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Moisture characterization of cementitious material properties: Assessment of water vapor sorption isotherm and permeability variation with ages

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HIGHLIGHTS

- Comparative study on various methods to stopping cement hydration was undertaken.
- The cement hydration was stopped before each test.
- Sorption isotherms of cementitious materials were evaluated according to their age.
- Water vapor permeability was experimentally measured according to the material age.
- The saturation zone (RH > 90%) was investigated.

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ABSTRACT

The water vapor adsorption desorption isotherm and the water vapor permeability of cementitious materials were experimentally evaluated in this study according to the evolving nature of the microstructure of this type of material due to their hardening over time (7 days and 28 days). For getting a constant microstructure during measurements, it was necessary to stop hydration. The cement arresting hydration technique was chosen based on a comparative study on various methods known in literature. Results show that the suitable method is immersion under air vacuum in acetone bath for 7 days, followed by drying in an air vacuum for 3 days (without acetone). Using adsorption and desorption isotherms allowed identifying the moisture storage capacity. This parameter varies according to the relative humidity and material age. Moreover, the range where the relative humidity values are important (saturation zone RH > 90%) was investigated by means of retention water measurements using the membrane extraction method. The results obtained show an important gap of water content from 90% of RH to saturation step. Consequently, the water vapor permeability tests highlight the evolution of this coefficient according to material age and its formulation.

1. Introduction

The coupled heat and moisture transfer in porous media has, recently, been a growing interest by the scientific community. Several macroscopic models of transfers were developed to predict the hygrothermal behavior of the porous materials [1–6]. The accuracy of these models is largely related to the quality of their input data consisting on the material properties obtained by experimental characterization. Indeed, in order to optimize the prediction uncertainties, an intensive experimental study should be initiated. Regarding the input data, two important parameters were distinguished; the moisture storage capacity and the water vapor permeability.

In this work, cementitious materials are particularly studied. The moisture properties of this kind of materials are directly related to their microstructure (porosity, tortuosity…) and their constituents. They depend, consequently, on their age. In order to identify experimentally these parameters for each age, the ongoing material hydration should be stopped. Because of the slowness of experimental test, the studied samples continue to hydrate and the materials properties will consequently change. The stopping hydration of cement consists on evacuating the remaining water in the samples. There are several techniques to stop hydration in the literature [7]. Most of them, if not all, are reported to be detrimental to composition or microstructural arrangement. What

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would be the most appropriate technique for our study case? In order to give an appropriate response to this problematic, a number of cement hydration stopping techniques were examined to find out the adopted method that affect slightly the microstructure or the cement paste and mortar hydration degree.

Konecny and Naqvi [8] found that compared to oven drying and freeze drying, solvent replacement with isopropanol produced samples where the fine pores were less damaged. Furthermore, they found that the pores of hardened cement pastes were damaged by freeze drying. In another study performed by Feldman and Beaudoin [9], the results showed that methanol and isopropanol generates less stress on the microstructure of the hardened cement pastes. Zhang and Glasser [10] showed that high vacuum drying increases the pore volume of hardened cement paste. Gallé [11] found that vacuum drying samples of hardened cement paste produced stresses and microcracks. Collier et al. [7] concluded that none of the arresting cement hydration methods studied in their work appeared to cause any noticed difference to the composition and microstructure of the hardened composite cement pastes studied. In this work, three techniques of stopping cement hydration are studied (see Section 2.1).

After having stopped the cement hydration at 7 days and 28 days age, the samples was analyzed in order to obtain the sorption desorption isotherm and the water vapor permeability according to the age. A porous building material's ability to store moisture can be experimentally determined by sorption and suction isotherms and mathematically described as the equations of state. These isotherms together are called the moisture retention curves. They are divided into three regimes. Fig. 1 shows the three regimes of moisture storage: the sorption or hygroscopic regime (Region A–C), the capillary regime (Region D) and the supersaturated regime (Region E). Based on the Fick's law where saturating concentration at the interfaces and C at infinity, allows admitting that the moisture content varies primarily with relative humidity, not with absolute humidity [5].

A part of this work is dedicated to the study of the capillary regime (zone D). Fig. 1 shows that the slope of the curves becomes steeper as the relative humidity (RH) is approaching 100%. At RH-values close to this limit, it is difficult to determine the corresponding moisture content accurately. Therefore, the sorption isotherms are considered valid for the descriptions of moisture retention up to the maximum hygroscopic moisture content which, in most definitions, corresponds to RH = 98% [12–14].

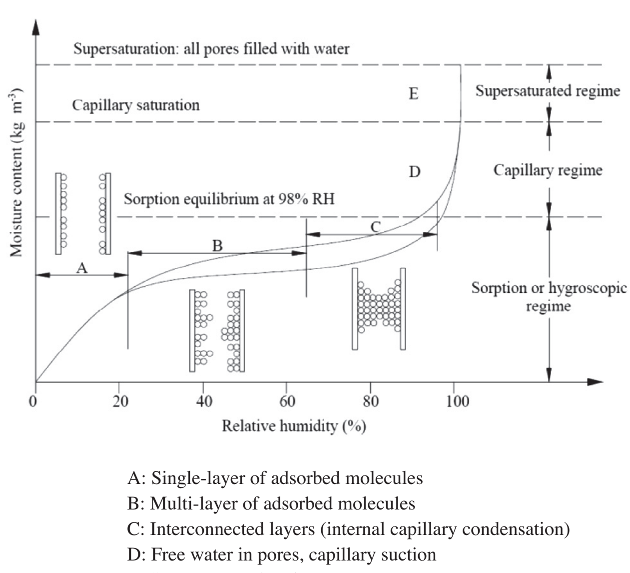

Fig. 1. Schematic diagram of the moisture storage according to a hygroscopic capillary-active building material.

The moisture storage capacity C_m is obtained from the slope of the sorption desorption isotherm. It represents an input parameter of coupled heat, air and moisture transfer models. The sorption desorption curve permits to characterize the porous material at the scale of pores concerning the water activity. The particular porous structures of cementitious materials determine their sorption behavior. A cementitious material is characterized by a microstructure change during hydration and drying [15–17]. Also, Espinosa et al. [18] concluded that the hygroscopic water content is directly proportional to the amount of hydrated hardened cement paste. This evolution of the microstructure affects the hydric behavior of the material [15,19]. In order to identify the influence of hydration on the moisture storage capacity of the material, the sorption desorption isotherm as well as the water vapor permeability were measured according to the material age; 7 and 28 days. Espinosa et al. [18] showed by Thermo-gravimetric analyses that the hydration degree increases during sorption desorption measurements. Because of this observation, the cement hydration must be stopped before each test. In addition, the water content of the cementitious material is greatly related to the amount of hydrates it contains. These hydrates are formed in the intergranular pores which the volume is determined by the amount of water contained initially. The capillary pores resulting from the intergranular spaces have a size which decreases with the water to cement ratio W/C [16]. Therefore, in the case of this study, tests of sorption desorption isotherms on cement pastes with different W/C ratios (0.3; 0.35 and 0.4) were carried out.

Trechsel [20] showed that the water vapor diffusion coefficient does not only depend on the nature of the material, but also on the vapor pressure applied to the sample. Indeed, the vapor permeability of the non-hygroscopic material is slightly affected by the relative humidity contrary to the hygroscopic materials [21]. Furthermore, the water vapor permeability changes with temperature. This sensitivity has been widely discussed in literature [22]. In this work, it was showed that in addition to all these parameters taken into account when measuring the water vapor permeability using the cup method, it is also important to define the age of the cementitious materials. The cup method is a well-adopted method for determination of water vapor diffusion coefficients for building materials.

2. Materials composition and experiments

Four types of materials have been studied. Two cement pastes with Three water to cement ratios (W/C) = 0.3; 0.35 and 0.4; and two mortars with W/C = 0.55 and Sand to Cement ratio (S/C) = 3. The first material of each formulation (paste 1, mortar 1) is based on Portland-cement CEM I 52.5 N with 95% part of clinker, and the second one with substituting a part of cement (30%) with blast furnace slag (paste L30, mortar L30) which characteristics are reported in Table 1. For each material, prismatic specimens 4 × 4 × 16 cm were cast according to the EN 197 and 998 European standards.

2.1. Hydration stopping techniques

To choose the relevant technique to stop hydration for this investigation, three techniques available in the literature were tested. These techniques are [7]:

Table 1
Physical properties of cement and blast furnace slag.

Cement	CEM-I 52.5N	Blast furnace slag	ORCEM
Clinker (%)	95	Initial setting time (min)	180
Initial setting time (min)	155	Specific mass (g/cm^3)	2.89
Specific mass (g/cm^3)	3.11	Specific surface (cm^2/g)	4500
Specific surface (cm^2/g)	3400		