



Importance of coil-overlapping for the effectiveness of hydroxypropylguars as water retention agent in cement-based mortars

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ABSTRACT

This paper is devoted to the role of hydroxypropylguars (HPG) on water retention (WR) capacities of cement-based mortars. WR and pore solution viscosity of mortars formulated with different dosage and kind of HPG were investigated. The results allowed us to rule-out that WR is only governed by pore solution viscosity. A sharp change in slope was observed on the curve WR vs. polymer dosage. This was attributed to the threshold dosage corresponding to the network formation of hydrocolloidal associated HPG molecules in pore solution. Below this dosage, no effect of HPG on WR was obtained. Above this dosage, HPG aggregates may stop the water flow by plugging the porous network of a thin HPG-enriched filter cake at the interface mortar-substrate. This study showed moreover that additional substitution of alkyl chain on HPG improves its effectiveness at low dosage. This was attributed to an enhancement of overlapping.

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1. Introduction

Hydroxypropylguars (HPG) are etherified derivatives of guar gum, a natural polysaccharide extracted from the seed endosperm of native plant from India *Cyamopsis tetragonolobus*. Due to their thickening effect, good solubility and thermal stability in solution, HPG are used in numerous industries such as textile printing, oil production or paper manufacturing [1]. HPG are also used in the field of building construction since they are introduced into mortar formulation as viscosity-enhancing and water retention admixtures.

Water retention (WR) is one of the key properties of mortar at fresh state. Indeed, water retention agent allows enhancing cement hydration by limiting the absorption of mixing water by the substrate. Thus, thanks to the WR agent, the mortar exhibits better mechanical and adhesive properties when it is applied in thin layer or on highly absorbent substrate [2,3].

Cellulose ethers (CE) are the most widely used WR agents. Therefore, up to now, research into the understanding of water retention in cement-based material is mainly focused on the effect of cellulose ethers. It appears that polymer molecular parameters, such as nature and content of substitution groups, and molecular weight, have a significant influence on WR [4,5].

Desbrière studied in the mid-90s the influence of cellulose derivatives to the limitation of the fluid loss in cement slurries during static filtration under strong differential pressure (70 bars) [6]. The author proposed two predominant mechanisms: the increase in the viscosity of the interstitial solution and the reduction of the permeability of the

cement cake. He reached these assumptions considering that the fluid flow through a porous media constituted by filter cake can be described by Darcy's law. Thus the fluid loss volume, the opposite of WR, is a function of the viscosity of the filtrate and the cake permeability.

Other authors use also Darcy's law to describe the flow of water in a porous media under a pressure gradient due to capillary suction or depression. In case of good water retention capacity, they assume that the amount of extracted water is low and hence the permeability of the sample remains almost constant. Thus water-soluble polymers improve WR by increasing the viscosity of the pore solution whatever the polymer molecular parameters and dosage [7,8]. In the same idea, many authors interpreted WR as the result of a reduction of the aqueous phase mobility due to an increase in viscosity when the polymer-to-cement ratio increases but without involving Darcy's law [9–11]. These authors explain also WR improvement in terms of sealing effect due to the formation of polymer water-impervious films in the paste (*i.e.* polysaccharides act as diffusion barrier to the water) [9–11].

Thanks to pulsed field gradient and NMR imaging investigations, Patural et al. [12] demonstrated that CE do not modify the water self-diffusion coefficient, even when an osmotic gradient is counteracting the diffusion flux. These results allow discarding the diffusion barrier hypothesis and showing that WR is governed by capillary suction.

Then, by means of NMR dispersion investigations, Patural et al. [13] showed that the surface diffusion coefficient of water is not modified by the presence of CE, in spite of strong effect on solution's viscosity. However, CE increase the fraction of mobile water molecules transiently present at the solid surfaces. It was found a linear correlation between this fraction of mobile water and WR.

In the same time, by comparing the flow between a CE solution and a simple oil of similar viscosity from imbibitions experiments, Marlière

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et al. [14] have obtained different behaviors with these two solutions. Indeed, the flow stops much before with the CE solutions which demonstrate that WR is not only governed by the fluid viscosity.

The second hypothesis of Desbrière [6] to explain WR is the reduction of filter permeability. According to this author, this is the major parameter and it may be explained by (i) a decrease in the porosity due to adsorption of macromolecular chains on cement; (ii) a physical plugging of the pore; (iii) an increase in the cake compaction due to modification of the local structure.

The influence of CE on the mortar's permeability is difficult to determine since it is very difficult to measure it in a direct way at the fresh state and water transport properties of hardened mortars differs strongly from the fresh material [15–17]. Nevertheless, several studies discussed the impact of CE on the microstructure and porous structure of cement-based material in both fresh and hardened state [3,4,18,19]. It appears that a specific porous structure is generated by CE.

However, Marlière et al. [14] suggested that WR is not influenced by cement-CE interactions within a given timeframe. Indeed, they have shown that the mortar WR curves (WR vs CE dosage) exhibit similar shape when the cement is substituted by sand or glass beads. Bülichen et al. [20] demonstrated also that MHEC effectiveness as water retention agent is not linked to its adsorption on cement.

In accordance with Desbrière [6], the authors cited above considered that WR is explained by a reduction of filter cake permeability due to the formation of hydrocolloidal associated 3D polymer networks at high dosage (*i.e.* above a certain threshold concentration known as the overlapping concentration) which physically plug the pores of the paste [20–22]. Marlière et al. [14] described a jamming effect explained by the size and the quantity of polydisperse aggregates. This theory is consistent with the thin CE-enriched filter cake at the mortar/substrate interface suggested by Patural et al. [12], and with the accumulation of CE observed at the interface with the substrate in tile adhesive mortar [3,23] or in the uppermost millimeter of self-leveling flooring mortar layer [24].

This short state of the art highlights that the understanding of WR mechanism induced by CE is crucial and interests many authors. The importance of colloidal interactions has emerged recently while the increase in pore solution viscosity is still often advanced. However, few studies have given attention to the impact of HPG on mortar WR although they were presented by Plank in 2004 as promising new class of water retaining agent in building materials [25]. Indeed, in comparison with CE, HPG exhibit comparable water retention abilities [26–28], similar delayed effect on cement hydration [27,28] and a manufacturing process less polluting [29]. Furthermore, the HPG use is already well-established in industrial construction chemistry.

The aim of this work consists in understanding the effectiveness of HPG as water retention agent and comparing its behavior with classical CE. The main issue is to determine if WR is due to an increase in pore solution viscosity or to the formation of hydrocolloidal aggregates in the case of HPG. Thus working with non-cellulosic polysaccharides can also allow us to establish how generalizable are the recent findings concerning CE. For instance, other polysaccharidic admixtures such as starch [5,26] and chitin derivatives [28] were found to act on WR in different ways.

The present work will be divided into two parts. The first one will consist in the study of the influence of pore solution viscosity on WR. The objective is to determine if a master curve appears whatever the admixture when WR is plotted against the pore solution viscosity. The second part will focus on the influence of the agglomerates formation due to coil-overlapping. To achieve this, the dosage and kind of HPG will be varied and the associate WR and viscosity of the extracted pore solution will be systematically characterized. Results will be compared to those obtained with classical cellulosic water retention agents. The influence of dosage on WR and the viscosity of admixed-mortars pore solutions will be first discussed.

2. Material and methods

2.1. Mineral and organic compounds

2.1.1. Mineral products

The investigated ordinary Portland cement (OPC) was a CEM I 52.5 R CE CP2 NF type cement according to EN 197-1 and NF P 15-318 standards. Oxide composition was determined by X-ray fluorescence spectroscopy (SRS3400, Bruker-AXS). Phase composition was quantified by means of Rietveld refinement method (Siroquant V2.5 software) after XRD analysis (D5000, Siemens). Chemical and phase compositions of the cement used are given in Table 1.

2.1.2. Organic admixtures

Seven polysaccharidic water retention admixtures were selected for this study: two hydroxypropyl methyl cellulose (HPMC1 and HPMC 2) and five hydroxypropylguars (HPG 1, HPG 2, HPG 3, HPG 5 and HPG 6).

Guar gum is a galactomannan consisting of a $\beta(1-4)$ -linked D-mannopyranose backbone, with random branchpoints of galactose via an $\alpha(1-6)$ linkage [30]. The ratio of mannose to galactose is about 1.8. HPG are obtained from the native guar gum via an irreversible nucleophilic substitution, using propylene oxide in the presence of an alkaline catalyst. As guar gum exhibits a high chemical reactivity and is soluble in cold water, the chemical modification of the native guar gum does not require hard reaction conditions of temperature and pressure, does not generate large quantity of by-products and needs weak purification procedure [29].

In order to compare HPG behavior with classical polysaccharidic admixtures, two cellulose derivatives were also tested. Cellulose is a polysaccharide composed of individual anhydroglucose units linked through $\beta(1-4)$ glycosidic bonds. Because of strong intermolecular hydrogen bridging bonds, cellulose is not soluble in water. Thereby, cellulose ethers are obtained by pretreating cellulose with base (alkali cellulose) in order to break hydrogen bonds and to make accessible the active groups for etherifying agents. In the case of HPMC, the substitution of the hydroxyl groups takes place by reacting alkali cellulose with the combination of two etherifying agent: propylene oxide and methylchloride.

The schematic structure of both molecules is shown in Fig. 1 (substituent positions are arbitrary). It appears that HPMC are linear polymers whereas HPG exhibit branched-chain structure. Table 2 presents a qualitative description of the admixtures used. The qualitative substitution degrees are provided by the manufacturers. The molar substitution ratio (MS_{HP}) is defined as the number of moles of hydroxypropyl groups per mole of anhydroglucose unit and is less than 3 for our molecules. The degree of substitution, noted DS_M , represents the amount of methoxyl groups per anhydroglucose unit and is about 1.8 for HPMC 1 and HPMC 2 according to the manufacturer.

All the HPG samples, provided by *Lamberti S.p.A*, have similar molecular weight, about $2 \cdot 10^6 \text{ g} \cdot \text{mol}^{-1}$. The only difference between HPG 1, 2 and 3 is the molar substitution ratio. HPG 5 and 6 exhibit additional substitution (short or long alkyl chains). The two HPMC samples exhibit the same substitution degrees. The only difference between HPMC 1 and HPMC 2 is molecular weight: about $0.25 \cdot 10^6$ and $1.10^6 \text{ g} \cdot \text{mol}^{-1}$ respectively.

2.2. Experimental methods of investigation

2.2.1. Preparation of mortars

Admixtures were previously dissolved in deionized water by magnetic stirring for 24 h in order to obtain a complete dissolution of all polymers. The concentrations of these polymer solutions (from 1 to $16 \text{ g} \cdot \text{L}^{-1}$) allow obtaining, in mortars, polymer dosages varying from 0.1 to 1.6% by weight of cement (bwoc).

Then, mortars were prepared according to the following mixture proportions: 30 wt.% of cement, 65 wt.% of siliceous sand and 5 wt.% of limestone filler. The dry mixture (*i.e.* cement, sand and filler) was

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