



Electrically induced chloride ion transport in alkali activated slag concretes and the influence of microstructure

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ABSTRACT

Chloride ion transport in alkali silicate powder and liquid activated slag concretes, and the influence of the material microstructure are discussed. Increasing the Na₂O-to-slag ratio (n) results in a reduction in the rapid chloride permeability (RCP) and non-steady state migration (NSSM) coefficients (D_{NSSM}) of solid sodium silicate activated slag concretes. Increasing the SiO₂-to-Na₂O ratio (M_s) of the activator beneficially influences the transport parameters of liquid sodium silicate activated concretes. The chloride transport parameters are related to the reaction product microstructure and composition evaluated using mercury intrusion porosimetry (MIP) and Fourier Transform Infrared (FTIR) spectroscopy respectively. A reduction in the critical pore sizes reduces the transport coefficients to a larger extent than porosity reduction, which is responsible for the better performance of solid sodium silicate activated concretes. The total chloride concentration determined from the charge passed and the ionic transference numbers is used to predict an apparent non-steady state transport coefficient.

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1. Introduction

The development and application of cement-less binders produced through alkaline activation of aluminosilicate materials such as ground granulated blast furnace slag, fly ash, and metakaolin is gaining prominence, fuelled by the need for sustainable construction materials [1,2]. Alkali hydroxides or silicates are generally used to activate aluminosilicate precursors to produce these sustainable binders [3–6]. These binders have been reported to achieve mechanical properties that are comparable to or better than those of OPC systems [7,8]. Among alkali activated binder systems, activated ground granulated blast furnace slag is one among the most studied materials. This is attributable to the well-accepted use of slag as a partial cement replacement material in concretes, and the fact that alkaline activation of slag also produces C-(A)-S-H gel as the reaction product [9], albeit with a lower Ca/Si ratio [3,10] and differences in C-S-H structure depending on the activator type [11,12].

Several studies have reported the mechanical performance of alkali activated slags with respect to the source material and the activating agent chemistry, activation processes, mixture parameters, and curing conditions [6,8,10,13–17]. However it is also imperative to understand the durability characteristics of these materials in order to facilitate its use in aggressive environments [18]. Durability studies of alkali activated slag binders have been reported with respect to their high temperature resistance [19], carbonation behavior [13], and alkali-aggregate reactivity

[20]. This study is focused on understanding the chloride ion transport resistance of alkali activated slag mixtures. A number of standardized techniques are available to evaluate the chloride ion penetration resistance of concretes. In the diffusion tests, the concentration gradients drive the transport whereas in the accelerated tests, the transport is typically driven by the application of an electric field. Accelerated tests such as the rapid chloride permeability test (ASTM C 1012) and the non-steady state migration test (NT Build 492 [21]) are among the commonly used methods to determine the chloride transport parameters of concretes. In this paper, both these methods are used to evaluate the chloride ion transport resistance of alkali activated slag concretes.

The material microstructure exerts a significant influence on the moisture and ionic transport. The starting material and activator type, chemistry, and dosage are crucial in dictating the type and amount of reaction products formed [22–24] and the pore structure, which influences the transport performance of the material. In general, alkali silicate solutions of varying moduli are used as activating agents for slag. In order to alleviate the concerns related to handling of these caustic agents, efforts are needed to develop methodologies where alkali powder activators are used. Such variations in the starting materials change the reaction product formation and thus the microstructure [24]. For instance, along with C-(A)-S-H gel as the main hydration product, hydrotalcite and AFm phases are also reported [25,26] in alkali activated slags. The chloride transport is influenced by some of these reaction products, especially the ones containing Al because of its capacity to bind chlorides. In this paper, the influence of microstructure and reaction product formation in alkali silicate powder and liquid activated slag concretes on their

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chloride transport resistance is quantified while a companion paper [27] specifically deals with identification of binding effects.

2. Experimental program

2.1. Materials and mixtures

Ground granulated blast furnace slag (Type 100) conforming to ASTM C 989 was used as the binding material in all the mixtures. The chemical composition of slag is shown in Table 1. The activating agents used are sodium silicate (in powder or liquid form) and NaOH (as pellets). The powder sodium silicate has a SiO₂-to-Na₂O ratio (or the modulus, M_s) of 1.95, and the liquid sodium silicate (waterglass) has an M_s of 3.26. The waterglass used has a solid content of 40%. The M_s values reported here are mass-based; however it needs to be noted that the mass-based and molar M_s values are very comparable since the ratio of molecular masses of SiO₂ and Na₂O is 0.97. Analytic reagent-grade NaOH facilitated the changes in the M_s of the sodium silicate activator in both the cases. The sodium silicate powder has a median particle size of 25 μm and a pH of 12.6 (for a 50% w/v slurry in water). In this paper, solid (or powder) sodium silicate activator implies that the activator is a combination of powder sodium silicate of M_s 1.95 and requisite amounts of NaOH required to bring the M_s of the activator to desired levels (between 0.6 and 1.5), and liquid sodium silicate (or waterglass) implies that the activator is a combination of waterglass of M_s 3.26 and NaOH solution required to bring the activator M_s to the desired levels (between 1.0 and 2.0). For the powder activated mixtures, the M_s values had to be reduced to these lower levels in order to obtain 28 day compressive strengths in the range of 20–30 MPa, while for the waterglass activated mixtures M_s values in between 1.0 and 2.0 produced much higher 28 day compressive strengths. The effect of activator dissolution that causes such a result has been detailed in [24]. The n values (Na₂O-to-slag ratio) used were 0.05 and 0.15 for the powder sodium silicate activated mixtures and 0.05 for the waterglass activated mixtures.

Several mixtures were proportioned as part of this study. Concretes containing two different slag contents (300 kg/m³ and 400 kg/m³) were proportioned and activated using powder sodium silicate whereas only the 400 kg/m³ slag content mixtures were activated using waterglass. The liquid-to-powder ratio for the mixtures was maintained at 0.40. The powder consists of the slag, powder sodium silicate and NaOH for the mixtures activated using powder sodium silicate. The liquid consists of the added water and the amount of water in the sodium silicate solution when waterglass is used as the activator. River sand was used as the fine aggregate and pea gravel with a nominal maximum size of 9.5 mm was used as the coarse aggregate. The specimens made using these mixture proportions were used for compressive strength and chloride transport studies after specified durations of curing in a moist chamber, and are shown in Table 2. In addition, mortar mixtures were proportioned with a paste volume fraction of 50%, to be used for compressive strength studies. Activated slag pastes were used for microstructural and pore structural characterization studies. These paste mixtures were also subjected to chloride transport tests with an aim of extracting the depth-dependent chloride profiles. All the paste, mortar, and concrete specimens were removed from the molds after 24 h and moist cured in a humidity chamber (>97%RH) until the time of testing.

Table 1
Chemical composition of slag.

SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	MgO	Na ₂ O	K ₂ O	SO ₃	LOI
36.0%	10.5%	39.8%	0.67%	7.93%	0.27%	0.80%	2.1%	3.0%

2.2. Test methods

2.2.1. Strength and transport test methods

The compressive strengths of the concrete cylinders were measured in accordance with ASTM C 39 and those of mortar cubes in accordance with ASTM C 109. Rapid chloride permeability (RCP, ASTM C 1202) and non-steady state migration (NSSM, NT Build 492) tests were carried out on 50 mm thick discs cut from 200 mm long cylindrical specimens. For the RCP test, the specimens were conditioned by vacuum saturation, and enclosed in a cell flanked by reservoirs that contain 0.3 N NaOH solution in the anodic compartment and 3% NaCl solution in the cathodic compartment. A 60 V potential difference was applied between the electrodes placed on both faces of the specimen, for a duration of 6 h. The total charge passed (in coulombs) at the end of 6 h of testing is reported as the RCP value. For the NSSM test, the specimens were preconditioned by vacuum saturation with calcium hydroxide solution. The catholyte and anolyte solutions used were 2 N NaCl and 0.3 N NaOH respectively. An initial voltage of 30 V was applied, and the initial current was recorded. The applied voltage and test duration were chosen based on the initial current, which are tabulated in [21]. The test duration was maintained at 24 h and the voltage at 30 V for all the cases. After the test duration, the specimens were axially split and sprayed with a 0.1 M silver nitrate solution. The depth of chloride penetration was measured based on the precipitation of white silver chloride. The non-steady state migration coefficient (D_{nssm}) in m²/s is given as:

$$D_{\text{nssm}} = \frac{RT}{zFE} \frac{x_d - \alpha \sqrt{x_d}}{t} \quad (1)$$

$$\alpha = 2 \sqrt{\frac{RT}{zFE}} \operatorname{erf}^{-1} \left(1 - \frac{2c_d}{c_0} \right) \quad (2)$$

$E = (U - 2)/L$, where U is the absolute voltage (V), L is the specimen thickness in m, z is the valence of the chloride ion, F is the Faraday constant (96,500 C/mol), R is the molar gas constant (8.304 J/mol-K), T is the average value of initial and final temperatures in K, x_d is the average value of the penetration depth in m, t is the test duration in seconds, c_d is the chloride concentration at which silver nitrate changes to silver chloride (0.07 N), and c_0 is the chloride concentration of the catholyte solution (2 N). The value of c_d chosen is the one generally used for ordinary Portland cement concretes. However, the calculations for the migration coefficients were repeated with c_d values of 0.05 and 0.1, but this did not change the D_{nssm} values to any significant extent. Hence a c_d value of 0.07 itself is used in this study.

2.2.2. Microstructural and pore structural characterization

Fourier Transform Infrared (FTIR) spectra of the activated pastes were obtained using an ATI Mattson Genesis FTIR spectroscope with a single reflection attenuated total reflectance (ATR) diamond crystal attachment. The spectra of the reaction products after 28 days were obtained in the wavenumber range of 4000 to 400 cm⁻¹ at a resolution of 1 cm⁻¹. Mercury intrusion porosimetry (MIP) was performed on small samples of oven dried alkali activated pastes using a porosimeter that can generate a maximum pressure of 414 MPa and evaluate a minimum pore diameter of 0.003 μm. The test was performed in two steps – the low pressure step evacuates the gases, fills the sample holder with mercury, and carries out the test up to 345 kPa, and the high pressure step reaches pressures of up to 414 MPa. The contact angle and surface tension values used were 130° and 0.485 N/m respectively. Lower values of contact angle have also been used in the MIP study of cement pastes [28], but the present study utilizes the commonly adopted value of 130°.

2.2.3. Particle induced X-ray emission (PIXE)

Particle induced X-ray emission (PIXE) is an ion beam analysis based technique that relies on the spectrometry of the characteristic

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