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1 INFLUENCE OF HYDROXYPROPYLGUARS ON FRESH STATE

2 PROPERTIES OF CEMENT-BASED MORTARS

3 Thomas Poinot, Alexandre Govin and Philippe Grosseau

4 **Biography:**

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14 **ABSTRACT**

15
16 Nowadays, modern factory-made mortars are usually formulated with polysaccharides in
17 order to improve water retention capacity, homogeneity and workability. Among all
18 polysaccharides, hydroxypropylguars have received very scant attention in technical literature.
19 This paper shows that hydroxypropylguars give excellent water retention properties to the
20 mortar. However, as with cellulose ethers that are the reference for this type of admixtures,
21 these molecules delay the hydration of cement. Moreover, the effect of the polymer
22 predissolution in the liquid phase has been studied. There is a real impact of the
23 predissolution on the water retention capacity of the mortar and the cement hydration kinetics.

24
25 **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, HydroxyPropylGuars (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 (CaCO₃) filler was also used.

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] (**Erreur ! Source du renvoi introuvable.-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 (**Erreur ! Source du renvoi introuvable.-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].

In this paper, six HPG powders from *Lamberti S.p.A* were studied. They have roughly the same molecular weight. **Erreur ! Source du renvoi introuvable.** gives a qualitative description of the HPG samples used in this work.

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

4

5 **Water retention measurements**

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

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

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

$$17 \text{WR (\%)} = \frac{W_0 - W_1}{W_0} \times 100$$

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

19 DTU 26.1 [11] specifies three classes of water retention (as measured by ASTM method) of a
20 fresh mortar. The first one (low water retention category) is for mortars with a water retention
21 lower than 86%. The second class (intermediate) corresponds to values ranging from 86% to
22 94%. The last one (strong) is defined by water retention higher than 94%, corresponding to
23 the required values.

24

1 A second method was also used to measure the water retention, which is the Standard DIN
2 18555-7 [12]. With this method, the freshly-mixed mortar was in contact with a filter paper,
3 thereby simulating the action of an absorptive substrate. DIN measurements had to be
4 performed 5 min after mixing for 5 min. The water retention capacity of a freshly-mixed
5 mortar was characterized by the retained mass of water after the capillarity action of the
6 absorbent substrate. The standardized apparatus is described in **Erreur ! Source du renvoi**
7 **introuvable.** The water retention was calculated using the following equation:

$$8 \quad WR (\%) = 100 - \frac{W_{fp}}{W_0} \times 100$$

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

11

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

15 Mortars were prepared according to the following mixture proportions: 30% of cement, 65%
16 of sand and 5% of filler (by weight). Admixture amount (0.1, 0.2 or 0.3% wt) was in addition
17 to the total dry mixture (i.e. cement, sand and filler). Dry mixture was blended in a shaker
18 (Wab, Turbula, Germany) for 15 min. Deionised water was added to have a water to cement
19 ratio $W/C = 1$. Mixing procedure was in accordance with EN 196-1 [13].

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

22

23 **Characterization of the cement hydration delay**

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

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

17

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

20 The liquid to solid (L/S) weight ratios used were equal to 10, 20 or 50. The solid was a mix of
21 cement and admixture powders which was blended in a shaker (Wab, Turbula, Germany) for
22 10 min. Polymer to cement (P/C) weight ratios equal to 0.5, 1.0, 1.5 and 2.0 % were studied.

23 The liquid used was a roughly saturated lime solution (20 mM).

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

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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):

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

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

EXPERIMENTAL RESULTS AND DISCUSSION

Impact of HPG on water retention in fresh mortar

Erreur ! Source du renvoi introuvable. 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.

1 Nevertheless, water retention values are so high that the comparison is difficult. Therefore,
2 authors decreased the polymer concentration (**Erreur ! Source du renvoi introuvable.**).
3 Clearly, the water retention capacity of the mortar depends on the amount of admixture.
4 Moreover, three groups stand out.
5 HPG 5 and 6 present the best results, which is significant at 0.1% (3.33 g.L⁻¹). At 0.2% (6.66
6 g.L⁻¹), the water retention capacity is almost maximum (strong WR category). HPG 2, 3 and
7 4 also allow good water retention to the mortar at 0.2% (intermediate WR category).
8 Nevertheless, for an amount as low as 0.1%, the increase is quite small compared to the
9 control. Finally, only the mortar with HPG 1 is in low WR category at 0.2%.
10 It is important to note that the results are significantly different according to the considered
11 HPG, although all these molecules belong to the same family, and the molecular weights are
12 approximately the same.

13

14 **Impact of HPG on hydration kinetics of cement in diluted suspension**

15 **Erreur ! Source du renvoi introuvable.** presents the conductimetric curves of admixed-
16 cement in almost saturated lime solution (20 mM), for ratios L/S = 20 and P/C = 2%.
17 Polymers studied are the six HPG and two cellulose ethers to get an idea of the comparative
18 behavior of HPG versus two molecules that are very commonly used.
19 This graph highlights the significant impact of these molecules on the kinetics of hydration.
20 Moreover, by focusing on the portlandite precipitation time (conductivity drop), it appears a
21 wide range of delays induced by the admixtures studied. HPG 5 and 6 exhibit superposed
22 curves and the weaker delay (increase of around 70 % in the portlandite precipitation time
23 compared to the control) while the most important delay is observed for HPG 1 (≈ 330 %).
24 It also appears that delays are between those obtained for the cellulose ethers studied.

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

6

7 The influence of the volume of lime solution is studied by varying the liquid to solid ratio, the
8 polymer to cement ratio being constant (2%) for each HPG (**Erreur ! Source du renvoi**
9 **introuvable.**). It seems to influence the portlandite precipitation time, and so the cement
10 kinetic hydration, for each polymer. However, this impact is the same for the neat cement.

11 On the contrary, the influence of the amount of polymer, studied by varying the polymer to
12 cement ratio with a constant liquid to solid ratio (20) for each HPG (**Erreur ! Source du**
13 **renvoi introuvable.**), is considerable. Indeed, a small increase in the amount of polymer
14 leads to a huge increase in the delay of hydration. This would suggest that the mechanism
15 responsible for the delay is an adsorption of the polymer, native or degraded, on the cement
16 phases, hydrates or anhydrous.

17

18 **Influence of predissolution on activity of HPG**

19 Water retention: Dry mortars are mixed with water in which the polymer was predissolved
20 for 24h at 3.33 g.L⁻¹ (**Erreur ! Source du renvoi introuvable.**) and 6.66 g.L⁻¹ (**Erreur !**
21 **Source du renvoi introuvable.**), corresponding to 0.1 and 0.2% by weight of dry mortar,
22 respectively. There is a significant impact on the water retention of the obtained mortar,
23 compared to the mortar obtained by the classic way, especially for HPG 1.

1 This impact is weaker for $[P] = 6.66 \text{ g.L}^{-1}$. Indeed, values without predissolution are very
2 high. So, it is not possible to improve water retention capacity with predissolution: a ceiling
3 effect is observed.

4 It is interesting to remark that HPG 1 at 6.66 g.L^{-1} is the worst water-retaining agent without
5 predissolution (low WR category) and becomes the better one with predissolution (strong
6 WR category).

7

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

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

23

FURTHER RESEARCH

1
2 Work with pure cement phases will overcome the complexity of the environment and
3 facilitate the identification of HPG interactions with cement. For example, adsorption of HPG
4 on these phases could be quantified.

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

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

SUMMARY AND CONCLUSIONS

11
12
13 The results of this experimental investigation exhibit the strong impact of the HPG on the
14 water retention capacity of portland cement-based mortars. Indeed, it has been shown in this
15 study the huge increase of water retention with the addition of HPG. Moreover, the values are
16 similar to those obtained with cellulose ethers. Nevertheless, the impact of these molecules is
17 very dependant of the concentration, so that a minimum rate is required to achieve high water
18 retention.

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

24 Finally, it has been demonstrated that predissolution of the polymer in the liquid phase
25 modifies its impact. Indeed, predissolution enhances the water retention capacity but

1 increases also the hydration delay induced by these polymers. However, the influence of
2 predissolution is different for each HPG studied.

3

4

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1

2

TABLES AND FIGURES

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

5 **Table 2**-Qualitative description of the HPG used.

6

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 ₃ S | 69.4 ± 1.2 |
| SiO ₂ | 20.2 ± 0.4 | TiO ₂ | 0.24 ± 0.01 | C ₂ S | 5.4 ± 0.9 |
| Al ₂ O ₃ | 4.8 ± 0.1 | P ₂ O ₅ | 0.05 ± 0.01 | C ₃ A | 8.0 ± 0.1 |
| SO ₃ | 3.5 ± 0.2 | MnO | 0.04 ± 0.00 | C ₄ AF | 8.7 ± 0.3 |
| Fe ₂ O ₃ | 2.9 ± 0.1 | K ₂ O | 0.01 ± 0.01 | Sulphates | 3.5 ± 0.2 |

7

8

Table 2-Qualitative description of the HPG used.

| | MS | Viscosity* (Pa.s) | Additional 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

9

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

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

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

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

- 1 **Fig. 5**–Water retention capacity of studied mortars measured by ASTM (A) and DIN (B)
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- 3 **Fig. 6**–Influence of polymer concentration on water retention (A=ASTM ; B=DIN).
- 4 **Fig. 7**–Conductimetric curves of cement admixed with polysaccharide in lime solution
5 (L/S=20, P/C=2%).
- 6 **Fig. 8**–Influence of volume of lime solution on portlandite precipitation time.
- 7 **Fig. 9**–Influence of polymer quantity on portlandite precipitation time.
- 8 **Fig. 10**–Influence of predissolution on water retention capacity with [P] = 3,33 g.L⁻¹.
- 9 **Fig. 11**–Influence of predissolution on water retention capacity with [P] = 6,66 g.L⁻¹.
- 10 **Fig. 12**–Influence of predissolution on portlandite precipitation time.
- 11

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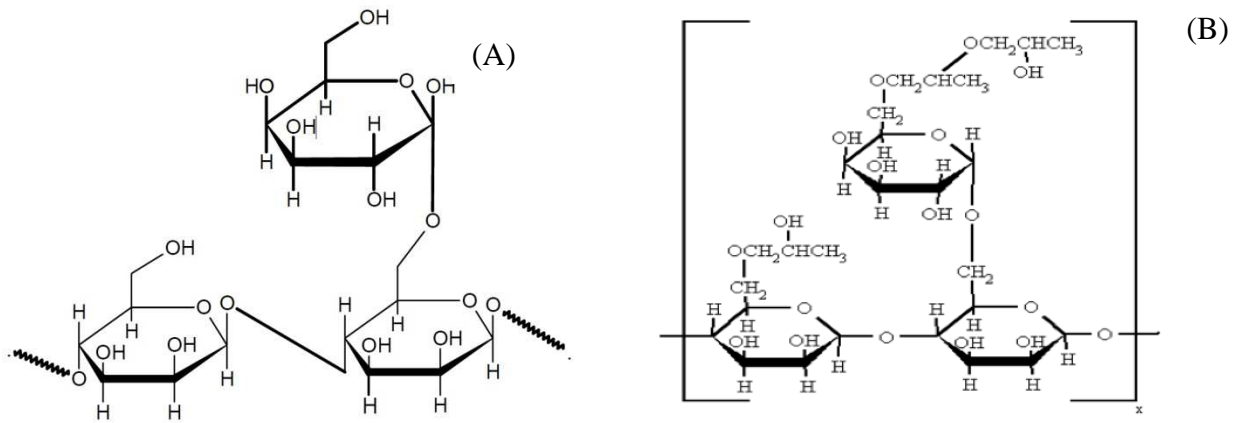


Fig. 1-Molecular structure of native guar gum (A) and hydroxypropylguar (B).

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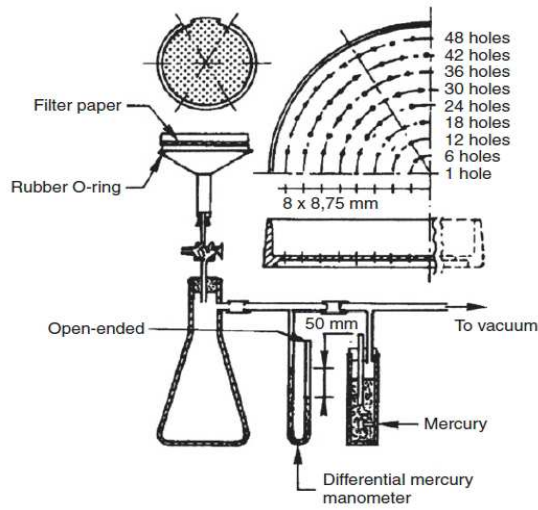


Fig. 2-Experimental device to measure water retention according to ASTM C91 standard.

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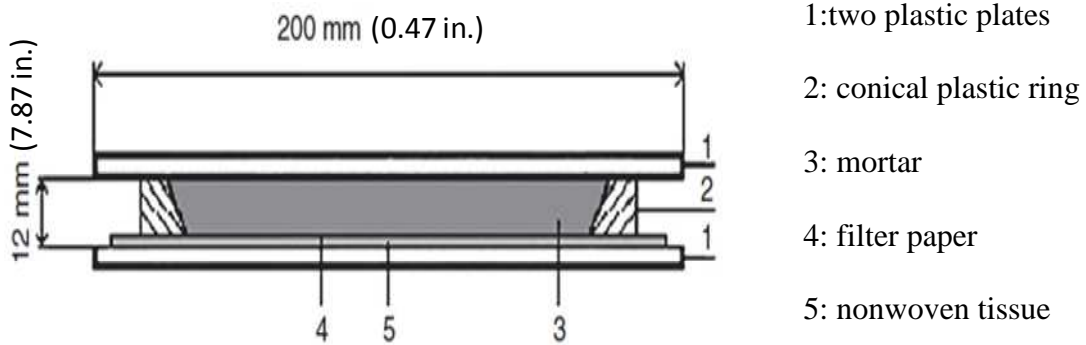
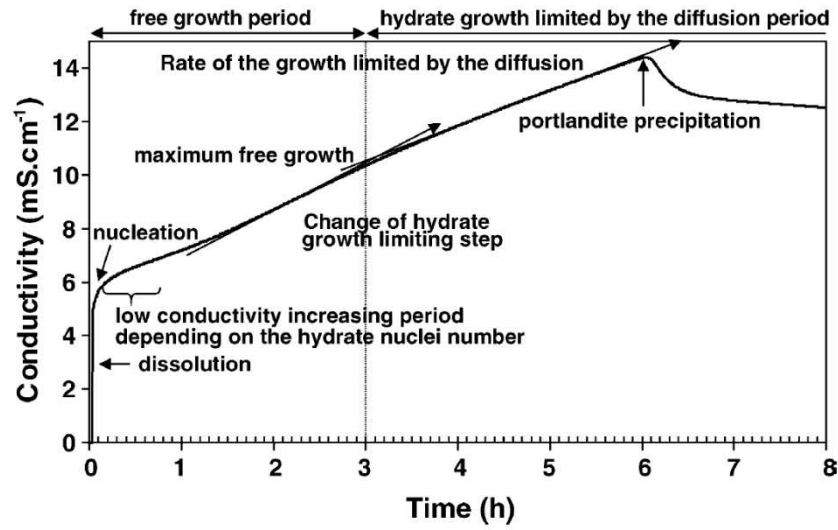


Fig. 3-Experimental device to measure water retention according to DIN 18555-7

standard.

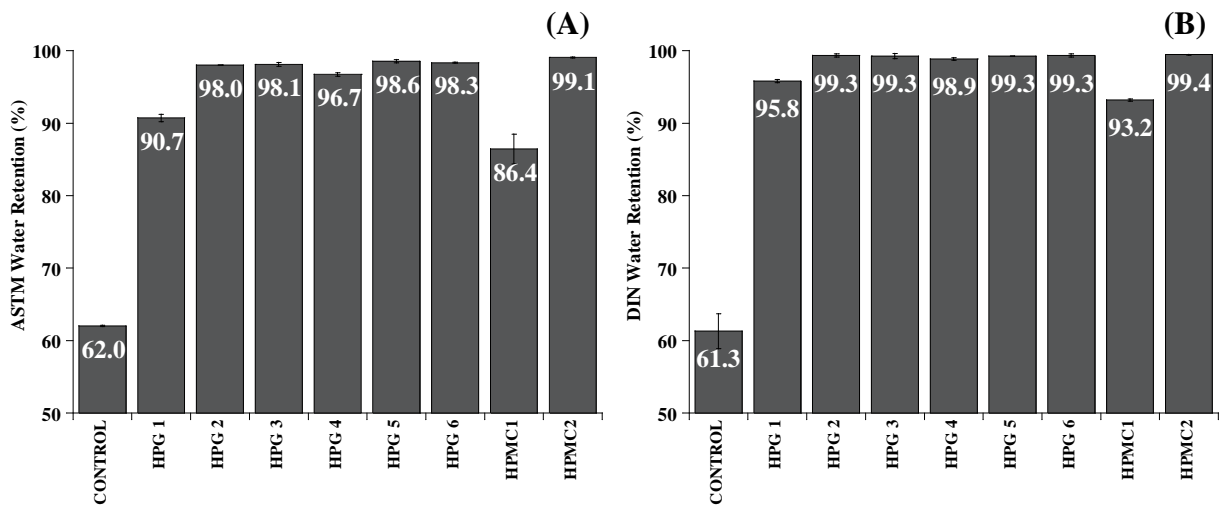
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3 **Fig. 4-Global view of hydration mechanism of cement in lime solution (liquid to solid**
4 **ratio equal to 20) on a conductimetric curve [14]**

5



6 **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).

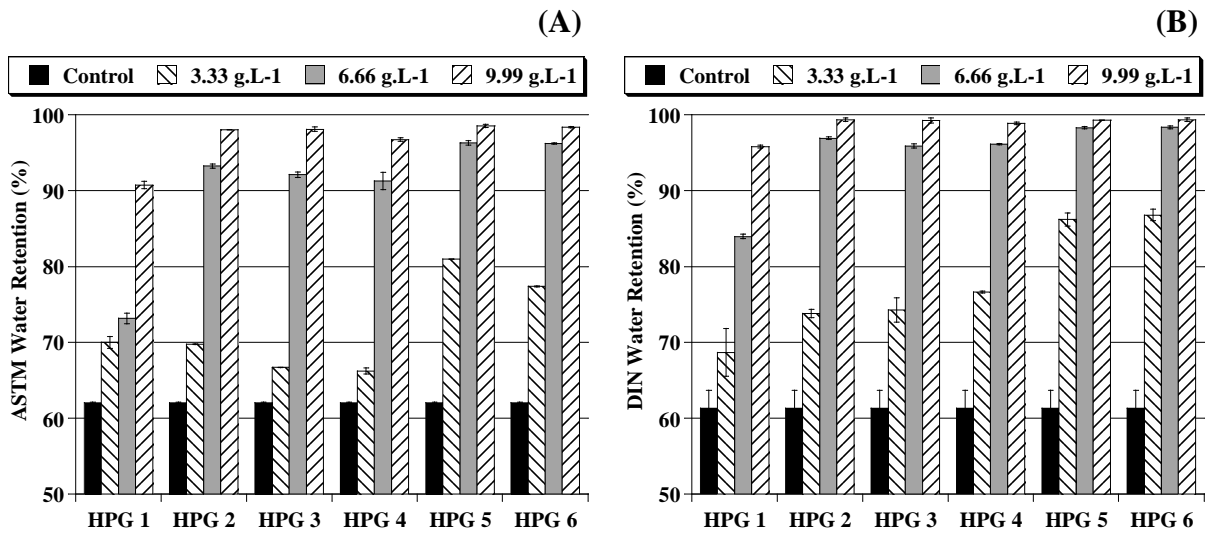


Fig. 6–Influence of polymer concentration on water retention (A=ASTM ; B=DIN).

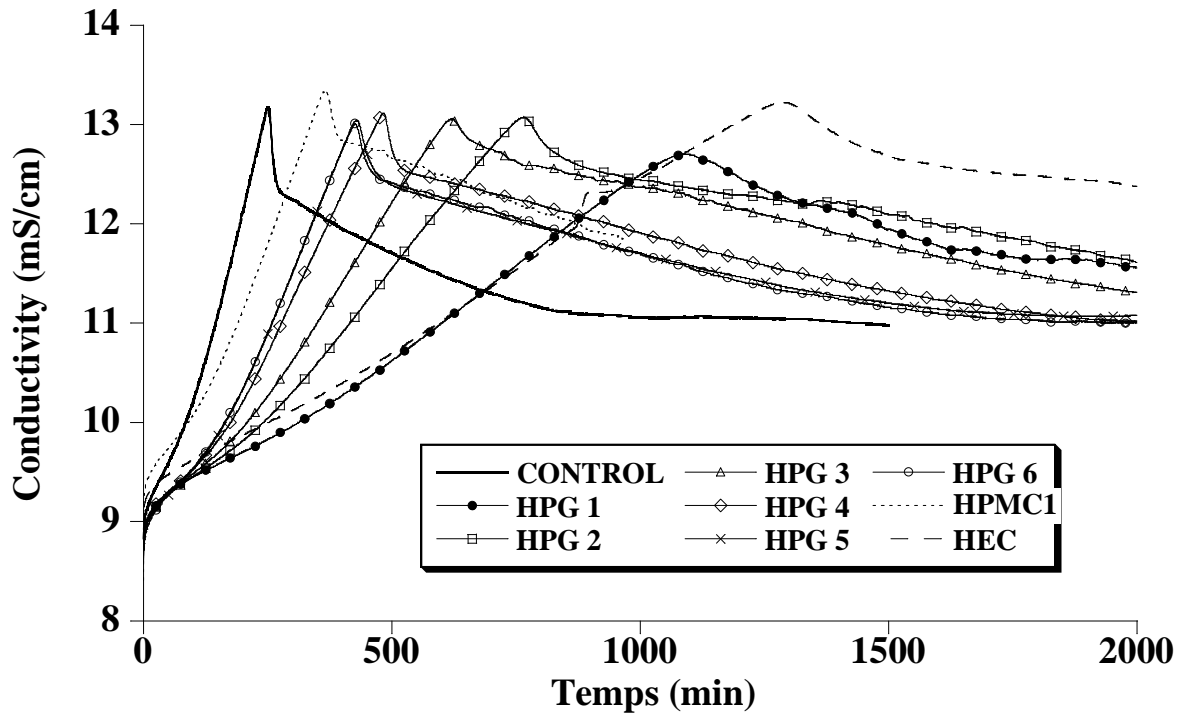
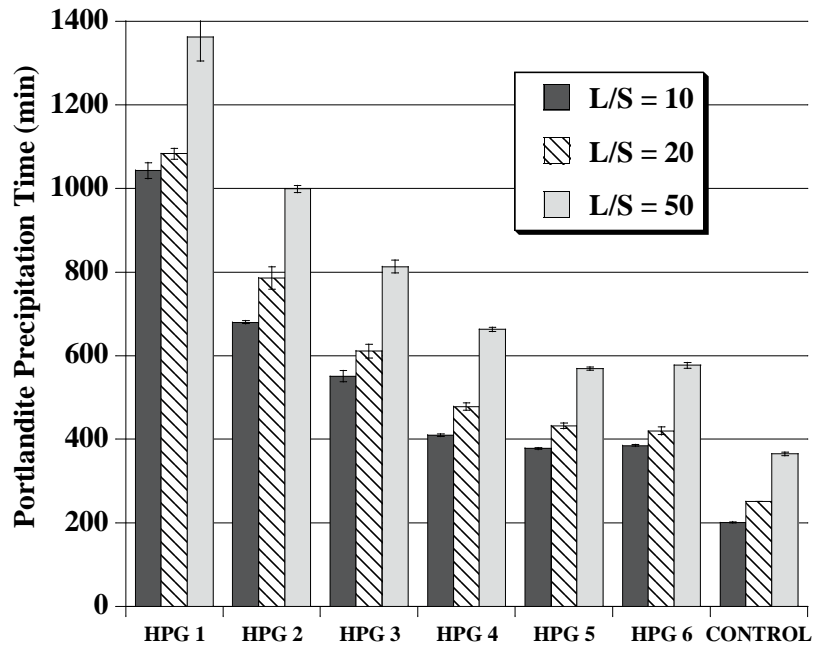


Fig. 7–Conductimetric curves of cement admixed with polysaccharide in lime solution (L/S=20, P/C=2%).

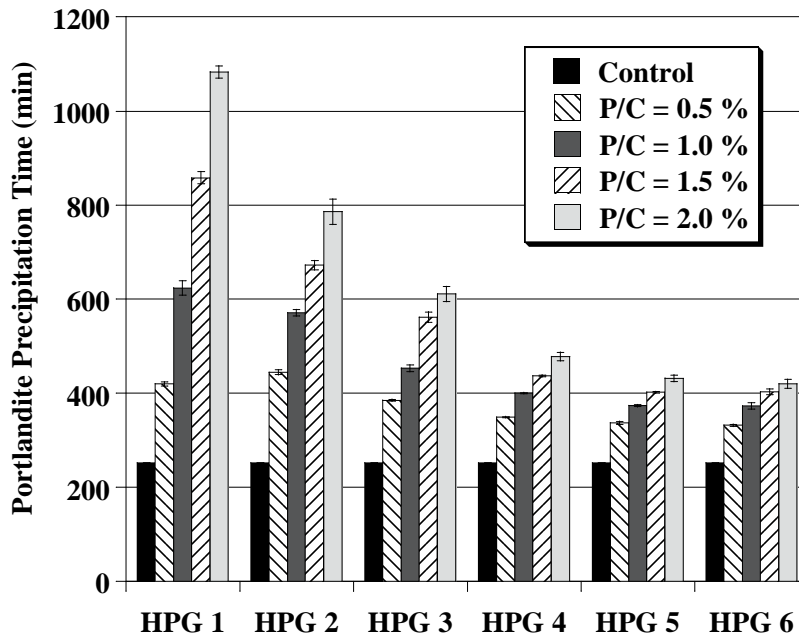


1

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

2

3



4

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

5

6

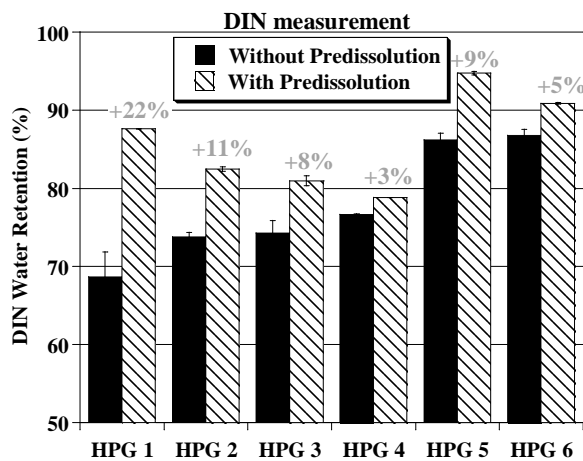
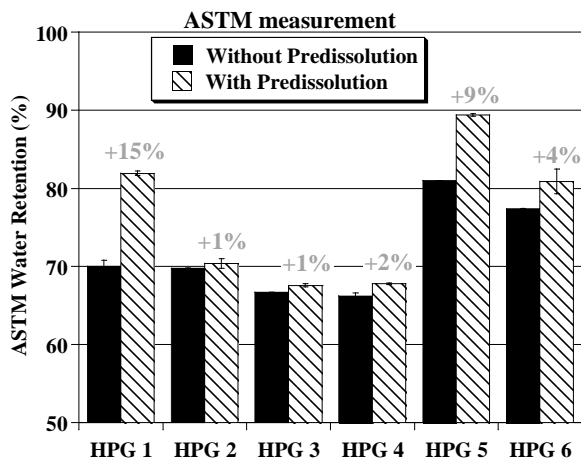


Fig. 10–Influence of predissolution on water retention capacity with [P] = 3,33 g.L⁻¹.

1

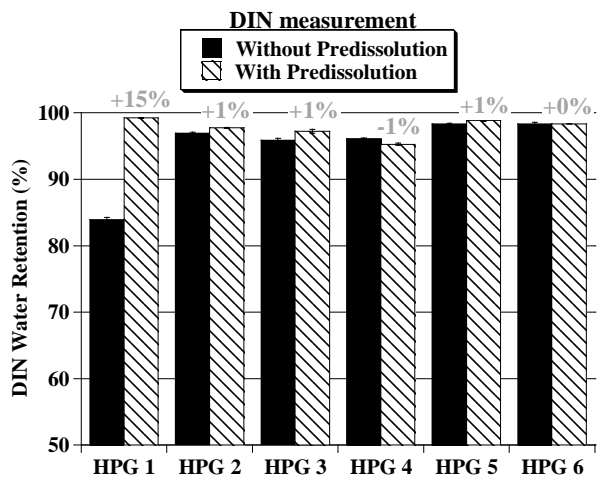
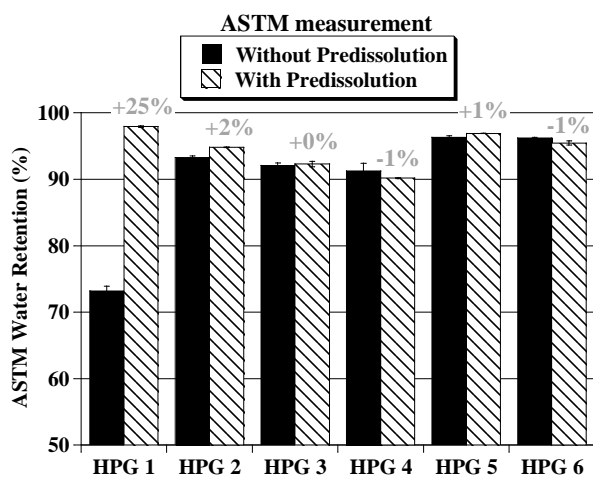
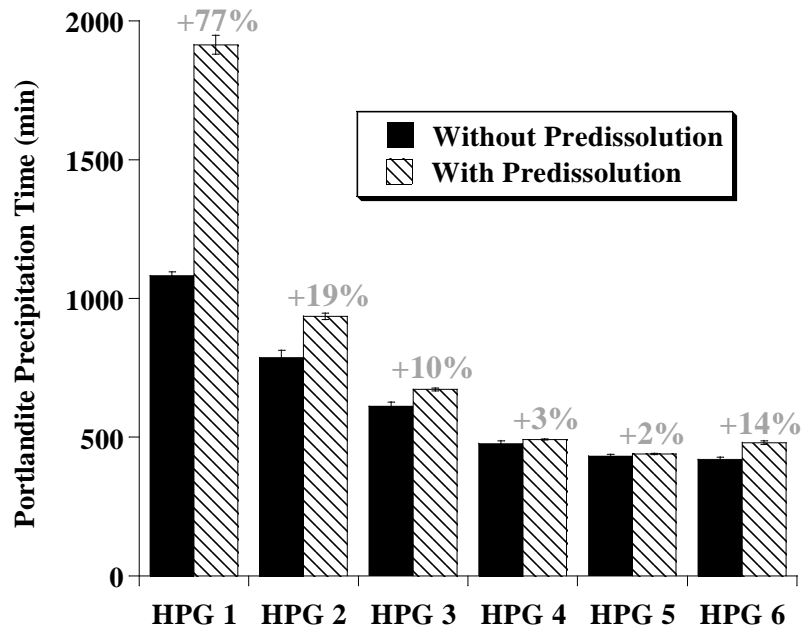


Fig. 11–Influence of predissolution on water retention capacity with [P] = 6,66 g.L⁻¹.

2



1

2

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

3

4