## ARTICLE IN PRESS

Cement and Concrete Research xxx (xxxx) xxx-xxx



Contents lists available at ScienceDirect

### Cement and Concrete Research



journal homepage: www.elsevier.com/locate/cemconres

## Wick action in mature mortars with binary cements containing slag or silica fume – The relation between chloride and moisture transport properties under non-saturated conditions

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#### ARTICLE INFO

Keywords: Transport properties (C) Durability (C) Chloride (D) Microstructure (B) Long-term performance (C)

#### ABSTRACT

Moisture and ionic transport under non-saturated condition is an important, but poorly understood transport phenomena particularly for mature systems containing supplementary cementitious materials. This paper investigates the moisture and chloride profiles of 3-year old mortars containing Portland cement (OPC), slag and silica fume (SF) after long-term (30–48 months) wick action exposure in 1.09 M NaCl solution. Moisture profiles were measured with <sup>1</sup>H NMR relaxometry and chloride profiles with microXRF. The measured profiles were discussed in relation to moisture dependent material properties such as chloride diffusion coefficients, moisture diffusion coefficients, and desorption isotherms. Results show that the combination of different cementitious materials, e.g. the cementitious binder, is the key factor affecting chloride penetration depth. The cementitious binder also strongly affects chloride diffusion coefficient, moisture diffusion coefficient and chloride binding properties, which are all important parameters for the prediction of chloride ingress.

#### 1. Introduction

Transport of moisture and ionic species are closely related to degradation of concrete structures [1]. These topics have been widely investigated under saturated conditions. But in many applications, the deterioration process occurs under unsaturated conditions, where the transport of ions and moisture are coupled. However, unsaturated transport is rarely studied experimentally, partly because of the complex moisture dependency and difficulties in decoupling ionic transport from moisture transport. Fundamental understanding of this is absolutely critical, for example in advancing service-life prediction models for structures in realistic exposure environments.

A method to study the interaction between moisture and ion transport is through wick action experiments [2]. Wick action is the transport of a solution (ionic solution or pure water) through a material, where one surface of the material is in direct contact with the solution and the opposite surface is exposed to a drying environment. If the relative humidity (RH) on the dry side is constant, the rate of absorption on the wet side will be similar to the rate of evaporation on the dry side when steady-state conditions are reached. If the experiment is performed with pure water, the moisture profile will hereafter be constant, and be dependent on the moisture diffusion coefficient and moisture sorption properties of the material.

Commonly in wick action experiments, the water contains ionic species which are transported with the moisture through the material. The ionic species will penetrate and precipitate either within the specimen, or at the surface of the specimen exposed to a drying environment, depending on the composition of the material and of the environmental conditions. In these cases, there is convective transport of ions with moisture transport, but also pure diffusive transport due to gradients in ionic concentration. Therefore, both the moisture diffusion coefficient and ion diffusion coefficient are important material properties for prediction of the ion profile through a structure [3]. The convective transport of ions with moisture transport is often regarded as the governing transport mechanism in cementitious materials, and pure ionic diffusion is assumed to be of minor importance [3-5]. This assumption has shown to be efficient for prediction of ion profiles for materials with a clear convective moisture transport and a moisture dependency in moisture transport [3]. However, the approach has limited abilities to predict the ion distribution for denser materials with

https://doi.org/10.1016/j.cemconres.2018.06.006

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Received 5 February 2018; Received in revised form 18 May 2018; Accepted 13 June 2018 0008-8846/@ 2018 Elsevier Ltd. All rights reserved.

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limited or no moisture dependency in moisture transport, such as cementitious materials at low water to binder ratio (w/b) or containing supplementary cementitious materials (SCMs) [6]. The interaction and binding of the ionic species with the solid phases in cementitious materials, and the RH on the dry side of the specimen, will also affect the ion profile [2].

Tuutti [7] made an early investigation of wick-action in cementitious materials. He discussed chloride concentration profiles in relation to w/b and drying condition for mortars with OPC and slag. It was concluded that ion transport in slag blends is less sensitive to w/b than mortars with OPC. Buenfeld et al. [2] made another early investigation of OPC mortars after 9 months wick action exposure. They included modeling of the obtained chloride profiles from measured material properties. However, the modeling was based on ion transport only with liquid moisture transport and does not take into account the diffusive ion transport and the moisture dependency of the chloride diffusion coefficient. This approach produced reasonable prediction of ion transport for OPC mortars with high w/b. But for denser materials with little or no liquid moisture transport, the diffusive ion transport and its moisture dependency need to be considered for accurate prediction of the ion profile. A more recent model [4] includes the diffusive transport of ionic species, but the ion diffusion coefficient was related to tortuosity of the liquid phase, which is problematic when modeling dense materials. In Baroghel-Bouny et al. [3], the measured ion profiles from wick action experiment by Francy [5] were predicted through a model that relates ion diffusion coefficient to the degree of saturation of the material. It was concluded that an accurate assessment of diffusive ion transport is crucial to achieve an accurate prediction of ion distribution in cases where liquid moisture transport is low. However, such data are rare. The need for more experimental research on unsaturated transport has been highlighted in a recent review [8], and in particular the effect of SCMs on transport properties.

In two recent publications by Olsson et al. [9] and Olsson et al. [6], the moisture dependency of ion diffusion and moisture transport properties for mortars with slag or silica fume (SF) were investigated. In this paper, measured moisture and chloride ion profiles after wick-action exposure of the same mortars are presented. The profiles are discussed and related to w/b, cementitious binder, drying conditions, and material transport properties such as ion diffusion coefficient, moisture diffusion coefficient and desorption isotherm of the mortars. The overall aim is to enhance the understanding of key parameters influencing non-saturated chloride transport under wick action in cementitious materials containing SCMs.

#### 2. Materials

In this study, four binders and two w/b were investigated. The investigated binders were ordinary Portland cement CEM I 42.5 N (OPC), OPC with 5% silica fume (SF), and OPC with 40% or 70% ground granulated blast furnace slag (slag), respectively (all by mass). These binders were used to produce mortars with siliceous sand (CEN-Standard Sand according to EN 196–1) of particle sizes 0-2 mm, at w/b 0.38 and 0.53. To get similar initial capillary volume in all systems, the exact w/b was calculated to obtain equivalent volume of paste in relation to the volume of water for all mortar mixes. This gave a slight variation in w/b due to differences in density between the OPC, SF and slag. A small amount of plasticizer ( $\sim 1 \text{ kg/m}^3$ ) was used in the w/b 0.38 mortars to improve workability. The composition of mortars and binders are given in Table 1 and Table 2, respectively.

The mortars were batched in a pan mixer and cast as blocks  $(600 \times 250 \times 250 \text{ mm}^3)$  in wood molds, and compacted with a vibrating rod. The blocks were demolded after 24 h and put into sealed containers with a limited amount of tap water for wet curing. The blocks were immersed in approximately 1 cm of water to limit the amount of water to minimize leaching. Cores of 94 mm diameter were drilled horizontally using a diamond corer through the blocks at the age

of 7 days. The cores were immediately placed back into their holes in the blocks for further wet curing. The reason for keeping the samples in their blocks was to limit direct exposure to water and therefore leaching. After 3 years of curing, the cores were cut into discs with thickness of 35 mm and again placed back into their blocks. The material used in this study was taken from the same blocks as those in previous studies [6, 9, 10], and was therefore expected to have similar properties. However, the difference in age between studies will affect the properties.

#### 3. Methods

#### 3.1. Wick-action exposure

In the wick-action experiment, disc specimens were exposed to 1.09 M (6% by mass) sodium chloride solution on one flat end of the disc, and to air at controlled relative humidity (RH) on the opposite end. Two RHs were used, 33% and 75%, which were generated by using saturated salt solutions of magnesium chloride (33% RH) and sodium chloride (75% RH) [11]. The air was circulated with a fan and the RH was regularly validated with RH sensors. The experiment was performed in sealed boxes at a temperature of 20 °C  $\pm$  0.5 °C, and with a carbon dioxide absorbent to avoid carbonation of the material.

The exposure to 1.09 M NaCl solution was carried out by mounting the flat end with a glass cup of similar diameter as the disc specimen, viz. 95 mm. The glass cups were filled with NaCl solution and the edge sealed with butyl tape to prevent side leakage. The cups were placed upside down in sealed boxes so that the NaCl solution was in contact with the top flat surface of the disc specimen. The specimens for wickaction exposure were taken from the blocks 1 month after cutting, i.e. at an age of approximately 3 years.

#### 3.2. Chloride profiles from microXRF

The specimens were exposed to wick-action for 30 months prior to ion profile determination. The weight of the cups was determined regularly to ensure that a steady-state flux was achieved during the exposure period. At the end of the exposure period, the glass cups were removed, and the specimens were split along the diameter with a wedge split. This method was considered to have the least risk of affecting the ion distribution on the analyzed surface. The two halves were assembled back together, wrapped in plastic sheets to prevent drying and stored for one to two weeks prior to micro X-ray fluorescence (microXRF) analysis. The effect of surface roughness is investigated in the work of Abdul Wahid [12]. This investigation, in combination with the fact that all samples were analyzed similarly, justifies comparison between profiles.

Chloride profiles were measured using an Orbis PC energy-dispersive microXRF system by Edax-Ametek. The microXRF is equipped with an 80 mm<sup>2</sup> Si(Li) detector with Be window for analysis of Na to U, and a motorised stage with 5  $\pm$  2 µm minimum step size. Analysis was carried out using a 2 mm spot size at 20 kV beam voltage, 30% dead time, 100 s live acquisition time and 25.6 µs time constant. More crucially, the analysis was carried out in an ambient environment (i.e. no vacuum) to prevent drying of the specimen that would otherwise influence the measured profiles. A 25 µm aluminium filter was employed to improve the detection of chlorine by removing overlapping rhodium spurious peaks.

In total 20 profiles were measured; one profile for each mortar and wick-action exposure condition (75% or 33% RH), i.e. 16 profiles, and a replicate analysis in 4 cases. The freshly split surface of each specimen was analyzed directly without any polishing or drying of the specimen to avoid re-distribution of chlorides. A spot size of 2 mm was chosen to ensure a substantial volume of analyzed material to obtain representative composition of the mortar. Analysis was carried out starting from the wet face exposed to NaCl, and then at every 2 mm

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