Influence of curing on pore properties and strength of alkali activated mortars

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HIGHLIGHTS

- Wet/ dry curing provides optimum strength and pore properties of AACMs.
- AACM mortar has similar strength as control PC mortar under wet curing.
- AACM mortar strength is much higher than PC mortar under wet/dry and dry curing.
- AACM gel pore volume is lower, capillary volume is higher than PC mortar.
- Cumulative porosity (gel and capillary) in AACMs is lower than PC mortar.

ARTICLE INFO

ABSTRACT

The paper investigates the effect of wet/dry, wet and dry curing on the pore properties and strength of an alkali activated cementitious (AACM) mortar. The pore characteristics were determined from the cumulative and differential pore volume curves obtained by mercury intrusion porosimetry. AACM mortars possess a bimodal pore size distribution while the control PC mortar is unimodal. AACM mortars have a lower porosity, higher capillary pore volume, lower gel pore volume and lower critical and threshold pore diameters than the PC mortar which indicate greater durability potential of AACMs. Wet/dry curing is optimum for AACM mortars while wet curing is optimum for the PC mortar. Shrinkage and retarding admixtures improve the strength and pore structure of the AACMs.

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1. Introduction

The use of alkali activated cementitious materials (AACM) in place of Portland cement (PC) has been recognized to have great potential in construction applications. There is the need for a viable alternative to PC because of the high carbon footprint generated during its production with a huge energy demand, which is not sustainable in the future. The carbon footprint is significant because of the large volume of Portland cement PC consumed worldwide, which is ranked second after the volume of water [1]. To put this into perspective, for each tonne of cement produced an equivalent tonne of CO_2 is emitted into the atmosphere. This translates to the emission of 400 Kg of CO_2 per 1 m^3 of concrete production [2]. In addition, the cement industry consumes between 12 and 15% of the total industrial energy use [3]. The electric energy consumption for the burning process during cement production is estimated to be 65 kWh/tonne while the thermal energy consumption for cement grinding is 2.72 GJ/tonne [3]. Clearly, there is a dire need for reducing this carbon foot print and energy demand.

Limited knowledge is available in literature on the pore properties of AACMs and geopolymers [4]. However, established knowledge on the pore properties of conventional concrete [5] shows their critical importance in controlling the durability and strength of concrete. The pore properties are equally important for AACMs and other porous ceramic materials. The refinement of concrete pore structure improves its compressive strength, resistance to diffusion of deleterious substances such as chlorides and CO_2, which affect its durability [6]. These deleterious substances which cause corrosion of steel in concrete are transported through the concrete pores by capillary absorption, hydrostatic pressure and diffusion [7]. Diffusion of the ionic elements (Cl^- and Na^+) is mainly through the pores of the cement paste matrix and not through the interface between cement paste and aggregates [8]. The interface between

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Notations

AACM	alkali activated cementitious materials		r	pore radius
PC	Portland cement		y	mercury surface tension (=0.48 N/m)
GGBS	ground granulated blast-furnace slag		ϕ	mercury contact angle (=140°)
ITZ	interfacial transition zone			
p	absolute applied pressure			

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the cement paste and aggregates accounts for up to 50% of the total volume of pores in hardened concrete but these were found to be discontinuous and isolated from each other, thereby preventing the penetration of harmful elements through them [8].

The little understanding of the pore properties of AACM concrete provided in current literature suggests that the pore size distribution of AACMs is bimodal under all curing conditions [2,4]. The pores of AACMs are separated into two zones (>1 μm and <0.02 μm ranges) unlike a similar grade of PC matrix which is observed to be unimodal ranging between 0.01 μm and 0.1 μm [2,4]. Literature suggests that the gel pores in AACMs are formed during the polymerization of aluminosilicate gel during curing [9]. The extent of gel pores formed under different curing regimes is not understood. The gel pores are defined to be within the range of 0.005–0.01 μm based on PC concrete research [9]. The large capillary pores which are orders of magnitude bigger than gel pores and are within the range of 0.01–100 μm based on PC concrete research [9]. *Yue and Jiaqi* [10] showed an inverse relationship between the volume of gel and capillary pores as hydration progresses in PC concrete. During the hydration process of concrete, the volume of capillary pores decreases while the gel pores increases. This results in a lower cumulative pore volume in time because the comparatively large capillary pores is partially occupied by the binder gel. Ultimately, a denser microstructure evolves as the hydration progresses. The influence of curing on the pore properties of AACMs such as the gel pores, capillary pores, critical and threshold pore diameters are not defined in literature. These aspects of pore properties of AACMs are reported in this paper.

Pore refinement of PC concrete is achieved by high humidity (>80% R.H) curing which provides prolonged hydration of cement at low or high temperatures [5]. In the case of AACMs, earlier research has shown a need for high temperature curing at 50–80 °C, such as steam or dry heat, for optimum geopolymerization reaction [2,11]. More recent work uses ambient temperature (20 ± 2 °C), which is practical on construction site, for curing AACMs [12,13]. The optimum levels of relative humidity required for AACM curing are not established. However, results indicate that "dry" curing at low relative humidity (e.g. 60% R.H.) produces high strength for AACMs unlike PC concrete which has maximum strength under wet curing (100% R.H.) [14,15]. This can be beneficial for practical use of AACMs since insitu curing in construction does not provide idealized wet conditions. Practical site conditions represent a balance between, wet, wet/dry and dry conditions by preventing moisture loss at early age while concrete is exposed to ambient conditions of wetting and drying in the longer term. The practical curing conditions wet/dry, dry and wet at ambient temperature applicable in the field, were adopted in this investigation to determine the benefits of early age moisture available for curing on the strength and pore properties of AACMs.

A potassium-based activator used in AACMs reduces the mean pore diameter more than a sodium-based activator [4] while the total porosity of an alkali activated blast furnace slag (BFS) is reduced by the inclusion of a high modulus (more concentrated) activator and low water content in the mix [2]. The influence of chemical admixtures such as retarder and shrinkage reducing admixtures on the pore properties of AACMs is not known. This aspect together with the influence of activator dilution on the pore properties of AACM mortar is investigated.

Mercury intrusion porosimetry (MIP) is the common test method for investigating the microstructure of concrete. This is performed by applying mercury under high pressure through concrete pores. The method is based on the "non-wetting" property of mercury on the walls of the concrete pores. Mercury intrusion into the concrete matrix is suitable for pores within the range of 0.003–400 μm [16]. This method is used for analysing the accessible pores within the AACM and the control PC mortar samples in this investigation.

This paper is part of a comprehensive durability investigation of AACMs (mortar and concrete) being undertaken by the authors. It characterises the basic pore-properties of the material to provide a deeper understanding of the durability properties of reinforced AACM concrete.

2. Experimental programme

2.1. Materials and mixes

The control PC mortar had a composition of 1: 2.1 (by weight) of CEM 1 cement to CEN standard sand with a water/cement ratio of 0.47. The CEM 1 cement used is 42.5 Portland cement and it was supplied by Frank Key group, Sheffield, UK. The PC mortar was produced in accordance with BS EN 196-1:2016 [17]. The corresponding AACM 1 and 2 mortar mixes comprised of GGBS binder, sodium silicate and hydroxide based activator, fine aggregate of 80% particle size passing 1 mm sieve, liquid/binder ratio of 0.47 (alkali activator + water), a shrinkage reducing admixture SRA and retarder R42. The fresh AACM 1 and 2 mortar mixes were designed to achieve a flow of about 15 cm using the flow table test method [18]. The shrinkage reducing admixture SRA was added to reduce the shrinkage of AACMs while retarder R42 was added to increase the setting time. AACM 1 and 2 mixes were investigated to provide optimum properties of the fresh and hardened material for practical applications. However, AACM 1a and 2a mixes were also prepared with the same mix proportions but without admixtures to provide data for direct comparison with the PC mix which also did not contain admixtures. The mix compositions for AACM 1, 2, 1a, 2a and the control PC mortars are presented in Table 1. Table 2 shows the chemical composition of Portland cement (PC) and ground granulated blast-furnace slag (GGBS) binders used in the tests.

The average 28-day strength of the AACM and control PC mixes were designed to be fairly similar under wet curing, based on trial mixes. Wet curing is the standard method for quality testing of concrete [5]. The different curing methods adopted in this research are detailed in Section 2.2.

Sodium silicate activator of molarity 6.5 mol/L and modulus 2% was used for the AACM mixes to provide optimum viscosity for controlling workability and setting time [19]. The molarity of NaOH activator used was 4.8 mol/L. The combined molarity of the activators was at the lower end of values used by other researchers [20] for a similar activator combination. The activator for AACM 2 mixes was diluted with water at 7.76% (Table 1). The retarder R42 is made from a blend of high grade Polyhydroxycarboxylic acid derivatives while the shrinkage reducing admixtures (SRA) is made from Alkyl-ether. Each admixture contained less than 0.1% chloride ion and 3.5% sodium oxide.

2.2. Casting and curing

The GGBS binder and saturated surface dry fine aggregate were placed in a 12 L, 3 speed Hobart mixer. They were mixed for 30 s at the lowest speed (option-1) to avoid dispersing the powder into the atmosphere. The liquid component containing the activator and retarder R42 were slowly added to the mix. Mixing continued for 2 min until a uniform texture was produced. The shrinkage reducing admixture SRA was then slowly added while mixing continued. The mortar was further mixed for