to simulate the industry production of CMU. PLC concrete was then demolded right after casting. Three curing schemes were investigated: (1) Sealed hydration in a plastic bag served as reference (0a) without water loss; (2) Initial air curing of 18 h in an environmental chamber of 50% relative humidity (RH) and 25 °C followed by 4-h carbonation and subsequent hydration (18a + 4c); (3) Initial air curing of 18 h in an environmental chamber of 50% relative humidity (RH) and 25 °C, followed by 4-h carbonation, water compensation and subsequent hydration (18a + 4c + sp). Water compensation through surface spray was introduced immediately after carbonation to restore all the curing-induced water loss. The spraying process was ceased

**Table 1**  
Chemical composition of PLC and OPC.

Cement	Constituent (%)								
	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	SO <sub>3</sub>	CO <sub>2</sub>
PLC	59.8	21.6	4.5	2.7	1.7	0.8	–	3.2	7.0
OPC	63.1	19.8	4.9	2.3	2.5	0.9	–	3.8	2.0

**Table 2**  
Mixture proportion of PLC concretes.

	Slab (g)	Mass (kg/m <sup>3</sup> )	Percent (mass%)
Portland limestone cement	88	241	13
Mixing water	35	96	5
SSD expanded slag	554	1502	82
Concrete sample	677	1839	100

upon reaching surface saturation, and required a few days for complete water compensation [9]. Subsequent hydration was carried out in a sealed bag at a relative humidity of 80 ± 5% and room temperature (24 ± 1 °C) until 24 h and 28 days for compressive strength tests following ASTM C140 [11]. Three rectangular specimens for each batch were tested and averaged with a compressive area of 127 mm × 38 mm.

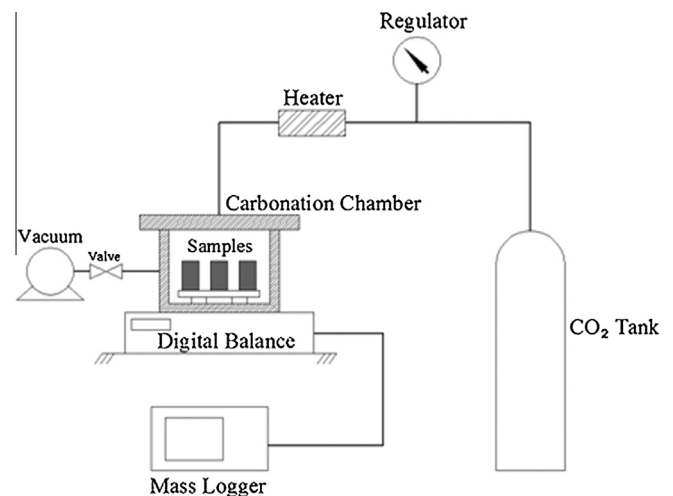
The fractured concrete samples were preserved in an acetone solution to stop hydration for microstructure analysis. Acetone exchange with water to stop hydration was reported as the least damaging method to preserve the microstructure [12]. Prior to analysis, the samples were pre-dried overnight at 60 °C.

A schematic of the carbonation setup is shown in Fig. 1. Within a few seconds, the pressure is reduced to 0.7 bars below atmospheric prior to carbonation by means of a vacuum. A heater installed at the inlet brings up the carbon dioxide gas of 99% purity to room temperature as it flows into the sealed chamber. A pressure regulator is connected to the system to retain the pressure at 0.1 MPa. The whole system is placed on a digital scale to monitor the mass increase due to the carbonation reaction.

**2.2. Measurement of CO<sub>2</sub> uptake in PLC concrete**

CO<sub>2</sub> uptake of carbonated PLC concrete was quantified by three methods: mass gain, mass curve, and furnace decomposition. Mass gain method estimates CO<sub>2</sub> uptake in concrete by comparing mass of samples before and after carbonation (Eq. (1)). Carbonation-induced water loss was collected by absorbent paper and added to the final mass. By treating the system as a closed system, it was imperative to include the evaporated water, which was initially inside the samples prior to carbonation.

$$CO_2 \text{ uptake}(\%) = \frac{(\text{Final mass} + \text{Mass of water loss} - \text{Initial mass})}{(\text{Mass of cement})} \quad (1)$$



**Fig. 1.** Schematic of carbonation setup.

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