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Research Paper

Experimental study and analytical model for pore structure of hydrated cement paste

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A R T I C L E I N F O

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A B S T R A C T

Mercury intrusion porosimetry (MIP) test is one of the techniques that have been widely used for analyzing the pore size distribution of hydrated cement paste and also for determining the pore structure parameters. This study presents the test results of the MIP experiments obtained for different cement paste specimens with the water–cement ratios of 0.3, 0.4, 0.5, 0.6 and 0.7 which had been cured for 1, 3, 7, 14 and 28 days. Thus, the effect of water–cement ratio and curing time on the pore structure of cement paste was investigated. The pore structure of samples is characterized by the porosity, pore size distribution as well as characteristic pore sizes. A thermodynamic pore fractal model was also used to calculate the pore fractal dimensions of specimens on the basis of MIP data. A mathematical model was developed to describe the pore size distributions of cement paste and related the pore size distributions with water–cement ratios and curing age. The procedure followed in this paper, i.e., determining the statistical distribution and analyzing the probability, can be used to interpret pore size data in other studies.

1. Introduction

Many important properties, such as strength (Chen et al., 2013; Kumar and Bhattacharjee, 2003a; Matusinovic et al., 2003; Ozturk and Baradan, 2008), corrosion resistance (Diamond, 1971), permeability (Atzeni et al., 2010; Cui and Cahyadi, 2001; Goto and Roy, 1981; Liu and Winslow, 1995), and shrinkage (Li et al., 2010; Meddah and Tagnit-Hamou, 2009) are closely related to the pore structures of cement-based materials. For the determination of usable relationship between pore structure and permeability it is necessary to choose the suitable pore structure parameters. It is generally accepted that the total porosity, as the basic structural parameter, appears not to be a significant measure for an evaluation of water permeability, because the water flow is influenced mainly by the pore size distribution rather than the total pore volume (Liu and Winslow, 1995).

Mercury intrusion porosimetry (MIP) is one of the most widely used methods for studying the pore structure characteristics of cement-based materials (Abell et al., 1999; Horpibulsuk et al., 2012; Kayyali, 1987; Winslow and Cohen, 1994). The MIP method has been used to investigate the influence of mineral admixture effect on the pore structure (Midgley and Illston, 1983) and the effects of carbonation on pore structure and diffusion properties of cement-based materials (Kumar and Bhatacharjee, 2004). Winslow and Liu (1990) found that the coefficient of chloride ion diffusion varied linearly with the critical pore radius as determined by the MIP, and the permeability followed a power–law relationship vs. the critical radius. However, there are some limitations of the MIP testing in determining pore size distributions in cement-based materials. The very fine pores in diameters from 1 to 3 nm are not intruded by mercury for most commercial porosimeters (Odler and Robler, 1985). Using image analysis, Diamond (2000) found that the pore shapes in hydrated cements are different from cylindrical pores assumed by the Washburn equation model. The assumptions in the Washburn equation model influence the result of pore size distribution in cement-based materials measured with the MIP. The "ink bottle" effect (Atahan et al., 2009; Olson et al., 1997) and the segmentation of capillary pores by the gel (Abell et al., 1999) produce the overestimation of fine pores and for the underestimation of larger capillary pores. During mercury intrusion under very high pressures, the mercury breaks down fragile walls within the cement microstructure (Ye, 2005). Diamond (2000) pointed out that the MIP measurements are useful only to provide threshold diameters and intrudable pore space measurements, which can serve as comparative indices for the connectivity and capacity of the pore system in hydrated cements.

Some researchers have maintained that fractal geometry can contribute to a description and interpretation of the microstructural complexity of cement-based materials (Atzeni et al., 2010). The applications deal with both the solid phases and the complementary porosity. Winslow (1985) recognizes the fractal character of the surfaces of cement pastes. Winslow (1985) guesses the fractality in the process of nucleation and growth of tricalcium silicate. Ficker et al. (2010) use the concept of fractal dimensions to describe the structure of pores defined through image analysis, while Winslow et al. (1995) apply it on the pore profile.

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A fractal character was recognized on materials similar to cement-based materials from a porosimetric point of view such as limes, soils, rocks and ceramics (Xu et al., 2008). In this paper, the fractal geometry is used to characterize the pore structure of cement paste.

In the present work mercury porosimetry measurements are made on Portland cement paste at five water–cement ratios (0.3, 0.4, 0.5, 0.6 and 0.7), hydrated for various periods of time up to 28 days. The main objective of the work is to determine the differences in the pore structures as a function of water–cement ratio and age of the paste.

2. Experimental procedures

2.1. Materials and sample preparation

The cement used in this work was ordinary Portland cement. The water used was potable laboratory tap water. Five water–cement ratios (0.3, 0.4, 0.5, 0.6 and 0.7) were used. The pastes were moist cured at 20 °C for periods of 1, 3, 7, 14, and 28 days. Moist curing consisted of wrapping the specimens with cling film and placing in plastic bags. After the required period of curing, paste samples of up to 5 g mass were taken from the middle central region of each cube and dried in a sealed cabinet at 40 °C under silica gel. This moderate form of drying

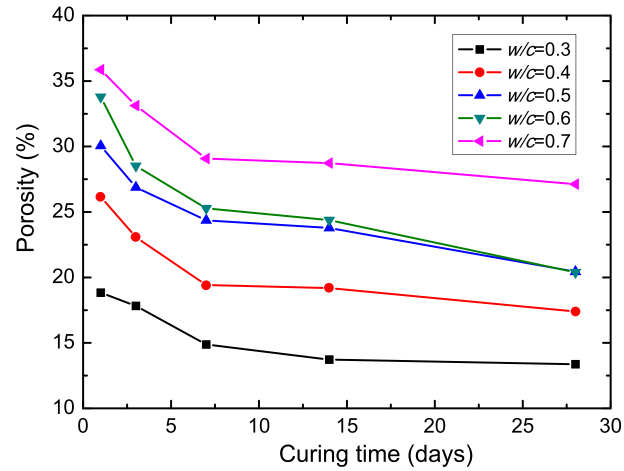

Fig. 1. Porosity of cement paste as a function of curing time.

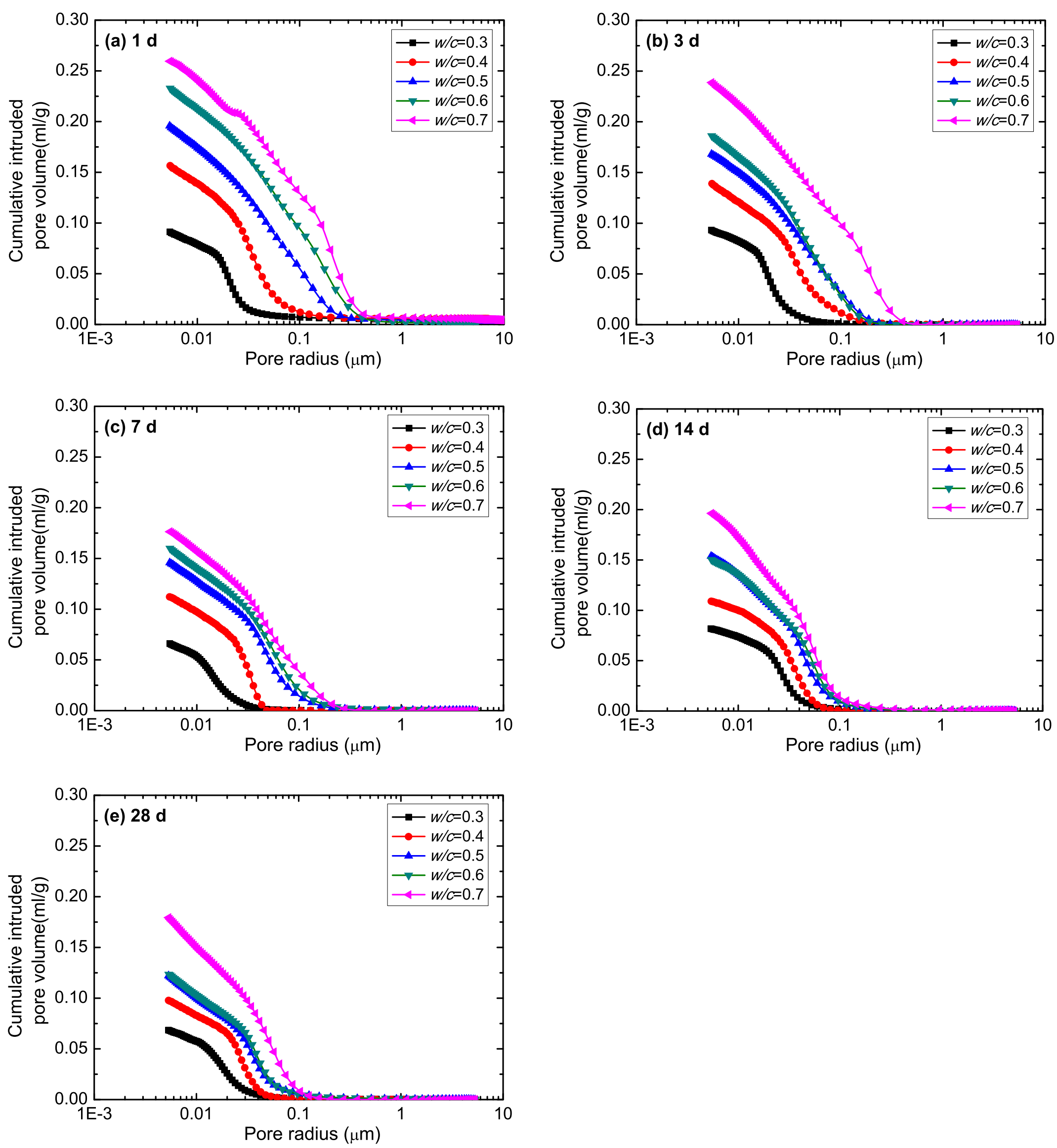

Fig. 2. Cumulative pore size distribution of cement paste at different ages, (a) 1 day, (b) 3 days, (c) 7 days, (d) 14 days, and (e) 28 days.

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