



Application of X-ray microfluorescence for the determination of chloride diffusion coefficients in concrete chloride penetration experiments



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HIGHLIGHTS

- Chloride diffusion/migration methods explored in context of concrete made with new binders.
- Efficient and rapid protocol for X-ray microfluorescence based Cl-profiling presented.
- A pronounced matrix effect was observed for characteristic Cl K α X-ray production.

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ABSTRACT

The reliable estimation of the rate of chloride penetration in concrete is a major parameter in service life predictions. Electro-chemical test methods have the advantage of being fast while more long-term test methods can provide conditions that better approximate the in-service environment. This study investigates the performance of commonly used standard test methods NT Build 443 “Accelerated Chloride Penetration” and NT Build 492 “Chloride Migration Coefficient From Non-Steady-State Migration Experiments” as applied to three different types of binder systems: high Ca content (portland cement concrete), moderate Ca content (alkali activated slag concrete), and low Ca content (alkali activated fly ash and metakaolin concrete). Trends in terms of measured results for migration/diffusion coefficients were consistent across both tests approaches. In addition to these standard tests, a micro X-ray fluorescence (μ XRF) method was effectively used to determine chloride diffusion coefficients, but the development of multiple calibration curves were required to account for matrix effects across the variety of binder systems.

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1. Introduction

For concrete with embedded steel reinforcement, appropriate cover depth is required to impede access of chlorides and thereby provide protection against corrosion. However, proper design and sufficient curing are not always provided, and in some cases, chlorides (from seawater or de-icing salts) can penetrate into the concrete and initiate corrosion. Due to the importance of chloride penetration phenomena, many testing protocols have been developed to assess the performance of concrete against the ingress of chloride ions, as summarized in reviews by Angst et al. [1] and Galan and Glasser [2]. The applicability of these existing tests to concrete binder systems consisting of alkali-activated materials (AAMs) remains an active area of research, as summarized in the review of Bernal et al. [3].

Two widely used approaches developed for portland cement based concrete were explored here, but as applied to concrete made using AAM binders: NT Build 443 [4] involves the measurement of the chloride concentration profile after ponding in a NaCl solution for 35 days, and NT Build 492 [5] involves the measurement of the chloride penetration front after application of a constant current in a migration cell. In concept NT Build 443 is similar to ASTM C1543 and ASTM C1556 [6,7], while NT Build 492 is similar to ASTM C1202 [8]. The primary difference between NT Build 443 and the ASTM C1543 and ASTM C1556 tests is that a higher concentration ponding solution is utilized (16.5 wt% NaCl vs. 15 wt% and 3 wt% for the ASTM procedures respectively). The primary difference between NT Build 492 and ASTM C1202 is that ASTM C1202 measures only current (charge passed) without any measurement of the depth of chloride penetration.

Chloride migration tests such as NT Build 492 and ASTM C1202 are both rapid tests and commonly used, however, the application of voltage causes heating effects and the electrical conductivity

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may be affected by the pore solution chemistry, which may or may not lead to reliable predictions for the rate of chloride penetration in portland cement concrete containing supplementary cementitious materials (SCMs) or concrete made with AAMs [3,9]. Alternatively, NT Build 443, ASTM C1543, and ASTM 1556 are chloride profile methods where samples of powdered concrete are collected at intervals of increasing depth by drilling or grinding. In this research program, the profile grinding approach is used as a baseline method to compare to the more rapid electrochemical approach.

Wet-chemical methods, such as titration, are most often employed to determine the chloride concentrations in materials obtained through profile grinding. This involves digestion of the powders either in an acid or water, followed by filtration of the solutions, and finally the titration. Since titration can be very labor intensive, alternatives such as bulk X-ray fluorescence (XRF) [10,11], laser induced breakdown spectroscopy (LIBS) [12], and laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) [13] have also been employed. Cross-contamination during the profile grinding step is a major concern, so another common approach is to break or cut a cross-section through the concrete and to collect profile measurements directly from the exposed surface. Several approaches to measuring chlorine concentrations have been successfully demonstrated, including LIBS [14], LA-ICP-MS [15], X-ray energy dispersive spectrometry (EDS) and/or X-ray wavelength dispersive spectrometry (WDS) with either a scanning electron microscope (SEM) [16–18], a synchrotron radiation source [19–21] or by proton induced X-ray emission (PIXE) [22,23]. The relatively recent development of capillary optics to collimate X-ray beams to diameters of 100 μm or finer has also facilitated the use of micro X-ray fluorescence (μXRF) for chloride profiling [24–30]. μXRF instrumentation is non-destructive, as opposed to the LIBS, LA-ICP-MS, and PIXE approaches, and more suitable for routine analyses as compared to a synchrotron based approach. Although procedurally μXRF is very similar to EDS and WDS by SEM, sample preparation/polishing is less of a concern given the scale of the incoming X-ray flux compared to the much finer electron beam. The approach to μXRF profiling explored here employs spot analyses restricted to the paste matrix, in an effort to minimize the time required to obtain a useful profile.

2. Experimental

2.1. Materials

The extent of chloride ingress in four different types of concrete binding systems were investigated: portland cement, an alkali activated coal combustion fly ash (AAF), an alkali activated metakaolin

(AAK), and an alkali activated ground granulated blast furnace slag (AAS). The compositions of the general-use portland cement, the coal combustion fly ash, the metakaolin, and the two varieties of ground granulated blast furnace slags are listed in Table 1 as determined by XRF. Both the coarse aggregate (maximum size of 14 mm) and the fine aggregate consisted of rounded particles from a natural deposit that originated from carbonate-rich fluviually sorted glacial sediments generated by the Laurentide ice sheet. Sodium silicate solution (29.4 wt% SiO_2 , 14.7 wt% Na_2O , and 55.9 wt% H_2O) and sodium hydroxide pellets (99.1 wt% NaOH purity, 0.01% NaCl) were incorporated in the AAM mixtures.

2.2. Mixture designs and sample preparation

The AAM mixture designs were the outcome of a round-robin testing program by RILEM Technical Committee 247-DTA Durability Testing of Alkali-Activated Materials. Labs participating in the round-robin testing all used the same sources of fly ash, metakaolin, and slag precursor materials. However, for the research presented here, an alternative source of metakaolin was used, and an additional slag source (Slag B) was also tested.

Portland cement concrete mixtures were made with water to cement ratios (w/cm) of 0.45 (OPC45) and 0.48 (OPC48). The mix designs for all of the concrete mixtures are presented in Table 2. The portland cement concrete mixtures were produced and cured in lime-water in accordance with ASTM C192 [32]. For the AAM concrete mixtures, sodium hydroxide solutions (when utilized) were prepared one day in advance and allowed to equilibrate to room temperature. The AAM concrete mixing procedure consisted of one minute of dry mixing of the precursor material with room temperature oven-dried aggregate in a 100 L flat pan mixer. After the initial mixing of dry ingredients, the water, sodium silicate solution, sodium hydroxide solution, or sodium hydroxide pellets were added over the next two-minute period, followed by mixing for another four minutes. Concrete was poured in $\text{Ø}100 \times 200$ mm cylindrical plastic molds and stored in 99% relative humidity (RH) curing boxes. After 24 h samples were demolded and sealed in plastic bags, except for the AAF mixtures which were left for 48 h in the molds due to their slower rate of strength gain. Previous experiments with trial batches determined that the ASTM C1611 flow test [33] for self compacting concrete provided a better assessment of the workability of the AAF and AAK mixtures as compared to the traditional ASTM C143 slump cone test [34]. The exothermic dissolution of the solid NaOH pellets in the AAK mixtures resulted in a significant increase in temperature of the fresh concrete, yielding a warm, viscoelastic (non-Newtonian) mixture resistant to shear deformation. To facilitate mixing, a vibrator was inserted into the pan mixer during the four minute mixing period for the AAK mixtures. For the AAS and AAF mixtures, the

Table 1
Chemical compositions of the precursors by XRF and CSA A3000 designations [31].

Oxide	Wt%				
	CSA A3000 GU cement	CSA A3000 Type F Fly ash	CSA A3000 Type N meta-kaolin	CSA A3000 Type S Slag A	CSA A3000 Type S Slag B
SiO_2	19.4	48.59	52.4	35.7	37.18
Al_2O_3	5.1	21.71	45.5	11.2	7.16
CaO	61.6	7.32	0.02	43.9	38.44
Fe_2O_3	3.26	8.03	0.39	0.3	0.66
K_2O	1.1	2.11	0.11	0.38	0.42
MgO	2.6	2.40	0.03	6.5	10.1
Na_2O	0.28	1.06	0.21	0.21	1.06
P_2O_5	–	0.64	0.08	0.02	0.02
SO_3	3.37	0.84	–	0.44	2.72
TiO_2	0.25	0.95	1.39	0.51	0.4
Other	0.94	1.12	–	0.28	–
LOI	2.7	5.26	0.98	0.31	1.04

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