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INFLUENCE OF HYDROXYPROPYLGUARS ON FRESH STATE PROPERTIES OF CEMENT-BASED MORTARS

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ABSTRACT

Nowadays, modern factory-made mortars are usually formulated with polysaccharides in order to improve water retention capacity, homogeneity and workability. Among all polysaccharides, hydroxypropylguars have received very scant attention in technical literature. This paper shows that hydroxypropylguars give excellent water retention properties to the mortar. However, as with cellulose ethers that are the reference for this type of admixtures, these molecules delay the hydration of cement. Moreover, the effect of the polymer predissolution in the liquid phase has been studied. There is a real impact of the predissolution on the water retention capacity of the mortar and the cement hydration kinetics.

Keywords: HydroxyPropylGuar, mortar, water retention, cement, hydration kinetics

INTRODUCTION

Traditionally, mortars are made from a mixture of sand, a binder such as cement or lime, and water. Modern factory-made mortars are complex materials in which are added several kinds of admixtures to exhibit various properties, from the fresh paste to the hardened material.

Polysaccharides are one of these admixtures. They are frequently introduced into mortar formulations in order to improve water retention capacity of the freshly-mixed materials, which enhances cement hydration and adhesion to the substrate. These products are also expected to act as viscosity-enhancing admixtures to prevent segregation and thus improve the homogeneity and workability of the mortar. However, the major drawback of these macromolecules in mortar formulation is the delay in the setting time of the cement [1].

Among all polysaccharides, cellulose ethers are the most widely used when high water retention capacity is expected. Nevertheless, **H**ydroxy**P**ropyl**G**uars (HPG) were recently presented as a promising new class of water-retaining agent [2]. HPG are already used in paint or paper manufacturing due to its high water retention capacity. HPG were also the subject of a lot of patents as admixture for mortars. However, these molecules have received scant attention by the academic community. As far as we knew, only Izaguirre *et al.* [3] have studied the effect of HPG in lime-based mortars.

Thus, the aim of this work is to study the impact of HPG on the properties of portland cement-based mortars at fresh state, in particular water retention capacity and the cement hydration kinetics at early age.

RESEARCH SIGNIFICANCE

Polysaccharides are commonly used in cement-based materials, but most studies focus on cellulose ethers or welan gum. So, studies about hydroxylpropylguars are very scarce in the technical literatures. However these molecules present very interesting properties, especially water retention properties, comparable to those obtained with cellulose ethers.

EXPERIMENTAL INVESTIGATION

Mineral products

Portland cement CEM I 52.5 R, according to EN 197-1 [4], was employed. Chemical analysis was performed three times by X-ray fluorescence spectroscopy (XRF). Thus, phase compositions (Table 1) were calculated using Bogue formula with CaO correction [5]. Sand was siliceous and conformed to standard NF EN 13139 [6]. Calcium carbonate ($CaCO_3$) filler was also used.

Table 1–Chemical and phase composition of the investigated cement.

Chemical composition (% wt)				Phase composition (% wt)	
Oxides	XRF	Oxides	XRF	Phases	XRF (Bogue)
CaO	66.1 ± 1.2	MgO	1.11 ± 0.02	C_3S	69.4 ± 1.2
SiO_2	20.2 ± 0.4	TiO_2	0.24 ± 0.01	C_2S	5.4 ± 0.9
Al_2O_3	4.8 ± 0.1	P_2O_5	0.05 ± 0.01	C_3A	8.0 ± 0.1
SO_3	3.5 ± 0.2	MnO	0.04 ± 0.00	C_4AF	8.7 ± 0.3
Fe_2O_3	2.9 ± 0.1	K_2O	0.01 ± 0.01	Sulphates	3.5 ± 0.2

Organic admixtures: HydroxyPropylGuars (HPG)

Guar gum is a galactomannan consisting of a (1-4) linked β-D-mannopyranose backbone, with random branchpoints of galactose via an alpha (1-6) linkage [7] (Fig. 1-A). The ratio of mannose to galactose is about 1.8. This polysaccharide is extracted from the seed endosperm of Cyamopsis tetragonolobus, a native plant from India. The extraction does not require chemical process, but a thermo-chemical one. So, unlike the manufacturing of cellulose, guar gum is obtained without producing any effluent or causing pollution.

HPG is obtained from the native guar gum via an irreversible nucleophilic substitution, using propylene oxide in the presence of an alkaline catalyst (Fig. 1-B). Due to a significant thickening effect, this compound is one of the most used derivatives, especially in textile printing, oil production, paper manufacturing [8].

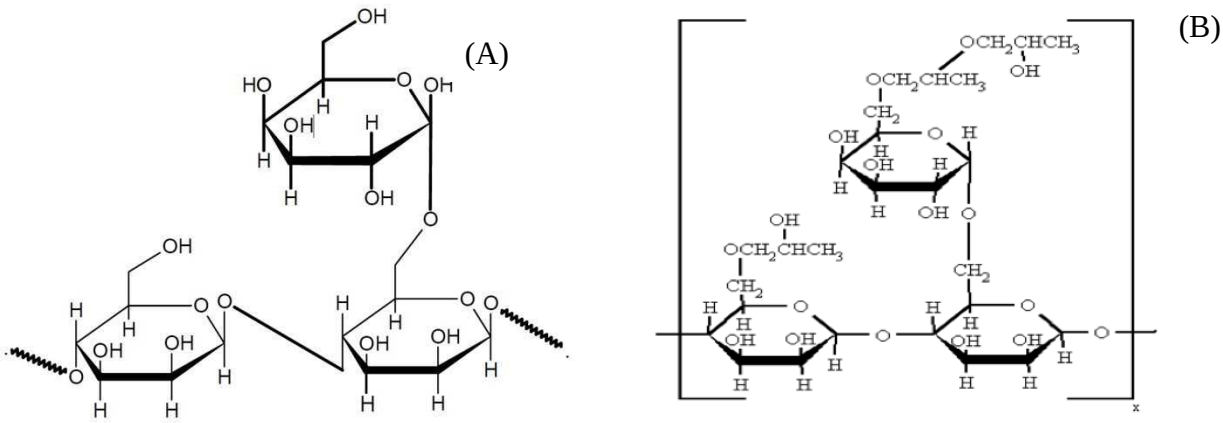

Fig. 1-Molecular structure of native guar gum (A) and hydroxypropylguar (B).

In this paper, six HPG powders from Lamberti S.p.A were studied. They have roughly the same molecular weight. Table 2 gives a qualitative description of the HPG samples used in this work.

Three cellulose ethers were also tested: two HydroxyPropylMethyl Cellulose (HPMC) named HPMC1 and HPMC2, and one HydroxyEthyl Cellulose (HEC). HPMC 1 has a lower molecular weight than HPMC 2, and thus a lower viscosity.

Table 2-Qualitative description of the HPG used.

	MS	**Viscosity* (Pa.s)**	**Additionnal substitution**
HPG 1	Low	1180	-
HPG 2	Medium	810	-
HPG 3	High	670	-
HPG 4	High	330	-
HPG 5	High	850	Shorter alkyl chain
HPG 6	High	660	Longer alkyl chain

* : apparent viscosity at shear rate = 5.1 s^{-1} of 10 $g.L^{-1}$ solution of HPG

Water retention measurements

Water retention is a very sensitive mortar property. Indeed, it must be important enough, to not disturb the hydration of the cement. It also limits the absorption of the mixing water by the substrate and thus provides good mechanical and adhesive properties to the mortar.

The water retention capacity of freshly-mixed mortar can be assessed using different tests where the removed water after suction or depression is measured [9].

A standard method to estimate the water retention capacity of a mortar is the test described in ASTM C1506-09 [10]. ASTM measurements had to be performed 15 min after mixing to measure the water loss of a mortar under depression. The standardized apparatus (Fig. 2) was exposed to a vacuum of 50 mm of mercury (6.6×10^3 Pa) for 15 min. Then, the water retention capacity, noted WR, was calculated using the following equation:

$$WR(\%) = \frac{W_0 - W_1}{W_0} \times 100$$

W_0 represents the initial mass of mixing water; W_1 is the loss of water mass after aspiration.

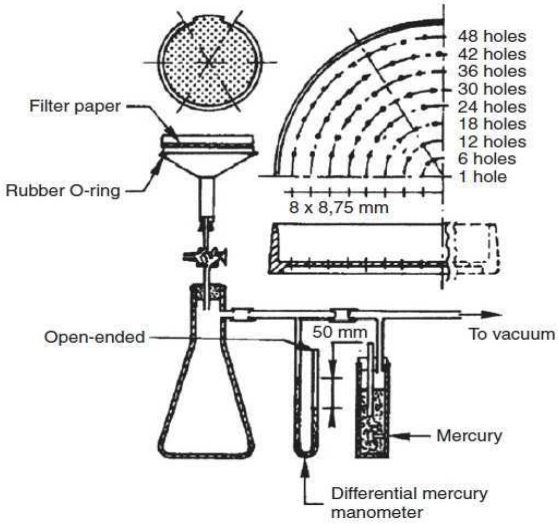

Fig. 2-Experimental device to measure water retention according to ASTM C91 standard.

DTU 26.1 [11] specifies three classes of water retention (as measured by ASTM method) of a fresh mortar. The first one (low water retention category) is for mortars with a water retention

lower than 86%. The second class (intermediate) corresponds to values ranging from 86% to 94%. The last one (strong) is defined by water retention higher than 94%, corresponding to the required values.

A second method was also used to measure the water retention, which is the Standard DIN 18555-7 [12]. With this method, the freshly-mixed mortar was in contact with a filter paper, thereby simulating the action of an absorptive substrate. DIN measurements had to be performed 5 min after mixing for 5 min. The water retention capacity of a freshly-mixed mortar was characterized by the retained mass of water after the capillarity action of the absorbent substrate. The standardized apparatus is described in Fig. 3. The water retention was calculated using the following equation:

$$WR(\%) = 100 - \frac{W_{fp}}{W_0} \times 100$$

Where W_{fp} is the quantity of water retained by the filter paper and W_0 is the initial quantity of water inside the studied mortar.

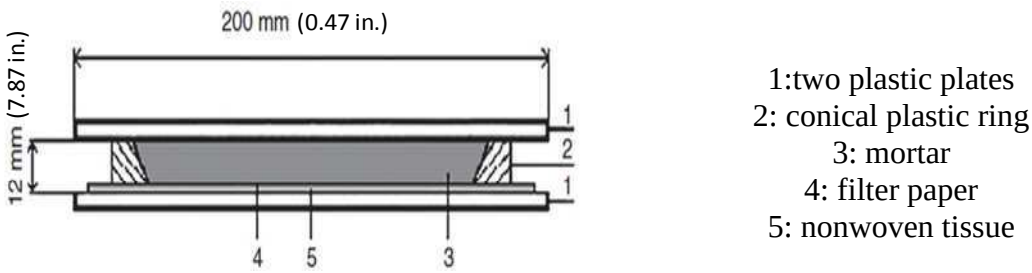

Fig. 3-Experimental device to measure water retention according to DIN 18555-7 standard.

All tests were carried out in triplicate and a controlled temperature of 23°C (73.4°F). Temperature had to be controlled because water retention and consistency are temperature-dependent.

Mortars were prepared according to the following mixture proportions: 30% of cement, 65% of sand and 5% of filler (by weight). Admixture amount (0.1, 0.2 or 0.3% wt) was in addition

to the total dry mixture (i.e. cement, sand and filler). Dry mixture was blended in a shaker (Wab, Turbula, Germany) for 15 min. Deionised water was added to have a water to cement ratio W/C = 1. Mixing procedure was in accordance with EN 196-1 [13].

Admixtures used for the water retention tests were the six HPG, HPMC1 and HPMC2. A control test was also performed with a mortar without admixture.

Characterization of the cement hydration delay

It is empirically established for years that polysaccharides induce a more or less important delay of cement hydration. But the mechanisms of interaction between this polymers and cement are still not well established and depend on the nature of the polysaccharide [14], [15]. To characterize the delay induced by the studied polymers on the cement hydration, conductivity measurements are performed. During cement hydration, ions are released during dissolution of anhydrous phases and others ions are consumed during the precipitation of hydrates. This overall evolution of ionic concentrations can be monitored by conductivity measurements [16]. This method appears as a powerful tool to monitor the hydration kinetics and lead to reveal the nucleation, growth and precipitation processes of hydrates such as C–S–H or portlandite (Fig. 4).

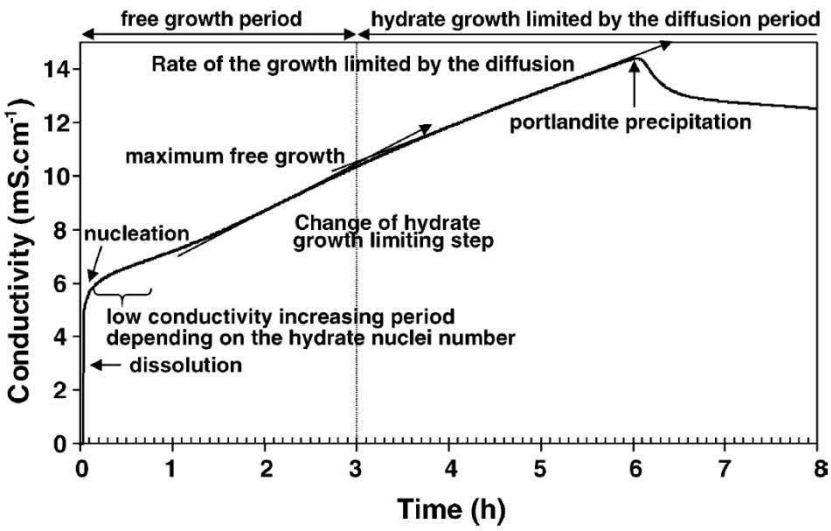

Fig. 4-Global view of hydration mechanism of cement in lime solution (liquid to solid ratio equal to 20) on a conductimetric curve [14].

Conductimetric measurements could be realized in lime suspension. This allows to obtain hydration kinetics close to that observed in cement pastes in spite of a high liquid to solid (noted L/S) weight ratio. The portlandite precipitation time is represented by an electrical conductivity drop. This phenomenon could be used to the determination of the hydration delay. Therefore, in this study, conductimetry enables to classify and to determine the relative retardation capacity of admixtures on cement hydration.

The experiments were performed in diluted suspensions, thermostated at 25°C (77°F) and continuously stirred. Each experiment was carried out in triplicate.

The liquid to solid (L/S) weight ratios used were equal to 10, 20 or 50. The solid was a mix of cement and admixture powders which was blended in a shaker (Wab, Turbula, Germany) for 10 min. Polymer to cement (P/C) weight ratios equal to 0.5, 1.0, 1.5 and 2.0 % were studied. The liquid used was a roughly saturated lime solution (20 mM).

The studied admixtures are the 6 HPG and two cellulose ethers: HPMC 1 and HEC. The control test is the neat cement.

Predissolution of the polymer

Typically, the polymer is mixed with solid cement. However, we also study the influence of the polymer predissolution in the liquid phase for 24h, before to introduce cement in the case of the conductimetric measurements, or before to mix with the dry mortar in the case of water retention characterization.

To obtain the effect of the predissolution, authors calculated as followed an increase percentage of the water retention value WR and of the portlandite precipitation time t_{CH} (in gray on the graphs):

$$Increase\ of\ WR\ percentage\ (\%) = \frac{WR(with\ prediss) - WR(without\ prediss)}{WR(with\ prediss)} \times 100$$

$$Increase\ of\ t_{CH}\ percentage\ (\%) = \frac{t_{CH}(with\ prediss) - t_{CH}(without\ prediss)}{t_{CH}(with\ prediss)} \times 100$$

EXPERIMENTAL RESULTS AND DISCUSSION

Impact of HPG on water retention in fresh mortar

Fig. 5 shows the water retention capacity of the studied mortars with an amount of polymer of 0.3% (9.99 $g.L^{-1}$), according to the adjuvant used and depending on the standard method (ASTM or DIN).

The results obtained by both methods are not identical but similar: HPG exhibit very high WR for this formulation (>90%), as announced by Plank [2]. The increase, compared to the control, is considerable. In addition, the results are quite comparable to those obtained with HPMC.

Moreover, with this formulation, mortars containing HPG 2 to 6 and HPMC 2 could be considered as high WR capacity according to the DTU 26.1. Mortars containing HPG 1 and HPMC 1 are in the intermediate category.

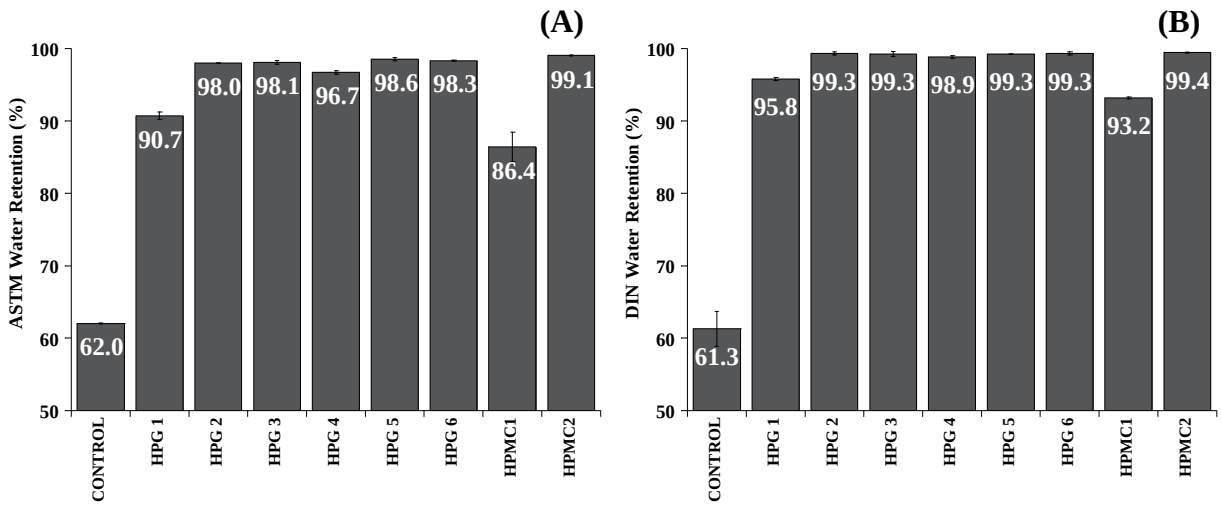

Fig. 5–Water retention capacity of studied mortars measured by ASTM (A) and DIN (B) methods with 0.3% of HPG (9.99 $g.L^{-1}$).

Nevertheless, water retention values are so high that the comparison is difficult. Therefore, authors decreased the polymer concentration (Fig. 6).

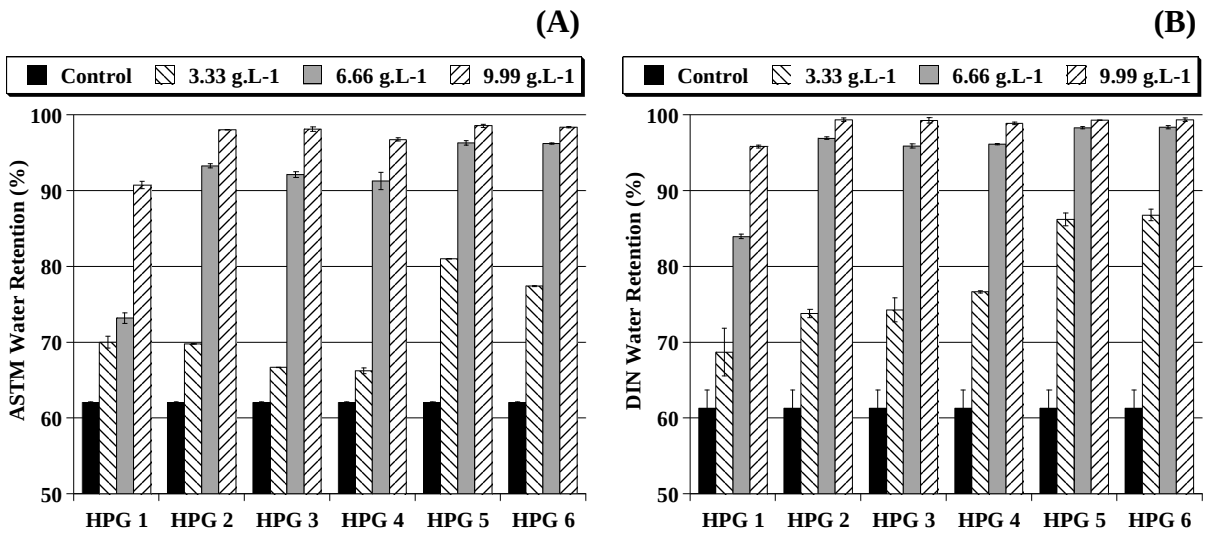

Fig. 6–Influence of polymer concentration on water retention (A=ASTM ; B=DIN).

Clearly, the water retention capacity of the mortar depends on the amount of admixture. Moreover, three groups stand out.

HPG 5 and 6 present the best results, which is significant at 0.1% (3.33 $g.L^{-1}$). At 0.2% (6.66 $g.L^{-1}$), the water retention capacity is almost maximum (strong WR category). HPG 2, 3 and 4 also allow good water retention to the mortar at 0.2% (intermediate WR category). Nevertheless, for an amount as low as 0.1%, the increase is quite small compared to the control. Finally, only the mortar with HPG 1 is in low WR category at 0.2%.

It is important to note that the results are significantly different according to the considered HPG, although all these molecules belong to the same family, and the molecular weights are approximately the same.

Impact of HPG on hydration kinetics of cement in diluted suspension

Fig. 7 presents the conductimetric curves of admixtured-cement in almost saturated lime solution (20 mM), for ratios L/S = 20 and P/C = 2%. Polymers studied are the six HPG and two cellulose ethers to get an idea of the comparative behavior of HPG versus two molecules that are very commonly used.

This graph highlights the significant impact of these molecules on the kinetics of hydration. Moreover, by focusing on the portlandite precipitation time (conductivity drop), it appears a wide range of delays induced by the admixtures studied. HPG 5 and 6 exhibit superposed curves and the weaker delay (increase of around 70 % in the portlandite precipitation time compared to the control) while the most important delay is observed for HPG 1 (≈ 330 %).

It also appears that delays are between those obtained for the cellulose ethers studied.

No period of low activity before the increase of the conductivity is observed as for some others polysaccharides (some cellulose ethers [15] or dextrines [14]) or others admixtures (setting retarding admixtures as sodium gluconate or some superplastifiants [17]). Here, a decrease of the slope is observed. Thus, these polymers may not influence the germination of the first hydrates, but their growth.

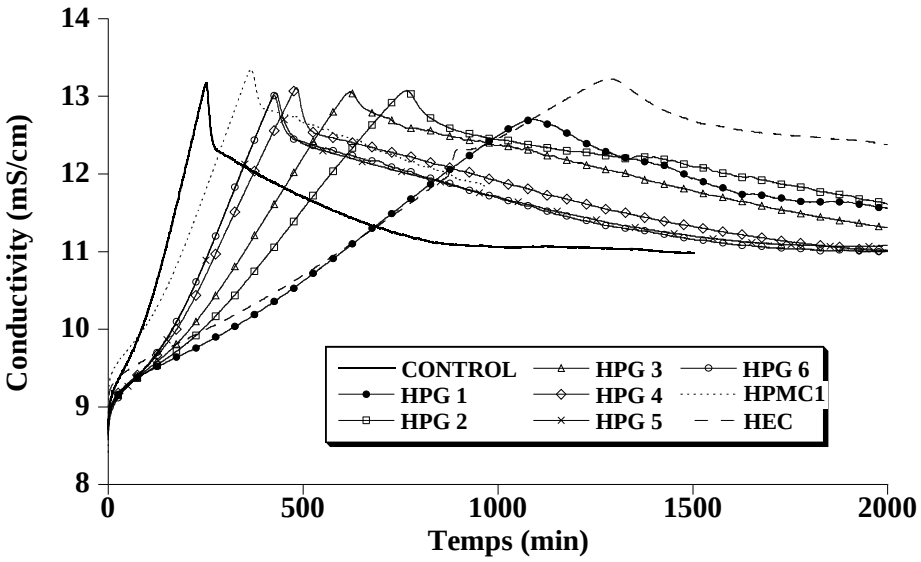

Fig. 7–Conductimetric curves of cement admixed with polysaccharide in lime solution (L/S=20, P/C=2%).

The influence of the volume of lime solution is studied by varying the liquid to solid ratio, the polymer to cement ratio being constant (2%) for each HPG (Fig. 8). It seems to influence the portlandite precipation time, and so the cement kinetic hydration, for each polymer. However, this impact is the same for the neat cement.

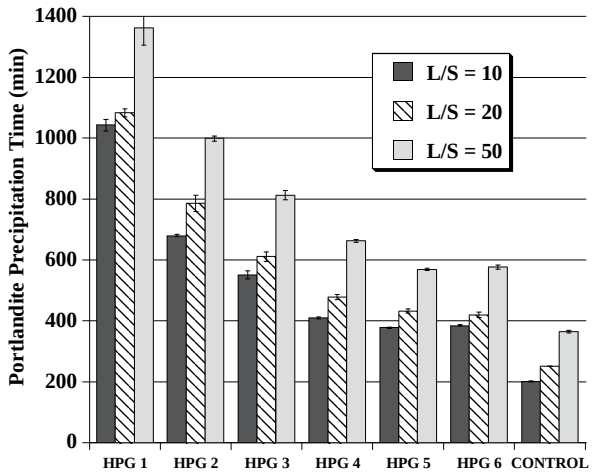

Fig. 8-Influence of volume of lime solution on portlandite precipitation time.

On the contrary, the influence of the amount of polymer, studied by varying the polymer to cement ratio with a constant liquid to solid ratio (20) for each HPG (Fig. 9), is considerable. Indeed, a small increase in the amount of polymer leads to a huge increase in the delay of hydration. This would suggest that the mechanism responsible for the delay is an adsorption of the polymer, native or degraded, on the cement phases, hydrates or anhydrous.

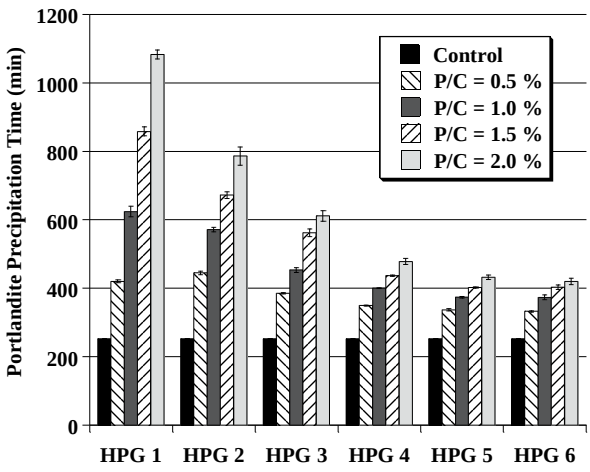

Fig. 9-Influence of polymer quantity on portlandite precipitation time.

Influence of predissolution on activity of HPG

Water retention: Dry mortars are mixed with water in which the polymer was predissolved for 24h at 3.33 g.L^{-1} (Fig. 10) and 6.66 g.L^{-1} (Fig. 11), corresponding to 0.1 and 0.2% by weight of dry mortar, respectively. There is a significant impact on the water retention of the obtained mortar, compared to the mortar obtained by the classic way, especially for HPG 1. This impact is weaker for [P] = 6.66 g.L^{-1}. Indeed, values without predissolution are very high. So, it is not possible to improve water retention capacity with predissolution: a ceiling effect is observed. It is interesting to remark that HPG 1 at 6.66 g.L^{-1} is the worst water-retaining agent without predissolution (low WR category) and becomes the better one with predissolution (strong WR category).

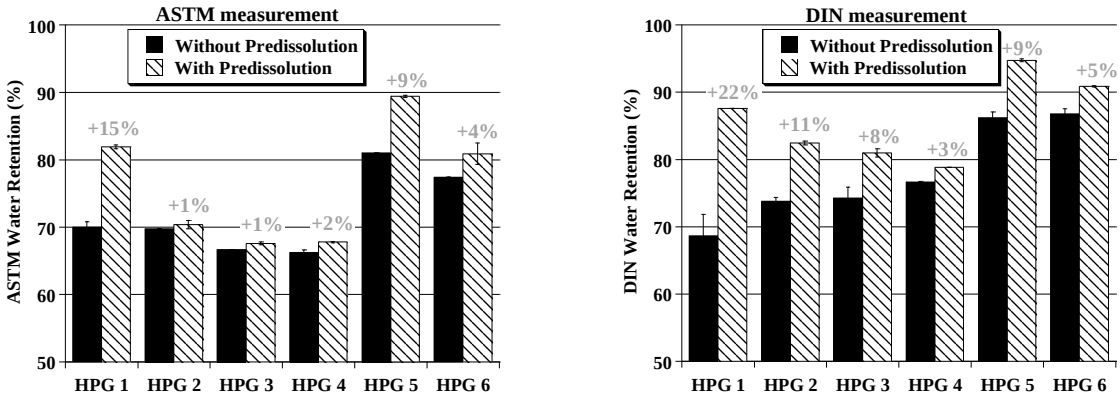

Fig. 10–Influence of predissolution on water retention capacity with [P] = 3,33 g.L^{-1}.

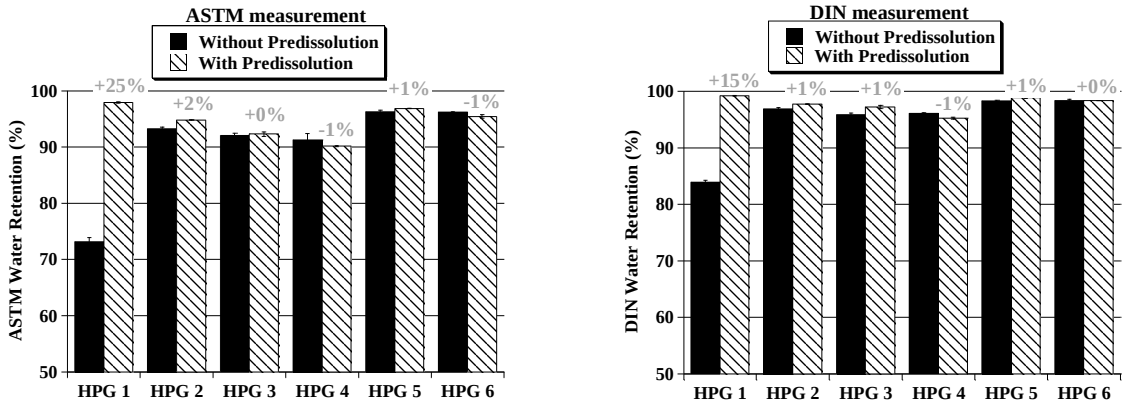

Fig. 11–Influence of predissolution on water retention capacity with [P] = 6,66 g.L^{-1}.

Hydration delay: Predissolution has also an impact on the hydration kinetics because the delay caused by the polymer is more important after predissolution (Fig. 12). This effect is very strong for HPG 1, with a huge increase in portlandite precipitation time of 77%, and weaker for other HPG: between 10 and 20% for HPG 2, 3 and 6, and lower than 5% for HPG 4 and 5.

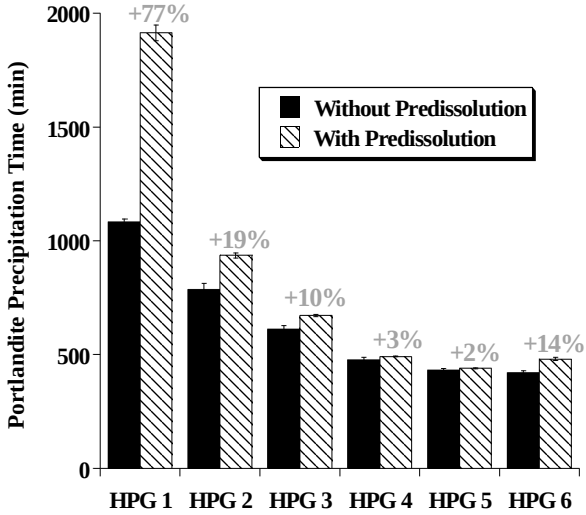

Fig. 12-Influence of predissolution on portlandite precipitation time.

These results show that the polymer is more active to delay the hydration of cement when it is already in solution. Two hypotheses can be considered to explain these results. The polymer could be degraded due to the high alkalinity of the medium. This degradation could lead to the formation of carboxylates by peeling reaction, molecules known to have a strong retarding effect [18]. The predissolution would promote this degradation, resulting in an increase of the delay. The second hypothesis would be a slowdown in dissolution kinetics of the polymer in the highly alkaline media or high ionic strength media. In that case, without predissolution, the polymer is not fully active to delay the cement hydration because the dissolution would be disturbed. On the contrary, predissolution leads to the complete dissolution of the polymer and therefore a maximum delay.

FURTHER RESEARCH

Work with pure cement phases will overcome the complexity of the environment and facilitate the identification of HPG interactions with cement. For example, adsorption of HPG on these phases could be quantified.

Moreover, it is essential to identify the degradation products of HPG under alkaline conditions. For this, a method developed at the laboratory will be performed [19], [20]. Thus, it will be possible to test their impact on the cement hydration kinetics.

HPG dissolution kinetics should also be determined, according to the pH and the ionic strength of the medium. This might help to know if this phenomenon could be responsible of the difference obtained with and without predissolution.

SUMMARY AND CONCLUSIONS

The results of this experimental investigation exhibit the strong impact of the HPG on the water retention capacity of portland cement-based mortars. Indeed, it has been shown in this study the huge increase of water retention with the addition of HPG. Moreover, the values are similar to those obtained with cellulose ethers. Nevertheless, the impact of these molecules is very dependant of the concentration, so that a minimum rate is required to achieve high water retention.

The conductimetric experiments show a variable influence according to the HPG considered on the kinetics of cement hydration. Delays observed are between those obtained with the cellulose ethers tested. As a first approximation, it seems that HPG influence the growth of hydrates by adsorption on some cement phases, and not their germination. But this conclusion should be handled with care and requires further researches.

Finally, it has been demonstrated that predissolution of the polymer in the liquid phase modifies its impact. Indeed, predissolution enhances the water retention capacity but increases also the hydration delay induced by these polymers. However, the influence of predissolution is different for each HPG studied.

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