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Transport properties of concrete after drying-wetting regimes to elucidate the effects of moisture content, hysteresis and microcracking

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ABSTRACT

Drying and wetting induce a number of microstructural changes that could impact transport properties and durability of concrete structures, but their significance is not well-established. This research examines pastes, mortars and concretes with different w/b ratios, binders, aggregate sizes, curing and conditioning regimes. 50 mm thick samples were dried to equilibrium at either 105 °C, 50 °C/7% RH, 21 °C/33% RH or gentle stepwise at 21 °C/93% RH → 3% RH, and then rewetted stepwise by humidification at 21 °C/33% RH → 86% RH and full saturation to produce varying degrees of damage and moisture content. Oxygen diffusivity and permeability, electrical conductivity, microcracking, accessible and total porosity were measured at different conditioning stages over 3-year period to better understand the effects of shrinkage, hysteresis and drying-induced damage on transport properties. The effect of supplementary cementitious materials (GGBS, SF) and implications of drying-wetting on concrete durability are discussed.

1. Introduction

Durability of concrete and concrete structures is not only dependent on proper selection of ingredients, mix design and site execution (placement, compaction, curing), but also on its interactions with the exposure environment. All degradation processes affecting concrete with the exception of mechanical damage involve the transport of ions and/or fluids through its porous microstructure. Therefore, durability of concrete depends largely on its ability to resist penetration of water and other aggressive species (chlorides, CO_2, oxygen, sulphates), with the main transport mechanisms being diffusion, permeation, electro-migration (conductivity) and capillary sorption. Understanding how microstructure influences these transport processes is absolutely critical for the development of more durable and sustainable concretes, and for reliable service-life management of structures.

Most structures in service experience substantial drying-wetting and temperature cycles. Consequently, concrete structures are rarely completely dried or fully saturated or in a state of constant and uniform hygrothermal conditions. Exposure to natural environments induces a number of important effects. For example, drying leads to removal of water from pores and hydrates, and consequent volumetric shrinkage and microcracking [1–12]. On rewetting, the microstructure regains water and swells. However, some of the changes that occurred on first drying are non-reversible [13,14]. The moisture state (which can be represented as moisture content, degree of saturation or equilibrium relative humidity) influences the type and rate of transport processes [9,15–18]. It is also well-known that cyclic drying-wetting induces sorption hysteresis where the hygral state of cementitious materials exhibits a complex path (history) dependent behaviour [19–21].

All these effects may impact transport processes and durability, but their relative contribution or significance is not well-established. This is partly because they occur simultaneously and it is very challenging to decouple/isolate them to enable a systematic study. Furthermore, the associated experiments are extremely time-consuming due to the slow processes involved, particularly for a representative (thick) sample. Cracks smaller than 0.1 mm, i.e. “microcracks” are of particular interest because it is very difficult to control and eliminate them by proper structural design and use of steel reinforcement [22]. One particular aspect that remains unclear is the importance of microcracking and other drying-induced microstructural changes on transport properties when cementitious materials undergo drying-wetting cycles. Very few studies have examined this and where available, they involve mainly dried pastes and mortars rewetted to fully saturated condition and tested for water permeability [2,23–25]. Studies on how moisture hysteresis influences transport properties of concrete, in particular those concretes containing supplementary cementitious materials (SCMs), are also lacking [20,26,27]. Furthermore, most studies have been carried out on small (crushed) or thin samples (to accelerate moisture equilibrium), but thin samples can be dried with little microcracking [7,28] and are not representative of typical concrete

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cover in real structures.

This paper presents an attempt to isolate and to better understand some of these effects by subjecting a range of pastes, mortars and concrete samples to several drying regimes (105 °C, 50 °C/7% RH, 21 °C/33% RH or stepwise at 21 °C/93% RH → 3% RH) to induce varying degrees of damage and moisture content. The micro-cracked samples were then reconditioned at gradually increasing relative humidity (21 °C/33% RH → 86% RH) and to full saturation. Oxygen diffusivity and permeability, moisture content, degree of saturation and accessible porosity were measured at various stages of each conditioning regime. Electrical conductivity and total porosity were determined at saturation. Microcracking was characterised using fluorescence microscopy and image analysis. The entire programme involved > 1440 transport measurements over a 3-year period. The questions that we wish to answer include: a) What is the nature of drying-induced microcracking? b) To what extent microcracks influence transport properties after accounting for moisture effects? c) What are the effects of moisture hysteresis on transport properties? and d) How do systems containing SCMs behave compared to those containing CEM I?

2. Experimental

2.1. Materials

The binders used were ordinary Portland cement CEM I 32.5 R, CEM III/B 42.5 N containing 70% ground granulated blastfurnace slag (GGBS) and CEM I blended with 10% silica fume (SF). Oxide composition and loss on ignition of the CEM I, CEM III/B and SF are shown in Table 1. The mineral composition (Bogue) of CEM I was 52.7% C_3S, 19.3% C_2S, 10.6% C_3A, 7.4% C_4AF by mass. Specific gravities of the CEM I, CEM III/B and SF were 3.06, 2.90 and 2.30 respectively. The fineness of CEM I and CEM III/B were 290.5 m^2/kg and 463.5 m^2/kg respectively. CEM III was factory pre-blended while silica fume was added to CEM I during concrete batching.

A polycarboxylate-based superplasticiser at dosage of 0.5% wt. binder was used to improve the workability of the low water/binder (w/b 0.35) mixes. Thames Valley sand (< 5 mm) and crushed limestone (< 10 mm) were used as fine and coarse aggregates respectively. Limestone was selected as coarse aggregate because of its high stiffness and low shrinkage. This increases the aggregate/paste property contrast, the amount of aggregate restraint and therefore the potential for microcracking. Sieve analyses showed that the sand complied with BS 882:1992 medium grading while the limestone complied with BS EN 12620:2002 + A1 overall grading. The specific gravity at saturated surface dry condition (SSD) and 24-hour water absorption were 2.54 and 0.52% for the sand, 2.71 and 0.88% for the limestone.

2.2. Samples

Eight concrete, mortar and paste mixes were prepared according to the mix proportions shown in Table 2. Mix proportioning was designed by absolute volume. The free water/binder (w/b) ratio was either 0.35 or 0.50, obtained by correcting batch water for aggregate absorption and water from the superplasticizer. Total aggregate volume fraction for concrete mixes was fixed at 68% to enable meaningful comparison between concretes. However, the mortars contained slightly lower aggregate volume fractions because of the difficulty in compacting mortars with high sand content, in particular for the lower w/b mix. It was important to ensure that the mixes were well compacted, otherwise excess air voids would be another variable influencing mass transport properties.

128 disc samples of 100 mm diameter and 50 mm thickness were prepared and tested. The sample thickness was chosen as representative of the cover to steel reinforcement in concrete structures. Batching was carried out by dry mixing cement and aggregates for 30 s in a pan mixer, and then adding water (with superplasticizer if required) and mixed for a further 3 min. Samples were cast in steel moulds and compacted in two layers on a vibrating table until no significant release of air bubbles was observed. All mixes produced were easily compacted and showed no evidence of bleeding or segregation. Inevitably, the microstructure of the cast and trowelled surfaces will be different to that of the bulk material due to wall/packing effects, as would be the case in real structures. Given the thickness of samples tested, the effects of surface heterogeneity were probably small and insignificant and no attempt was made to isolate these.

2.3. Curing and conditioning

The compacted samples were covered with wet hessian fabric and plastic sheet at room temperature for the first 24 h to prevent drying. Afterwards, samples were demoulded and sealed-cured at room temperature to ages of 3 and 90 days. Sealed curing was achieved by wrapping each sample with at least 6 layers of cling film and sealing in polythene bags. Periodic weighing of the sealed samples found no significant weight change. The estimated degree of hydration using the backscattered electron microscopy method for similar CEM I systems after sealed curing are 0.62 (3 days) and 0.84 (90 days) at w/b 0.5, and 0.63 (3 days) and 0.72 (90 days) at w/c 0.35 [29]. After curing, samples were unwrapped and subjected to four conditioning regimes to produce samples with varying degrees of microcracking and moisture content. Each regime consists of several drying and wetting stages as summarised in Fig. 1. Prior to conditioning, the curved side of each disc was sealed with two layers of waterproof tape to induce unidirectional drying. This was deemed to be a more realistic approximation to the way in which structures dry or wet in service.

Conditioning regime A was the least severe, involving gradual stepwise drying at 21 °C from 93% RH to 86% RH, 76% RH, 66% RH, 55% RH, 33% RH and 3% RH. Regimes B, C and D were more severe and consisting of an initial drying stage at 21 °C/33% RH, 50 °C/7% RH and 105 °C respectively. These produce samples with varying degrees of microcracking and moisture content. Subsequently, the dried samples were rewetted by humidification at gradual step-wise increase of relative humidity from 33% RH to 55% RH, 66% RH, 76% RH and 86% RH at 21 °C, and finally full vacuum saturation. Saturated salt solutions were used to produce the required RH at 21 °C. These were KNO_3 (93% RH), KCl (86% RH), NaCl (76% RH), $NaNO_3$ (66% RH), Mg $(NO_3)_2$ (55% RH) and $MgCl_2$ (33% RH) [30,31]. Silica gel was used to achieve 3% RH. Conditioning was carried out in either an enclosed box in temperature controlled lab (± 1 °C) or in an oven, both contained fans to circulate air and soda lime to avoid carbonation.

Samples were frequently weighed and tested for transport properties when mass equilibrium at each drying or wetting stage was

Table 1
Oxide composition and loss on ignition of the binders used.

Binder	Oxide composition (%)									Loss on ignition (%)
	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	Na_2O	K_2O	SO_3	Cl-	
CEM I, 32.5R	63.4	20.6	5.6	2.4	1.6	0.2	0.7	2.9	< 0.1	2.1
CEM III/B 42.5N (contains 70% GGBS)	48.0	29.2	8.9	1.2	4.8	0.2	0.6	2.6	< 0.1	1.4
Silica fume	0.15	98.70	0.31	0.02	0.04	0.09	0.30	–	–	0.47