



# Effects of CO<sub>2</sub> surface treatment on strength and permeability of one-day-aged cement mortar



Xiaoying Pan<sup>a</sup>, Caijun Shi<sup>a,\*</sup>, Xiang Hu<sup>a</sup>, Zhihua Ou<sup>b</sup>

<sup>a</sup> College of Civil Engineering, Hunan University, Changsha 410082, China

<sup>b</sup> School of Civil Engineering, Hunan University of Technology, Zhuzhou 412007, China

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## ABSTRACT

Many of the degradation problems of concrete can be attributed to the weak surface layer. Surface treatment of concrete has been proven a simple and effective method to enhance the durability of concrete. In the present study, two series of portland cement mortar specimens with w/c ratios of 0.4 and 0.3 were prepared. CO<sub>2</sub> treatment was applied to the mortars 24 h after casting. The results indicated that CO<sub>2</sub> treatment resulted a carbonated layer of less than 1.6 mm in thickness, slightly increased compressive strength, but significantly reduced the water permeability, water-vapor transmission and chloride migration of the mortars. Environmental Scanning Electron Microscopy (SEM), Fourier Transform Infrared (FT-IR) Spectroscopy and thermogravimetric analysis (TGA) results indicated that calcium silicate hydrate with low Ca/Si ratio and CaCO<sub>3</sub> were the products in the carbonated surface layer.

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## 1. Introduction

Surface treatment is one of the effective methods to improve the durability of concrete [1–3]. It has been widely used for marine structures and bridge decks. The most common commercial surface treatment techniques include polymer coatings and hydrophobic impregnation. Calcium carbonate precipitation has been adopted recently for concrete surface treatment due to its good stability. It can be introduced through bacterial-induced mineralization, in which calcium carbonate forms from microbial cells and biochemical activities, or hydrolysis of dimethyl carbonate (DMC).

Several studies [4–7] indicated bacterial carbonate precipitation was an effective surface treatment technique. It decreased the water absorption, gas permeability, and chloride permeability by 20–80%, 17–33% and 39–51%, respectively. Achal et al. [4] showed that the bacterial treatment could reduce porosity of the mortar by 50%. However, the cultivation of bacteria on concrete surface requires careful control [8], and the nutrient solutions for the bacteria development are expensive [9].

Dimethyl carbonate (DMC) solution can hydrolyzes slowly and forms carbonate and methanol (CH<sub>3</sub>OH) at room temperature and pressure [5,6,10,11]. Amidi and Wang [11] showed that the DMC solution treatment could reduce the water absorption by

5–45%, depending on the treatment process and cumulative treatment time. DMC has high biodegradability and low toxicity, but the treatment process should follow specific procedure and cannot show good protection without multiple treatments [11].

Recently, accelerated carbonation of freshly moulded concrete and recycled aggregates have attracted wide attention [12–20]. Unhydrated cement clinker is also very reactive with CO<sub>2</sub> [21,22]. Concrete blocks after 2–4 h of CO<sub>2</sub> curing achieved similar strength to those after 24 h of steam curing [12,13]. In addition, CO<sub>2</sub> cured blocks also possessed lower porosity, water absorption and shrinkage. Monkman et al. [14] showed that carbonation curing enormously improved the freeze/thaw durability. Shao et al. [15] combined steam and carbonate curing to improve the durability of concrete pipes. They demonstrated that the early carbonation led to a reduction in calcium hydroxide near the surface while maintaining a pH above the corrosion threshold value at the core. Whereas, carbonation curing is generally used for dry plain concrete because the diffusion rate of CO<sub>2</sub> in saturated capillary pores is about 10,000 times slower than that in unsaturated capillary pores [16].

This study is to develop a simple, economic and environmental technique for surface treatment of precast concrete products. The current paper is the first part of a series study. It investigated how the CO<sub>2</sub> surface treatment affected the strength and permeability of the cement mortar at one-day age. The changes in microstructure were characterized by TGA, SEM, and FT-IR. Further

\* Corresponding author.

E-mail address: [cshi@hnu.edu.cn](mailto:cshi@hnu.edu.cn) (C. Shi).

research on the effects of CO<sub>2</sub> surface treatment on long-term performance will be reported in another paper [23].

## 2. Materials and methods

### 2.1. Materials

A P. I. 42.5 Portland cement with a specific surface area of 336 m<sup>2</sup>/kg was used. The chemical composition of used cement is given in Table 1.

Natural river sand with a density of 2610 kg/m<sup>3</sup> was used as fine aggregate. The grade of the sand is shown in Table 2. An industrial grade CO<sub>2</sub> with a purity of >99% was used.

### 2.2. Mixture proportion and sample preparation

All the mortars were prepared with a constant sand-to-cement ratio of 1.5. Two water-to-cement (w/c) ratios of 0.3 and 0.4 were used. The cement mortars were mixed in a rotary mixer for 3 min, then cast in different moulds, and consolidated using a vibration table. Cubic specimens of 40 × 40 × 40 mm were cast for compressive strength and water absorption testing; cylinder specimens of Φ75 × 100 mm and Φ110 × 100 mm were made in PVC molds for water vapor transmission and rapid chloride migration (RCM) testing respectively. All freshly cast specimens were covered with a plastic sheet and cured in a room at T = 20 ± 1 °C and RH ≥ 98% for 24 h.

### 2.3. CO<sub>2</sub> treatment

All cubic specimens were demolded 24 h after casting. One end of the cylinder specimens with PVC moulds was cut off to get sizes of Φ75 × 75 mm and Φ110 × 75 mm, as shown in Fig. 1. All these specimens were then placed in a pressurized CO<sub>2</sub> chamber. The chamber was vacuumed before the CO<sub>2</sub> gas injection. The CO<sub>2</sub> treatment time was 3 h or 6 h, as shown in Fig. 2. The CO<sub>2</sub> pressure in the chamber was kept at 0.2 MPa during the treatment. A RH of 70% in the chamber was maintained using over-saturated calcium nitrate solution. All faces of the cubic samples, but only one or two ends of cylinders were in contact with CO<sub>2</sub> gas.

### 2.4. Testing methods

#### 2.4.1. Compressive strength

Compression test was conducted using a machine with 120-kN maximum loading capacity. Load was applied at a constant rate of 0.6 MPa/min until the specimens failed. The average values of three samples were reported.

#### 2.4.2. Water absorption

The water absorption test was conducted in accordance to ASTM C1585-13 [24]. Cubic samples 40 × 40 × 40 mm were firstly vacuum-dried at 60 °C for 48 h. Then, two-component epoxy was applied to seal five faces of the samples to ensure that water was only absorbed through the bottom face. The samples were placed on stainless steel rids in a tray such that the free water level was about 5 mm above the bottom face. The mass of water absorbed per unit area before immersion and subsequently after intervals of 10 min, 20 min, 40 min, and 90 min was recorded with a

resolution of 0.001 g. The average values of three specimens were reported. The initial water absorption coefficient was determined based on the slope of the water absorption ratio vs. square root of time since the beginning of water absorption. It reflects the water transport properties and the pore structure characteristics of cement paste [25].

#### 2.4.3. Water vapor transmission

Immediately after CO<sub>2</sub> treatment, cylinder samples of Φ75 × 75 mm were cut into Φ75 × 15 mm disks as shown in Fig. 1. All the disk samples were kept in a controlled room with RH = 50% and T = 25 °C for 3 days before the water vapor transmission testing. Wet-cup method was used following ASTM E96-2005 [26]. Pure water and water absorbent silica gel was used to achieve a constant high and low humidity at about 90% and 40% respectively by measurement for each region. Thus, the water would vapor and migrate from the high humidity region to the low humidity region through the mortar sample. The mass loss from the plastic cup was recorded, and the slope of the mass loss vs. time curve was determined.

#### 2.4.4. RCM testing

Similar to the water-vapour transmission test, the cylinder samples of Φ110 × 75 mm were cut into Φ110 × 50 mm disk immediately after CO<sub>2</sub> treatment for RCM testing. The excessive water on the specimen surface was wiped off with a brush. Then the specimens were placed in a vacuum (−0.1 MPa) container for 1 h, then filled up with saturated Ca(OH)<sub>2</sub> solution for another 18 ± 2 h. The catholyte solution was 10% NaCl, and the anolyte solution was 0.3 mol/L NaOH. The testing time and voltage were chosen following Chinese Standard of GBT50082-2009 [27]. After the testing, samples were split and sprayed with 0.1 mol/L AgNO<sub>3</sub> solution to determine the penetration depth of chloride ions. The chloride migration coefficients of the specimens were calculated following the standard. All mortar samples were tested at the same time, and the average values of three specimens were presented.

#### 2.4.5. Thermogravimetric analysis

To understand the influence of CO<sub>2</sub> treatment on the surface layer of the early-age mortar samples, powders were taken from outer surface layer of 2 mm at an interval of 0.2 mm using a computer numerical control (CNC) machine. The powder samples were first dried in an oven at 60 °C for 24 h, then sieved through a 45 μm sieve. TGA was carried out using the Netzsch STA 409 PC. The samples, weighing approximately 10 mg, were heated from 20 to 1000 °C in a nitrogen atmosphere at a heating rate of 10 °C/min. The mass loss data were used to calculate the amount of CaCO<sub>3</sub> present in the samples.

#### 2.4.6. Fourier transform infrared spectroscopy (FT-IR) analysis

Powder samples from outer 0.2 mm layer of the mortars were used for FT-IR analysis. A powder sample of pre-dried IR-grade KBr was used to get thoroughly mixed disks. Transmission infrared spectra of the disked samples were recorded using commercial Thermo Scientific IS10 FT-IR workstation. The wavenumbers range was 2000–400 cm<sup>−1</sup> at a resolution of 2 cm<sup>−1</sup>. Each sample was scanned 32 times.

**Table 1**  
Chemical composition of cement (w%).

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	SO <sub>3</sub>	Na <sub>2</sub> O <sub>eq</sub>	LOI
Percentage	21.91	5.30	3.67	64.5	1.51	0.62	0.19	2.03	0.59	2.49

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