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New insights into wood and cement interaction [◇]

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Abstract

This work deals with the influence of poplar extractives and poplar alkaline degradation products on the hydration of cement. The wood chemical composition was characterized to determine the impact of soft alkaline conditions on wood-fiber. Some of the constituents are hydrolyzed and converted into carboxylic acids which are identified and quantified. The monitoring by electrical conductivity clearly showed that wood extractives are less effective than wood alkaline degradation products to inhibit the hydration of cement.

Keywords: Alkaline degradation, Electrical conductivity, Extractives, Hydration of cement, Setting retardation, Wood-cement composites.

Introduction

Many works have been devoted to the use of vegetable fibers as aggregates in cement. Wood-cement composites were specifically studied since these aggregates enhance soundproofing and thermal insulation properties. However, several drawbacks have hindered the development of these composites. The main difficulty is the chemical incompatibility between wood and ordinary Portland cement (OPC) which retards cement setting and hardening. The chemical structure of wood is complex. It is composed of cellulose, hemicelluloses, lignins and extractives. Biblis and Lo (1968) reported that among the extractives simple sugars were powerful retarders. However other compounds were also found to adversely affect the hydration of cement reactions such as sugar acids (Ficher et al. 1974), hemicelluloses (Simatupang 1986) and phenolic compounds such as tannins (Miller 1988, Miller and Moslemi 1991).

Most of the previous works deal with the effect of several wood species or wood constituents on cement **setting** or **hardening** by means of adiabatic or isothermal calorimetry. However, the mechanism of the wood-cement interaction is not yet fully understood. The present study aims at the investigation of the effect of wood components on the **hydration** of cement and the behavior of wood in such systems. Precisely, the effect of cement and a soft alkaline medium was studied on poplar wood by quantifying the content of sugar, lignin and extractives in the leaching and analyzing the degradation products by gas chromatography (GC). The influence of extractives and alkaline degradation products on the hydration of cement was explored by means of electrical conductivity.

Materials and Methods

Raw materials

A gray Portland cement CPA CEM I 52.5 was used in this study. The designation follows the French standard NF P 15-301. Chemical composition and mineralogical phases of the cement were calculated by the Bogue

approximation (Taylor 1990) and these are listed in Table 1. Poplar (*Populus hybrid* I214) powder was prepared by milling wood and screening through a 150 and a 200 μm mesh. The powder between the two screens was used as wood sample and was stored at 20 °C and 60 % of relative humidity. Each experiment was conducted in triplicate.

Wood soaks preparation

The soaks were prepared by mixing wood (3 g) either with 150 ml of deionized water (Millipore, mQ, USA), either with lime solution (1 g.l^{-1}) or with cement suspension (7.5 g in 150 ml of water, water to cement weight ratio $\text{W/C}=20$ (w/w)), at 25 °C under stirring. This two-phase solution was filtered at different lengths of time ranging from 1 to 24 h. Then the wood was dried at 103 °C for 48 h and weighed. The weight loss was then calculated.

Electrical conductivity

Electrical conductivity measurement was performed in dilute suspensions with a W/C ratio of 20 (w/w). Conductivity equipment consisted of a 25 °C thermostated reactor, two stainless steel annular electrodes and a conductimeter (Tacussel CD810, France). The constant cell was first calibrated with a 0.1 M KCl solution. Cement (5 g) was mixed with 100 ml of the previously described wood soaks. The reference used for lime soaks was a 0.2 g.l^{-1} lime solution. It corresponded to the chemical composition of the lime solution after 1 h of leaching with poplar. After 24 h of leaching, the lime soaks corresponded to a 0.06 g.l^{-1} lime solution. However, the conductivity curve was identical. For experiments requiring both cement and wood, wood was introduced 30 seconds before the cement with a wood to cement weight ratio (wood/C) equal to 0.4 (w/w).

Determination of wood chemical composition

The amount of poplar extractives were quantified according to the ASTM D1105-56 standard. The content of Klason lignin and each sugar were obtained by acid hydrolysis of poplar according to the ASTM D1106-56 standard. The hydrolyzates were neutralized by Ba(OH)_2 , then centrifuged and filtered. The sugar concentrations were determined by means of HPLC. The apparatus was equipped with a pump (WATERS 916, USA) and a refractometer (WATERS 410, USA). A specific column for sugar analysis (SUPELCO, Supelcogel Pb 7.8 mm x 30 cm, USA), conditioned in furnace at 80 °C, was used. Deionized water, in-line degassed, was used as eluent and the flow was maintained at 0.5 ml.min^{-1} . Calibration of the apparatus was performed by the injection of

HPLC grade sugar samples. The acid soluble lignin contents, in acid hydrolyzates, were estimated by the determination of the total phenolic compounds by colorimetry (Singleton and Rossi 1965 and Alberto et al. 2001). The catechin was used as standard and the results were expressed by mg of catechin.l⁻¹.g⁻¹ of wood. The crystallinity index of cellulose was determined by X-ray diffraction according to the method proposed by Segal et al. (1959) and Fan et al. (1980).

Trimethylsilylation of nonvolatile carboxylic acids, Gas Chromatography-Mass Spectrometry (GC-MS) analysis

The previous soaks (100 ml) were evaporated down to 2 ml by a rotary evaporator. A 0.2 ml sample of the liquors was then taken to be analyzed for nonvolatile carboxylic acids. The mixture was then converted into ammonium salts, evaporated and dried. Xylitol (0.06 mg) was introduced as an internal standard. The trimethylsilylation reaction was realized by introducing 0.9 ml of pyridine and 0.1 ml of BSTFA under nitrogen atmosphere and by mixing the constituents for 4 h. A volume of 1.5 µl was injected and analyzed by GC-MS. Separation was carried out on a gas chromatograph (SHIMADZU GC-17A) equipped with a MS detector (SHIMADZU QP-5000) at 70 eV. The temperature program began with a step at 100 °C for 1 min followed by a heat rate of 20 °C.min⁻¹ up to 240 °C and finally a step at 240 °C for 2 min. The injector as well as the detector temperature were set to 260 °C. The concentration of acetic and formic acids were measured out by ionic chromatography (DIONEX GP50 pump, DIONEX CD20 detector and DIONEX AS11HC column).

Results and discussion

Influence of leaching conditions on wood mass loss

The kinetic of poplar mass loss induced by the extraction in water and in lime solution for time ranging from 1 to 24 h is presented in Figure 1. The mass losses, equal to 2.15 % in water, are independent of the leaching time, contrary to those associated with the leaching in lime solution. These values are higher than those obtained in water and ranging from 2.2 to 3.9 %. Consequently, the amount and the origin of molecules solubilized in solution are controlled by the leaching conditions and will be assigned later. Their respective effects on the hydration of cement were followed by conductivity measurement.

Measurement on cement hydration by electrical conductivity

The electrical conductivity measurement is a quite simple and reproducible method to follow up the hydration of cement compared to isothermal calorimetry. Damidot (1990) and Nonat et al. (1997) reported that this method allows to investigate the influence of admixtures on the nucleation and growth of cement hydrates and to simulate cement paste conditions with the help of saturated lime solutions. We interpreted the electrical conductivity curves according to the explanation proposed by Comparet et al. (1997). As soon as cement was introduced into the cell, the electrical conductivity raised as a consequence of cement dissolution (Figure 2). Hydrates then nucleated on the grain surface, which led to a lower increase in the conductivity. Conductivity then accelerated more quickly due to the nuclei growth. Meanwhile, cement dissolution still continued. The conductivity rose up to the critical supersaturation with respect to portlandite and decreased rapidly due to its precipitation.

The hydration of cement by water eluates is shown in Figure 3. During the first minutes, the conductivity reached is similar to that of neat cement. The dissolution of cement is thus not affected by the chemical composition of water soaks. The duration of the plateau is slightly extended. The growth of hydrates is a bit slowed down by the chemical composition of water soaks since the slope of the conductivity curve is weaker than for neat cement. Consequently, the portlandite precipitation is delayed of about 1.8 h. Moreover, the effect of water wood eluates on the hydration of cement is independent of the leaching time. This result can be related to the constant mass loss obtained in water.

Figure 4 depicts the evolution of conductivity measured out in lime soaks. The alkaline wood eluates significantly modify the hydration. After the constant dissolution of cement, a conductivity plateau appears which varies from 3 to 6.5 h for leaching time ranging from 1 to 24 h. According to Comparet et al. (1997), the duration of this step is inversely proportional to the number of hydrate nuclei that precipitated during the initial hydration. Wood soaks in lime

solution seems to limit the number of precipitated hydrates. The slope of the curve is equal to $1.3 \text{ mS.cm}^{-1}.\text{h}^{-1}$, $1 \text{ mS.cm}^{-1}.\text{h}^{-1}$ and $0.9 \text{ mS.cm}^{-1}.\text{h}^{-1}$ for the reference, the wood soak leached for 1 h and 24 h in lime solution respectively. The growth of hydrates is then slowed down by the chemical composition of the solutions. The modifications of hydration cause a strong delay in portlandite precipitation which depends on the leaching time. The supersaturation with respect to portlandite is reached for hydration times ranging from 9.6 h (1 h of leaching) to 13.8 h (24 h of leaching). The kinetic of the cement hydration is, then, inversely proportional to the leaching time and to the mass loss.

The effects of the 24 hours-extraction in water and in lime on the mass loss and the retardation of portlandite precipitation are listed in table 2. The mass loss, equal to 2.2 % in water, was assigned to the amount of poplar extractives solubilized. It induces a portlandite precipitation delay of about 1.8 h. In alkaline solution, the mass loss is equal to 3.9 % and the delay of portlandite precipitation is extended to 7.8 h. These values were allocated to the global effect of extractives and alkaline degradation products. Therefore, the difference between leaching in water and in lime solution was attributed to alkaline degradation products. Although the difference in mass loss is only equal to 1.7 %, the portlandite precipitation is delayed of about 6 h. It point out that degradation products are more efficient than extractives to inhibit the cement hydration. The chemical composition of poplar was analyzed after 24 h of leaching to confirm the allocation of the mass loss.

Influence of leaching conditions on wood chemical composition

The chemical composition of wood was examined according to the ASTM D1105-56 and ASTM D1106-56 standards on natural and 24 hours-leached poplar. The alcohol/alcohol-benzene extraction reveals a decrease of about 2.2 % in the extractive content after leaching (Table 3). Moreover, the amount of extractives solubilized in the soak is independent of the

solvent (water or alkaline media). The Klason lignin content and the acid soluble lignin content are not affected significantly by deionized water or alkaline conditions. The breakdown of lignin is mainly the result of cleavage of various ether bonds, especially at the α and β position, but requires drastic conditions (Lai 1991). The effect of alkaline media is enhanced for carbohydrates except for the cellobiose content that is constant. As expected for most hardwoods, arabinose and galactose contents are negligible and will not be discussed. The average content of xylose in natural poplar, equal to 12.4 %, remains unchanged after the water leaching. However, this rate falls to 10.7 and to 9.8 % for leaching in lime and in cement suspension respectively. This sugar, being representative of glucuronoxylan content in poplar, indicates that hemicelluloses are unchanged in water and hydrolyzed under soft alkaline conditions. The decrease in the mannose and glucose contents, which are representative of glucomannans, confirms this result. However, the crystallinity index of cellulose, determined by X-ray diffraction, raises with alkaline treatment. According to Knill and Kennedy (2003), the glycosidic (1-4)-linkage between the β -D-glucopyranose units in cellulose is alkali stable. Nevertheless the dissolution of short-chain units detached from the reducing ends of the cellulose molecules can occur. It leads to an additional reduction of the glucose rate. These results highlight that in water the mass loss is only due to the solubilization of a part of wood extractives. But under alkaline conditions such as cement, hemicelluloses and amorphous cellulose are hydrolyzed besides the solubilization of extractives. Cement environment is slightly more aggressive towards polysaccharides than a 1 g.l⁻¹ lime solution. The wood composition, after leaching in water and in lime, allows to assign the difference of mass loss to the degradation of polysaccharides by alkaline media. Consequently, the alkaline degradation products are more effective than the poplar extractives solubilized in the eluates to inhibit the cement hydration.

In order to confirm it, natural poplar and poplar leached in water and lime solution for 24 h were directly introduced into the conductivity cell with deionized water. The critical supersaturation with respect to portlandite, reached by the system “natural polar-cement”, is retarded of 5.1 h (Figure 5). When poplar was previously leached by water or lime, this delay is reduced to 3.5 h because of the absence of extractives from wood. The acceleration of the hydration (1.6 h) was conformed to the retardation produced by the water soaks containing the extractives. The delay of about 3.5 h was mainly allocated to alkaline degradation products of wood since wood extractives were previously removed by water or lime. Consequently, the poplar extractives are twice less efficient than hydrolysis products. These results are consistent with those obtained with wood soaks and those found by Young (1968). Indeed, he proposed that products from alkaline degradation of sugar were more efficient as retarding compounds than sugars themselves on C_3A hydration. The compositions of wood soaks were analyzed to identify the active molecules.

Chemical composition of soaks

The HPLC analysis (method described earlier) carried out on lime soaks did not reveal the presence of sugars in solution. This result could be explained by the soft alkaline conditions which were strong enough to decompose the water-soluble hemicelluloses into acidic compounds. The degradation mechanism of polysaccharides under alkaline conditions was intensively studied in the pulping industry. According to several authors, polysaccharides were converted into carboxylic acids by alkaline treatment (Whistler and BeMiller 1958; Sjöström 1977; Alen et al. 1984; Niemelä and Sjöström 1986). The chemical composition of eluates was monitored by GC-MS according to the method described earlier (Figure 6). Most of the 18 molecules detected resulted from polysaccharides degradation by peeling reaction.

The results of the analysis at two leaching times and in two solvents are shown in Table 4. The soak, carried out in water for 1 h, is consisted of many carboxylic acids with a concentration lower than 1 mg.l⁻¹ excepted for formic (4.2 mg.l⁻¹), acetic (18 mg.l⁻¹), malic (2.5 mg.l⁻¹), benzoic (2.7 mg.l⁻¹) and *p*-salicylic (5.6 mg.l⁻¹) acids. According to Hillis (1975), the degradation of polysaccharides might occur during wood drying and produces carboxylic acids. When leaching time is extended to 24 h, the content of carboxylic acids is unchanged as the conductivity measurements and the mass loss. After 1 h of leaching in lime, the concentration of acids grows compared to those determined in water (1 h). This increase is enhanced after 24 h of leaching since the concentration of acetic, glycolic, pyruvic, glyceric, malic, benzoic, hydrocinnamic, *o*- and *p*-salicylic acids are multiplied by 38, 13, 9.5, 3.5, 3, 3.8, 13, 6 and 5 respectively. The higher concentration of carboxylic acids in lime confirms the decreasing content in wood sugars due to the alkaline degradation of polysaccharides and the rise in mass loss. However, neither glucoisosaccharinic acid nor xyloisosaccharinic acid, the main degradation acids of hexosans and pentosans (Sjöström 1977; Alen et al. 1984; Niemelä and Sjöström 1986), were detected. This result might be due to softer alkaline conditions than those applied in these study. In spite of its high concentration in lime solution, the acetic acid, resulting from the cleavage of O-acetyl groups and glucuronoxylan chain, is considered as a poor accelerator at low concentration and a poor retarder at higher concentration (Young 1972). A solution of 1 g.l⁻¹ of acetic acid, inducing a delay lower than 0.5 h can not explain the experimental retardation of about 7.8 h. Several authors referred to the retarding effect of some of the detected organic molecules. Taplin (1960) supposed that the active group was HO-C-C=O. He identified glycolic, pyruvic and malic acids which have this chemical function as very efficient retarders. Other authors also obtained these results (Wilding et al. 1984; Ramachandran and Lowery 1992). Wilding proposed the following

classification of benzoic acids: *o*-salicylic acid > benzoic acid > phenol. He suggested that *o*-salicylic acid is active on cement as well as malic or pyruvic acids.

Conclusion

The experiments concerning the chemical composition of the eluates and poplar show that wood polysaccharides are indeed hydrolyzed under alkaline conditions such as in lime solution with a concentration of 1 g.l⁻¹ and in cement suspension. These polymers are converted into numerous carboxylic acids, which are known to be powerful retarders. The electrical conductivity measurement demonstrate that the effect induced by wood extractives is three time less efficient than alkaline degradation products on the hydration of cement. Accordingly, the hydrolysis products must be taken into account to understand the wood and cement interaction.

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

| Chemical composition | Weight % |
|--------------------------------|----------|
| SiO ₂ | 19.98 |
| TiO ₂ | 0.29 |
| Al ₂ O ₃ | 5.47 |
| Fe ₂ O ₃ | 2.72 |
| CaO | 63.03 |
| MnO | 0.03 |
| MgO | 2.32 |
| Na ₂ O | 0.46 |
| K ₂ O | 0.93 |
| P ₂ O ₅ | 0.26 |
| LOI | 0.42 |
| SO ₃ | 5.14 |
| <i>Bogue approximation</i> | |
| C ₃ S | 64 |
| C ₂ S | 9 |
| C ₃ A | 10 |
| C ₄ AF | 8 |

Fig. 1 Wood mass loss / initial dry wood weight ratio associated with leaching time in water

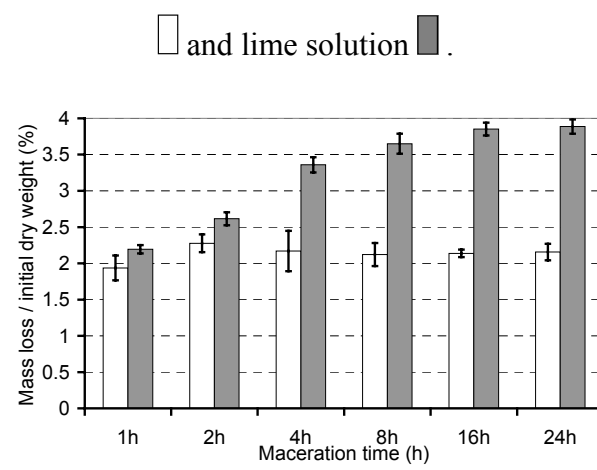


Fig. 2 Interpretation of the electrical conductivity curve of cement.

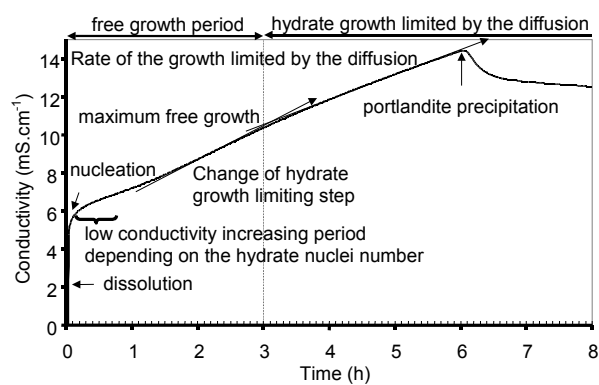


Fig. 3 Effect of poplar leaching time in water on cement hydration.

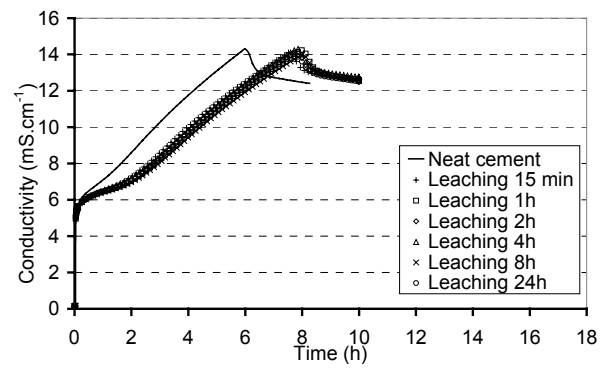


Fig. 4 Effect of poplar leaching time in lime solution on cement hydration.

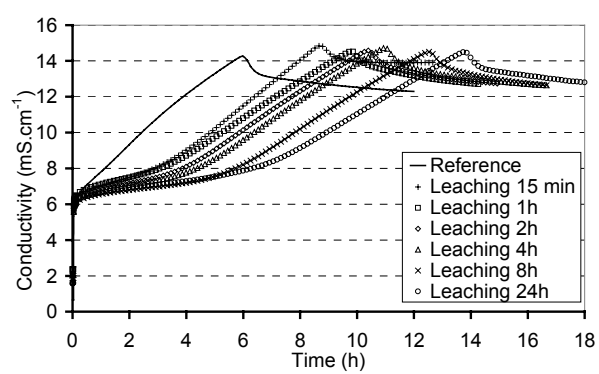


Table 2 Effect of a 24 hours-extraction in water and in lime on mass loss and retardation of portlandite precipitation. * due to extractives; ** due to extractives and alkaline degradation products.

| Leaching | Mass loss (%) | Precipitation delay (h) |
|------------|---------------|-------------------------|
| In water | 2.2 * | 1.8 |
| In lime | 3.9 ** | 7.8 |
| Difference | 1.7 | 6.0 |

Table 3 Wood chemical composition according to ASTM standards for natural poplar, leached poplar in water, in lime solution and in cement suspension for 24 h (in percent of initial dry wood weight).

| | Poplar | Leached poplar in water | Leached poplar in lime solution | Leached poplar in cement solution |
|--|------------------|-------------------------|---------------------------------|-----------------------------------|
| Alcohol/alcohol-benzene extractives (%) | 5.7 ± 0.3 | 3.3 ± 0.5 | 3.3 ± 0.7 | 3.7 ± 0.2 |
| Klason lignin (%) | 23.5 ± 0.6 | 24.2 ± 1.1 | 22.0 ± 0.4 | 22.2 ± 1.1 |
| Acid soluble lignin (mg of catechin.l ⁻¹ .g ⁻¹ wood) | 32.8 ± 1.5 | 34.7 ± 2.3 | 32.8 ± 1.0 | 34.0 ± 1.3 |
| Cellobiose (%) | 4.8 ± 0.1 | 4.5 ± 0.5 | 4.5 ± 0.2 | 5.1 ± 0.2 |
| Glucose (%) | 35.7 ± 0.1 | 35.6 ± 0.5 | 34.1 ± 0.5 | 30.7 ± 0.7 |
| Xylose (%) | 12.4 ± 0.1 | 12.2 ± 0.3 | 10.7 ± 0.5 | 9.8 ± 0.2 |
| Mannose (%) | 3.9 ± 0.6 | 3.9 ± 0.3 | 2.8 ± 0.3 | 2.5 ± 0.3 |
| Galactose (%) | 0.17 ± 0.02 | 0.25 ± 0.09 | 0.09 ± 0.04 | 0.31 ± 0.03 |
| Arabinose (%) | 0.022 ± 0.01 | n.d. | 0.06 ± 0.04 | 0.06 ± 0.006 |
| Crystallinity Index (%) | 71.0 ± 1.1 | 70.5 ± 0.6 | 80.6 ± 0.5 | 75.3 ± 0.8 |

Fig. 5 Effect on time to reach supersaturation with respect to portlandite of natural poplar, leached poplar in water (24 h), leached poplar in lime solution (24 h).

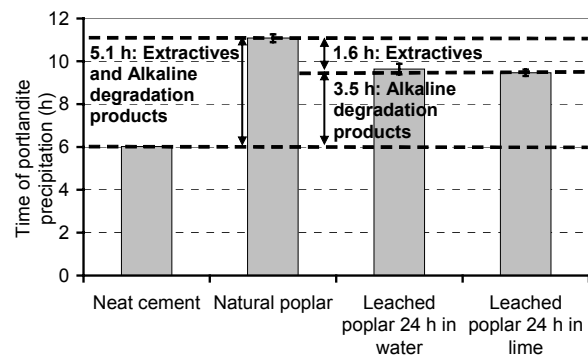


Fig. 6 Chromatogram of the trimethylsilylated soaks obtained by leaching poplar in lime solution for 24 h. 1: acid (a.) lactic; 2: a. glycolic; 3: a. pyruvic; 4: diethylene glycol ethyl ether; 5: a. malonic; 6: diethylene glycol; 7: a. benzoic; 8: glycerol; 9: a. succinic; 10: a. glyceric; 11: a. maleic; 12: a. hydrocinnamic; 13: a. malic; 14: a. *o*-salicylic; 15: a. *p*-salicylic; 16: a. vanillic.

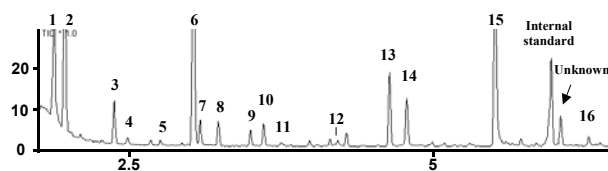


Table 4 Chemical composition of natural poplar soaks in water and lime solution for 1 and 24 h.

| Class | Molecule name | 1 h in water (mg.l ⁻¹) | 24 h in water (mg.l ⁻¹) | 1 h in lime solution (mg.l ⁻¹) | 24 h in lime solution (mg.l ⁻¹) |
|-----------------|-------------------------------|---------------------------------------|--|--|---|
| Alcohols | Diethylene glycol ethyl ether | 1.2 ± 0.1 | 1.5 ± 0.3 | 1.4 ± 0.1 | 2.2 ± 0.1 |
| | Diethylene glycol | 1.9 ± 0.4 | 2.4 ± 0.4 | 15.2 ± 2.1 | 21.1 ± 1.3 |
| | Glycerol | 0.4 ± 0.1 | 0.5 ± 0.1 | 0.7 ± 0.1 | 1.1 ± 0.1 |
| Aliphatic acids | Formic acid | 4.2 ± 0.5 | 5.17 ± 0.7 | 6.5 ± 0.3 | 9.0 ± 0.3 |
| | Acetic acid | 18.0 ± 2 | 19.6 ± 2 | 662 ± 12 | 773 ± 10 |
| | Lactic acid | 1.2 ± 0.1 | 1.6 ± 0.1 | 2.7 ± 0.2 | 3.7 ± 0.5 |
| | Glycolic acid | 0.6 ± 0.1 | 0.8 ± 0.1 | 2.8 ± 0.2 | 10.2 ± 1.0 |
| | Pyruvic acid | 0.2 ± 0.01 | 0.2 ± 0.04 | 0.53 ± 0.03 | 1.9 ± 0.1 |
| | Malonic acid | 0.3 ± 0.04 | 0.4 ± 0.1 | 0.5 ± 0.1 | 1.2 ± 0.1 |
| | Succinic acid | 0.7 ± 0.1 | 1.2 ± 0.1 | 1.1 ± 0.1 | 1.4 ± 0.02 |
| | Glyceric acid | 0.3 ± 0.04 | 0.4 ± 0.03 | 0.78 ± 0.1 | 1.4 ± 0.02 |
| | Maleic acid | 0.3 ± 0.04 | 0.4 ± 0.02 | 0.4 ± 0.06 | 0.8 ± 0.08 |
| | Malic acid | 2.5 ± 0.2 | 2.2 ± 0.1 | 2.8 ± 0.2 | 6.9 ± 0.2 |
| Benzoic acids | Benzoic acid | 2.7 ± 0.3 | 3.6 ± 0.2 | 5.7 ± 0.3 | 13.7 ± 1 |
| | Hydrocinnamic acid | 0.2 ± 0.0 | 0.2 ± 0.04 | 1.6 ± 0.4 | 2.6 ± 0.1 |
| | <i>o</i> -Salicylic acid | 0.8 ± 0.1 | 1.0 ± 0.1 | 1.8 ± 0.1 | 5.8 ± 0.2 |
| | <i>p</i> -Salicylic acid | 5.6 ± 0.4 | 7.3 ± 0.3 | 11.8 ± 0.4 | 35.6 ± 0.6 |
| | Vanillic acid | 1.1 ± 0.1 | 1.5 ± 0.1 | 1.7 ± 0.2 | 2.4 ± 0.1 |

Caption of Tables

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

Table 2 Wood chemical composition according to ASTM standards for natural poplar, leached poplar in water, in lime solution and in cement suspension for 24 h (in percent of initial dry wood weight).

Table 3 Chemical composition of natural poplar soaks in water and lime solution for 1 and 24 h.

Caption of Figures



Fig. 1 Wood mass loss / initial dry wood weight ratio associated with leaching time in water  and lime solution  .

Fig. 2 Interpretation of the electrical conductivity curve of cement.

Fig. 3 Effect of poplar leaching time in water on cement hydration.

Fig. 4 Effect of poplar leaching time in lime solution on cement hydration.

Fig. 5 Effect on time to reach supersaturation with respect to portlandite of natural poplar, leached poplar in water (24 h), leached poplar in lime solution (24 h).

Fig. 6 Chromatogram of the trimethylsilylated soaks obtained by leaching poplar in lime solution for 24 h. 1: acid (a.) lactic; 2: a. glycolic; 3: a. pyruvic; 4: diethylene glycol ethyl ether; 5: a. malonic; 6: diethylene glycol; 7: a. benzoic; 8: glycerol; 9: a. succinic; 10: a. glyceric; 11: a. maleic; 12: a. hydrocinnamic; 13: a. malic; 14: a. *o*-salicylic; 15: a. *p*-salicylic; 16: a. vanillic.