



The effects of the early carbonation curing on the mechanical and porosity properties of high initial strength Portland cement pastes



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HIGHLIGHTS

- 1 h of carbonation improves the mechanical resistance of the paste, in relation to the references.
- 1 h of carbonation does not affect the degree of polymerization of the C–S–H.
- The 24 h carbonation at low RH conditions, affects the hydration process, limiting the final mechanical resistance.
- The slight higher porosity of the P_{C1h} paste occurs due to a superficial structural change caused by the carbonation.
- Increasing the carbonation time 1–24 h increases the porosity of the paste P_{C24h}, caused by the intense carbonation.

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ABSTRACT

Mechanical and porosity related properties of high initial strength sulfate resistant Portland cement (HS SR PC) pastes subjected to early age carbonation curing were investigated. This work presents the characterization through mechanical properties, of carbonated HS SR PC pastes and of non-carbonated references. They were treated at two different times of carbonation (1 h and 24 h), at the best conditions of capturing, previously determined by the authors. The mechanical properties were characterized by compressive strength, tensile strength by diametrical compression, elastic modulus and Poisson's coefficient. It was found that 1 h of carbonation improved the mechanical properties, while the increase of carbonation time to 24 h, decreased significantly the same properties in relation to the reference specimens. The porosity related properties were characterized by total absorption, absorption by capillarity, gas permeability, and mercury intrusion porosity. It was noticed that the increase of the carbonation time from 1 h to 24 h, increased substantially the absorption properties. Despite the mechanical properties of the 1 h carbonated paste were better than those of the reference, the absorbed water content of the carbonated specimen was slightly higher than that of the reference.

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1. Introduction

In a previous work, the authors concluded that the CO₂ capture by an early high strength and sulfate resistant Portland cement (HS SR PC) paste, increased the amount of CO₂ captured in the paste studied, only after a brief hydration period of 6 h without carbonation, which corresponds to the initial setting time of the paste with 0.7 W/C ratio, in an environment with relative humidity of 60% and temperature of 25 °C [1]. This was proved by thermogravi-

metry, comparing samples treated with and without CO₂ treatment [2]. Considering the same issue, the literature reports that the carbonation curing of cementitious materials can improve the mechanical and durability properties in some operating conditions [3,4].

The importance of the CO₂ treatment of cementitious materials as concrete, mortar and paste is the possibility to develop materials with a better performance than when using conventionally curing in water, besides considering the environmental and economic benefits to capture CO₂ from the atmosphere [5–7].

According to the mechanical results obtained by the authors [2] for cylinders of HS SR PC pastes, with 14 days of hydration, treated with CO₂ in a concentration of 20% in volume at five different times of carbonation (1, 2, 4, 8 and 12 h), it was noticed that the time of

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exposure to CO₂ is crucial for its capture capacity without damaging the cementitious matrix. The increase of the exposure time to CO₂ to 24 h, despite increases substantially the amount of captured CO₂, decreases significantly the mechanical resistance of the material, a problem that was solved when the matrix was treated during only 1 h [2].

The aim of this work is to characterize, by mechanical property, porosity and permeability measurements after 28 days of conventional curing, the HS SR PC pastes subjected to CO₂ curing in the two carbonation times (1 and 24 h) previously studied by the authors [2]. Carbonated cement pastes are compared with a conventionally hydrated reference paste by mechanical properties as compressive strength (elastic modulus, and Poisson coefficient) and tensile strength by diametrical compression, as well as by porosity properties studied by total absorption, absorption by capillarity, gas permeability and mercury intrusion porosity.

The choice of the HS SR PC, according to previous studies of the authors [8,9], is related to the possibility to use this technology in the precast concrete industry, which uses this type of cement in large amounts. The carbonation process is used during curing time due to the higher porosity induced when it is done in this way, as well as because the simultaneous availability of a very reactive Ca(OH)₂ at early stages, which will improve and increase the ability to capture CO₂ in the matrix.

2. Materials and methods

In this study, a high initial strength and sulfate-resistant Portland cement (HS SR PC) [10] was used to prepare the paste specimens, which allows to reach high strengths at early hydration stages. This kind of cement may have a maximum of 5% of carbonates addition and aggregation of blast furnace slag or pozzolanic materials [11]. The chemical composition, determined by XRF is presented at Table 1.

Deionized water was used to prepare the pastes, which were mixed during 30 s in a mechanical mixer in an environmental temperature of 23 °C (±2 °C).

The pastes were prepared using a water to cement ratio (W/C) of 0.7, were immediately cast in two types of molds. Despite this ratio is higher than the miscibility limit found by the authors in closed systems in equilibrium [9], this was done purposely because this operating condition was essential to guarantee water in the pores during the carbonation process. The low relative humidity of 60% promotes accelerated water evaporation, which decreases the initial W/C ratio in the paste and due to this fact; actually, no water segregation was noticed during and after casting.

Table 1
Chemical composition of the cement.

Compound	Content/%
CaO	66.92
SiO ₂	16.45
Al ₂ O ₃	5.00
SO ₃	4.44
Fe ₂ O ₃	3.30
TiO ₂	0.40
K ₂ O	0.35
MnO	0.28
SrO	0.25
ZnO	0.03
ZrO ₂	0.02
LOI	2.55

Table 2
Conditions of the carbonation treatment.

Specimens	Number of specimens for each experiment	Age for the initial of the carbonation treatment (h)	Carbonation time (h)	Temperature (°C)	Relative humidity (%)	CO ₂ concentration (%)
P _{REF}	4	–	–	25	100	–
P _{C1h}	4	6	1	25	60	20
P _{C24h}	4	6	24	25	60	20

- 36 cylinders with 25 mm of diameter and 50 mm of height for the compressive strength, absorption by capillarity and mercury intrusion porosity experiments.
- 36 discs with 50 mm of diameter and 25 mm of height for the tensile strength by diametrical compression, total absorption and gas permeability experiments.

For each experiment 12 specimens were separated, where:

- 4 specimens were conventionally hydrated during 28 days, immersed in water saturated with lime. These specimens are referenced as P_{REF}.
- The hydration process of this cement was studied in a previous paper of the authors, in order to know quantitatively how the content of each main hydrated phase was being produced as a function of time [8]. Initial setting data of the pastes at different W/C ratios were obtained by non conventional differential thermal analysis (NCDTA) and Vicat needle analysis. From these data it was seen that at 6 h of hydration, for W/C ratio equal to 0.7, the hydration was at the mid of the accelerating period as well as at the beginning of the setting process [9]. In this hydration time it was noticed from [8] the existence of a significant content of calcium hydroxide, as well from [9] a mechanical consistency of the paste that could allow its handling without damaging its shape. Consequently, it was decided to initially hydrate 8 specimens for 6 h without any carbonation in a controlled climatic chamber with RH = 100%, after which they were demolded and immediately placed in a controlled climatic chamber maintaining the best conditions of capture established by the authors [1], which are RH = 60%, 25 °C and a volumetric concentration of 20% of CO₂ in the chamber. The specimens were treated with CO₂ during 2 different times of carbonation: 4 specimens treated during 1 h of carbonation being referenced as P_{C1h} and 4 specimens treated during 24 h of carbonation being referenced as P_{C24h}, according to Table 2. After each carbonation treatment, the specimens were cured in an ambient with RH = 100% until 28 days after respective preparation. These carbonation times were selected from the results obtained in a previous paper of the authors [2], in which the compressive strengths after 14 days of hydration of similar specimens carbonated for 1, 2, 4, 8 and 12 h after being hydrated for 6 h in water saturated ambient, were respectively, 23.06, 21.25, 17.62, 17.03 and 15.91 MPa.

The mechanical tests of compressive strength and tensile strength by diametrical compression, were performed in a SHIMADZU machine, model UH – F1000kN with a loading speed of 0.01 mm/min, following the respective Brazilian NBR 5739 and NBR 9778 standard methods [12,13].

The tests of total absorption and absorption by capillarity, were performed according to the Brazilian NBR 9778 and NBR9779 standard methods [14,15], respectively.

The voids index is calculated according to the Brazilian NBR 9778 of the total absorption test [14], given by the equation:

$$V.I = \frac{Msat - Md}{Msat - Mi} \times 100$$

where

Msat is the mass of the saturated sample after immersion and boiling.

Md is the mass of the dried sample in a heater.

Mi is the mass of the saturated immersed sample after boiling for 2 h

Before the test, the samples were dried in a heater in a temperature of 35 °C during 3 days until constant mass.

The nitrogen gas permeability test was performed in a nitrogen gas permeameter used at LABEST/COPPE/UFRJ [16] with a controlled pressure of 0.2 MPa, which simplified schematics is shown in Fig. 1. To perform this experiment, all the samples were correctly cured to avoid any cracks, except the carbonated samples with 24 h, in which cracks could occur. For the apparatus, the lateral leaks (which could disturb the results) were prevented by the use an aluminum wrap in the lateral area, positioning the samples inside a silicone rubber cylindrical ring to guarantee the lateral isolation as well, before being placed in the test chamber. The experiment consists on measuring the time during which a drop of water entrained by the nitrogen flow passes through the 5 ml volume height of a pipette. The drop of water is injected at the bottom of the pipette as shown in Fig. 1 after attained a stable nitrogen flow. The gas permeability was calculated using the modified Darcy's law for compressible fluids.

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