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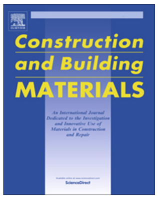

Experimental and statistical study on the irregularity of carbonation depth of cement mortar under supercritical condition

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HIGHLIGHTS

- We experimentally and statistically studied irregularity of carbonation depth.
- We systematically carried out supercritical carbonation tests of cement.
- We used imaging processing method to obtain irregular carbonation depth.
- We first consider power spectral density (PSD) of irregular carbonation depth.

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ABSTRACT

The heterogeneity of a cement-based material results in a random spatial distribution of carbonation depth. Currently, there is a lack of both experimental and numerical investigations aiming at a statistical understanding of this important phenomenon. This paper presents both experimental and numerical supercritical carbonation test results of cement mortar blocks. The carbonation depths are measured along the carbonation boundary by the proposed rapid image processing technique, which are then statistically studied by calculating, e.g., their probability density and power spectral density (PSD). The results indicate that the distribution of the carbonation depth can be approximately represented by a lognormal distribution function and the PSD has quantitative correlation with some of the statistic parameters used in the simulations. In particular, the effects of the autocorrelation lengths and the coefficient of variation of porosity, which are used to define the random porosity field, on the irregularity of carbonation depth are analyzed numerically in details and validated by experimental results. The study has shown that using a random field of porosity with due consideration of spatial correlation and variance, the irregularity of carbonation depth can be realistically captured by the numerical model. The numerical results confirm that lognormal distributions represent the random nature of carbonation depth well and the average and variance of the irregular carbonation depth increase with the increase of carbonation time, autocorrelation length and coefficient of variation of porosity. The study also offers a potential method to numerically calibrate some of the statistic parameters required by a numerical carbonation model through comparing the PSD with that from experimental tests. Overall the methodology adopted in the paper can provide a foundation for future investigations on probability analysis of carbonation depth and other similar work based on multi-scale and -physics modelling.

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1. Introduction

Carbonation of cement-based materials is a complex multi-physics process [1–5], involving chemical reactions of CO_2 with CH and C-S-H; gas-liquid two phase flow; dispersion and diffusion of CO_2 in water and temperature propagation. Extensive research has been carried out on natural and accelerated carbonation, including the reviews on carbonation of cement-based materials [6,7] and the life prediction model of cement-based materials under natural carbonation [8,9]. However, when the temperature and pressure exceed 304.12 K and 7.38 MPa, which are their respective critical values, CO_2 is in a supercritical fluid state that has a similar density of fluid and can diffuse through porous materials, such as cement-based materials, like a gas [10]. When the

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state of CO_2 is between supercritical fluid state and natural atmospheric environment, the carbonation of cement-based materials belongs to accelerated carbonation. Techniques have been developed in recent years to take advantages of the above carbonation processes to, e.g., modify composition and microstructure of cement-based materials [11–13] and to improve material properties using CO_2 curing [14–16]. The techniques have also important applications in CO_2 capture and storage [1], carbonation of hazardous water materials [17–19] and treatment of recycled concrete [20–22].

Carbonation depth is one of the most important characteristics that are used to define the extent of the chemical process taken place during carbonation. Experimental research has shown that under both natural [23] and supercritical [3,24] conditions, the boundary topography of a carbonation zone is irregular, characterized by a random distribution of depth along the boundary with distinctive maximum and minimum. However, current theoretical and numerical models are almost exclusively based on the assumption that the materials are isotropic and homogenous [25], resulting in an uniform carbonation depth [26]. There are very limited research on the irregularities of carbonation depth, including Huang, et al. [27] and Ruan, et al. [28]'s studies on the carbonation process of concrete where a non-uniform distribution of carbonation depth was observed by considering the influence of aggregates.

It can be concluded that that it was the heterogeneity of cement-based materials that contributes mostly to the observed randomness [29]. This includes the presence of coarse aggregates [30,31], the carbon dioxide gas diffusion paths caused by distribution of cracks [31,32] and the randomly distributed porosity of cement mortar [24]. To study the irregularity of carbonation depth, Huang, et al. [27] found that the variation of carbonation depth increased with increased use of coarse aggregates. Jiang, et al. [33] proposed a normal distribution model for carbonation depth in fatigue-damaged concrete and found that the probability distributions of carbonation depth were comparable with realistic frequency distribution histograms. However, apart from revealing the random nature of carbonation depth, there is a lack of quantitative assessment on its random distribution in the current literature.

How to quantitatively describe the topography of rough carbonation front is one of the challenging issues preventing an insight understanding of this important phenomenon. A quantitative description of this randomness front is important in many aspects in terms of not only providing a fundamental understanding of the carbonation process, but also providing a quantitative tool for evaluating material property changes due to carbonation and assessing, e.g., occurrence of steel corrosion due to CO_2 penetration. A literature review by the authors showed that power spectral density (PSD) is a powerful quantitative tool to measure a randomly distributed subject. For instance, researchers have used PSD to quantitatively characterize the topography of a rough surface so that the roughness could be further considered in estimating its impact on the mechanical and chemical properties of a material. PSD is a mathematical tool that decomposes a random signal into contributions from different spatial frequencies (wavevectors). Mathematically, PSD is a Fourier transform of the autocorrelation function of a signal, which contains just the power (and not the phase) across a range of wavevectors [34,35]. There are some applications of the spectral analysis in civil engineering, such as stochastic content of aggregate shape profiles [36], inelastic torsional response of buildings [37] and monitoring of corrosion of rebar embedded in mortar [38]. The primary utility of PSD in the above studies is that it contains unbiased statistical information on the randomness of objectives [39]. However, to the authors' best knowledge, there have been no attempts made to use PSD to describe carbonation depth. In addition, since PSD can provide a quantitative description of a random distribution, it can be used in a carbonation analysis to quantitatively determine the influences of materials, geometric and spatial parameters that define, e.g., randomness of porosity, on carbonation depth through comparisons between the PSD of experimental results and that of numerical models.

Due to the complex nature of the problem, this paper attempts, as a pioneer work, to focus on investigating the irregularity of carbonation depth of cement mortar caused by randomly distributed porosity before it can be developed further to include aggregates and micro cracks. Both supercritical carbonation experiments and multi-physics numerical simulations are carried out. The numerical simulations are based on the multi-physics model developed and validated previously by the authors [24], by which random porosity of cement mortar can be considered. An image processing technique is proposed in this paper for the spectral analysis of the experimental results. The probability density function and the PSD of carbonation depth from both experimental and numerical studies are found and discussed. The effects of some of the statistics factors, such as the autocorrelation spatial lengths and the coefficient of variation of porosity, on the carbonation depth are also discussed in this paper.

2. Experimental investigation on the irregularity of carbonation depth

2.1. Specimens preparation and mercury intrusion porosimetry

Cement mortar cubes with a dimension of 100 mm × 100 mm × 100 mm were cast for mercury intrusion porosimetry (MIP) to measure the average porosity of the material. The mix design proportions are given in Table 1. The specified 28-day cubic compressive strength of the cubes is 30 MPa. Ordinary Portland cement type P.O 42.5 was used as the binder for the mixtures. Normal river sands with fineness modulus of 2.7 were used as the fine aggregates.

Twenty-one cement mortar cubes were cast for the MIP and supercritical carbonation tests. After casting, the test specimens were covered with plastic sheets, and left in the casting room for 24 h. The specimens were then demolded and placed into a standard curing room with a constant temperature of 20 °C and humidity of 95% until the 28-day strength was achieved. Eighteen of the cured cubes were divided into 3 groups, each of which had six samples, for the supercritical carbonation test. The remaining three cubes were used for the MIP.

MIP is a widely-used technique for characterizing the distribution of pore sizes in cement-based materials. Three small cubic samples of approximately 1 cm^3 were taken at different locations of the cement mortar cubes mentioned above and tested by mercury intrusion porosimetry to obtain the average porosity of the cement mortar cube. The samples were then immersed in absolute ethyl alcohol to avoid further hydration. The threshold pressure was 7.11 Pisa for MIP. Fig. 1 shows cumulative mercury intrusion versus pore size diameter of the three small samples. The porosities were estimated at 11.5%, 10.8%, 13.5%, respectively, for samples A, B and C, which gave an average of 12.0%. The average value will be used in the following Sections as the average porosity of the cement mortar before carbonation. During the MIP, It was also found that the size of the pores ranged from 3 nm to 90 μm.

2.2. Supercritical carbonation tests: Procedure and setup

The supercritical carbonation tests were performed using a closed-cycle carbonation system that includes a sealed chamber,

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