



## The pore structure and permeability of alkali activated fly ash

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### HIGHLIGHTS

- ▶ We study the pore structure and water permeability.
- ▶ We discuss the relationship between the permeability and pore structure parameters.
- ▶ A modified model is proposed to predict the water permeability.

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### ABSTRACT

This study correlates the pore structure and water permeability of paste made of alkali activated fly ash (AAF) with varying silica and alkali content, curing age and curing temperature. The pore structure of AAF paste was examined by mercury intrusion porosimetry (MIP) and environmental scanning electron microscopy (ESEM), and compared with the pore structure of Portland cement paste. The results reveal that the pore structure of AAF paste with high silica content significantly differs from that of cement paste, resulting in different water permeability. For equal reaction age, the permeability of AAF paste was higher than that of cement paste with  $w/c = 0.4$ , an effect (that was) especially pronounced at later ages. However, a longer curing time at elevated temperature could significantly decreased the water permeability of AAF paste. MIP results show a consistent relationship between water permeability and total porosity, effective porosity and pore threshold diameter, respectively. Furthermore, a modified model incorporating all hereby experimentally derived parameters, is proposed to predict the water permeability of AAF.

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### 1. Introduction

Utilization and further development of alkali activated fly ash (AAF) and alkali activated slag (AAS) as potential construction materials are known and reported to have lower environmental impact compared to ordinary Portland cement, mainly due to the reduced emission of greenhouse gas during production and application [1,2]. The reaction product of alkali activated materials (AAMs) is a highly connected three-dimensional aluminosilicate gel [3], which provides sufficient mechanical strength comparable to that of calcium silicate hydrate (CSH) gel in cement-based materials. Although the utilization of AAM has economic and environmental advantages, the durability of this construction material in terms of service life is still largely unknown.

In order to assess durability, and further predict service life of AAM as a construction material, the understanding of water

transport and its correlation with microstructure parameters is a crucial step. Water transport, pore structure characteristics and relevant ion transport are closely linked to durability issues, especially for reinforced concrete structures. It is well known that water can be a carrier of  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$  or  $\text{CO}_2$  that can penetrate into the concrete bulk material, interact with the reaction products and/or reach the steel/paste interface. The result is an altered matrix microstructure and/or corrosion initiation which will further lead to degradation of the reinforcement. To this end, water transport and water permeability are considered to be of significant importance for durability related issues [4]. Many studies discussing the effect of various relevant parameters on the water permeability of cement-based materials have been reported [5–7]. However, such investigations are limited with regard to AAM.

The most important parameter that influence the water and ion transport is the materials' pore structure, particularly the pore volume, pore size distribution, connectivity and shape of the pores [8]. The pore structure of alkali activated fly ash and metakaolin has been studied by using gas sorption porosimetry [9] and mercury intrusion porosimetry (MIP) [10]. Lloyd et al. characterized the

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pore structure of AAM by Wood's metal intrusion porosimetry [11]. Valuable information was obtained, revealing the difference in pore structure of AAM and cement-based materials. Given the distinctly different pore structure of AAF, the water transport process is logically expected to be very different. However, to our best knowledge, no data is available on the relationship between pore structure and water permeability, and the durability of this material.

Therefore, the objective of the present study is to investigate the water permeability of AAF and to examine the correlation to pore structure parameters. The pore structure of AAF was characterized by the MIP and environmental scanning electron microscopy (ESEM); the coefficient of water permeability was calculated by using Darcy's law.

## 2. Materials and methods

### 2.1. Materials

The Class F fly ash (according to ASTM), produced in The Netherlands, was used in this study. Table 1 presents the chemical composition of fly ash determined by X-ray fluorescence spectrometry (XRF). The fly ash was activated with a series of activating solutions with different silica and sodium oxide content. The chemical composition of each solution is given in Table 2. Sodium hydroxide (analytical grade, >98% purity), sodium silicate solution (Na<sub>2</sub>O: 8.25 wt.%, SiO<sub>2</sub>: 27.5 wt.%) and distilled water were used to prepare the activating solutions. These solutions were further used as the "mixing water" for casting the AAF samples.

### 2.2. Sample preparation

AAF specimens were cast by mixing fly ash with the activating solutions, maintaining constant water to fly ash mass ratio of 0.35. Two different curing conditions were applied in this study to investigate the effect of continuous heat curing on pore structure development and water permeability:

*Curing condition I:* after casting (and before testing), the specimens were sealed and cured in an oven at 40 °C for 7 days; afterwards, curing continued at 20 °C (relative humidity >98%) until for 3 or 6 months. Thus in curing condition I three ages were monitored (i.e. 7 days, 3 and 6 months).

*Curing condition II:* after casting the specimens were sealed and cured in an oven at 40 °C for 28 days. Thus the investigated age for this curing regime was just 28 days.

For MIP tests, samples were tightly sealed in plastic bottles. At a certain curing age, the samples were first crushed (by hammer) into small pieces with dimensions of 1–2 cm<sup>3</sup>. Then these pieces were immersed in liquid nitrogen to cease the chemical reaction and placed in a vacuum freeze-dryer at –28 °C until constant weight. This quick freezing and drying process at low temperatures allows the remaining liquid solution transforming into ice micro-crystal and further removed by sublimation without a significant damage to the microstructure [12]. For the permeability tests, the samples (in the form of slurry) were cast in PVC cylinders with an inner diameter of 95 mm. The thickness of these samples was around 10 mm. After casting, these samples were cured in sealed

**Table 2**  
Chemical composition of alkali solutions for 1 kg fly ash.

Sample (SiO <sub>2</sub> –Na <sub>2</sub> O)	SiO <sub>2</sub> (mol)	Na <sub>2</sub> O (mol)	H <sub>2</sub> O (g)
0.5–1.5	0.5	1.5	350
1–1.5	1.0	1.5	350
1–1.3	1.0	1.3	350
1–1.0	1.0	1.0	350
1.5–1.5	1.5	1.5	350

containers. At each curing age, three samples were taken out from the sealed mould and immersed in water, followed by vacuum saturation for around 17 h.

### 2.3. Methods

In this study, MIP and ESEM were employed to characterize the pore structure of AAF.

#### 2.3.1. MIP

MIP is widely used to determine both the porosity and pore size distribution of cement-based materials for decades [13,14]. In this study, the porosity and pore size distribution of AAF were examined by Micromeritics Poresizer 9500. The measurement was conducted in two stages. The first stage is at low pressure: from 0 to 0.0036 MPa. The second stage is the high pressure running from 0.0036 to 210 MPa and followed by an extrusion running from 210 to 0.14 MPa. The Washburn equation [15] was used to calculate the diameter of pores intruded by mercury at each pressure step. In formula form:

$$D = \frac{-4\gamma\cos\theta}{P} \quad (1)$$

where  $D$  is the pore diameter ( $\mu\text{m}$ ),  $\gamma$  is the surface tension of mercury (mN/m),  $\theta$  is the contact angle between mercury and the AAF materials ( $^\circ$ ) and  $P$  is the applied pressure (MPa). The surface tension used here is 0.485 N/m, and the contact angle is 132° based on a study reported in [16]. According to the Washburn equation, the pore size ranging from 350  $\mu\text{m}$  to 0.007  $\mu\text{m}$  can be detected.

The "total porosity", "effective porosity" and "threshold (critical) pore diameter" are deduced from the MIP results as shown in Fig. 1. The volume of mercury intruded at the maximum experimental pressure per gram sample multiplying the bulk density of the material is known as the "total porosity" (open pores in material). During the intrusion, the continuous pores, including the "ink-bottle" pores [13], can be detected from the intrusion curve. After the intrusion, the mercury is extruded from the specimen by lowering down the pressure. However, the mercury in the "ink-bottle" pores will stay inside. The volume of mercury that remains inside during the extrusion is called "ink-bottle" porosity. The total porosity minus the "ink-bottle" porosity is defined as "effective porosity". The pore diameter corresponding to the highest rate of mercury intrusion per change in pressure or in diameter ( $dV/dP$  or  $dV/dD$ ) is known as the "critical" or "threshold" pore diameter [17].

It was argued that the MIP method could not reflect the true pore size distribution of cement-based materials [18], mainly due to the different pore shapes and "ink-bottle" effect. On the other hand, the permeability and diffusion characteristics of materials

**Table 1**  
Chemical composition of fly ash used in this study (wt.%).

Oxides	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>
Fly ash	48.36	31.36	4.44	7.14	1.35	1.64	0.72	1.24	1.90	1.18

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