Construction and Building Materials 49 (2013) 407-416

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Pore structure in concrete exposed to acid deposit

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HIGHLIGHTS

• Development of pore structure in concrete exposed to acid deposit is studied.

• The pore structures of interior concrete are determined by using MIP, SEM, and CT.

• The probabilistic distribution model is suggested for porosity of the concrete under various exposure conditions.

• Relation between porosity and the mass change is analyzed.

ARTICLE INFO

Article history: Received 26 March 2013 Received in revised form 15 August 2013 Accepted 29 August 2013 Available online 20 September 2013

Keywords: Concrete Acid deposit Porosity Multiscale SEM MIP CT

ABSTRACT

Pore structure has a significant effect on the mechanical response and durability of concrete. To well understand damage evolution of concrete exposed to the acid deposit, the pore characteristics inside the concrete were studied from multi-scale levels. To simulate the acid deposit, acid solutions with pH level of 1.0, 1.5 and 2.5 were deposed by the mixture the sulfate and nitric acid in the laboratory. The pore structures of concrete under various deterioration states were examined by mercury intrusion porosimetry (MIP) test and scanning electron microscopy (SEM). Contents of the chemical elements in the concrete samples were measured by Energy Dispersive Spectra (EDS) analysis. Computed tomography (CT) test was carried out to examine the meso-structure in concrete at the desired exposure ages. The CT digital images were processed and analyzed by Pro-Plus software. According to the probabilistic analysis of the porosity, it is indicated that the porosity in all the concrete specimens under various damage conditions obeys to the normal distribution. A distribution density function of the porosity in concrete specimens was suggested, and it was revealed that the mean and variance values of the porosity decrease linearly with the conditioning age. It was illustrated that the porosity has a slight increase at the initial exposure period, and decreases gradually with the elongation of the conditioning age. The higher acidity of the conditioning environment was, the more obvious changes of the porosity in concrete occurred. The evolutions of pore character are discussed from both the micro- and meso- levels, which can well discover the development mechanism of macro- behavior of concrete exposed to acidic environment.

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1. Introduction

Durability of concrete structures in aggressive environments has become a major concern to the scientific community over the past several decades. At the present time, early age deterioration of the world's infrastructure is motivating the understanding of deterioration process of the construction materials. Some work has been done to investigate the mechanical performance of concrete in severe environment, and knowledge has been obtained [1–4]. However, the performance of the concrete (such as permeability, mechanical behavior, and durability.) is critically affected by the characteristics of the pore structure. The intensity of the interactions with the aggressive agents, and external loads always

* Corresponding author. Tel.: +86 13998546368. E-mail address: fanyf72@yahoo.com.cn (Y.-f. FAN). lead to the change of the pore structure inside concrete [5]. The results revealed that the change in micro structure, chemical content and pore structure are the critical reasons resulting in the deterioration of mechanical behavior of concrete. In order to discover the deterioration process of concrete servicing in the acidic environment, the understanding of the pore characteristics inside the concrete under various damage states is a crucial step.

Many research studies have been examined the microstructure of cementations materials [6-11]. Zhang carried out experiments on the effect of curing on the degree of capillary porosity of concrete in a tropical environment [12]. Duan studied the pore structure of concrete incorporating metakaolin (MK) when exposed to two types of curing conditions, seawater and fresh water. The results showed that the seawater curing condition further causes a corresponding low porosity at early days and a corresponding high porosity at later days [13]. Mendes' study indicated that a higher







^{0950-0618/\$ -} see front matter © 2013 Elsevier Ltd. All rights reserved. http://dx.doi.org/10.1016/j.conbuildmat.2013.08.075

Table 1Mix proportion of the concrete.

Cement (kg/m ³)	Sand (kg/m ³)	Coarse aggregate (kg/m ³)	Water (kg/m ³⁾	w/c	w/b	Undisturbed fly (ash/kg)	Reducing-water (agent/kg)	Slump (mm)
450	678	1040	159	0.353	0.304	60.0	12.8	180

Table 2

Conditioning details.

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	Series	Specimen dimension (mm)	Solution acidity	Immersion time (days)
	CA-1 CA-2 CA-3 CB-1 CB-2	$\begin{array}{c} 10 \times 10 \times 10 \\ 10 \times 10 \times 10 \\ 10 \times 10 \times$	pH1.0 pH1.5 pH2.5 pH1.5 pH2.5	0,3,7,10 0,3,7,10 0,3,7,10 0,5,10,15,20,30,40 0,5,10,15,20,30,40

porosity and coarser capillary pore size distribution (PSD) in paste compared to paste in concrete [14]. Chen conducted an experimental study to evaluate the effect of curing conditions on the porosity of concretes made with high slag blast furnace cement (HBFC) and ordinary Portland cement (OPC) [15]. Up to now, few studies considered the development of pore character in concrete with the action of acid deposit. Few results about the porosity distribution character in concrete suffering various damage conditions were reported.

The main objective of this study was to investigate the pore structures of concrete exposed to acid deposit from multiscale levels. The acid solutions with pH level of 1.0, 1.5 and 2.5 deposed by the mixture the sulfate and nitric acid in the laboratory were considered. After being exposed to the acidic solution for the desired periods, the concrete samples were picked out. The removed specimens were first dried for about two to three days, followed by the physical, computed tomography (CT), and scanning electron microscopy (SEM)/energy dispersive spectra (EDS) test. The CT digital images were processed and analyzed by Pro-Plus software. The threshold value is suggested for the identification of the pore. The developments of pore structure and porosity inside the concrete samples are examined. Pore distribution and porosity of concrete specimens suffering various deterioration processes are obtained. The relation between the pore characteristic and physical performance of concrete are discussed.

2. Experimental programme

2.1. Specimen preparation

Mix proportion of the concrete mixtures is shown in Table 1. The concrete mix was proportioned using the procedure recommended in the present code [16]. Cubic specimens with the dimension of 150 mm \times 150 mm \times 150 mm were cast. After being demoded after 24 h, the specimens were stored in the curing room for 28 days. Then, the compressive strength was tested according to the procedure recommended in the current standard [17]. The tested 28-day compressive strength of the designed concrete is 40 MPa. In order to obtain the pore distribution in concrete from micro-level, 10 mm \times 10 mm \times 10 mm cubic samples were drilled from the specimen.

2.2. Accelerated corrosion procedure

Two types of tests are usually used to simulate concrete structures attacked by environmental conditions: site and accelerated corrosion tests [2]. Although long-term exposure site test [18] can best simulate field conditions, it takes much longer time to achieve desired deterioration. Therefore, accelerated corrosion test was used in most of the past studies and is also adopted herein.

Submerging and spraying are the two main ways to accelerate concrete deterioration caused by acid rain in the laboratory. Based on the previous study [2,3], although the two methods are comparable and both give reliable results, the submerging method is more suitable for cement concrete, and is therefore adopted for accelerated ageing in this study. Since the acid rain is due to sulfuric acid in most parts of China, sulfuric acid type acid rain is simulated in this study. Acidic solutions with pH level of 1.0, 1.5 and 2.5 were deposed by the mixture of sulfate and nitric acid solutions (molar ratio is 9:1) in the laboratory, respectively. Acidity of the solution was recorded by PB-10 sartorius acidometer. The pH levels of the acid solutions were detected by the digital pH meter [19]. To keep the pH level of the mixed solution constant, nitric acid solution was added periodically. At the same time, the solution was stirred thoroughly to reduce differential concentrations of the acid inside the solution container. The specimens were divided into four groups denoted by series CA-1, series CA-2, series CA-3, series CB-1 and CB-2 (Table 2). Series CA is the $10 \text{ mm} \times 10 \text{ mm} \times 10 \text{ mm}$ cubic samples, and Series CB is the 150 mm \times 150 mm \times 150 mm cubic specimens. Series CA-1 through CA-3 was conditioned in solutions with pH levels of 1.0, 1.5 and 2.5, which covers practical range of the acidity of acid rain worldwide (Fig. 1). Series CB-1 and CB-2 were conditioned in solutions with pH levels of 1.5 and 2.5 respectively. Ageing effects were taken into consideration for all groups.

After being exposed to the acidic solution for the desired periods listed in Fig. 2, the samples were picked out. The removed specimens were firstly dried for about two to three days, followed by the physical, CT and SEM/EDS test.



Fig. 1. Concrete specimens exposed to the acid rain solution with various pH levels.



Fig. 2. 16 slice spiral CT scanner.

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