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Effect of polysaccharides on the hydration of cement paste at early ages

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Abstract

This work deals with the relative efficiency of polysaccharides and their influence on cement hydration. Several parameters such as the structure, concentration, average molecular weight, and soluble fraction value of polysaccharides were examined. Cement hydration was monitored by isothermal calorimetry, thermogravimetry (TGA), and infra-red spectroscopy (FTIR). Results clearly show that retardation increases with higher polysaccharide-to-cement weight ratio (P/C). Low molecular weight starch showed enhanced retarding effect on the hydration of cement. The retardation effect of polysaccharides is also dependant on the composition of cement.

Keywords :. Hydration ; Polymers ; Polysaccharides ; Retardation ; Cement Paste

1. Introduction

Organic admixtures have been widely used in concrete and mortar for several decades, providing advanced specific properties needed during the construction. The European market of coatings is changing to the advantage of the remodelling market. Mortars for this application require a water retention value of at least 95% to prevent water absorption. To obtain this value, polysaccharides which can be equally classified as water-reducers and set-retarding agents, are frequently used. These admixtures can be either native bio-polymers (e.g. starch) or substituted polymers (e.g. cellulose ethers). Dewacker et al. [1] suggested that a blend of polysaccharides such as cellulose and starch was a good water retention agent.

These admixtures are also set-retarding agents which improve workability duration and modify cement hydration. Yamamuro [2] showed that a polysaccharide derivative containing an ionic functional group and a hydrophobic group increases the viscosity of cement suspensions. In the Shotcrete process, Ghio et al. [3,4] showed that concretes formulated with polysaccharides are easier to pump and to spray at high shear rates than unadmixed concrete. Moreover, concrete is more cohesive and viscous at low shear rates. Tanaka et al. [5] patented an additive that contains at least one sulfated polysaccharide to improve fluidity and workability and that supplies higher final compressive strength. Hayakawa et al. [6,7] reported that cellulose ether improves binding between cement matrix and aggregates.

Previous investigations on physico-chemical aspects were restricted to the effect of monosaccharides or sugar acids on cement or single phase hydration [8-11]. Influence of such admixtures on Calcium aluminate phase (C_3A) was studied by Young [8] who showed that, interaction of sugars on C_3A prevents rapid formation of the cubic phase C_3AH_6 and promotes formation of the hexagonal phase C_4AH_{13} . Colleparidi et al. [9,10] found that addition of glucose, gluconate or lignosulfonate stabilizes ettringite in the C_3A -gypsum system. It was also noted that glucose retards gypsum consumption and ettringite formation.

Many studies on the hydration of silicate phase in presence of admixtures were devoted to the determination of calcium hydroxide content. According to Milestone [11] the retarding effect is as follows: sugar acids > sugars > lignosulfonates. Scanning electron microscopic (SEM) examination showed that in presence of these admixtures, calcium silicate phase (C_3S) particles are covered by flaky calcium silicate hydrate (CSH), instead of acicular hydrates [11].

Bensted et al. [15] investigated the effect of lignosulfonate on the hydration of cement and found that retardation is linked either to a preferential complexation with calcium and silicon or to an introduction of organic admixtures into a precipitated gel. This latter hypothesis postulates that the retarding effect would come from the formation of a less permeable gel coating around cement grains. However the mechanism of cement-admixture interaction is yet to be fully understood.

The present study was undertaken to determine the relative efficiencies of polysaccharides and to reveal the mechanism of interaction between cement particles and the admixture at the early stages of hydration.

2. Experimental

2.1. Raw materials

White Portland cement (C_1): CPA CEM I 52.5, and grey cement (C_2): PMES 42.5 were used in this study. The designation follows French standard NF P 15-301. Chemical composition and mineralogical phases of the two cement samples were calculated by the Bogue approximation [12], and these are listed in Table 1.

Five powder polysaccharides, a cellulose ether (CE), a starch ether (SE), a native starch (NS), a white dextrin (WD), and a yellow dextrin (YD) both prepared from NS, were tested. Properties of these polysaccharides are given in Table 2.

2.2. Methods of investigation

Isothermal microcalorimetry, TGA and FTIR spectrometry were used to monitor kinetics and mechanism of cement hydration.

Each experiment was conducted in triplicate. In each case, cement and polysaccharides were mixed for 2 periods of 2 minutes using a shaker-mixer (Wab, Turbula, Germany). Unless otherwise stated, polysaccharide to cement weight ratio (P/C) was equal to 0.5 (w/w).

Deionized water to cement ratio (W/C) was approximately 0.3 and was adjusted in order to have a constant workability. The W/C values of C₁, C₁+CE, C₁+SE, C₁+NS, C₁+WD, C₁+YD, C₂, C₂+YD were 0.282, 0.265, 0.342, 0.295, 0.295, 0.267, 0.215, 0.225 respectively. Cement pastes were obtained by using a mixer (Technotest, France) for one minute at low speed (60 rpm) and four minutes at high speed (120 rpm). Samples were stored in a water vapour saturated desiccator and under nitrogen atmosphere. Cement hydration was stopped by breaking and grinding hydrated samples and washing them with anhydrous ethanol. Samples were ground in an agate mortar until each particle diameter became lower than 100 µm. This step was reproduced at different lengths of time ranging from 1 to 24 hours.

Thermal analysis was performed on a TG-DSC 111 thermogravimetric apparatus (Setaram, France). All experiments were conducted from 20°C up to 800°C (heating rate of 10°C/min) under dynamic argon atmosphere on a approximately 25 mg samples.

The amount of portlandite CH(%) was obtained directly from TG curves by Eq.1

$$CH(\%) = WL_{CH}(\%) \times \frac{MW_{CH}}{MW_H} \quad (1)$$

where $WL_{CH}(\%)$ corresponds to weight loss in percent occurring during CH dehydration, MW_{CH} and MW_H are molecular weights of portlandite and water respectively.

Infra-red spectra were collected with a spectrometer (Biorad FTS 185 Digilab, USA) equipped with a Harrick scattering reflexion accessory. Analysis was conducted on hydrated samples diluted at 5% in KBr. Spectra were converted by the Kubelka-Munk algorithm.

Admixtures can be classified in retarders or accelerators category using isothermal microcalorimetry [13]. It enables the hydration process to be monitored continuously. It was conducted on a C80 calorimeter (Setaram, France). Temperature of the calorimetric block was maintained at 25°C and the experiments were conducted on a 250 mg sample at a W/C ratio of 0.4. Cement and water mixing was done outside the calorimeter according to the procedure described by Sauvat et al. [14] and Ramachandran et al. [15]. Consequently, only the exothermic hydration peak was encountered.

3. Results and discussion

3.1. Hydration of C₁

Investigations were first focused on C₁ since this kind of cement is frequently used in coating formulas. The calorimetric curve of neat cement exhibits two separate exothermic peaks. According to Sauvat et al. [14] they correspond to C₃A and C₃S hydration peaks respectively whereas Bensted et al. [16] suggested the reverse order. Retarding ability of polysaccharides on cement hydration is obtained from curves of heat evolution rate. Three different times were defined as proposed by Sauvat et al. [14], t_1 , t_2 , t_3 which correspond to minimum flux time, second peak time and third peak time respectively. Heat released in 24 hours of hydration was calculated by curve integration from t_1 to t_{24h} (Table 3). Intensity and shape of the exothermic peaks are not significantly modified by CE and NS addition. Intensity of heat flow for cement formulated with SE is lower. The cement admixed with YD presents a

larger wetting peak which induces a shift of the minimum heat flow to 10 hours. Moreover, the heat released during the first 24 hours of hydration is smaller. These results confirm previous studies regarding effects of set-retarding agents on cement hydration [14,15]. Ramachandran et al. [15] found that sucrose is an efficient retarder which extends induction period for several hours. Similar result was obtained with YD in the present study.

Previte et al. [17] suggested that alkali-stable and high molecular weight sugars are more efficient to retard hydration. It should be noted that oligomers used by these authors had a maximum chain length of 3 units. To extend this previous study, the present results focus on polysaccharides with a much higher degree of polymerization. Polysaccharides with a strong set-retarding ability i.e., SE and YD have a lower average molecular weight than CE and NS. A weak average molecular weight favors alkali-degradation of polysaccharides [18-19]. Several authors have shown that products from decomposition of sugar are more effective than native sugars [15,20].

Differential Thermal Analysis (DTA) and TGA were used to study the effects of sugars and hydroxycarboxylic acids on hydration of different cement phases. As Bensted et al. [21] and Odler et al. [22] suggested, it is possible to assign the first two peaks at 120°C and 145°C to ettringite and gypsum decomposition respectively (Fig. 1a). Beyond 4 hours of hydration, decomposition peaks of CSH and ettringite overlap. It is also possible to monitor gypsum consumption until it completely disappears since no other phase interferes with it. Decomposition peak of CH occurs between 420°C and 460°C.

The C₁+SE formula revealed a smaller amount of ettringite and a smaller gypsum consumption than C₁ (Fig. 1b). However the admixture YD accelerated gypsum consumption and ettringite formation (Fig. 1c) as compared with C₁. It can be at the origin of the wetting peak broadening on the calorimetric curve. Between 4 and 8 hours of hydration, the ettringite

phase seems to be stabilized and gypsum is no longer consumed for both formulas. Moreover, decomposition peak of portlandite is less intense for C₁+SE and C₁+YD. Further information is obtained by calculation of portlandite content as specified by Eq. 1. Portlandite formation depends strongly on the type of polysaccharide introduced in cement (Fig. 2). YD stops portlandite formation during 8 hours. Beyond this period the formation rate increases and finally portlandite content is very close to those of pure cement. Conversely, CE and NS have had no significant effect on gypsum consumption, on ettringite as well as on portlandite formation (Fig. 2).

A smaller amount of portlandite could be associated with a greater carbonation as proposed by Silva et al. [23] for cement admixed with ethylene/vinyl acetate copolymer. Weight losses occurring during decarbonation were compared. Carbonation does not significantly increase upon polysaccharide incorporation. Thus, at a given time the presence of admixtures YD and SE induces a smaller content of portlandite.

CSH formation was monitored by infra-red spectroscopy since monitoring by thermal analysis at early ages is not possible. CSH is usually observed by a gradual shift of band centered at 925 cm⁻¹ to 970 cm⁻¹, which is assigned to the asymmetrical SiO₄ stretching frequency [21]. It indicates polymerization of silicate units SiO₄⁴⁻ with formation of CSH phase [24]. CSH has already been formed for C₁ and C₁+CE at 8 hours of hydration (Fig. 3). On the contrary, C₁+SE and C₁+YD revealed no shift attributed to CSH formation.

Results obtained from these studies are consistent. CE has no significant effect on hydrate formation. YD extends induction period, favours ettringite formation at very early ages and retards CSH and CH formation. SE slows down ettringite formation and gypsum consumption and also delays considerably CSH and CH formation.

3.2. Influence of SE and YD concentration on cement hydration

Results obtained by isothermal microcalorimetry for an increasing amount of polysaccharides are listed in Table 4. It shows that t_1 and t_2 are not significantly modified by an increase of SE concentration. Time t_3 is not defined for SE concentration over 0.5%. As YD concentration rises, t_1 and t_2 are shifted to later times. In both formulas, evolution of heat released is inversely proportional to concentration. Results of C_1+YD are consistent with those of Ramachandran et al. [15] who proposed a linear relation between concentration of retarders and duration of induction period.

3.3 Study on the admixture YD

3.3.1 Influence of cement composition on retarding ability of YD

In order to determine the effect of cement composition and especially C_3A content on set-retarding ability of polysaccharides, cement C_2 was studied. For this experiment admixture YD was used, as it has a strong retarding effect on cement hydration. As illustrated in (Fig. 4a), gypsum was totally consumed for pure C_2 after 16 hours of hydration. Introduction of YD revealed a decomposition peak at $145^\circ C$ attributed to gypsum up to 24 hours (Fig. 4b).

Portlandite formation rate is higher for C_1 than C_2 (Fig. 5). It can be associated with a higher C_3S content for C_1 . C_3A amount seems to be an important parameter since 0.5% of YD totally inhibits portlandite formation up to 48 hours. Present results are similar to those of Singh [24]. Monosi et al. [26] found that lignosulfonate totally stops C_3S hydration up to two weeks without C_3A . The inhibiting effect lasts only one day with C_3A . It reveals the importance of C_3A content in cement which acts as a sink for retarders [15].

C₂ hydrated with 0.5% YD suggests an excess of admixture with respect to cement. P/C₃A ratio for this formula is 0.2 instead of 0.045 for C₁+0.5% YD. In order to verify the fact that an excess of YD with respect to C₂ was introduced, an experiment on C₁+3% YD was carried out (ratio P/C₃A equal to 0.25). Results obtained with this formula are similar to C₂+0.5% YD, i.e., portlandite formation was completely inhibited.

3.3.2 Influence of dextrinization on set retarding ability of starch compounds

WD and YD were obtained from NS by dextrinization which is a combination of an acid and a thermal treatment. It allows starch solubilization by a decrease of average molecular weight. Soluble fraction value increases with degree of conversion of starch to dextrin (Table 2). Thermal analysis is performed on C₁ with 0.5% of admixture. YD delays portlandite formation whereas NS and WD have no effect (Fig. 6).

Average molecular weights and soluble fraction of starch derivatives are given in Table 2. At a given P/C ratio, YD concentration in liquid phase is higher than WD and NS concentration. Portlandite rate formation is more dependent on soluble fraction than on molecular weight of compounds. Dextrinization of NS is a predominant parameter on the retarding ability of starch derivatives.

5. Conclusions

Results obtained by thermal analysis show that the introduction of a small amount of polysaccharide (0.5% by weight of cement) induces a strong modification of cement

hydration. Retarding ability is dependent on polysaccharide variety and is as follows: NS < WD < CE < YD < SE.

A mechanism which describes cement-polysaccharide interaction is difficult to establish. The first step of C₃A hydration leading to ettringite is accelerated by YD and retarded by SE. These polysaccharides then stabilize ettringite and gypsum content. This observation will induce a delay in conversion of ettringite to monosulfoaluminate phase. From these results, the action of polysaccharides on C₃A hydration seems more restricted to growth of hydrates than to nucleation. Consequently the origin of retardation could be linked to an adsorption of admixtures on the first hydrates forming a less permeable coating. As far as silicate hydration is concerned SE and YD (most effective polysaccharides) slow down and delay formation of both CSH and portlandite respectively.

Polysaccharide retarding effect depends on cement composition. C₃A content might be considered as a key parameter. Extent of retardation is higher for cement with low C₃A content which is in accordance with authors [15,25-26]. The retarding effect depends also on the chemical structure of admixtures. With respect to starch compounds, YD with a higher soluble fraction than NS and WD is more effective to retard cement hydration. A study with polysaccharides which have only one variable parameter, like degree of polymerization or degree of substitution, would produce valuable results in terms of admixture selection and properties prediction. Experiments on pure cement phase would provide useful information. It could be a first step to model pure phase hydration in presence of polysaccharide.

Acknowledgements

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Table 1

Chemical composition	C ₁	C ₂
SiO ₂	22.2	21.9
TiO ₂	0.2	0.4
Al ₂ O ₃	4.4	3.8
Fe ₂ O ₃	0.3	4.3
CaO	67.2	62.8
MnO	0.01	0.04
MgO	0.5	2.2
Na ₂ O	0.4	0.4
K ₂ O	0.05	0.39
P ₂ O ₅	0.11	0.26
SO ₃	3	2.6
Bogue approximation	C ₁	C ₂
C ₃ S	66	51
C ₂ S	14	24
C ₃ A	11	2.6
C ₄ AF	1	13.2

Table 1 Chemical composition of cement and potential phases as determined by Bogue approximation.

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Table 2

Admixture	Solubility at 25°C	Mw (Daltons) ^a	Substituent ^b
CE	Yes	$(11.6 \pm 0.1)10^6$	CH ₃ , (CH ₂) ₃ -OH and (CH ₂) ₂ -OH
SE	Yes	$(1.48 \pm 0.05)10^6$ $(173 \pm 5)10^3$	(CH ₂) ₃ -OH
NS	Nd	$25.2 \cdot 10^6$ $(290 \pm 6)10^3$	-
WD	5% < S < 35%	$(11 \pm 1)10^3$	-
YD	S > 90%	$(4.5 \pm 0.1)10^6$ $(8.5 \pm 0.5)10^3$	-

^a determined by Gel Permeation Chromatography

^b determined by Pyrolysis-Gas Chromatography-Mass Spectrometry

S : soluble fraction value (producers data)

Table 2 Properties of polysaccharides.

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Table 3

Formula	t_1 (hours)	t_2 (hours)	t_3 (hours)	Q_{24h} (J/g)
C_1	2.4 ± 0.3	7.2 ± 0.3	11.2 ± 0.6	99 ± 5
C_1+CE	3.5 ± 0.5	9.5 ± 0.7	13.5 ± 0.7	97 ± 5
C_1+SE	2.2 ± 1.5	8.2 ± 2.1	14.5 ± 4.1	60 ± 8
C_1+NS	2.9 ± 0.2	7.7 ± 0.1	11.4 ± 0.1	94 ± 7
C_1+YD	7.9 ± 0.4	18.2 ± 1.1	-	9 ± 1

Table 3 Times and heat released in 24 hours as a function of polysaccharide added.

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Fig 1a

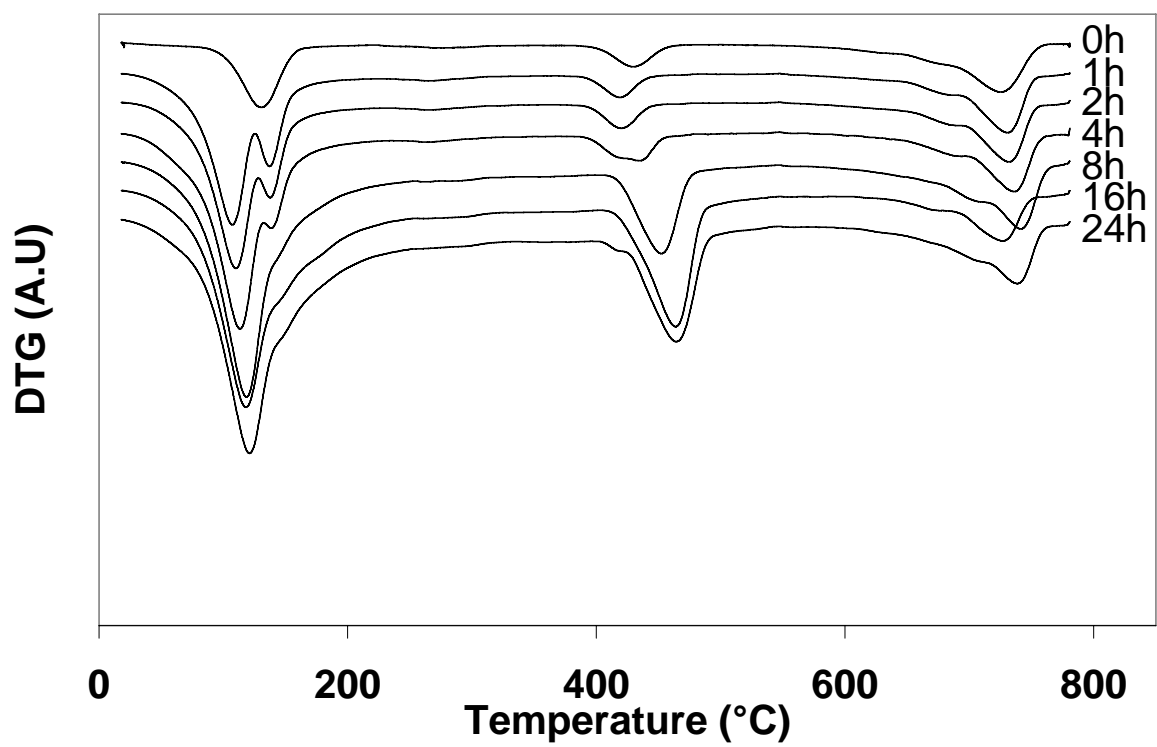


Fig 1a DTG curves of C₁

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Fig 1b

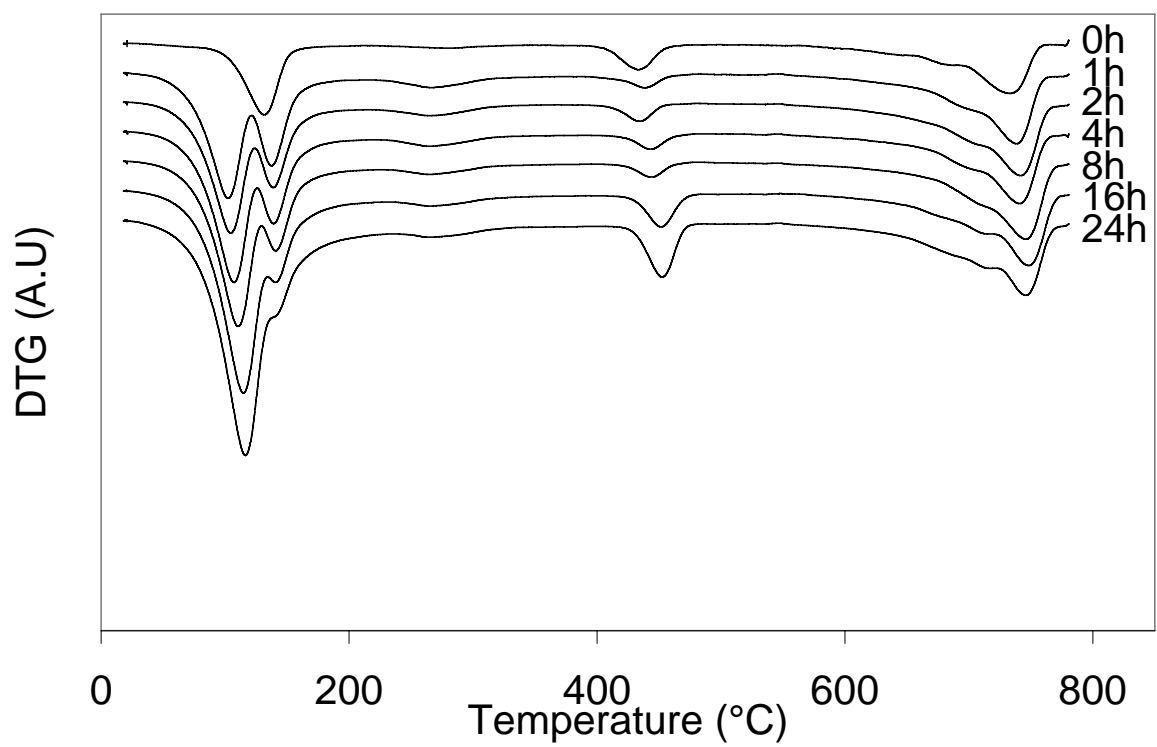


Fig 1b DTG curves of C₁+SE

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Fig 1c

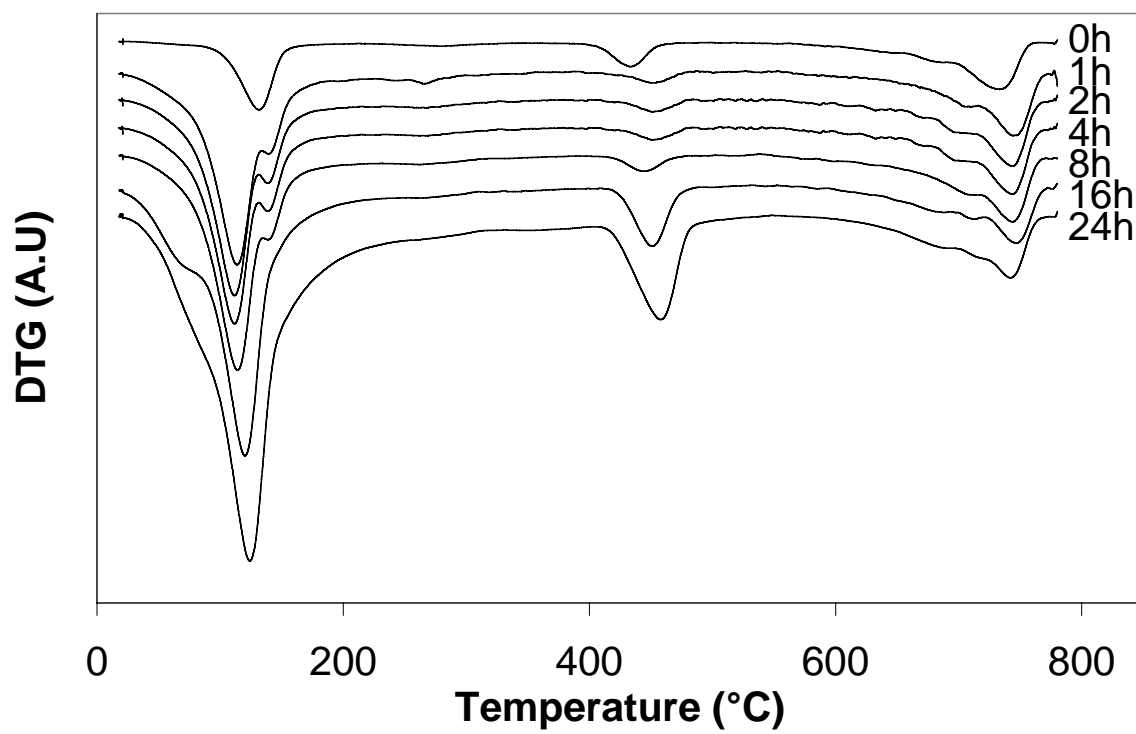


Fig 1c DTG curves of C₁+YD

A.Peschard, A.Govin^{*}, P.Grosseau, B.Guilhot, R.Guyonnet

Fig 2

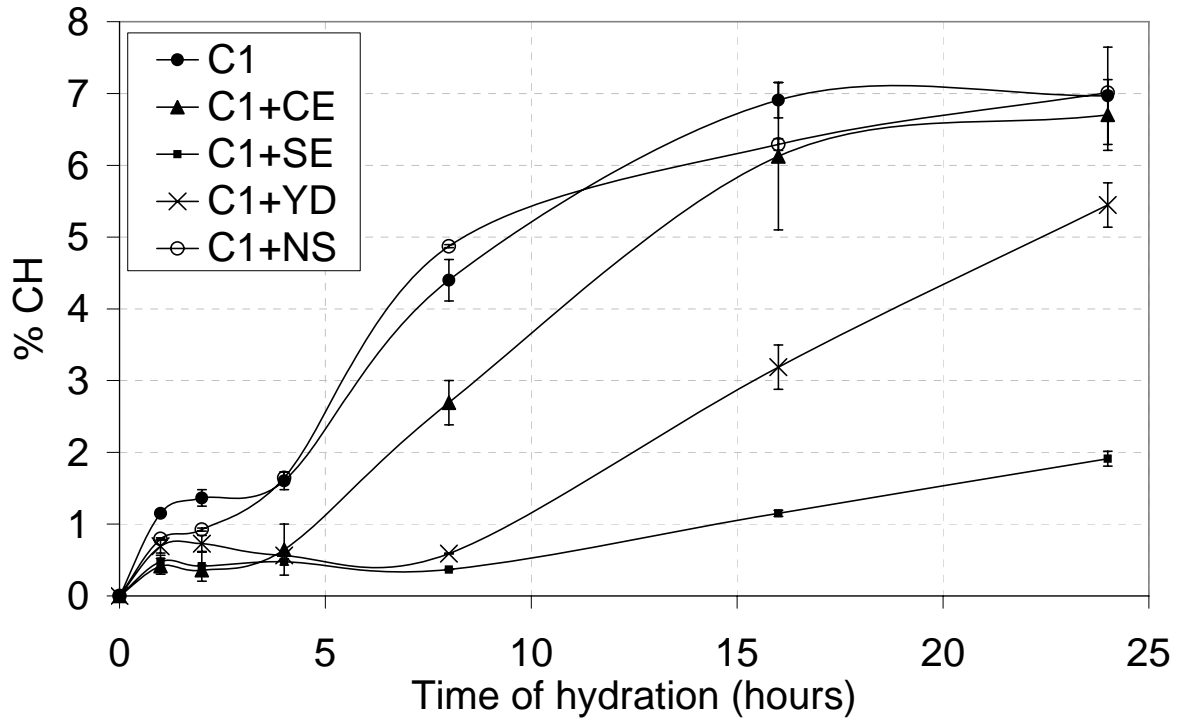


Fig 2 Evolution of portlandite content in admixed cement pastes

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Fig 3

Kubelka-Munk

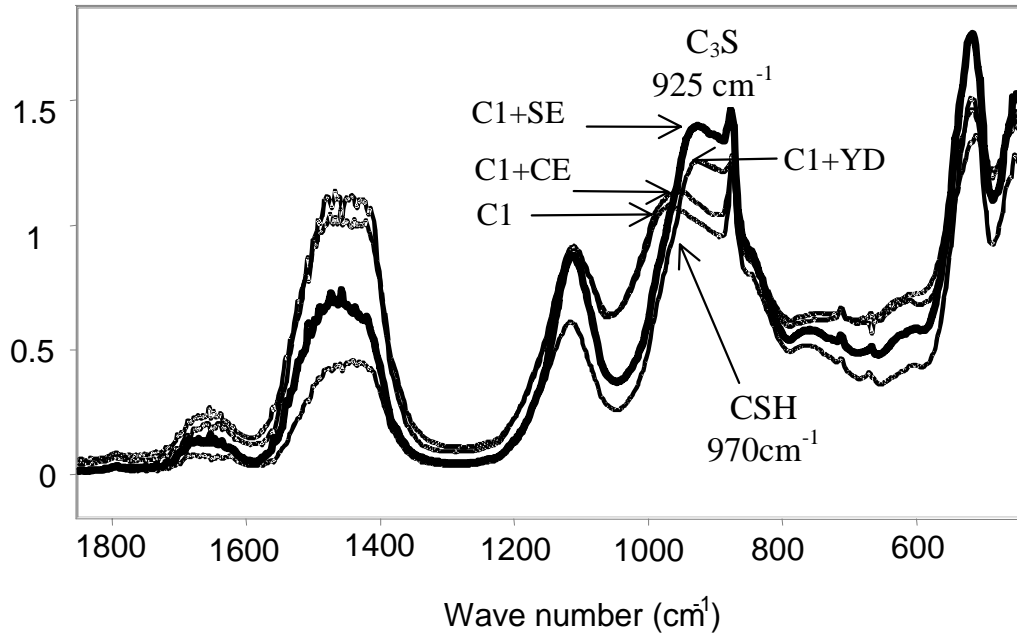


Fig 3 FTIR spectra of hydrated admixed cements.

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Table 4

Formula	t ₁ (hours)	t ₂ (hours)	t ₃ (hours)	Q _{24h} (J/g)
C ₁	2.4 ± 0.3	7.2 ± 0.3	11.2 ± 0.6	99 ± 5
C ₁ + 0.5% SE	2.2 ± 1.5	8.2 ± 2.1	14.5 ± 4.1	60 ± 8
C ₁ + 1.2% SE	3 ± 0.2	7.8 ± 0.4	-	45 ± 5
C ₁ + 1.5% SE	3 ± 0.2	7.9 ± 0.3	-	28 ± 5
C ₁ + 2% SE	3.8 ± 0.3	7.9 ± 0.4	-	8 ± 1
C ₁ + 0.1% YD	2.2 ± 0.2	8.0 ± 0.3	12.2 ± 0.5	78 ± 5
C ₁ + 0.3% YD	4.6 ± 0.3	15.0 ± 0.5	-	48 ± 5
C ₁ + 0.4% YD	7.2 ± 0.3	16.4 ± 0.8	-	37 ± 4
C ₁ + 0.5% YD	7.9 ± 0.4	18.2 ± 1.1	-	9 ± 1

Table 4 Times and heat released in 24 hours for increasing concentration of SE and YD.

A.Peschard, A.Govin^{*}, P.Grosseau, B.Guilhot, R.Guyonnet

Fig 4a

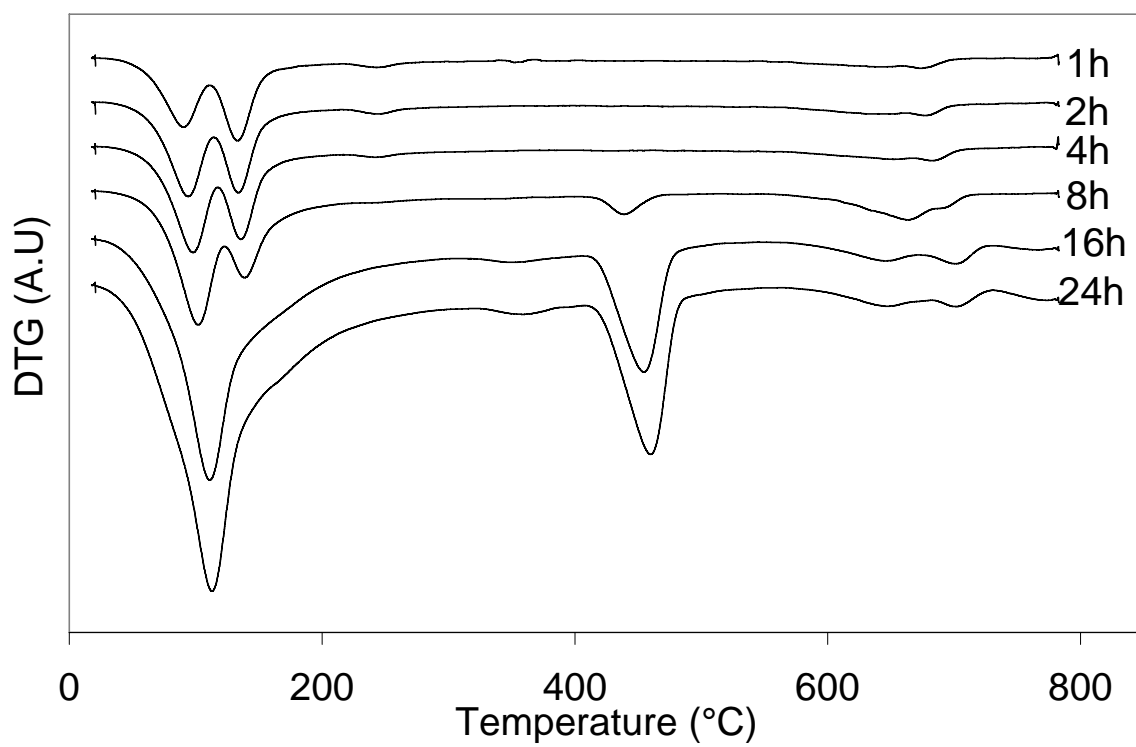


Fig 4a DTG curves of C₂

A.Peschard, A.Govin^{*}, P.Grosseau, B.Guilhot, R.Guyonnet

Fig 4b

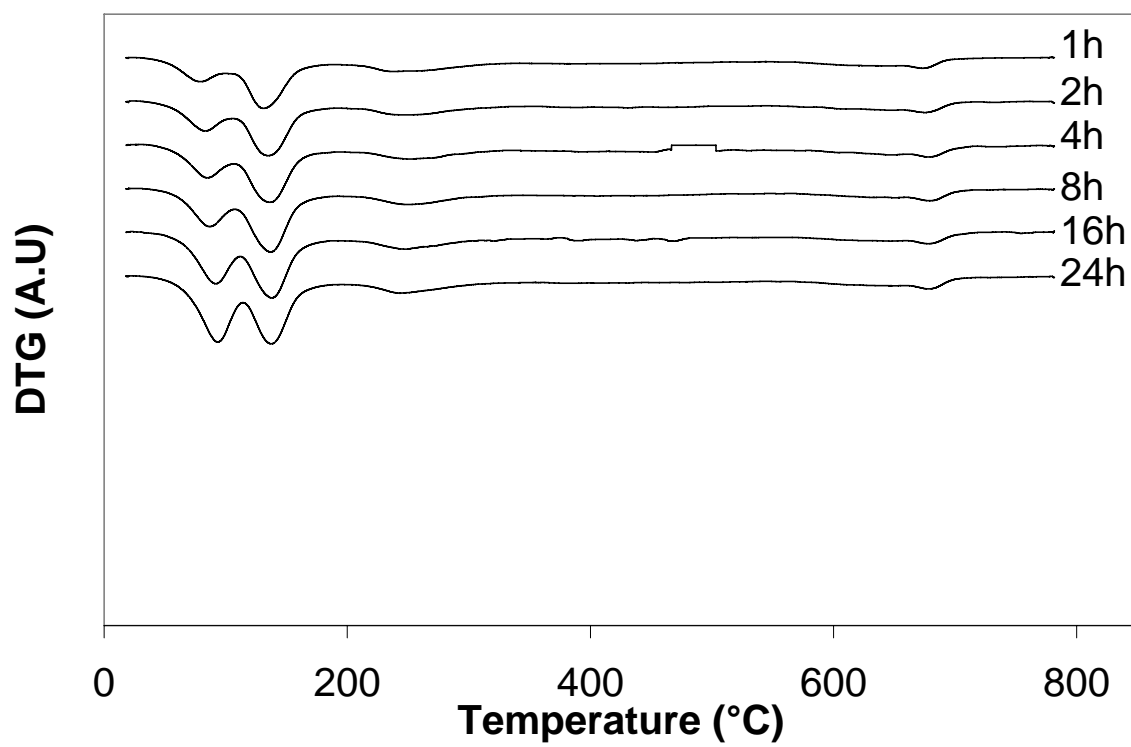


Fig 4b DTG curves of C₂+YD

A.Peschard, A.Govin^{*}, P.Grosseau, B.Guilhot, R.Guyonnet

Fig 5

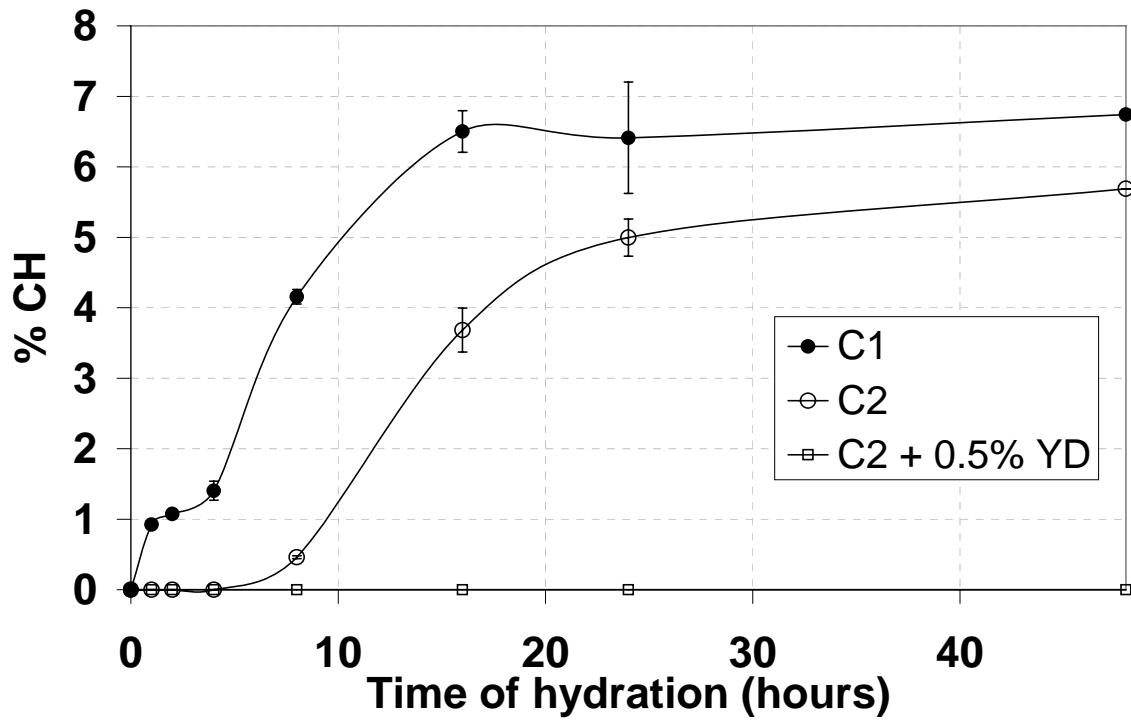


Fig 5 Effect of cement composition on the retarding ability of YD.

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Fig 6

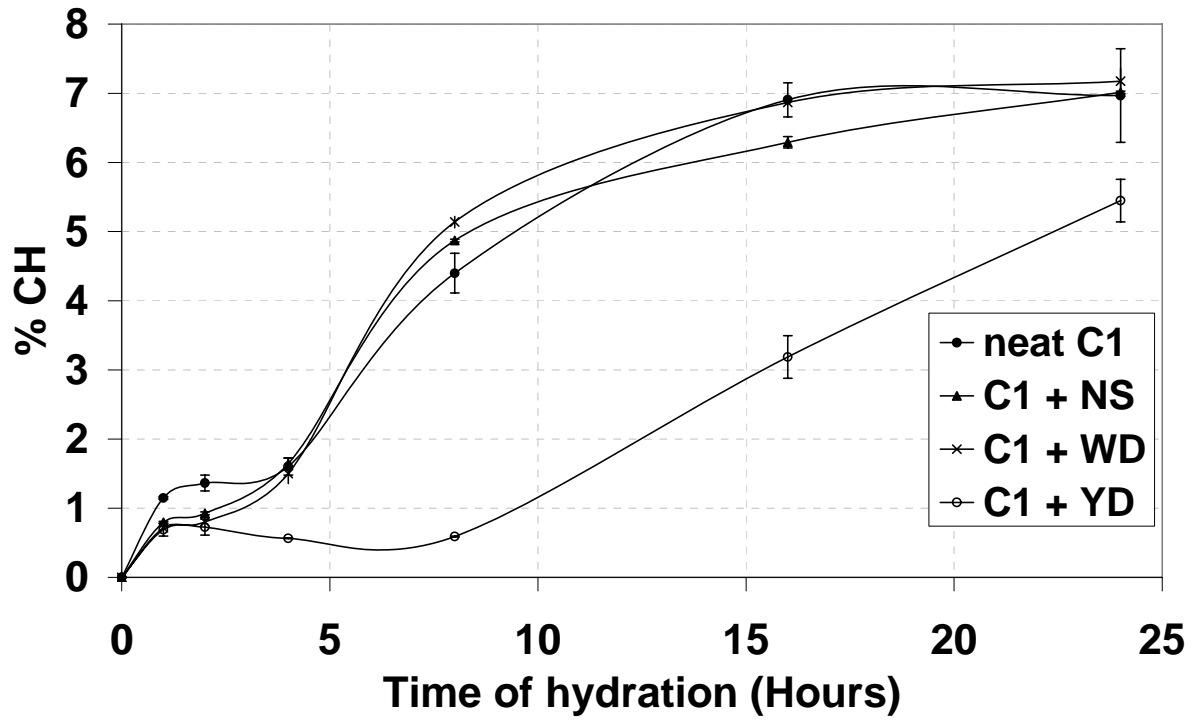


Fig 6 Effect of starch dextrinization on the evolution of CH formation in C₁.

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Captions

Table 1 Chemical composition of cement and potential phases by Bogue approximation.

Table 2 Properties of polysaccharides.

Table 3 Times and heat released in 24 hours as a function of polysaccharide added.

Fig 1 DTG curves of a C_1 , b C_1+SE , c C_1+YD .

Fig 2 Evolution of portlandite content in admixed cement pastes

Fig 3 FTIR spectra of hydrated admixed cements.

Table 4 Times and heat released in 24 hours for increasing concentration of SE and YD.

Fig 4 DTG curves of a C_2 , b C_2+YD .

Fig 5 Effect of cement composition on the retarding ability of YD.

Fig 6 Effect of starch dextrinization on the evolution of CH formation in C_1 .