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3D porosity distribution of partly calcium leached cement paste

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HIGHLIGHTS

• The 3D porosity distributions of calcium leached specimens are presented.

• Large porosities over 60% are observed on the leached parts of three specimens.

• Very sharp porosity gradients are observed on the leaching fronts.

The averaged porosity results are verified using the Gravimetric method.

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1. Introduction

Calcium leaching of cement-based materials is a chemical degradation caused by long-term contact with the low pH water or solution. It is important for applications in radioactive waste disposal containers, underground pipes, dams, and water tanks. In the calcium leaching processes, pore space plays a very important role. Firstly, the dissolution and the diffusion of calcium ion happen in the pore solution. Secondly, the dissolved calcium from skeleton will generate new pore space [1-5]; and these new pores will increase the effective diffusion coefficient or permeability, so further increase the leaching speed [6-10]; furthermore, the new porosity will decrease the stiffness and strength of the cement matrix [11-13]. Thirdly, in modeling research of calcium leaching, initial porosity is a key parameter for modeling, and the increased porosity is a key result showing the degree of leaching [3,9]. So how to effectively characterize the porosity evolution is very

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important for calcium leaching research.

ABSTRACT

Calcium leaching of cement-based materials is one of durability concerns in concrete structures. Calcium leaching leads to high porosity and porosity gradient near the wet surfaces of cement-based materials. In this research, the 3D porosity distributions of partly leached cement pastes with different water-to-cement ratios are characterized for the first time, and the porosity evolution caused by calcium leaching are discussed. The results are verified using the average porosities of the partly leached specimen. From the quantitative porosity results, large porosities over 60% are observed on the leached parts. and very sharp porosity gradients are observed on the leaching fronts. The results of current research can be used for the verification of calcium leaching models and for understanding the leaching mechanism. © 2013 Elsevier Ltd. All rights reserved.

> Gravimetric method [2,3,15] and mercury intrusion porosimetry (MIP) [1,4] have been used to guantitatively characterize the porosity of calcium leached cement-based materials. Both of them can only give the average porosity information of the whole characterized sample without spatial resolution showing the heterogeneity information, so they cannot show the gradual leaching trends. Scanning electron microscopy (such as in [14,16]) has been applied to characterize the porosity evolution of calcium leached cement-based materials, while it is still very hard to give the spatial distribution of porosity. X-ray Computer Tomography (CT) [17-19] has been applied to characterize the pore structure of leached specimens through simply segmenting the gray-scale values (GSV), while the spatial resolution for micro-pore is not yet sufficient even using the synchrotron micro-tomography with highest spatial resolution, so that the porosity is greatly underestimated. In the pioneer work of Withjack [20], a dual CT scan method to characterize the porosity has been proposed. The method has been applied to characterize the porosity distribution of different rocks [20,21].

> In this research, to understand the mechanism of calcium leaching, partly leached cement pastes with different water-to-cement (w/c) ratios are characterized by the dual CT scan method with one scan on dry specimen and the other scan on saturated







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specimen. The 3D porosity distributions of partly leached cement pastes are characterized.

2. Method of porosity distribution measurement

The proposed method is based on X-ray CT images. The principle of X-ray CT imaging is briefly introduced here, which has been discussed extensively elsewhere [22,23]. Basically, X-ray imaging measurements taken around an object from different directions produce cross-sectional images of an object. Lambert–Beer's law is used to relate the intensity of transmitted radiation and the intensity of incident radiation to the object described by its linear attenuation coefficient and the distance traveled through the object. The transmitted X-ray beams have a modulated intensity dependent on the overall linear attenuation characteristics of the intervening material. This varying intensity image is referred to as a projection, and the projection information is then manipulated to produce a reconstructed image of a slice of the sample, CT image. The resulting CT image is a spatial distribution of the linear attenuation coefficients, which is expressed by GSV, with brighter regions corresponding to higher values of the coefficient and darker regions to lower ones.

The principle of the dual CT scan method was first established by Withjack, which is mainly based on the additivity of linear attenuation coefficient [20]. If a voxel contains void space, its measured CT values will be a weighted average of the end-member values for air and the solid material. If water or water solution replaces air, the net attenuation will rise due to water being more attenuating than

air, and the increased CT value is proportional to the percentage of void space. Thus, porosity distribution can be obtained by imaging a sample twice with its pore spaces empty and filled with water or solution. This principle will be further expressed using the following physical equations.

Under a dry state, the total linear attenuation coefficient μ_d of a voxel containing cement matrix material and air can be expressed by:

$$\mu_d = \phi \mu_a + (1 - \phi) \mu_m \tag{1}$$

where ϕ is the porosity of the voxel, and μ_m and μ_a are the linear attenuation coefficients of the cement matrix material and air respectively. Similarly, under a saturated state, the total linear attenuation coefficient μ_s of the voxel containing cement matrix material and water or water solution can be expressed by:

$$\mu_s = \phi \mu_w + (1 - \phi) \mu_m \tag{2}$$

where μ_w is the linear attenuation coefficient of the dilute pore solution. Rearrange Eqs. (1) and (2), then, porosity of the each voxel can be obtained by:

$$\phi = \frac{\mu_s - \mu_d}{\mu_w - \mu_a} \tag{3}$$

In micro tomography systems, the final CT images is generally expressed by GSV but not CT numbers. Because GSV is proportional to linear coefficient with equivalent imaging conditions, porosity of the each voxel can be expressed by GSV of the sample in saturated state G_s , in dry state G_d , GSV of pore solution G_w , and GSV of air G_d :



Fig. 1. (a) Typical 2D slices of the saturated and (b) dry specimen with w/c ratio of 0.23, (c) histogram of (a) and (b); (d) typical 2D slices of the saturated and (e) dry specimen with w/c ratio of 0.35, (f) histogram of (d) and (e); (g) typical 2D slices of the saturated and (h) dry specimen with w/c ratio of 0.53, (i) histogram of (g) and (h).

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