Modification of cement hydration at early ages by natural and heated wood

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Abstract

Heat treatments of wood are widely used for the reduction of wood swelling and dimensional instability of wood–cement composites. The effect of natural and heated wood on the hydration of cement at early ages was investigated by isothermal calorimetry, thermogravimetry (TGA) and Fourier transform infrared (FTIR) spectroscopy. The addition of wood strongly delays and inhibits the hydration of the silicate phases. Consequently, the amount of portlandite is lower in composites than in neat cement. Approximately 30% of the inhibition of portlandite precipitation is due to an increase of calcium carbonate content in cement paste. The absorption of a part of water by wood produces a decrease in gypsum consumption. Nevertheless, the ettringite formation is not significantly affected since a diffusion of sulfate ions from wood occurs.

Keywords:

Hydration; Cement; Wood; Heated wood; Retardation

I. Introduction

Thanks to the low thermal conductivity and the good acoustic absorption capacity of wood–cement panels, these composites have been developed for thermal insulating and noiseproof walls. These materials present better nailing, durability and fire resistance properties than wood-based particleboards. Moreover, the wood–cement composites are an alternative way to promote the wood industry by-products. However, several difficulties such as dimensional instability due to moisture variations, efflorescence emergence and set retardation, hinder the development of these composites. The set retardation stems from chemical incompatibility between wood, organic material, and the inorganic binder. According to many authors, the effects of wood on cement setting may depend on various factors *i.e.* the geographical location of the tree, its felling season, its species and its various chemical constituents [1], [2], [3], [4], [5] and [6]. Biblis and Lo reported that the setting time could be related to the amount of soluble reducing sugars [2]. Simatupang, Miller and Moslemi attributed the inhibitory effect of wood on cement setting to the water-soluble hemicelluloses [3] and [4]. Some condensed tannins and gallotannins have also a significant inhibitory effect on cement setting [5] and [6]. Ficher *et al.* proposed that the detrimental molecule to cement hardening was saccharinic acid resulting from alkaline degradation of soluble sugars [7]. Whistler *et al.* suggested that wood polysaccharides could be hydrolyzed by alkali media in a large amount of hydroxy-acids and organic acids [8], which are known to be set-retarding agents [9], [10] and [11].
Previous investigations on physico-chemical aspects were restricted to the effect of monosaccharides or sugar acids on cement or single phase hydration [12], [13] and [14].

Influence of such admixtures on aluminate phase (C_3A) was studied by Young [12] who showed that interaction of sugars with C_3A prevented rapid formation of the cubic phase C_3AH_6 and promoted the formation of the hexagonal phase C_4AH_{13}. Collepardi *et al.* [13] found that the addition of glucose, gluconate or lignosulfonate stabilized ettringite in the C_3A–gypsum system. It was also reported that glucose retarded the gypsum consumption and the ettringite formation. Milestone [14] classified the retarding ability of various admixtures on the hydration of the silicate phase (C_3S). He determined the calcium hydroxide content and a set retarder efficiency which is as follows: sugar acids > sugars > lignosulfonates.

Most of the studies involving modification of cement setting by wood focused on the global behavior of the hydration and the resulting strength of the composite. The objectives of this study were to improve the knowledge of the influence of natural and heated wood particles on the hydration of cement at early ages and to propose a mechanism of interaction between cement and wood materials.

II. Experimental details

II.1. Raw materials

A gray Portland cement (C), CPA CEM I 52.5, was used in this study. The designation follows the French standard NF P 15–301. The chemical composition and mineralogical phases of the cement were calculated by the Bogue approximation [15] and are listed in Table 1.

The wood species used for this study was Poplar sapwood (*Populus hybrid* I214). Three kinds of fibers were tested: natural Poplar (P), Poplar heated at 240 °C (240) and Poplar heated at 260 °C (260). Thermal treatment was carried out according to the retification® [16]process. This treatment consisted of a mild pyrolysis of wood under a nitrogen atmosphere. The swelling of wood is strongly decreased whereas the fungal resistance is enhanced. Treatments were carried out on wood shavings of approximately 2-mm length, stored in a climatic chamber at 20 °C and 60% relative humidity.

II.2. Methods of investigation

Isothermal microcalorimetry, thermogravimetry and FTIR spectrometry were used to monitor kinetics and to determine the mechanism of cement hydration. In each case, cement and wood were mixed for two periods of 2 min using a shaker-mixer (Wab, Turbula, Germany) and the experiments were carried out in triplicate.

Isothermal microcalorimetry carried out on a C80 calorimeter (Setaram, France), enabled the hydration process to be monitored continuously and the admixtures to be classified as retarders or accelerators [10]. The experiments were conducted on a 1-g sample at a deionized water-to-cement weight ratio (W/C) of 40% and a wood-to-cement weight ratio (wd/C) between 2.5% and 7.5%. Wood powder (150–200 µm) was preferred to shavings because of the small size of the cell and the higher specific surface area. Cement and water were mixed outside the calorimeter according to the Sauvat *et al.* [17] and Ramachandran and Lowery [11] procedures. Consequently, only the exothermic hydration peak was encountered. The temperature of the calorimetric block was maintained at 25 °C.

The natural and the heated wood were sieved between 2-mm and 1.4-mm screens. The fraction of shavings between the two sieves was used in the cement pastes. For samples prepared with natural Poplar, two wd/C ratio: 7.5% and 20% were employed. A single ratio fixed at 20% was used for the heat treated Poplar samples. Cement and water were mixed (mixer Technotest, France) with a W/C ratio equal to 0.4 (w/w), for 1 min at low speed (60 rpm) and 4 min at high speed (120 rpm). During hydration, samples were stored in a water-vapor-saturated desiccator and under a nitrogen atmosphere. The hydration of cement was stopped by breaking and grinding hydrated samples and washing them with anhydrous ethanol. In order to avoid modification of the thermogravimetric signal by wood thermal decomposition, the wood was fully removed from the cement pastes by successive screenings. The cement samples were then ground in an agate mortar until each particle size became lower than 100 µm. This step was repeated at different lengths of hydration time ranging from 1 to 48 h.

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

Application of Equations. (1) and (2) below enable the calculation of the amount of portlandite (CH) and calcium carbonate from TG curves.

$$CH(\%) = WL_{CH}(\%)x\frac{MW_{CH}}{MW_{H}} \tag{1}$$

$$CaCO_3(\%) = WL_{CaCO_3}(\%)x\frac{MW_{CaCO_3}}{MW_{co_2}} \tag{2}$$

where $WL_{CH}(\%)$ and WL_{CaCO_3} correspond to weight loss (%) occurring during CH dehydration and $CaCO_3$ decomposition, respectively. MW_{CH}, MW_H, MW_{CaCO_3} and MW_{co_2} are the molecular weights of portlandite, water, calcium carbonate and carbon dioxide, respectively.

Infrared spectra were collected with a spectrometer (BioRad FTS 185 Digilab, USA) equipped with a Harrick scattering reflection device. Analysis was carried out on hydrated samples (24 h) diluted at 5% in KBr. Spectra were converted by the Kubelka–Munk algorithm.

III. Results and discussion

III.1. Calorimetric investigation

The calorimetric curve of pure cement exhibits two distinct peaks (Figure 1). According to Sauvat *et al.* [17], they correspond to C3A and C3S hydration peaks respectively whereas Bensted *et al.* [18] suggested the reverse order in a very high C_3A containing cement. The shape of the curve, the time values and the thermogravimetric results suggest the first allocation. Three different times were defined as proposed by Sauvat et al., t_1, t_2 and t_3, which correspond to the minimal flux time, the first peak time and second peak time, respectively. The heat released in 48 h of hydration was determined by signal integration between t_1 and t_{48} h (Table 2). The t_1 time, corresponding to the induction period increases from 0.4 h for pure cement to 1.2 h for natural Poplar-containing paste. Alberto et al. presented similar results with a tropical hardwood [19]. The heated Poplars slightly lengthen the induction period and the t_2 time compared to natural wood. The hydration of the silicate phase is significantly delayed and inhibited by wood. The natural Poplar increases the t_3 values and reduces the maximums of the corresponding heat flows. These phenomena are amplified by heated wood and are proportional to the treatment temperature. Consequently, the corresponding heat released values are much lower.

III.2. Thermogravimetric and infrared spectroscopy investigations

Several authors used differential thermal analysis (DTA) and thermogravimetric analysis (TGA) to investigate the effects of admixtures such as sugars or polysaccharides, on the hydration of the cement phases [12], [13], [14] and [20]. The thermogravimetric curve of the cement pastes exhibits five weight losses. The two first peaks, at 115 and 135 °C were assigned to ettringite and gypsum decomposition, respectively (Figure 2a). However, the decomposition peaks of ettringite and C–S–H overlap beyond 4 h of hydration. The gypsum consumption can be monitored until its complete disappearance since no other phase interferes with it. When the system becomes undersaturated with respect to sulfate, ettringite is converted into the monosulfoaluminate (MSA) by dissolution and reaction with the excess of C_3A. This hydrate is detected beyond 16 h of hydration and its decomposition occurs at 185 °C [13]. The thermal decomposition of CH and $CaCO_3$ phases occurs between 420–480 °C and 600–750 °C, respectively.

The gypsum consumption is slower for C + 20% P formula than for pure cement (Figure 2b) since, the full consumption occurs beyond 8 h of hydration. However, the amount of ettringite is equal for C and C + 20% P. This result seems paradoxical because ettringite results from

consumption of gypsum. The presence of sulfate ions held in the wood was then investigated by ionic chromatography. The result showed that sulfate ions contained in wood are quickly solubilized in water. The same effect as the natural wood on ettringite and gypsum content is observed with heated Poplar at 260 °C (Figure 2c). However, during the first 8 h of hydration, the consumption of gypsum is slightly accelerated compared with C + 20% P formula. Therefore, the amount of ettringite formed during this time is enhanced. After treatment, the heated wood is less hygroscopic than natural wood [21]. Consequently, the amount of water available for the hydration of cement is higher for C + 20% 260 than for C + 20% P. This result is consistent with Bensted observation [22] who showed that a high value of the W/C ratio accelerated the gypsum dissolution and promoted ettringite formation. In the first 8 h of hydration, the ability of wood to promote C_3A. hydration is as follows: C + 20% 260 > C + 20% 240 > C + 20% P due to the modification of the hygroscopic behavior of the fiber by the heat treatment.

In pure cement, the decomposition peak corresponding to the monosulfoaluminate (MSA) appears between 16 and 24 h of hydration (Figure 2a). Its intensity still increases beyond 48 h, which leads to a decrease in ettringite content. For the three composites, the MSA is not observable until 48 h of hydration on the thermogravimetric curves. Ettringite seems stabilized and its conversion into MSA is delayed. Seligmann and Greening observed the same phenomena by hydrating cement with a saccharose solution [23]. Collepardi et al. found that the addition of glucose, gluconate, or lignosulfonate stabilized ettringite in the C3A–gypsum–lime system [13].

The decomposition peak of portlandite is less intense for the three composites than for C. Further information can be obtained by the calculation of portlandite content by Equation. (1). The formation of portlandite is stopped during the first 4 h and strongly inhibited beyond 48 h of hydration by natural Poplar (Figure 3). This inhibition could be linked to the partial absorption of water by wood particles, which leads to a decrease in the W/C ratio. Nevertheless, the Figure 3 shows that a W/C of 0.33 promoted the CH precipitation below 16 h of hydration. Consequently, the inhibition of CH formation is not due to the decrease in W/C ratio. When cement is hydrated in presence of heat treated wood (240 and 260 °C), the induction period of CH is extended up to 8 h of hydration. Moreover, the amount of portlandite depends on the heat temperature of wood and is inversely proportional to the treatment strength. These results are consistent with the calorimetric investigation.

Silva *et al.* proposed that the smaller amount of portlandite observed for cement admixed with ethylene/vinyl acetate copolymer (EVA), could be associated with a greater carbonation of the cement pastes [24]. Figure 4 depicts the evolution of $CaCO_3$ content in cement pastes during hydration. The amount of carbonate phases is strongly increased in the composites. This result is confirmed by infrared spectroscopy in the range 1420–1480 cm−1 (Figure 5). It is possible to correlate the portlandite deficit, ΔCH (mol/g of cement), and the carbonate excess, $\Delta CaCO_3$ (mol/g of cement). These values are calculated by the Equations. (3) and (4),

respectively. $$\Delta CH(mol\,/\,g\,\,of\,\,cement) = \frac{CH_{composite}(\%) - CH_{cement}(\%)}{100\,\,x\,\,MW_{CH}} \qquad (3)$$

$$\Delta CaCO_3(mol\,/\,g\,\,of\,\,cement) = \frac{CaCO_{3composite}(\%) - CaCO_{3cement}(\%)}{100\,\,x\,\,MW_{CaCO_3}} \qquad (4)$$

where $CH_{cement}(\%)$, $CaCO_{3\ cement}(\%)$, $CH_{composite}(\%)$ and $CaCO_{3\ composite}(\%)$ correspond to the amount of portlandite and calcium carbonate in cement and in wood cement composites, respectively. MW_{CH} and MW_{CaCO_3} are the molecular weights of portlandite and calcium carbonate, respectively (Figure 6).

For the three composites, the slope is equal to −0.3. The correlation between CH deficit and CaCOa excess is about 30%. This value is not high enough to explain completely the CH deficit. In a forthcoming study, it is shown that polysaccharides coming from wood fibers are hydrolyzed by cement media and converted into numerous carboxylic acids which are known

to be powerful retarders [25]. Although the main retarding effect on cement hydration is attributed to alkaline degradation products, the inhibition of portlandite formation is extended by the carbonation of the pastes. Wood alkaline degradation could induce a carbon dioxide release. This CO_2 leads to a carbonation of the portlandite. Consequently, the amount of $CaCO_3$ in the paste increases. This hypothesis is consistent with the observation of the NaOH carbonation during a pulping process [26]. In this case, NaOH solution is used to hydrolyze the wood hemicelluloses.

Because it is not possible to monitor the C–S–H formation by thermal analysis at early age, it was monitored by infrared spectroscopy. The bands at 925 and 525 cm⁻¹ in dry cement undergo changes during hydration. The shift of the Si–O asymmetric stretching (v_3) vibration to higher wave numbers (970 cm⁻¹) and the change in the relative intensity of the Si–O out-of-plane bending vibration (v_4) indicate the polymerization of the silicate units (SiO_4^{2-}) [27].

After 24 h of hydration, Figure 5 shows that C–S–H was formed for pure cement. The broad band centered on 930 cm⁻¹ indicates that the C–S–H formation is delayed for the three composites. In 24 h old samples, the Si–O stretching band was shifted by only 5 cm⁻¹. This low shift suggests that there was very little polymerization of the silicates in the wood–cement composites, as proposed by Mollah *et al.* [28]. This result is confirmed by the slight change of the relative intensity of the Si–O bending vibration at 525 cm⁻¹.

III.3. Influence of natural Poplar wood-to-cement ratio on hydration

Table 3 shows the value of t_1, t_2 and t_3 obtained by calorimetry according to the increase in wood-powder content. The t_2 value is unchanged from C to C + 7.5% P. It suggests that the kinetics of the C_3A hydration is similar even if for a low amount of wood it seems to be slightly accelerated. Wood particularly delays and inhibits the silicate phase and extends the length of induction period. The t_3 value increases from 8.8 to 12.2 h for wood content ranging from 2.5% to 7.5%. This result confirms that the hydration of silicate phases is significantly delayed by natural wood.

The thermogravimetric curve of C + 7.5% P reveals an acceleration of the gypsum consumption compared with C + 20% P. The corresponding amounts are close to those obtained for pure cement. The presence of sulfate ions in wood explains the higher amount of ettringite in C + 7.5% P formula than in pure cement. Moreover the gypsum being strongly consumed, the ettringite content increases compared with C + 20% P.

The evolution of the portlandite content is shown in Figure 7. The induction period of CH in the C + 7.5% P formula is higher than in pure cement and lower than in cement admixed with 20% of natural fiber. The amount of calcium carbonate increases (Figure 8) whereas the amount of portlandite decreases with increasing natural Poplar content. The correlation between ΔCH and ΔCaCO₃ for the two composites is reasonably similar (Figure 9). The slope of the curve is equal to −0.32 and −0.27 for 7.5% and 20% of wood, respectively. This result confirms that approximately 30% of the CH deficit is linked to its carbonation. This value is also slightly dependent of the wood content. The infrared spectroscopy reveals that 7.5% of natural Poplar inhibits the C–S–H formation.

Conclusion

The thermogravimetric investigation reveals a strong modification and delay of silicate phase hydration by wood particles. The corresponding induction periods are lengthened by the introduction of wood. The phenomenon is extended by heat treated Poplar. In composites, the amount of precipitated portlandite is lower than in pure cement. An increase in the amount of calcium carbonate phase explains partially (30%) this inhibition. Infrared spectroscopy shows a strong delay in the precipitation of C–S–H when cement is hydrated with wood.

The hygroscopicity of natural wood particles tends to decrease the amount of free water. This phenomenon slows the dissolution and consumption of gypsum. The expected lack of sulfate ions is compensated by the dissolution of sulfate ions from Poplar fibers. Consequently, the amount of ettringite in wood–cement pastes is equal to or higher than in neat cement.

Heat treatments improve dimensional stabilization and reduce the hygroscopicity of the wood fiber. Consequently, heated Poplar promotes gypsum dissolution or consumption and ettringite formation compared with natural Poplar. In the presence of natural and heat treated wood fibers, the ettringite is stabilized and its conversion into monosulfoaluminate is delayed.

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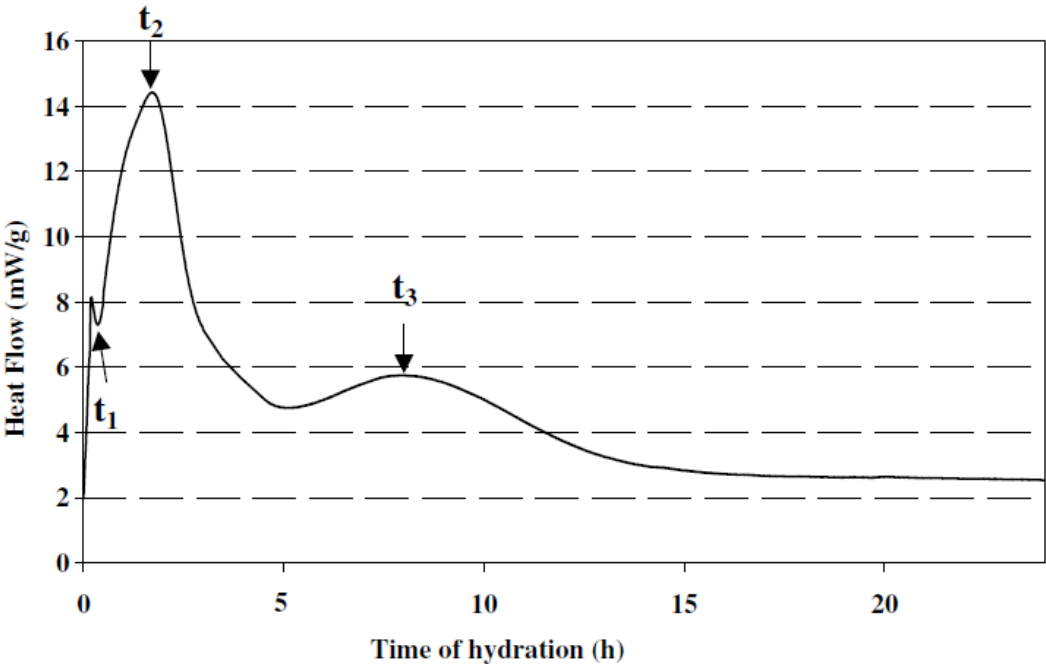
Figures

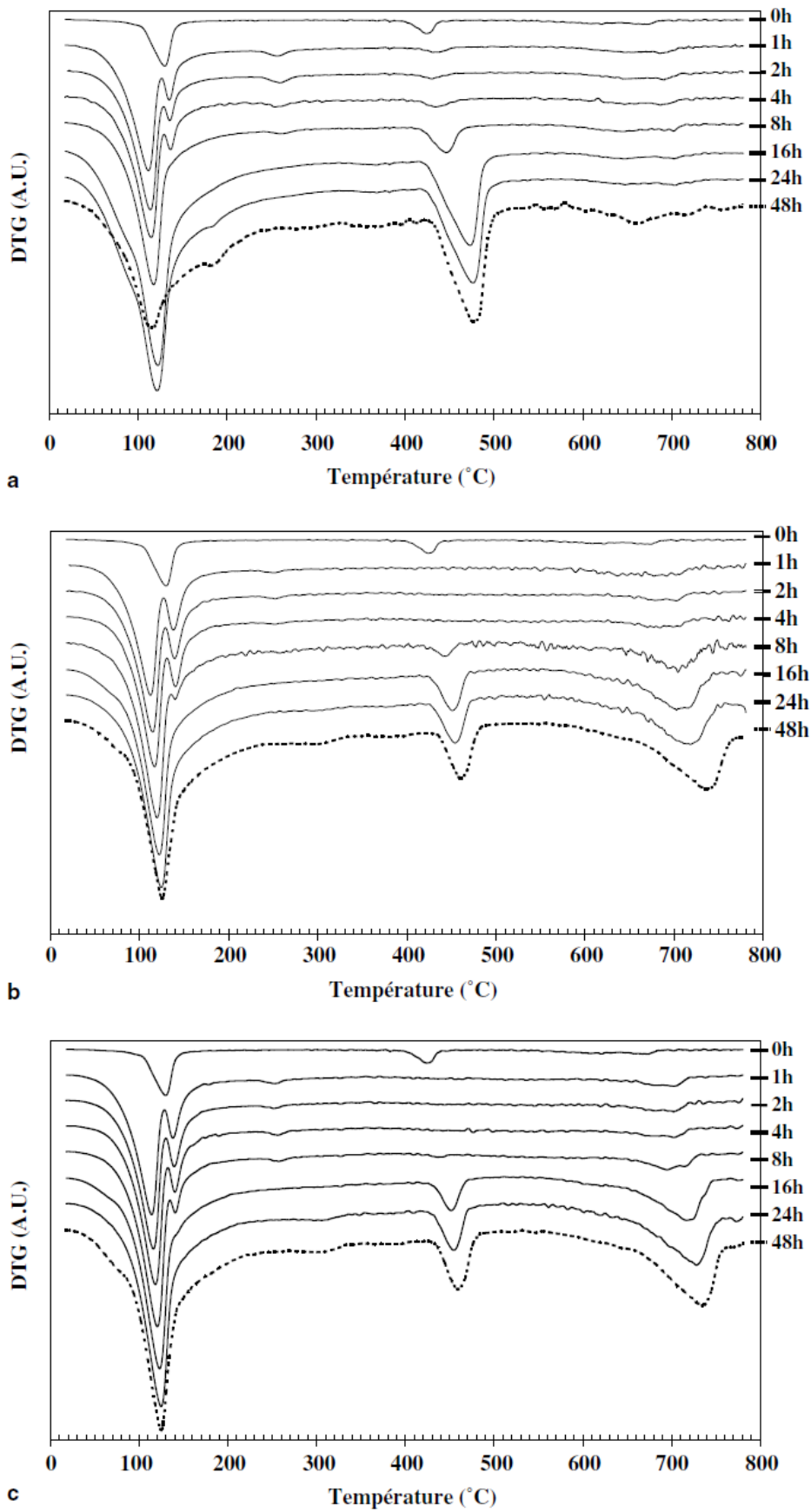*Figure 1: Three characteristic times of a calorimetric curve according to Sauvat et al. [17].*

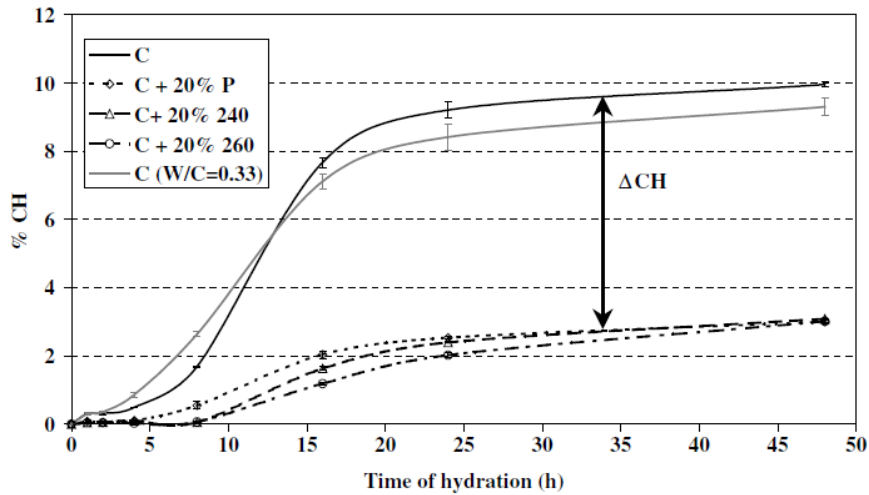*Figure 2: DTG curves of (a) pure cement, (b) C + 20% P and (c) C + 20% 260.*

Figure 3: Influence of natural Poplar, heated Poplar and W/C ratio on the portlandite content in cement pastes.
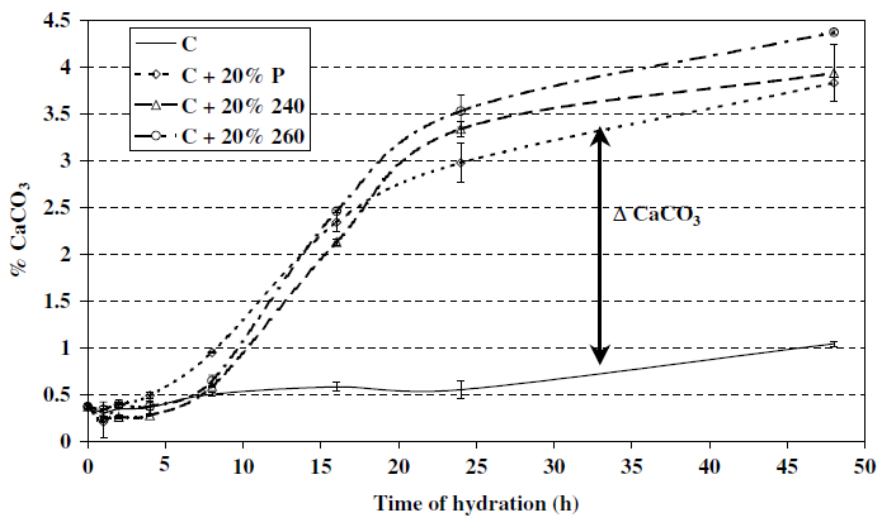

Figure 4: Influence of natural and heated Poplar on the calcium carbonate content in cement pastes.
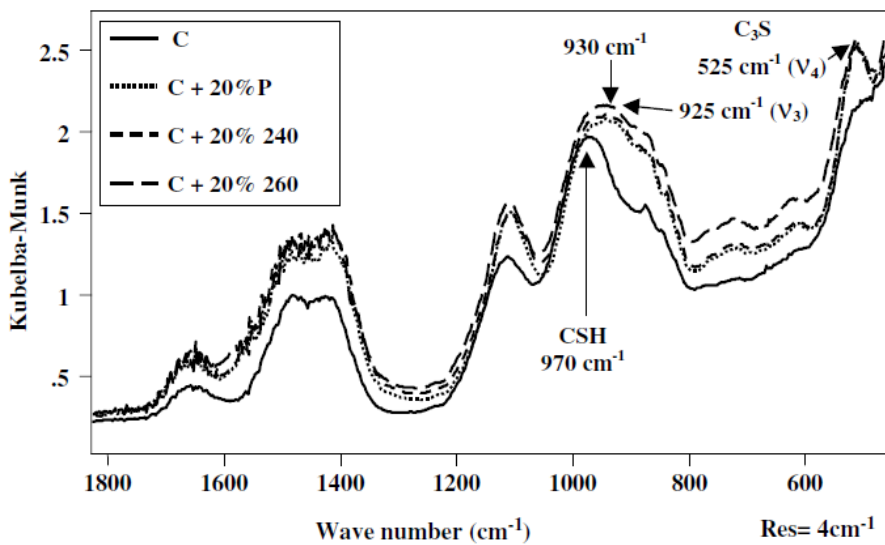

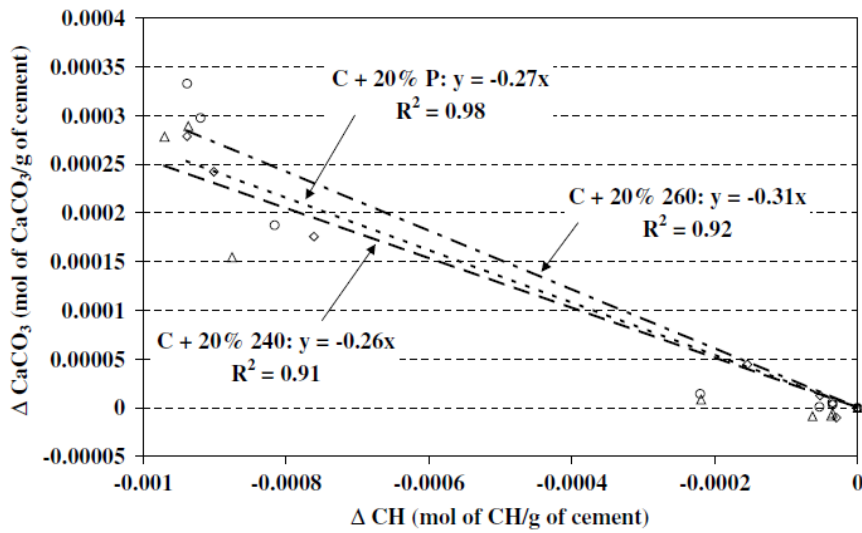*Figure 5: FTIR spectra of admixed cement pastes hydrated for 24 h.*

Figure 6: Correlation between the increase in CaCO₃ content and the decrease in CH content in wood-containing pastes.
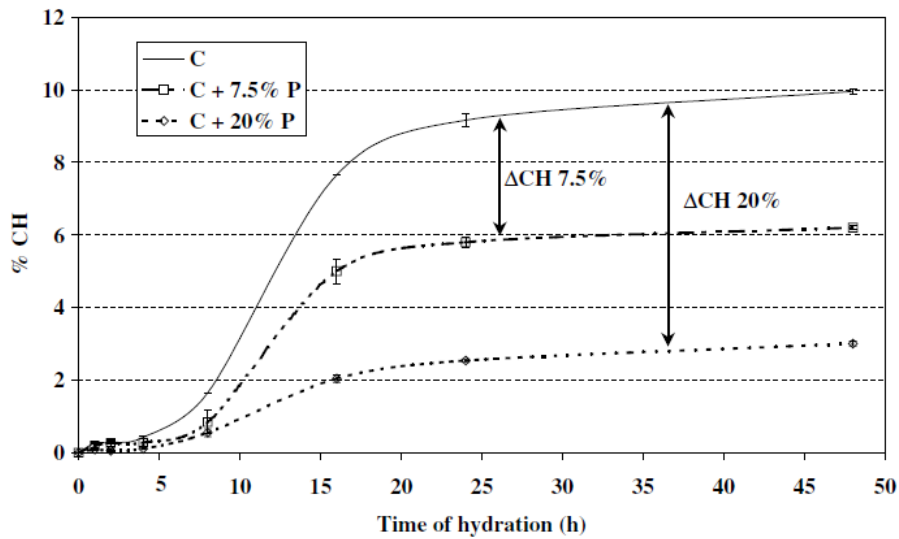

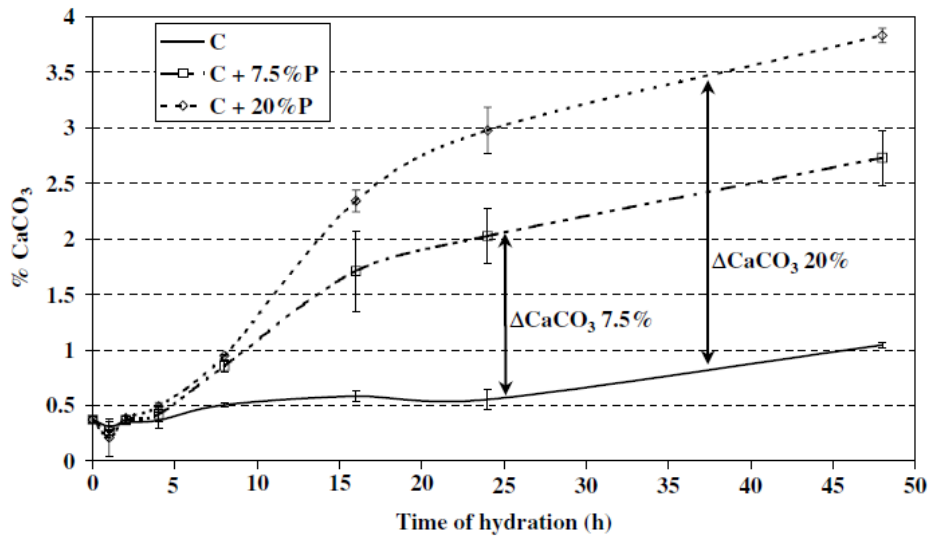*Figure 7: Influence of the natural wood-to-cement ratio on portlandite content in cement pastes.*

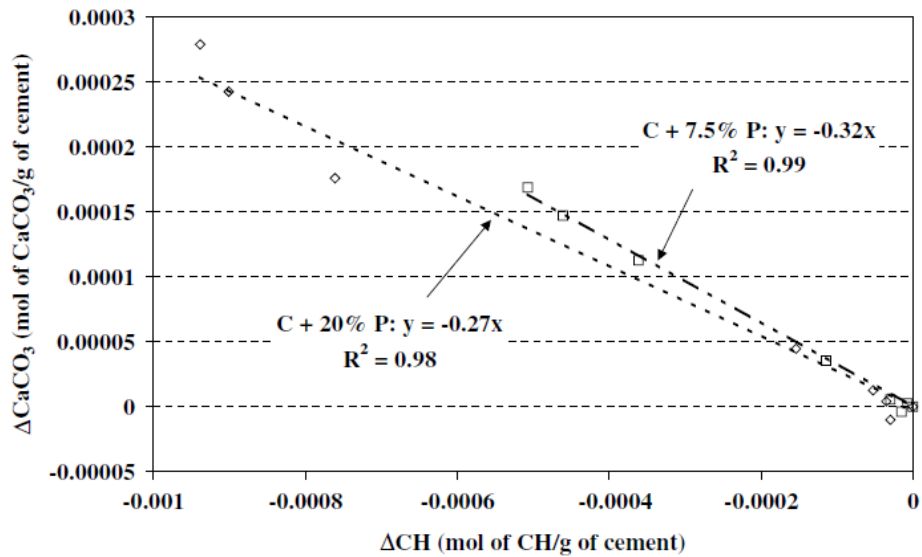*Figure 8: Influence of the natural wood-to-cement ratio on calcium carbonate content in cement pastes.*

Figure 9: Influence of the natural wood-to-cement ratio on correlation between the increase in CaCO$_3$ content and the decrease in CH content.

Tables

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

Chemical composition	Weight % of components
SiO_2	19.98
TiO_2	0.29
Al_2O_3	5.47
F_2O_3	2.72
CaO	63.03
MnO	0.03
MgO	2.32
Na_2O	0.46
K_2O	0.93
P_2O_5	0.26
SO_3	5.14
Bogue Approximation	
C3S	64
C2S	9
C3A	10
C4AF	8

Table 2: Influence of natural and heated wood on characteristic times and heat released in 48 h

	$T_1(h)$	$t_2(h)$	$t_3(h)$	$Q_{48\,h}\,(J/g)$
C	0.4 ± 0.1	1.8 ± 0.1	8.1 ± 0.2	360 ± 7
C + 7.5% P	1.2 ± 0.2	1.8 ± 0.1	12.2 ± 0.3	250 ± 25
C + 7.5% 240	1.3 ± 0.1	1.9 ± 0.2	13.9 ± 0.2	190 ± 12
C + 7.5% 260	1.6 ± 0.1	2.2 ± 0.2	Undetermined	140 ± 9

Table 3: Influence of the natural wood-to-cement ratio on hydration times and enthalpy

	$T_1(h)$	$t_2(h)$	$t_3(h)$	$Q_{48\,h}\,(J/g)$
C	0.4 ± 0.1	1.8 ± 0.1	8.1 ± 0.2	360 ± 7
C + 2.5% P	0.8 ± 0.1	1.4 ± 0.1	8.8 ± 0.2	340 ± 10
C + 5% P	0.9 ± 0.1	1.5 ± 0.2	9.5 ± 0.3	310 ± 8
C + 7.5% 260	1.2 ± 0.2	1.8 ± 0.1	12.3 ± 0.3	250 ± 25