



Link between microstructure and tritiated water diffusivity in mortars: Impact of aggregates



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ABSTRACT

The present study aims to investigate the influence of microstructure, particularly the effect of aggregates content and interfacial transition zone (ITZ), on tritiated water (HTO) diffusivity in mortars.

To this end, three different series of mortars were prepared and HTO diffusion tests were conducted. Variables are water-to-cement ratio, sand volume fractions, and particle size distribution. In parallel, the microstructure of these materials was characterized by water porosimetry, mercury porosimetry, and by backscattered electron microscopy associated to images analysis.

It was observed that at low sand content (0%–50%), diffusion properties of mortars are dominated by aggregates dilution effect. Beyond 50% of standard sand, other effects related to the large number of sand grains appear, such as air voids and porous areas mainly due to the difficulty of obtaining well-compacted materials.

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1. Introduction

Concrete durability continues to be a subject of considerable interest, especially with the use of cement-based materials on structures increasingly demanding on term of sustainability. Diffusion is considered one of the main transport phenomena that cause migration of aggressive solutes and radionuclides in a porous media according to most studies [1–6]. In order to enable more effective prediction of structures service life, the understanding of the link between microstructure and transport properties in cement-based materials needs to be enhanced.

Unlike hardened cement paste (hcp), mortars and concretes contain aggregates, which may have an impact on transport properties. The so-called interfacial transition zones (ITZs) is an area of cement paste adjacent to sand or gravel grain where the microstructure is disturbed by the presence of this aggregate. Such zones have often been claimed to be a region of weakness because it contains, on average, more pores and fewer cement particle than the bulk. ITZ average detectable porosity has been recorded to 30%. In addition, micro-cracks often initiate and propagate predominantly in the ITZ [7]. This well-known behavior leads to the common view of the ITZ as the weak link in mortars and concrete. Consequently, it is often assumed that ITZ has also an impact on transport properties by facilitating the penetration of deleterious ions into concrete. Some previous studies suggested that the aggregate and ITZ behave as a “hard core/soft shell” system where aggregates were represented by hard spheres surrounded by a constant thickness soft shell representing ITZ [8–14]. That is why it is expected that

adjacent ITZs overlap and form a percolating cluster at higher aggregates content, which may significantly affect bulk transport properties. However, available experimental investigations on the effect of aggregates content and interfacial transition zones on mass transport properties did not always follow this expectation and seems to be ambiguous. Some studies found that diffusivity decreased with increasing sand content suggesting that tortuosity induced by aggregates have stronger influence than the interconnection of ITZs [15–22]. Other studies suggested otherwise and showed that the bulk transport properties of concretes and mortars increase with adding aggregates or observed that above a certain sand content, ranging from 35% to 60%, ITZs become connected and thus create channel for easy diffusion [23–26].

In general, available studies on the effect of aggregates content and ITZs do not provide a clear answer about a deleterious impact to overall mass transport. Some other studies questioned the general existence of the ITZ in concrete [27,28]. They found, based on images analysis and SEM observations, that the higher content of detectable pores is within only some local areas of bulk adjacent to aggregate surfaces and that the overall average of the incremental pore space detected seems to be small. They showed also that the extra pore space originally associated to wall effect in ordinary concrete is mostly filled up by deposits of calcium hydroxide and by deposits of C–S–H passing through solution, leading to modest residual average pore content within the ITZ.

The conflicting results that can be found in literature exhibit the need to enhance understanding of microstructure effect, especially aggregates content and ITZs on concrete transport properties. Moreover, available experimental investigations were carried out with different techniques (diffusion cells, electric field, penetration profiles) and different diffusing elements (chloride, oxygen, carbon dioxide, and HTO)

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in different water saturation degree. The physico-chemical interaction of diffusing elements with cement matrix seems also not yet obvious and must be taken into account. It was suggested that HTO, the liquid form of tritium, has negligible interaction with cement paste hydrates [1,18], which allows the measurement of effective transport parameters of the material. Its diffusion is purely molecular and Fick's law can then be applied. However, only a few studies dealt with HTO within mortars and concretes.

In this context, the present study is undertaken to enhance the understanding of the links between microstructure and HTO diffusivity in saturated mortars. The effect of aggregates via ITZ on transport properties and materials durability is studied.

To this end, several series of HTO diffusion tests were conducted on mortars with various water-to-cement (w/c) ratio, sand volume fractions, and particle size. Materials microstructure was also characterized by water porosimetry, mercury porosimetry, and by backscattered electron microscopy associated to images analysis.

2. Materials and methods

Three different series of mixtures were prepared. Series S1 is mortars with w/c ratio varying between 0.3 and 0.5 at the same sand content (50%). In series S2, five different sand volume fractions were tested (0%–60%) at 0.4 w/c. S3 included three formulations of different grain size: fine sand (0.125/0.3 mm) labeled SF, standardized (EN 196-9) sand (0.08/2 mm) labeled SN, and a coarse siliceous sand (0.63/2 mm) labeled SG. Grain size distributions (Table 1) were defined so that a great difference on calculated ITZ fraction volume exist between the three formulations.

Natural siliceous sand (98.5% silica) with specific gravity of 2.63 and 24 h-absorption of 0.4% was used for all formulations. S1 and S2 mortars were cast with standardized sand (SN). S3 mortars were prepared at w/c = 0.4 and with 50% of sand. Regarding our tests time scale, there is no possibility of alkali silica reaction with the sand used.

Mortars were prepared by mixing Portland cement (European grade CEM I 52, 5 N CE PM-ES-CP2 NF) with the required quantity of tap water in a mechanical mixer. The cement oxide composition is CaO = 66.6%, SiO₂ = 22.4%, Al₂O₃ = 2.96%, Fe₂O₃ = 2.33%, SO₃ = 2.13%, MgO = 0.95%, Na₂O = 0.1%, K₂O = 0.15%. Mixtures preparation was based on the standard of normalized mortar fabrication procedure NF EN 196-1. The mixing time of the components was 240 s for all formulations.

At low w/c ratio (w/c = 0.3 and w/c = 0.35) and at high amount of sand grains (S2_60% and S3_SF), a modified polycarboxylic ether polymers-based superplasticizer admixture was used in order to improve workability. Mixture proportions are given in Table 2. The amount of mixing water, sand volume fraction, and grain size were varied to prepare specimens with different porosity and pores distributions.

2.1. Samples preparation and curing

Cylindrical samples (70 × 11 cm) were cast and mortars with high sand contents and/or low w/c ratio were compacted in two layers using a vibrating table in order to evacuate air bubbles. The intensity and duration of vibration was adjusted according to the workability of the mix throughout preventing segregation. Some samples with high

Table 2

Mixture proportions of mortar samples (SN for standardized sand, SF for fine sand, and SG for Coarse Sand).

Series	Mixture ID	Water/Cement	Sand volume fraction	Particle size	Glenium27 (% wt. Cement)	
S1	S1_0,3	0.3	50%	SN	0.50%	
	S1_0,35	0.35	50%	SN	0.50%	
	S1_0,4	0.4	50%	SN	–	
	S1_0,5	0.5	50%	SN	–	
	S2	S2_0%	0.4	0	SN	–
S2	S2_10%	0.4	10%	SN	–	
	S2_30%	0.4	30%	SN	–	
	S2_50%	0.4	50%	SN	–	
	S2_60%	0.4	60%	SN	0.50%	
	S3_SF	0.4	50%	SF	0.50%	
	S3	S3_SN	0.4	50%	SN	–
	S3_SG	0.4	50%	SG	–	

sand fraction and fine sand was difficult to compact even with the use of superplasticizer and vibrating table.

The molds with fresh mortars were sealed with plugs to avoid evaporation. Samples are demolded after 24 h and immersed in a saturated lime solution for 3 months period at 20 ± 1 °C. After the curing period, the cylindrical samples (70 × 11 cm) are sectioned from the center to produce three 6 mm thick discs. Sectioning was done using diamond abrasive cutter at a low feed rate of 5 mm/s just before transport experiments in order to avoid carbonation and drying. Care was taken to select specimens that are free of apparent defects on either surface.

2.2. Porosities measurements

2.2.1. Free water porosity and bulk density measurements

Oven drying at 105 °C has remained, for long time, the most widely used technique for porosity measurement. However, some studies suggested that, at this temperature, pore structure can be altered. In fact, some hydrates like ettringite and CSH lost significant amount of non-evaporable water and therefore water porosity is overestimated [30].

In this study, the samples were dried in an oven at 60 °C until constant weight was reached. Duplicate samples were tested for each formulation and the mean value is reported as result. Samples were not vacuum-saturated before drying. Water porosity θ and bulk density ρ can be calculated as follows:

$$\theta = \frac{M_s - M_d}{M_s - M_w} \quad (1)$$

$$\rho = \frac{M_d}{M_s - M_w} \quad (2)$$

where M_s is the sample water saturated mass (kg), M_d is the sample dried mass (kg), M_w is the sample mass when immersed in water.

2.2.2. Mercury intrusion porosimetry (MIP)

Cubes of mortars measuring 10 ± 1 mm were cut from the center portion of cylindrical samples and used for MIP tests. These cubes were first frozen (–196 °C) by immersion in liquid nitrogen. After freezing, samples were introduced for 7 days in a freeze-dryer in

Table 1

Particle size distribution of the used siliceous sand.

	Percentage retained at sieve size (in mm)												
	0.08	0.125	0.16	0.2	0.25	0.315	0.5	0.63	0.8	1	1.25	1.6	2
Standardized sand (%)	99	–	87	–	–	–	67	–	–	33	–	7	0
Fine sand (%)	–	96.7	86.1	63.7	22	2.3	–	–	–	–	–	–	–
Coarse sand (%)	–	–	–	–	–	–	–	98.4	84.2	54.6	29.8	15.3	3.1

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