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# Microstructure, permeability and mechanical properties of accelerated shotcrete at different curing age

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## HIGHLIGHTS

- High $w/b$ and volume of fly ash prolonged the setting time of pastes with accelerator.
- Hydration process of accelerated shotcrete was studied by using XRD, TG–DSC and SEM.
- It had good correlation between pore structure and permeability of shotcrete after curing 90 d.
- The model was established of shotcrete in term of $w/b$, fly ash and curing age.
- The relationship between strength and pore structure or permeability was discussed.

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## ABSTRACT

Given the different hydration processes of ordinary concrete, shotcrete with low-alkali accelerator not only has short final setting time and high early age mechanical properties but also different hydration products. Shotcrete microstructure was studied at different curing ages by X-ray diffraction, thermogravimetry–differential scanning calorimetry, and scanning electron microscopy to investigate its hydration process. The pore structure and permeability of shotcrete were also tested. The mechanical properties of shotcrete were then studied. Results showed that under the effect of accelerator, the retarded action of gypsum disappeared in the cement–accelerator–water system. $C_3A$ hydrated quickly to form calcium aluminate hydrate (CAH) crystals, and a mesh structure was formed by ettringite, albite, and CAH. A large amount of hydration heat improved the hydration rate of the cement clinker mineral and the density, thus leading to high mechanical properties at the early curing age. The setting time of pastes increased with increasing water–binder ratio and fly ash (FA) dosage. Thus, the hydration process and the microstructure and morphology of the hydration products changed. Excessive water and FA increased the percentage of pores in shotcrete, which directly caused poor permeability. The permeability and compressive strength were poor when the FA dosage was 30% compared with that of 20%. Based on the investigation, a simple 0.5 power relationship between compressive strength and splitting tensile strength of mixture S2, meanwhile, there was an exponential relationship between mechanical performance and permeability or porosity, respectively.

## 1. Introduction

Sprayed concrete is concrete which is conveyed under pressure through a pneumatic hose or pipe and projected into place at high velocity, which simultaneous compaction, condensation and hardening [1–3]. Compared with ordinary concrete, shotcrete has short setting time and high early age mechanical properties [4]. Therefore, shotcrete is widely used in different fields, such as tunnel support, rapid repair, slope support, gas and oil wells, and other underground structures [5–8].

The hydration process changes when using an accelerator in concrete; thus, the microstructure, permeability, and mechanical properties of shotcrete is different from that of ordinary concrete. Robert [9] proposed that the microstructure of shotcrete depends on raw material composition, hydration process, and curing age. The permeability and mechanical properties are determined by the microstructure [10].

The hydration of Portland cement (PC) has been widely investigated. Andrew [11] and Rikard [12] analyzed the mechanical property development and early hydration physical chemistry of PC paste by X-ray diffraction (XRD), infrared spectroscopy (IR), scanning electron microscopy (SEM), Vicat techniques, and ultrasound

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reflection. Deschner [13] demonstrated that pozzolanic reaction and increased strength was observed with portlandite consumption because of the pozzolanic reaction developed after 28 d of hydration. Zeng [14] reported the application of mercury intrusion porosimetry (MIP) and nitrogen adsorption/desorption (NAD) methods to analyze the relationship between the pore structure of cement paste and water–binder ratio (*w*/*b*), fly ash (FA) content, and age. Hamlin [15] built the microscope structure model of calcium silicon hydrate (CSH) gel through theoretical analysis.

However, only a few investigations have been carried out on the hydration process and properties of shotcrete and fiber reinforced shotcrete. Barbara [16] considered that ettringite and CSH are the main hydrates formed during hydration of low-alkali cement consisting of 60% cement, 40% microsilica, and 4.8% accelerator. Gwenn [17] and Paglia [18] studied the hydration mechanism of ordinary PC (OPC) with calcium sulfoaluminates and modeled the change process of the hydration products. Christopher [5], Won [19], and Park [20] investigated the mechanical properties and permeability of shotcrete with different types of accelerator.

The mineral composition, hydration product morphology, and pore structure was examined by X-ray diffraction, thermal analysis, scanning electron microscopy, and mercury intrusion porosimetry to study hydration process and properties of shotcrete. Permeability was also tested for Auto Clam, initial surface absorption, and rapid chloride migration (RCM). Thereafter, the compressive and splitting tensile strength tests were performed.

## 2. Materials and experiments

### 2.1. Materials

An OPC and FA complying with Chinese standard GB175-2007 [21] and GB/T1596-2005 [22] were used in this study. Their characteristics were examined by using chemical analysis, particle size analysis, and XRD. The chemical and physical properties of the cementitious materials were obtained by using X-ray fluorescence spectrometry (BRUKER-AXS S4PIONEER, BRD). Figs. 1 and 2 show the particle size distribution and XRD result of the cementitious materials. The fine aggregate used in this study was natural river sand with a fineness modulus of 3.5, an apparent density of 2.688 g/cm$^3$, and a water absorption of 1.2%. The coarse aggregate used was gravel with continuous grading from 5 to 10 mm. The density and absorption were 2650 kg/m$^3$ and 0.61%, respectively. Both aggregates complied with the GB50086-2001 requirement [23]. The low-alkali accelerator (AC) used contained $NaAlO_2$ and $Ca_2SiO_4$ ($C_2S$) as the major substances. The chemical characteristics of the cementitious materials and accelerator are listed in Table 1.

### 2.2. Specimen preparation

Shotcrete specimens were prepared with three *w*/*b* ratios (0.38, 0.43, and 0.49) and four FA amounts (0%, 10%, 20%, and 30%). The addition of FA (FA/b) was noted as the mass ratio between FA and cementitious materials. The six mixtures are illustrated in Table 2. The sand to total aggregate ratio of 0.50 and the mixing amount of superplasticizer and accelerator was 1% and 4% of the cementitious material, respectively.

The concrete was first sprayed by using the dry-mix method with large slabs of 1 m × 0.5 m × 0.12 m (Fig. 3). After 3 h, the slabs were removed and taken into the tunnel for 7 d of curing. Subsequently, large slabs were cut into specimens with the size of 400 mm × 100 mm × 100 mm by using automatic rock cutting machine. Afterwards, the prism specimen was cut into three cube samples with length of 100 mm. The specimens were moist cured at 20 ± 2 °C for 21 d and then dry cured until testing [24]. The cutting mode for the shotcrete specimens is illustrated in Fig. 3.

### 2.3. Testing method

#### 2.3.1. Setting time

Pastes of three *w*/*b* ratios and four FA amounts at different curing ages were used for the setting time, which was measured according to ASTM C191-08 [25]. The mixture properties of the pastes are illustrated in Table 3.

#### 2.3.2. Microstructure

Microstructure characterization included XRD analysis, thermal analysis, and hydration product morphology.

XRD analysis was used to examine the mineral phase composition of the hydration products at different curing ages. After being removed from the ethanol, the pastes were first pulverized in ethanol by an agate mortar and passed to an 80 μm sieve as vacuum filtrating. The powder was dried at 60 °C for 8 h in a vacuum drying oven. The XRD patterns of the pastes were tested by using XRD, with a Cu K$\alpha$ source, a scanning range of 7–60°, and a speed of 2° per minute.

Thermal analysis, particularly the thermogravimetric (TG)–differential scanning calorimetry (DSC) hyphenated method, is an effectively method for quantifying and identifying hydration products, including ettringite, *AFm*, and portlandite. The samples for thermal analysis were prepared similar to the preparation for XRD analysis. TG–DSC analysis was conducted by using a simultaneous thermal analyzer with a uniform heating rate of 10 °C per minute from 25 to 900 °C in a gas flow atmosphere.

SEM was used to observe the microstructure and morphology of the hydration products. After compressive strength testing, few pieces of the samples were taken out from the shotcrete specimen and immersed into ethanol to terminate hydration. Before testing, the samples were removed from the ethanol, air dried at room temperature (RT) for 10 min, and then dried at 60 °C for 8 h. A platinum coating was subsequently applied onto the surface e of the samples.

#### 2.3.3. Pore structure

The pore structure of the shotcrete specimen was evaluated by the MIP method. The bulk samples with a diameter from 3 to 6 mm were taken out of the ethanol for immersion for 24 h, dried at RT for 10 min, and then dried at 105 °C for 6 h.

#### 2.3.4. Permeability

Permeability is a method for characterizing the difficult degrees and abilities of permeation, diffusion, and migration by gas, liquid, and ions under pressure, chemical potential, and electric field. Permeability is an important part of concrete durability. The permeability study included air permeability, water absorption, and the RCM method.

*2.3.4.1. Air permeability.* Air permeability was tested by using an Auto Clam permeability instrument (Fig. 4) with a 100 mm cubic shotcrete specimen [26,27]. Before testing, air was implanted inside the instrument by syringe through the pipe until the air pressure was more than 500 mbar (1 mbar = 500 Pa). After starting the test, the air pressure was recorded per minute. Given the linear relationship between the natural logarithm of air pressure and time, the slope (*K*) of the liner could be used as an index for evaluating air permeability (shown in Table 4).

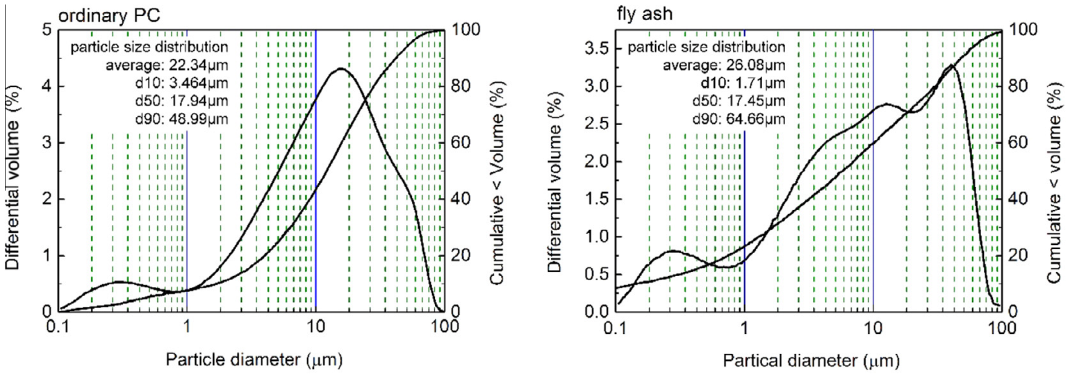

**Fig. 1.** Particle size distribution of ordinary PC and fly ash. $d_{10}$, $d_{50}$, and $d_{90}$ are the 10th, 50th, and 90th percentile by volume of the particle size distribution, respectively.