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The effect of heat-curing on transport properties of low-calcium fly ash-based geopolymer concrete

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HIGHLIGHTS

- Thermal curing has significant effect on transport properties of low calcium FA-based GPC.
- Optimum thermal curing for FA-GPC is found to be oven curing at 75 °C for 18–24 h.
- Proper thermal curing reduces the porosity and sorptivity coefficient of FA-GPC.
- Proper thermal curing increases the compressive strength and electrical resistivity of FA-GPC.
- Thermal curing reduces the surface and bulk resistivity of Portland cement concrete.

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ABSTRACT

The aim of this study is to evaluate the transport properties of class F fly ash-based geopolymer concrete cured at various conditions. Twelve different heat curing regimes including three temperatures of 60, 75 and 90 °C and four curing durations of 8, 12, 18 and 24 h, as well as ambient curing, were imposed to the specimens. The material properties such as compressive strength, elastic modulus, ultrasonic pulse velocity, absorption, volume of permeable voids, pore size distribution and resistivity were evaluated against the reference Portland cement (OPC) concrete. Experiments showed that proper curing conditions such as curing at 75 °C for 18–24 h yield a geopolymer concrete with low volume of permeable voids and low sorptivity coefficient. The reduced volume of permeable voids and less continuous capillaries, attained by applying the proper curing regime, leads to an increased electrical resistivity and compressive strength of the low-calcium fly ash-based GPCs.

1. Introduction

The term 'geopolymer' was first introduced by Davidovits in 1979 to represent the inorganic polymers resulting from geochemistry. Geopolymers like other polymers are macromolecules with definite size and molecular weight [1]. For instance, the polymerization number of K-poly(sialate-siloxo) type geopolymer $(—Si—O—Al—O—Si—)_n$ is stated by Davidovits [2] to be in the range of $512 < n < 8000$. This yields a molecular weight of $60{,}000 < MW < 850{,}000$ or a particulate dimention of 5–15 nm (50–150 Å) as measured by Kriven et al. [3]. Geopolymer consists of nanoparticulates which are seperated by nanoporosity whose features are of the order of 3–10 nm [2]. It is the accumulation of these geopolymeric micells (individual nanoparticulates) that forms the geopolymer matrix.

The production of silico-aluminate based geopolymer requires source materials that are rich in silica and alumina content, such as fly ash, slag, metakaolin, etc. In the geopolymerization process of the fly ash-based geopolymer concrete (GPC), the reaction of aluminosilicate source and alkali polysilicates results in the formation of the geopolymer nanoparticulates with three-dimensional polymeric structure consisting of Si—O—Al—O bonds. Positive ions (e.g. Na^+, K^+) are present in the framework cavities to balance the negative charge of Al^{3+} in IV-fold coordination [4].

Although the geopolymer microstructure and properties have been investigated for about 50 years now, the study on fly ash (FA)-based geopolymer is very limited as this type of geopolymer concrete was first introduced in 1993 [5] and further studied by other researchers in late 1990 s [6]. A review of the literature indicates that most of the existing studies on the behaviour of fly ash based geopolymer concrete have focused on the mechanical properties and micro structural characteristic. There are very few studies on transport properties and durability characteristics of this

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class of geopolymer concrete and the long-term performance of this new material has not yet been extensively proven [7].

The pore structure of a material is important in determining the transport properties and, thus, the durability characteristics. Sorptivity provides an indication of the pore structure and their connectivity (capillary network) which is a major factor influencing aggressive ions penetration into the concrete when exposed to the severe environments (e.g. marine environment). Lower sorptivity delivers a higher resistance to concrete towards water absorption. A high sorptivity coefficient indicates the existence of a highly connected porous structure or low tortuosity of the pore network. McCarter et al. [8] reported that minimising the capillary sorptivity is essential in order to reduce the ingress of aggressive ions (e.g. chloride or sulfate) into concrete, which can cause serious damage.

The electrical resistivity of water saturated concrete is another measure of the concrete pore connectivity. Concrete resistivity is a volumetric property that indicates the ability to transport electrical charge through the material [9]. As it is graphically illustrated in Fig. 1, the concrete containing less interconnected pores with a more tortuous path will have a higher electrical resistivity as it would be more difficult for ions to pass through these kinds of pore network. Accordingly, the measurements of concrete electrical resistivity can provide a good indication of its permeability since both properties are mainly controlled by the tortuosity of the pore network. More compacted microstructures which tend to have more complex and refined pore networks result in a higher tortuosity and a lower permeability, whereas, less-dense microstructures resulting for instance from a higher water to binder ratio would have greater porosity, lower tortuosity and higher permeability [10]. The higher the porosity the lower the resistivity, due to higher volumetric fraction of pores [9]. Hence, the concrete electrical resistivity provides a reasonable indications of the pore connectivity and therefore of the concrete's resistance to penetrations of aggressive substances (e.g. salt water having cl^- or atmospheric CO_2) [9].

Although the pore network connectivity and the moisture content in the pore network is the major factor determining the electrical resistivity of concrete, however, the existence and motion of ions such as Na^+, K^+ and OH^- in the pore solution should also be considered. In concrete, electrolytic conduction is the main phenomenon of electricity transport since aggregates conductivity is very low [11]. Thus, the ions concentration in the pore network plays a significant role in concrete electrical resistivity measurements.

Moreover, the concrete resistivity can be used as a measure of corrosion rate of embedded steel reinforcement [12–18]. Generally, a low concrete resistivity is correlated to rapid chloride penetration and to a high corrosion rate [15]. Browne [12] previously studied the relationship between corrosion rate and electrical resistivity of concrete and found that the concrete resistivity must be kept above the range of 5–10 kΩ cm to prevent corrosion of reinforcement steel. In many particular cases, it has been shown that concretes with high resistivity have a low chloride diffusion coefficients [16,17,19]. Gonzalez et al. [13] suggested that the corrosion rate was negligible when concrete resistivity was higher than 10 kΩ cm.

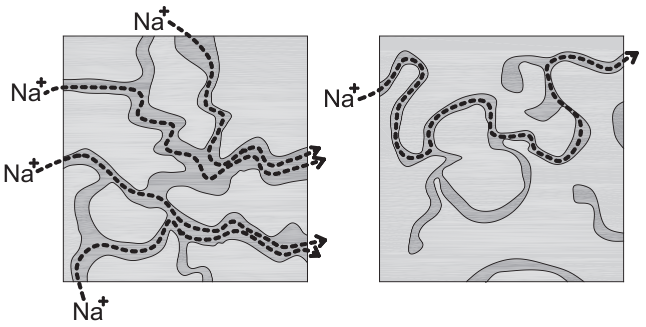

Fig. 1. Schematic representation of ion flow (Na^+ in here) through saturated concrete mixtures: (left) highly porous microstructure; (right) dense microstructure [10].

The geopolymerisation process depends on many parameters, one of which is the curing temperature. The curing temperature affects the degree of geopolymerisation and the formation of a dense molecular structure. This could affect the transport properties and durability of the final product. This study is aimed to experimentally investigate the influence of heat curing regime on mechanical and transport properties of low-calcium fly ash-based geopolymer concrete such as water absorption, porosity, pore size distribution, sorptivity and electrical resistivity. A Portland cement concrete (OPCC) mix has also been prepared as a reference point for the sake of comparison. The OPCC mix is a counterpart to the geopolymer concrete having the same amount of aggregate and binder.

2. Experimental procedures

2.1. Constituent materials

The binder used in this study to make geopolymers is combination of three constituent aluminosilicate source materials, namely, 70% class F fly ash, 20% ultra-fine fly ash and 10% slag (by mass). The low-calcium type fly ash branded as Blue Circle Fly Ash by Boral was sourced from Eraring Power Station in New South Wales, Australia. The ultra-fine fly ash branded as Kaolite High-Performance Ash (HPA) by Cement Australia was obtained from Callide Power Station in Queensland, Australia. Finally, ground granulated blast furnace slag (GGBFS) was supplied by Australian Steel Mill Services, Port Kembla, New South Wales, Australia.

Scanning electron microscopy (SEM) images of the raw materials are given in Fig. 2. All micrographs are taken under the same magnification. The chemical composition of the binders, as determined by X-ray fluorescence (XRF) analysis, is summarized in Fig. 3. The amorphous content of the raw materials was also measured through the X-ray diffraction (XRD) test using the spike method (5 wt.% Zinc oxide been used). The amorphous content of fly ash, Kaolite HPA and GGBFS was found to be 85.2%, 81.2% and 95.8%, respectively. The crystalline phases in fly ash samples consists of Mullite ($Al_{2.17}O_{4.89}Si_{0.78}$), Quartz ($SiO_2$), Magnetite ($Fe_3O_4$) and Hematite ($Fe_2O_3$) whereas the crystalline phases in slag were Gypsum (CaH_4O_6S) and Hatrurite (Ca_3O_5Si).

The particle size distribution of the binders was determined using the laser diffraction technique with a Malvern Mastersizer 2000 instrument, and the result is presented in Fig. 4.

The alkaline activator used is a mixture of aqueous solution of sodium hydroxide (NaOH) and commercially available sodium silicate (Na_2SiO_3) grade D in a mass ratio of 2.5:1 (Na_2SiO_3 to NaOH). The sodium hydroxide solution used was obtained by dissolving the technical grade NaOH pellets of 98% purity in tap water. These pellets have a molecular weight of 40, a specific gravity of 2.1 and a pH of approximately 14. The concentration of sodium hydroxide solution used is 12 molar (M) consisted of 480 grams (g) of NaOH pellets per litre of NaOH solution or 361 g of NaOH pellets per kg of NaOH solution. The sodium silicate solution has a chemical composition by mass of 14.7% Na_2O, 29.4% SiO_2 and 55.9% H_2O with a modulus ratio (M_s) of 2 ($M_s = SiO_2/Na_2O = 2$), specific gravity of 1.53 and a pH of 12.9 (values provided by supplier, PQ Australia). Both alkaline solutions were prepared and mixed together 24 h prior to usage.

Coarse aggregate used is crushed basalt sourced from Dunmore quarry in New South Wales, Australia, with a maximum nominal size of 10 mm, the specific gravity of 2.8 and water absorption of 1.6%. The fine aggregate used is Sydney sand with a specific gravity of 2.65 and water absorption of 3.5%. All aggregate was first kiln dried at 105 °C for a period of 48 h and then prepared to saturated surface dry (SSD) prior to batching. The drying procedure, which removes all the moisture content of the aggregate and also the organic particles in the sand, could help to adjust more accurately the mix water and consequently enhance the consistency of the GPC.

In order to make the conventional concrete for the purpose of comparison, ordinary Portland cement conforming to the requirements of ASTM Type I was used.

2.2. Mix compositions, preparation and curing

Two concrete mixes, one geopolymer and one Portland cement concrete, have been investigated in this study. The OPC concrete is a counterpart of the GPC having exactly the same amount of aggregate and binder. The GPC mix has 388 kg/m^3 of