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Mercury intrusion porosimetry characterization of micropore structures of high-strength cement pastes incorporating high volume ground granulated blast-furnace slag



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HIGHLIGHTS

• We experimentally characterized the microstructures of high volume GGBFS-blended high-strength cement pastes.

• The total porosity increased over the GGBFS ratios in the specimens at early ages.

• The total porosity decreased over the GGBFS ratios in the specimens at later ages.

• A GGBFS replacement ratio of 65% produced the densest pore structures.

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ABSTRACT

In this study, we characterized the micropore structures of high-strength cement pastes incorporating high volume ground granulated blast-furnace slag (GGBFS). Using mercury intrusion porosimetry, we measured the pore-size distributions of cement pastes with different GGBFS replacement ratios at ages of 3, 7, 28, and 91 d. The test results indicated that the total porosity increased with increasing GGBFS ratio in the specimens at early ages, whereas it decreased at later ages. The replacement ratio of 65% produced the densest pore structure, which was consistent with compressive strength test results.

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1. Introduction

Recent worldwide efforts to reduce greenhouse gases have required development of eco-friendly concrete in the construction industry. In order to reduce the cement content in concrete, which is known to account for approximately 7% of all global carbon dioxide emissions [1], the increased use of concrete incorporating high volume ground granulated blast-furnace slag (GGBFS) is expected. The current practice, which generally allows GGBFS replacement ratios in the range of 30–60% [2], is still followed, even though there is strong demand for the use of higher volume GGBFSblended concrete. This continued use of low GGBFS replacement ratios is because the strength development of concrete with higher

* Corresponding author. E-mail address: schoi@cau.ac.kr (S. Choi). GGBFS replacement ratios is rather limited at early ages because of the latent hydraulic reaction of GGBFS [3,4].

Many studies have attempted to improve the strength development of high volume GGBFS-blended cementitious materials at early ages by employing low water-to-binder ratios. Gupta [5] found that when 70% of ordinary Portland cement (OPC) was replaced with GGBFS and silica fume at a water-to-binder ratio of 0.2, the early-age and later-age strengths increased by 10% and 17%, respectively, as compared to the control mix. Wang et al. [6] reported that when 50% of OPC was replaced with GGBFS, silica fume, and limestone powder at a water-to-binder ratio of 0.16, the compressive strength at 90 d increased to 175.8 MPa. Yazici [7] investigated the curing of concrete incorporating pulverized fly ash and GGBFS. The results indicated that when up to 80% of the cement was replaced with GGBFS, the compressive strength increased by 25–63% with steam curing and 9–61% for autoclaving as compared to standard curing. Zhang et al. [3] used nano-silica in



order to overcome limitations in high volume slag-blended cement such as the delayed setting time and the insufficient strength development at early ages. As compared to the reference concrete, which incorporated 50% slag, the initial and final setting times were reduced by 95 min and 105 min, respectively, when 2% nano-silica was included. In addition, the compressive strength increased by 22% at 3 d and 18% at 7 d. Other studies investigated the hydration of GGBFS-blended cementitious materials. For example, Luan et al. [8] studied the hydration of GGBFS-blended cement and characterized the hydration products. Zhou et al. [9] suggested that as the GGBFS content in the binders increases, the porosity of the GGBFS-blended cement pastes increases because the hydration rate of GGBFS is relatively slow owing to the latent hydraulic properties of GGBFS. Chen and Brouwers [10] proposed the hydration model of GGBFS-blended cement paste, in which the interactions between GGBFS and calcium hydroxide (CH) are considered based on stoichiometry, so that the chemical compositions of the unhydrated GGBFS and OPC can be correlated with the quantities of hydration products and the composition of calcium-silicate-hydr ate (C-S-H).

Several different factors that affect the strength development of high volume GGBFS-blended cementitious materials with low water-to-binder ratios have been investigated in previous studies. However, the hydration characteristics and the corresponding micropore structures of materials incorporating various GGBFS replacement ratios have seldom been addressed, even though they have a significant influence on the strength development of highstrength, high volume GGBFS-blended cementitious materials. In this study, we evaluated the effect of GGBFS replacement ratio on the pore structures of GGBFS-blended cement pastes with a considerably low water-to-binder ratio of 0.2 and analyzed variations in porosity and threshold diameter, at different ages. In addition, setting time, rate of heat development, and compressive strength were measured. The relationship between the porosity and compressive strength of the specimens was estimated.

2. Experiment

2.1. Materials

GGBFS-blended cement-paste specimens were fabricated using OPC and GGBFS. The OPC used in the test was Type I Portland cement, satisfying ASTM C150. The OPC had a Blaine fineness and density of $3270 \text{ cm}^2/\text{g}$ and 3.18 g/cm^3 , respectively. GGBFS satisfying ASTM C989 had a Blaine fineness and density of $4330 \text{ cm}^2/\text{g}$ and 2.89 g/cm^3 , respectively. Table 1 shows the chemical compositions of the OPC and GGBFS used in the experiment. The mineral composition of the OPC through Bogue's equation [11] was 58.1%C₃S, 15.0% C₂S, 8.1% C₃A, and 9.2% C₄AF. Fig. 1 shows the particlesize distributions of the OPC and GGBFS used in the experiment. The distributions were measured in accordance with ISO 13320

| Chemical compositions | of | OPC | and | GGBFS |
|-----------------------|----|-----|-----|-------|
|-----------------------|----|-----|-----|-------|

| | OPC (mass%) | GGBFS (mass%) | |
|--------------------------------|-------------|---------------|--|
| SiO ₂ | 20.80 | 34.00 | |
| Al_2O_3 | 4.93 | 16.40 | |
| Fe ₂ O ₃ | 3.50 | 0.50 | |
| CaO | 62.40 | 37.20 | |
| MgO | 1.61 | 6.29 | |
| K ₂ O | 0.90 | 0.45 | |
| Na ₂ O | 0.33 | 1.33 | |
| SO ₃ | 2.21 | 2.71 | |
| H ₂ O | - | _ | |
| LOI | 2.74 | 0.17 | |
| Sum | 99.42 | 99.05 | |

LOI: loss of ignition.



Fig. 1. Particle-size distributions of OPC and GGBFS.

[12] using a laser diffraction particle-size analyzer (Partica LA-950V2, Japan HORIBA).

2.2. Mixture proportions and test methods

Table 2 shows the mixture proportions of the specimens in the experiment. The cement in the specimens was partially replaced with GGBFS at replacement ratios of 35, 50, 65, and 80% by weight. The water-to-binder ratio was 0.2 so that the results could be related to the characteristics of high-strength materials. The superplasticizer used was 0.53% of the binder by weight (63.6 g) and the total water used in all the samples was 2400 g. The number following the letter "S-" in the specimen name indicates the replacement percent of cement with GGBFS by weight, i.e., 35, 50, 65, and 80%.

The materials were mixed in accordance with ASTM C305. The setting time was determined using a Vicat needle in accordance with ASTM C191. The needle test was conducted in a room at 23 °C and $50 \pm 5\%$ relative humidity (RH). An isothermal calorimetry test was conducted in accordance with ASTM C1702. A TAM-AIR isothermal calorimeter (TA Instruments) was employed to measure the heat flow of the specimens with time. Approximately 4 g of each specimen was placed in a 20 mL glass ampoule, and the heat flow of the specimens was measured for 48 h at a constant temperature of 20 °C.

The mixtures were cast into $40 \times 40 \times 160$ mm molds, which were placed for 24 h under the standard curing condition at constant temperature and RH (23 ± 1 °C; 100% RH). After demolding, the paste bar specimens were cured under the standard water curing condition at 23 ± 1 °C. At ages of 3, 7, and 28 d, the compressive strength test was conducted in accordance with ISO 679. Each bar, which was $40 \times 40 \times 160$ mm in size, was split into two bars. Compressive strength was measured for each split bar, which was $40 \times 40 \times 80$ mm in size. For the MIP experiment, the specimens were split into $10 \times 10 \times 10$ mm cubes and dried at 60 °C for 24 h. Using the Micromeritics AutoPore IV9500 system, the

| Table 2 | |
|---------------------|-------------------|
| Mixture proportions | of the specimens. |

| Specimen | OPC (g) | GGBFS (g) | S.P.* (g) | Water (g) | w/b** (-) |
|----------|---------|-----------|-----------|-----------|-----------|
| S-35 | 7800 | 4200 | 63.6 | 2400 | 0.2 |
| S-50 | 6000 | 6000 | 63.6 | 2400 | 0.2 |
| S-65 | 4200 | 7800 | 63.6 | 2400 | 0.2 |
| S-80 | 2400 | 9600 | 63.6 | 2400 | 0.2 |

* Superplasticizer.

Water-to-binder ratio.

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