



Quantitative measurement of the influence of degree of saturation on ion penetration in cement paste by using X-ray imaging



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HIGHLIGHTS

- TXM is a fast imaging method to study in-situ ion penetration in non-saturated samples.
- Important insights about combined ion transport mechanisms have been observed by TXM.
- Sample with 40% DoS shows a 40x increase in P_c at 2 d over the saturated sample.
- A model was developed to estimate the penetration coefficient of non-saturated samples.
- Iodide shows a similar penetration performance to Cl in samples with different DoS.

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ABSTRACT

The external penetration of ions into concrete plays an important role in long-term durability of concrete structures. In addition, the degree of saturation (DoS) of the paste has a large impact on rate and mechanisms of fluid penetration into concrete. There is little knowledge of the dynamic process of fluid penetration into these materials because few experimental techniques are able to give quantitative spatial measurements of the fluid movement without damaging the sample. This paper uses laboratory transmission X-ray microscopy (TXM) to investigate the in-situ penetration of an iodide based tracer into cement paste with water-to-cement ratio of 0.40 conditioned at four different relative humidities (RH). This technique can non-destructively image the distribution of the dissolved ions at 8.8 μm resolution in minutes. In addition, micro X-ray fluorescence (μXRF) imaging and conventional gravimetric measurements were used to compare I and Cl penetration rate and validate the results from TXM. Mathematical models have been proposed to account for ion penetration in partially-saturated samples. The results show that samples that were ponded for two days after being conditioned in 35% and 70% RHs (DoS of 40.8% ($\pm 0.1\%$) and 68.8% ($\pm 0.5\%$) respectively) show a 40 \times and 10 \times increase respectively in the penetration coefficient over the saturated sample. Furthermore, important insights about combined ion transport mechanisms have been observed from concentration profiles by TXM.

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1. Introduction

Concrete structures face many threats to their service life. A number of durability problems are caused by external fluids penetrating into cement-based materials [1–4]. These fluids can contain potentially harmful ionic species, can lead to damage from corrosion, sulfate attack, alkali silica reaction, bulk freeze thaw, and salt scaling [5–6]. Therefore, the ion transport properties of cement-based materials play an important role in the durability performance of concrete structures [7–8].

Concrete that is partially saturated has a more rapid and more complicated method of ion transport [9–13]. For concrete with very low relative humidity (RH) or low degree of saturation (DoS), many pores are empty and these empty pores will draw in outside fluid from capillary action when wetted. However, more work is needed to understand how materials conditioned at different RH or different DoS impact this process.

The most widely used method to investigate fluid ingress into partially-saturated samples is through gravimetric measurements according to ASTM C 1585 and CEN EN 1925. This test method determines the one-dimensional rate of fluid penetration in cement-based materials by measuring the increase in the weight of a specimen as a function of time. However, this method only measures the total cumulative fluid uptake into the material and

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does not provide information about the spatial distribution of the fluid in the specimen [12,14–15]. This test makes assumptions about the geometry of the concrete pore network to estimate a depth of penetration [9–10,14]. Furthermore, this method does not account for the porosity, initial DoS of the sample, and the effect of the fluid viscosity and surface tension on penetration [11].

Recently, neutron and X-ray imaging techniques have been used to visualize fluid penetration in cement-based materials [8,12,14–17]. Both of these methods are based on the same principle of radiation attenuation when passing through matter. However, X-rays interact with the electron shell of the atom while neutrons interact with the nucleus. One drawback of neutron imaging is that it requires a high energy source that is typically only available at synchrotron beamlines. This reduces the availability of the method. Neutron imaging is also sensitive to hydrogen atoms and is not able to measure specific ions. This may or may not be important depending on the information needed.

Transmission X-ray microscopy (TXM) has been used to learn more about moisture movement in cement-based materials [7–8,12,16,18–19]. Previous studies using this method have tried to image water penetration into the paste and mortar samples [8,12,19]. Unfortunately, X-rays have a weak interaction with light atoms such as hydrogen. This limits the detection sensitivity of water in the method [8,20]. Furthermore, experiments that use TXM to investigate water ingress cannot provide spatial information about the ions and these publications have not been validated by other experimental techniques.

In this work, the moisture penetration is imaged with the aid of using potassium iodide (KI) as a tracer. Iodine is strongly X-ray attenuating because of its high atomic number. Furthermore, iodide and chloride ions are similar in size (iodide radius is 206 pm versus a chloride radius of 167 pm). This property makes iodide an excellent tracer for fluid transport study in concrete [21–24]. As the tracer penetrates into the cement paste it is possible to see the corresponding changes in X-ray absorption or gray value of the material in the radiograph. These images can be captured in less than a minute. This allows time resolved images to be taken and a rate of ion penetration can be calculated. Since this technique is non-destructive, the same sample can be monitored over time or in different conditions. The method requires minimal sample preparation and the technique can image at a spatial resolution of 200 nm to 20 μ m with laboratory equipment.

This work aims to use TXM to investigate in-situ iodide penetration in paste samples with water-to-cement ratio (w/c) of 0.40. These samples were conditioned in four different RHs to examine the effect of DoS on the ion penetration progress. The penetration of Cl and I in paste samples was also determined gravimetrically and with micro X-ray fluorescence (μ XRF) at these same conditions and then compared with the TXM results. μ XRF provides individual compositional maps which can be combined into a single map that simultaneously provides unique compositions and location [25–29]. Furthermore, μ XRF imaging was used to compare I and Cl penetration concentration profiles in paste samples.

2. Experimental methods

2.1. Mixture proportion and sample preparation

The cement is an ASTM C 150, Type I. The chemical composition of cement with bulk XRF is provided in Table 1. The paste mixture had a w/c of 0.40 and the mixture proportion is given in Table 2. Cylindrical micro vials with inside dimension of 9.5×46 mm were used to cast the samples. A lid was used to seal each vial. The vials were partially filled up to 30 ± 1 mm. Twelve samples were made for each RH condition (three samples to measure KI penetration

Table 1

Chemical composition of cement with bulk XRF.

Oxide content (%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	TiO ₂	SO ₃	L.O.I
Type I	20.40	5.03	2.95	62.89	2.08	0.35	0.35	0.28	3.05	2.09

Table 2

Cement paste mixture proportion.

Mixture	Water (g)	Cement (g)	w/c
P-40	340	850	0.40

Table 3

Porosity and DoS of paste samples at different RH.

RH	Porosity (%)	DoS (%)	D _i (%) [*]
≈0%	39.8 ± 0.6	0 ^{**}	0
35%	42.8 ± 0.6	40.8 ± 0.1	17.5 ± 0.3
70%	40.2 ± 0.1	68.8 ± 0.5	27.7 ± 0.4
100%	38.5 ± 0.2	100	38.5 ± 0.2

^{*} D_i is the initial DoS of the sample in volume fraction.

^{**} It has been assumed that an approximate DoS of 0% achieved in this condition.

with TXM, six samples for gravimetric measurement of KI and NaCl penetration, and three samples for measuring porosity and DoS). All samples were cured for 35 d in a sealed condition at 23 °C to remove the impacts of early hydration on the measurements. Based on previous publications by other researchers these samples would be expected to have a 70% degree of hydration [30–31].

After 35 d curing, the samples were demolded and conditioned at four different RHs (≈0%, 35%, 70%, and 100%). The 35% and 70% RH samples were stored in sealed containers above saturated MgCl₂ and KI salt solutions at 23 °C according to ASTM E 104 to reach mass equilibrium, respectively. An RH sensor was used for each container to monitor and verify the RH. The samples were placed in the container and their mass was measured daily until there was less than 0.03% change in mass loss over a 24 h period. It was then assumed that the moisture in the pore networks had sufficiently equilibrated with the environment. The 35% and 70% RH samples reached mass equilibrium after 50 d and 30 d, respectively.

The 100% RH samples were submerged in tap water inside a sealed container. The tap water was first boiled and allowed to cool to ambient temperature according to ASTM C 1202. These samples were then measured every 24 h until there was less than 0.03% mass change. This was reached after 5 d. The ≈0% RH samples were stored in a sealed container above saturated MgCl₂ solution (35% RH) at 23 °C for 40 d and then dried in an oven at 50 °C for 7 d until there was no measurable change in mass. For these samples they were assumed to be at approximately 0% RH and consequently a DoS of 0%. Because there may still be some moisture in the pores these samples are labeled as RH ≈0% and DoS ≈0%.

Since the 35% RH samples required 50 d to reach mass equilibrium, all samples were kept in a sealed container at 23 °C until 50 d in order to test all samples at the same age. The mass of the samples was monitored during the storage before testing and the mass change was found to be negligible.

2.2. Determining DoS

The samples for porosity and DoS were left in their respective environments and the 100% RH sample was kept in a sealed container within an environmental chamber at $97 \pm 1\%$ RH at 23 °C until they were tested. The porosity and DoS of the samples were

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