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# Water vapor sorption isotherms, pore structure, and moisture transport characteristics of alkali-activated and Portland cement-based binders

M. Babae<sup>\*</sup>, A. Castel

Centre for Infrastructure Engineering and Safety, School of Civil and Environmental Engineering, University of New South Wales, Sydney, Australia

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## ABSTRACT

Moisture transport plays a key role in determining the different durability-related features of cementitious materials. In this paper, moisture sorption in a range of low-calcium (geopolymer-type) and calcium-rich alkali-activated binders are studied and compared with that of Portland cement-based binders. Through the analysis of water vapor sorption isotherms (WVSI) and mercury intrusion porosimetry (MIP) test results, two vastly different pore structures were observed. Fly ash-based geopolymer-type binders showed a very porous structure where a large volume of mesopores coexisted with a significant volume of macropores. Alkali-activated slag binders, however, had a very fine pore structure, with a relative lack of large macropores. The different pore structure of fly ash-based and slag-based binders led to amplification of pore blocking and cavitation in blended systems by the addition of slag. Analysis of the sorption kinetics showed the prominent effect of the presence of calcium in the matrix to reduce the permeability.

## 1. Introduction

The moisture content of concrete influences several other concrete properties, such as electrical resistivity and the oxygen diffusivity, both of which are of significant importance during the propagation phase of corrosion. The transport of moisture through a convective process during wetting/drying cycles also facilitates the penetration of aggressive agents such as chloride ions in marine environments that can lead to the eventual depassivation of reinforcement. Modeling moisture transport, as a result, is a fundamental component of any comprehensive durability model and requires some material-specific information regarding both the rate of moisture adsorption/desorption and the relationship between the moisture content and the environmental relative humidity, which is established by water vapor sorption isotherms (WVSI) at a fixed temperature. WVSI proved to be extremely useful for the amount of information they provide regarding the pore structure in nanoporous materials, the sorption transport rate, and water binding mechanism [1,2]. In addition, pore structure and permeability implications from WVSI studies correlate well with other phenomena, such as carbon dioxide and chloride diffusion.

Although moisture transport and WVSI in Portland cement-based binders have been studied by many researchers in the past (see for example [1–4]), there is a lack of information in the literature about alternative binders, such as alkali-activated binders. Regarding the

study of WVSI (and moisture transport) in alkali-activated binders, only two relevant studies could be found in the open literature. Boher et al. [5] reported the WVSI of four metakaolin-based geopolymers fabricated from various alkaline solutions at different modulus ratios. An almost constant slope of WVSI for RH levels of between 20% and 70% followed by a sharp increase in the slope of WVSI for RH > 70% was observed. This suggests the presence of a considerable volume of large pores that coexist with a large volume of small nanopores. This pattern is considerably different from OPC binders, where a network of connected capillary pores and smaller gel pores form the pore structure. This has also been reported by de Burgh et al. [6], who studied the main features of the WVSI of two heat-cured alkali-activated blended FA and GGBS binders with GGBS contents of up to 35%. Furthermore, a lack of low relative humidity hysteresis, which was characteristic of a lack of water binding capacity, was reported by de Burgh et al. [6] for the investigated mixes.

When it comes to pore structure analysis, however, more studies could be found in the literature (see e.g. [7–10]). The majority of them investigated the pore structure of aluminosilicate networks fabricated of metakaolin or fly ash by employing gas adsorption/desorption, mercury intrusion porosimetry (MIP), or Wood's metal intrusion and the general agreement found in all these publications was that the aluminosilicate networks are highly porous with considerable pore blocking in the network. The effect of calcium in the matrix of blended

<sup>\*</sup> Corresponding author.

E-mail addresses: [sm.babae@gmail.com](mailto:sm.babae@gmail.com), [mahdi.babae@unsw.edu.au](mailto:mahdi.babae@unsw.edu.au) (M. Babae).

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systems on the pore structure has attracted comparatively less attention. Provis et al. [8] employed X-ray microtomography that allowed direct assessment of the pore structure and tortuosity of blended FA and GGBS binders. While they observed a general improvement (i.e. finer pore development with higher tortuosity) in the pore structure as a consequence of GGBS addition, their findings were restricted to microscale pores due to technical limitations.

In this study, the WVSI of one heat-cured paste sample with 50% GGBS replacement and three ambient-cured blended FA and GGBS pastes with 25, 50 and 75% GGBS replacement are evaluated using a sorption apparatus. The measured WVSI along with the two WVSI that were previously published by de Burgh et al. [6] are quantitatively analysed to give a better understanding of the nanopore structure of alkali-activated binders, in particular, to assess the effect of GGBS addition, alkali concentration, and curing regimes on the pore structure. The main features of the WVSI of alkali-activated binders are discussed and compared with those of Portland cement-based binders from the literature. The sorption kinetics of two ambient-cured alkali-activated binders with low and high calcium content are studied for the first time and compared with Portland cement-based binders. Major conclusions are made with regard to the type of sorption process (i.e. diffusive (Fickian) or anomalous (non-Fickian)), in light of the differences observed in the pore structures. The permeability of low-calcium geopolymer-type binders and calcium-rich binders are compared and key implications for durability design of alkali-activated binders are deduced.

## 2. Experimental program

### 2.1. Materials and mix proportions

Three different types of Australian-sourced low-calcium fly ash (FA) were used in this study to manufacture alkali-activated pastes: Eraring fly ash obtained from the Eraring power station in New South Wales, and Callide and Gladstone fly ashes from Callide and Gladstone power stations in Queensland, respectively. Ground granulated blast-furnace slag (GGBS) was supplied in two batches (namely A and B) by Australian Steel Mill Services (ASMS), Port Kembla, New South Wales. To prepare Portland cement-based control paste samples, Australian general-purpose cement (Type GP, SL and HE to AS 3972) manufactured at the Boral Berrima Cement Works (NSW, Australia) was used. To produce the cement, 7.5% limestone mineral addition was used. The chemical compositions of the raw precursors as determined by X-ray fluorescence (XRF) analysis are shown in Table 1. The amorphous content of the raw materials used for alkali-activated paste fabrication was also measured through the X-ray diffraction (XRD) test using the spike method (using 5 wt% zinc oxide). The amorphous content of

Eraring FA, Callide FA, Gladstone FA, GGBS A, and GGBS B was found to be 85.2%, 81.2%, 79.1%, 95.8%, and 100%, respectively. The crystalline phases in fly ash samples consisted of mullite ( $\text{Al}_{2.17}\text{O}_{4.89}\text{Si}_{0.78}$ ), quartz ( $\text{SiO}_2$ ), magnetite ( $\text{Fe}_3\text{O}_4$ ), and hematite ( $\text{Fe}_2\text{O}_3$ ) (in Callide and Gladstone FA). The crystalline phases in GGBS A were gypsum ( $\text{CaH}_4\text{O}_6\text{S}$ ) and hatrurite ( $\text{Ca}_3\text{O}_5\text{Si}$ ), with some traces of quartz, whereas GGBS B had some traces of quartz as the only crystalline phases.

The particle size distribution of the solid precursors (except GGBS (A)) was determined using the laser diffraction technique with a Malvern Mastersizer 2000 instrument (Fig. 1). The powders were dispersed in water and ultrasonication at 20 kHz was used before the analysis. As is shown, Callide FA and then Gladstone FA were the finest, followed by GGBS(B) and Eraring FA. A considerable volume of the Callide and Gladstone FA particles were about 5–6  $\mu\text{m}$  in diameter, as opposed to 30  $\mu\text{m}$  for the Eraring FA particles.

A mixture of sodium hydroxide (NaOH) solution and grade D sodium silicate solution were used as the activator. The sodium hydroxide white pellets with a purity of at least 98% were supplied by Ajax Finechem under the commercial name of UNIVAR A-302. The sodium hydroxide solution used was prepared by dissolving technical grade NaOH pellets in tap water. These pellets have a specific gravity of 2.1  $\text{g}/\text{cm}^3$  and a pH of approximately 14. Grade D sodium silicate, which was supplied by PQ Australia under the commercial name of Vistrol D-A53, has a chemical composition of  $\text{Na}_2\text{O} = 14.7\%$ ,  $\text{SiO}_2 = 29.4\%$  and  $\text{H}_2\text{O} = 55.9\%$  (by mass). The sodium silicate solution used was a thick adhesive liquid with a viscosity of 400 cps at 20 °C, with a specific gravity of 1.53  $\text{g}/\text{cm}^3$  and a pH of 12.9 (values provided by the supplier, PQ Australia). NaOH and sodium silicate solutions were mixed in proportions to form alkaline solutions at different concentrations and modulus ratios ( $M_s =$  molar ratio of  $\text{SiO}_2/\text{Na}_2\text{O}$ ), according to Table 2. After mixing, the alkaline activator was allowed to cool to ambient temperature and to equilibrate prior to preparation of the specimens.

Water vapor sorption isotherms of HCP (hardened cement paste), AAP1, and AAP2 mixes have been previously investigated by de Burgh & Foster [2] and de Burgh et al. [6], and their different aspects were further analysed here in comparison with the other mixes. To perform complementary tests required to study the material-specific properties; these mixes were refabricated and cured according to the conditions listed in Table 2.

Mix AAP3 was designed and manufactured solely to investigate the effect of a GGBS addition on the WVSI and the pore structure, in conjunction with AAP1 and AAP2 mixes. For this purpose, the water-to-binder ratio was kept at almost the same level as those of AAP1 and AAP2. The alkalinity of the activator, represented by the percentage of sodium oxide (by mass of FA + GGBS), was also not very different among AAP1, AAP2, and AAP3, which further facilitated drawing a conclusion as to the effect of having more calcium ions in the structure.

**Table 1**

Chemical compositions of Eraring, Callide, and Gladstone FA, and GGBS by X-ray fluorescence (XRF) analysis.

	General-purpose cement <sup>a</sup>	Eraring fly ash (wt%)	Callide fly ash (wt%)	Gladstone fly ash (wt%)	GGBS (A) (wt%)	GGBS (B) (wt%)
SiO <sub>2</sub>	19.1	65.9	50.1	47.9	32.4	35.0
Al <sub>2</sub> O <sub>3</sub>	5.8	22.10	26.72	25.7	13.1	14.1
Fe <sub>2</sub> O <sub>3</sub>	2.68	3.44	12.66	14.7	0.41	0.36
CaO	62.2	1.65	3.52	4.11	41.5	40.9
MgO	1.31	0.69	1.52	1.36	4.99	5.51
Na <sub>2</sub> O	0.02	0.59	0.60	0.81	0.30	0.29
K <sub>2</sub> O	0.53	1.81	0.60	0.67	0.33	0.30
TiO <sub>2</sub>	0.28	0.90	1.45	1.39	0.60	0.59
P <sub>2</sub> O <sub>5</sub>	NA	0.11	1.19	1.21	0.02	0.02
Mn <sub>3</sub> O <sub>4</sub>	NA	0.07	0.20	0.19	0.43	0.55
SO <sub>3</sub>	2.89	0.08	0.23	0.19	2.58	1.15
Loss on ignition (LOI)	4.5	1.36	0.62	0.69	2.17	NA

<sup>a</sup> From [2] (NA: not available).

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