

Effect of saccharides on the hydration of ordinary Portland cement



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HIGHLIGHTS

- The influence of saccharides on OPC was investigated.
- HPAEC and GC were used to characterised monomeric sugars and uronic acids.
- The influence of the fibre leachates on ordinary Portland cement was characterised.
- The pH of the leachates was correlated with the GAA and GLA acid concentration.

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ABSTRACT

Recently, the use of natural fibres as a sustainable alternative for reinforcements in cement-based materials has increased significantly. However, these lignocellulose fibres containing saccharides can have important retarding effects on cement hydration. The objective of this study is to characterise the effect of different organic compounds present in lignocellulose fibres on the cement hydration reactions. For a better understanding of this process, sugars such as fructose, glucose and sucrose, lignin and cellulose have been added to a cement paste as well as leachates of fibres. Experimental results show that glucose, mannose and xylose in fibre leachates had a significant impact on the cement hydration, slowing down the hydration for up to 2 days.

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1. Introduction

For many years, wood fibres, in various forms, have been mixed with cement to make composite materials such as wood wool cement boards (WWCB) and wood cement bonded boards (WCBB), collectively referred as cement wood composites. WWCB are manufactured with wood fibres, binder and additives: The wood is stored for 6–12 months in order to minimise the influence of sugars before it is cut to wood wool. Thereafter, wood is wetted, mixed with cement, placed into a mould, compressed and dried [1]. Two types of woods from regional forestry are very common to manufacture WWCB, which are spruce and poplar wood [2]. The binder is generally ordinary Portland cement, but magnesia cement, also known as *Sorell cement*, can be used as a binder as well [1]. WWCB shows a good resistance to decay and insects, a low density as well as good acoustical and thermal insulating properties [3]. The uses have focused primarily on these advantages of these composites. They are mostly used in parking decks, basement ceilings, floor

units, loft conversion or timber frame construction as sound barriers for acoustic absorption [1].

The use of a cellulosic material as filler or reinforcement in these composite materials has significantly increased over the past decade thanks to important the improvements in process technology, better economic incentives and increased sustainability concerns such as renewability and recycling of wood materials [4–8]. Nowadays, a large amount of inorganic and organic waste is generated with a huge environmental impact (waste dumps, pollution, etc.) [9,10]. These waste resources can be used to develop sustainable construction materials, for instance, cement fibre composites, where wood can be replaced by organic waste fibres like coir, hemp or oil palm fibres [11].

However, the development of cement wood composites has been slowed down by a lack of understanding of the mechanism involved in the reaction between cement and organic fibres [12]. Previous researchers have shown that not all wood types are compatible with cement, because generally, there is a retardation of cement hydration lowering the strength of the composite material below the requirements or even causing the disintegration of WWCB boards after compression [13–15]. The cause is saccharides contained in wood and the alternative fibres. [16]. Natural fibres

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contain different saccharides with different solubility in water due to the different structures of the saccharides and the fibres themselves. Those that can be dissolved create leachates, that can be analysed explaining how much and which saccharides, particularly monomeric sugars, are leaching from the fibres and how they could have an impact on the cement-fibre composite. The quantity and type of leached saccharides depend on the type of fibres and their growing conditions. However, not all types of sugar have the same inhibitory effect. In 2014, Na et al. [16] reported that glucose and sucrose have a greater retarding effect than others sugars.

The effect of saccharides on cement hydration can be explained by different phenomena. Firstly, the calcium binding capacity is important to consider, because general studies about organic retarders have shown that they have strong Ca^{2+} chelating groups which can prevent C-S-H gel formation [16–24]. Another effect is that sugars act through nucleation poisoning/surface adsorption forming semipermeable layers on the cement grains. They can also interact with different clinker minerals differently, for example, sucrose reacts with C_3S but does not react directly with C_3A and also accelerate ettringite formation, which it shows at early stages [17]. Another aspect to consider is the instability of some saccharides in a highly alkaline environment such as cement paste ($\text{pH} \sim 13$). The degradation products were more efficient than the original wood extractives at inhibiting cement hydration [16]. By-products such as sugar acid anions or calcium succinate cations appear to be more effective retarders than the sugars themselves [18]. In 2009, Simatupang [25] characterised other degradation products of saccharides mixed with cement as dihydroxy-butyric acid, gluco-saccharine acid, gluco-meta saccharine acid, lactic acid and mannose. Organic acids like uronic acids can suppress cement hydration and damage cement hydration products [26,27]. Several studies have investigated the effect of sucrose and glucose on cement hydration by using calorimetry, showing that sucrose had a strong retarding effect on cement hydration for up to several months [19,20,22].

These investigations show that hydration of cement in the presence of fibres is far more complex than the sum of hydration reactions of the individual minerals with saccharides. This study focuses on the influence of different little investigated natural fibres on cement hydration. Using leachates is a novel way to investigate this interaction. The studied fibres are bagasse, coconut husk fibres (coir), hemp, empty fruit bunches from oil palm trees and water hyacinth as well as spruce wood. Spruce wood is taken as a reference since it is commonly used for the production of cement fibres composites and is known to have relatively little influence on cement hydration [28]. The influence of saccharides

on OPC hydration is investigated by adding pure fructose, glucose, sucrose, lignin or cellulose to cement pastes. Chemical characterization of the leachates is performed in order to explain the interaction between OPC and fibres. The most problematic leached organics are monomeric sugars (arabinose, galactose, glucose, mannose, xylose) and galacturonic and glucuronic acids [29–32]. The monomeric sugars and the organic acids concentration are characterised by high performance anion exchange chromatography (HPAEC). The concentration of uronic acids is measured by gas chromatography (GC). The chemical composition of natural fibres is determined by HPAEC following the Tappi standards. Cement hydration was studied by calorimetry to determine the most dominant factor affecting the hydration of cement. The aim of this article is to compare saccharides with leachates of fibres and explain differences of various saccharides and leachates on cement hydration.

2. Materials and methods

2.1. Materials

Fructose (purity of 99.0%), glucose (purity of 99.5%), sucrose (purity of 99.5%), lignin were provided by Sigma-Aldrich and microcrystalline cellulose (20–160 μm particle size) was produced by Merck KGaA, Germany. Six types of organic fibres were studied (Fig. 1). Bagasse, coir, hemp, oil palm – empty fruit bunch and water hyacinth were provided by Wageningen Food & Biobased Research, the Netherlands, and spruce wood was provided by Knauf Insulation, the Netherlands. Their general chemical composition measured in this study, and comparison values from literature, are shown in Table 1. The chemical composition of natural fibres was determined by Tappi T222, Tappi UM250, Tappi T264 and HPAEC. Measurements were done two times. Spruce wood and natural fibres samples were stored in plastic bags at room temperature until use. CEM I 52.5R (OPC) from ENCI, the Netherlands, was used in this study as a binder. The chemical composition of the OPC is given in Table 2.

2.2. Leachate preparation

Fibres were dried at 60 °C to constant mass and soaked for 2 h at 80 °C in distilled water (water/fibres ratio of 5:1). Filtrate and fibres were separated with filter paper. The pH of the fibre leachates was determined by a pH Meter (Metrohm 780) after the leachates were cooled down to room temperature. Then, cement was mixed with the fibre leachate instead of water. The leachates were compared with cement mixtures with additionally added saccharides. Plain OPC mixed with water is used as a reference.

2.3. Calorimetry measurements

Calorimetry was performed with a TAM Air Isothermal calorimeter at a constant temperature of 20 °C. All sugars, lignin, cellulose and fibre leachates were mixed with OPC and water. The percentage of sugars and lignin mixed with OPC were chosen as follows: 1.0, 0.5 and 0.2 wt%, the amount of cellulose was 1.0 wt% (all based on cement). For glucose, weight fractions of 0.1, 0.05, 0.02 and 0.01 wt% were also

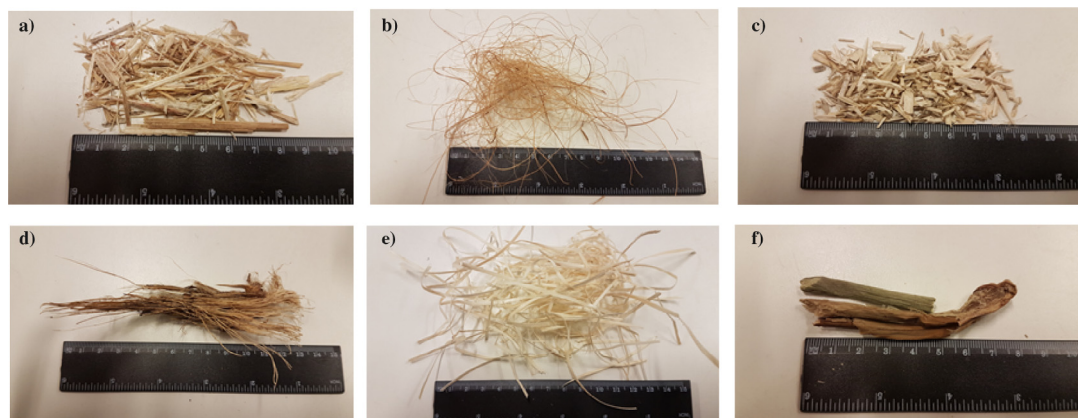


Fig. 1. Natural organic fibres: a) Bagasse; b) Coir; c) Hemp; d) Oil palm; e) Spruce; f) Water hyacinth.

Table 1
Chemical composition of natural fibres used in this study and presented in literature [18,38–50].

Fibres		Cellulose [%]	Hemicellulose [%]	Lignin [%]	Ash [%]	Extractive [%]
Bagasse	Literature	30.0–55.2	16.8	19.8–25.3	1.1–5.0	13.8
	Analysis	34.2 ± 0.9	40.1 ± 0.8	11.9 ± 0.3	3.9 ± 0.03	18.6 ± 0.15
Coconut	Literature	32.0–43.4	0.2–31.1	20.5–45.8	2.2–4.8	4.2
	Analysis	36.6 ± 0.15	37.0 ± 0.15	22.2 ± 0.05	1.9 ± 0.04	5.7 ± 0.05
Hemp	Literature	55.0–68.0	10.9–16.0	4.0–10.0	–	3.9
	Analysis	37.4 ± 1.9	41.5 ± 1.8	17.2 ± 0.2	2.5 ± 0.12	4.9 ± 0.05
Oil palm	Literature	29.0–65.0	12.0–38.0	13.0–37.0	1.0–6.0	8.6
	Analysis	32.8 ± 1.93	42.7 ± 1.99	14.9 ± 0.21	7.8 ± 0.02	11.3 ± 0.1
Spruce wood	Literature	47.0	25.3	27.7	–	1.7
	Analysis	41.6 ± 1.08	37.3 ± 1.1	19.4 ± 0.01	0.4 ± 0.02	2.2 ± 0.02
Water hyacinth	Literature	20–53	14–35	10	20	17.8
	Analysis	37.9 ± 3.62	37.6 ± 3.62	6.6 ± 0.04	16.8 ± 0.17	23.6 ± 0.21

Table 2
Chemical composition of ordinary Portland cement CEM I 52.5R used in this study.

Chemical composition	[%]
CaO	64.60
SiO ₂	20.08
Al ₂ O ₃	4.98
Fe ₂ O ₃	3.24
K ₂ O	0.53
Na ₂ O	0.27
SO ₃	3.13
MgO	1.98
TiO ₂	0.30
Mn ₂ O ₄	0.10
P ₂ O ₅	0.74
Cl ⁻	0.05

measured. The water/cement ratio was kept constant for all prepared mixes ($w/c = 0.5$). After mixing, the samples were placed in the calorimeter to observe and determine the cement hydration behaviour. The measurements were done three times. The standard deviation of calorimetry results is about 5%.

2.4. Concentration of monomeric sugars

The concentration of the monomeric sugars in the different leachate solutions was determined by high performance anion exchange chromatography (HPAEC) after H₂SO₄ hydrolysis. The solution is freeze-dried (1–4 ml of leachates) and vigorously mixed in Pyrex test tubes with 150 μ l of ice-cold H₂SO₄ (12 mol.L⁻¹) in a water bath at 30 °C for one hour. Samples were then removed from the water bath and mixed with an H₂SO₄ (4%) solution. Samples are then placed in an autoclave for 60 min at 120 °C. After being cooled down, 5 mg of ribose was added to each sample as an internal standard. Hydrolysed leachates were diluted with deionized water at

a different ratio (from 1:10 to 1:100) prior to analysis. Measurements were done with a Thermo Scientific Dionex ICS-5000 system. Samples were separated on a Dionex CarboPac PA1 carbohydrate column.

2.5. Concentration of uronic acid

The concentration of uronic acids was measured by gas chromatography (GC) after methanolysis according to Sundberg [33]. Samples were lyophilized and then hydrolysed in 2 M methanolic HCl at 100 °C for 5 h, followed by addition of pyridine and sorbitol as an internal standard at room temperature. After evaporation under N₂, samples were silylated over night with HMDS (hexamethyldisilazan, Sigma-Aldrich) and TMCS (trimethyl-chlorosilan, Sigma-Aldrich) in pyridine, injected into a GC/FID system (7890A, Agilent Technologies) at 260 °C, and separated on an HP-5 column (30 m, 0.32 mm ID, 0.25 μ m film thickness) using the following temperature program: 150 °C for 1 min, 150 °C to 220 °C at 4 °C/min, 220 °C to 320 °C at 20 °C/min, 320 °C for 6.5 min. Calibration of was done using identically treated authentic standards of D-glucuronic and D(+)-galacturonic acids

3. Results and discussions

3.1. Effect of saccharides on the hydration of cement

Figs. 2–4 depicts the calorimetry measurements of the effect of pure saccharides and lignin on cement hydration. Figs. 2a–4a show the heat flow during the exothermic reaction of OPC over time. Figs. 2b–4b show the total, cumulatively released heat during that reaction. The heat flow shows the kinetics of the hydration, indicating retardation in reactions. The total, cumulative heat gives an estimate of how far the system has hydrated. For the released heat of the different cement pastes, there is a general trend that the initial heat within the first hours of hydration was higher with saccharides and lignin compared to the reference. A possible reason for that could be an increased initial dissolution of phases

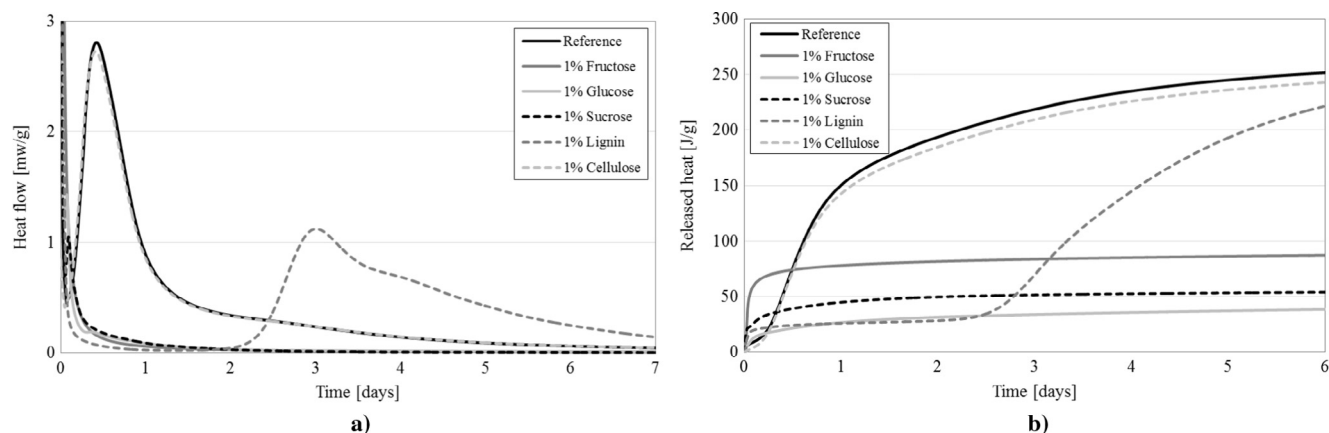


Fig. 2. Effect of 1% sugars, lignin and cellulose on the cement hydration of OPC paste at 20 °C: a) Heat flow, b) Released heat.

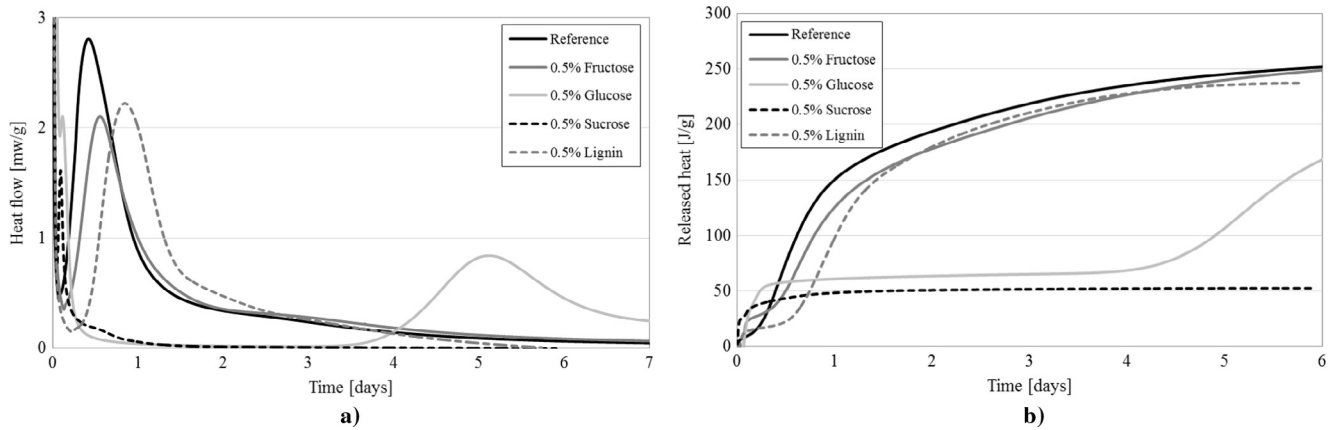


Fig. 3. Effect of 0.5% sugars, lignin and cellulose on the cement hydration of OPC paste at 20 °C: a) Heat flow, b) Released heat.

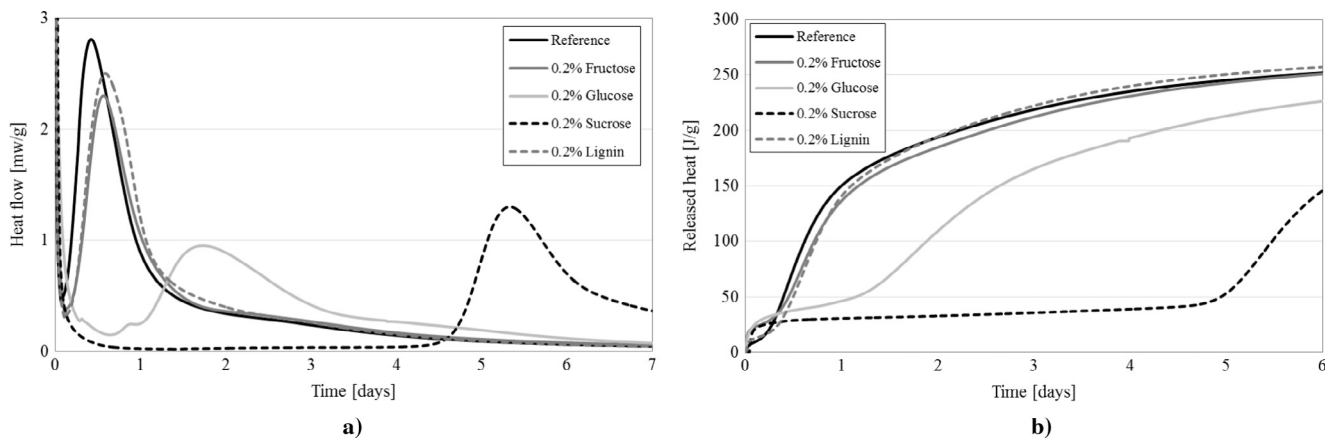


Fig. 4. Effect of 0.2% sugars, lignin and cellulose on the cement hydration of OPC paste at 20 °C: a) Heat flow, b) Released heat.

including sulphate phase promoting the initial formation of ettringite. The higher the concentration of sugars, the later the maximum heat flow of the main hydration occurs due to cement retardation. It can be explained because sugar decreases the concentration of Ca^{2+} ions in cement pore solution and delays the formation of hydration products. Fig. 2a shows that the addition of 1% cellulose to the cement paste cured at 20 °C does not have a retarding effect, but it causes the slight acceleration of cement hydration. A likely reason for this is that it provides additional crystallisation seeds for the hydration products which can speed up the reaction slightly [34]. The released heat after 24 h (Fig. 2b) is almost the same as for the reference. Since cellulose does not interact with cement to cause retardation, no further characterization with cellulose was done in this study.

Lignin does have a strong impact on cement hydration, but the retardation is low compared to simple sugars [35]. The addition of 1% of lignin to the cement paste delays the maximum of the cement hydration for 63 h (Fig. 2a) [36]. The cement paste with 0.5 and 0.2% of lignin was slowed down for only 10 and 6 h, respectively.

The highest retarding effect has been observed in cement mixtures with sugars at a concentration of 1% (fructose, glucose and sucrose). No hydration was observed within the first seven days after mixing the samples.

Among the tested sugars, fructose, has the lowest impact on cement hydration. The addition of 0.5 and 0.2% of fructose had a much lower impact on the maximum of the cement hydration than other sugars with the same concentrations.

Additions of 0.5 and 0.2% of glucose to the cement paste (Figs. 2a, 3a and 4a) resulted in a retardation of the cement hydration peak of respectively five and two days. Since glucose has a great impact on the cement hydration and knowing that glucose is often generated by cellulosic fibre in water [37,38], lower concentrations from 0.01 to 1% of glucose have also been used in this study (Fig. 5a). Fig. 5b shows the released heat after 24 h is the same for the reference as for 0.01 and 0.02% of glucose. The relation between the glucose concentration and the retardation of the cement hydration is depicted in Fig. 6. Moreover, calorimetry of almost every mixture shows a first small peak after the initial wetting, showing that the cement hydration starts with C_3S reacting before the main hydration starts [39,40]. The addition of sucrose changes the microstructure as well as a surface of the hydrating cement particles and hydration products. It caused the formation of a temporary barrier leading to a further hydration [22].

Sucrose has the highest impact on the cement hydration peak (Fig. 4a) since 0.2% of sucrose delayed the hydration for five days. Sucrose is a disaccharide combining glucose and fructose monomer units [41]. The reason for the different behaviour of the simple sugars is likely that each sugar has different stabilities in a high pH environment like cement and that they react differently with different cement minerals [42]. Sucrose is absorbed in multiple layers at cement surfaces. Cement hydration is strongly influenced by interactions between sucrose molecules and their alkaline degradation and the silicate and aluminate species at hydrating cement particle surfaces [42]. Sucrose has the biggest retarding effect at the same concentration as compared to other saccharides due to

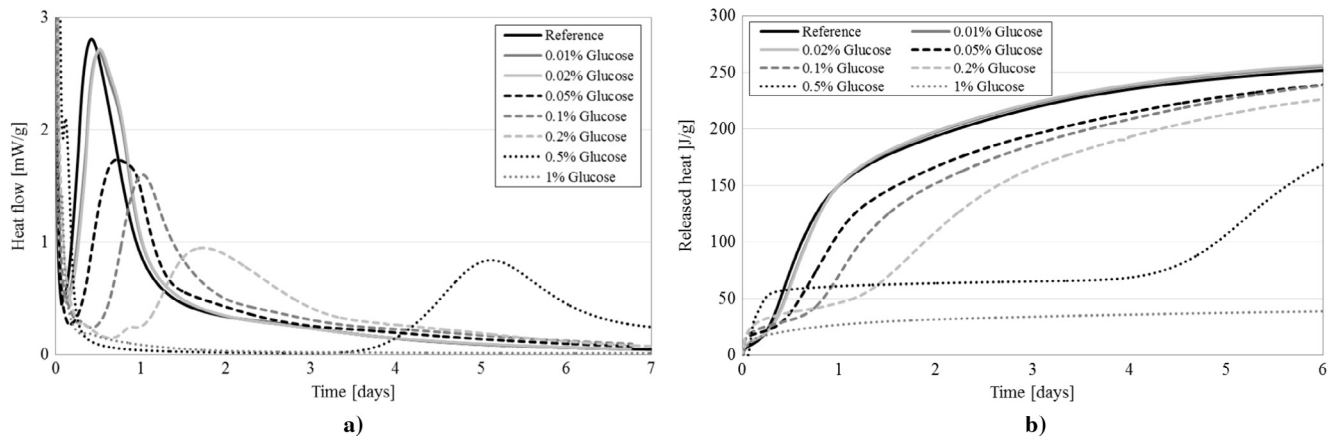


Fig. 5. Effect of glucose on the cement hydration of OPC paste at 20 °C: a) Heat flow, b) Released heat.

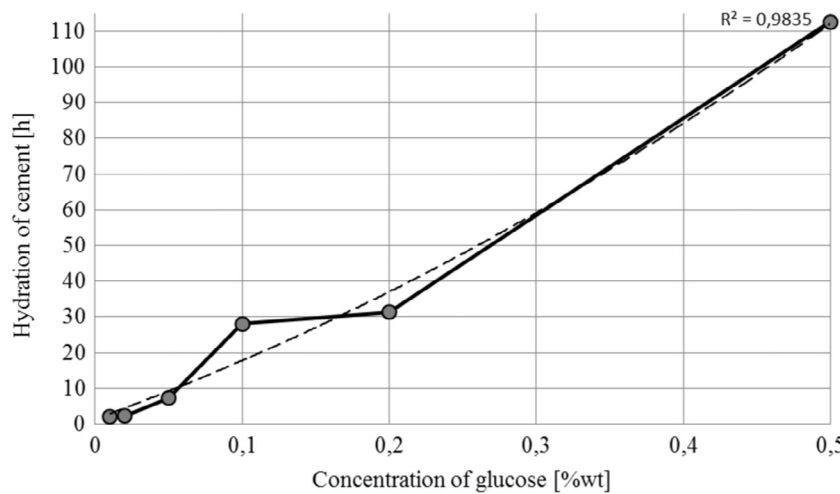


Fig. 6. The relation between a concentration of glucose and the retardation of the cement hydration peak.

its chemical structure. It is a disaccharide and has structure inducing a stronger steric hindrance than other saccharides [20].

3.2. Effect of fibre leachates on the hydration of cement

Fibre leachates were added to the cement instead of water and calorimetry results were obtained, showing the potential influence of different natural fibres on the cement hydration, (Fig. 7a and b).

The highest retardation effect is observable with bagasse leachate, showing more than three days without any cement hydration peak. Leachates of oil palm and water hyacinth show a similar behaviour, delaying the maximum of the hydration by approximately 47 h. Leachate of coir and hemp had a lower effect on the cement hydration (approximately one hour delay of the hydration maximum).

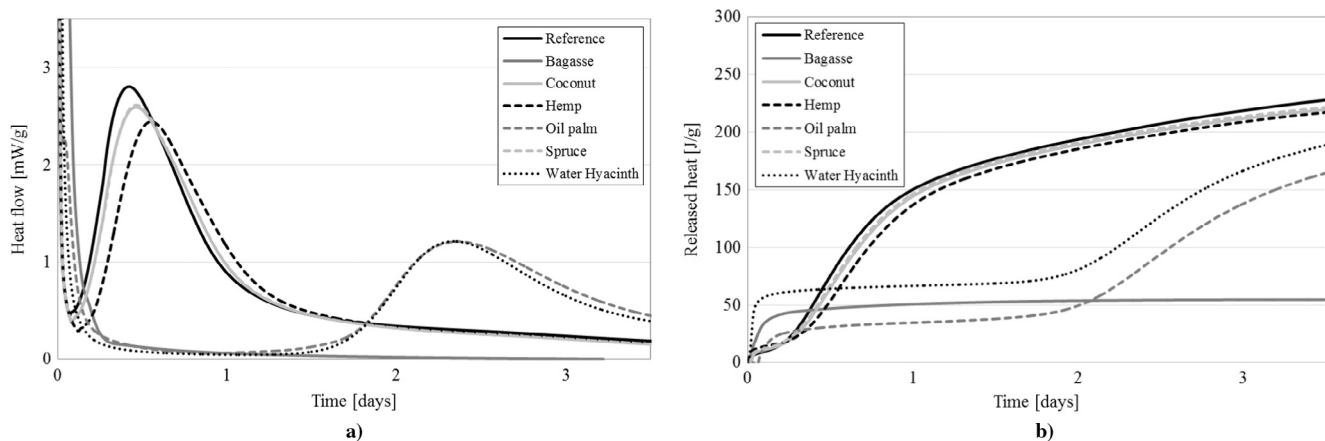


Fig. 7. Effect of solution of fibre extractive on the cement hydration of OPC paste at 20 °C: a) Heat flow, b) Released heat.

Table 3
Results of pH measurement of the leachates.

Fibre	pH
Bagasse	4.9
Coconut	6.2
Hemp	6.7
Oil palm	5.8
Spruce	6.6
Water hyacinth	5.1

As expected the leachate of spruce showed almost no retardation, it had the lowest effect on the cement hydration. This hydration peak is very close to the reference one.

3.3. Composition and pH of the leachates

The chemical composition of leachates was determined by HPAEC in order to evaluate the link between the leached organics and cement retardation. Table 4 shows monomeric sugars concentration of the different leachates. Five monosaccharides were measured, namely arabinose, galactose, glucose, xylose and mannose. Those five monomeric sugars are the main hydrolysis products of cellulose and hemicellulose and are also components of the plant sap [31]. These monosaccharides can be found primarily in the hemicellulose structure but the concentration of these sugars is highly dependent on the fibres.

The glucose concentration in the bagasse leachate was very high compared to other fibres (7.4 mg/ml). A high glucose content is expected because bagasse is the remaining waste from sugar cane that is harvested for sugar production. It also causes the long retardation in the cement hydration, because glucose has a very high impact on cement hydration as shown in Section 3.1, due to a disruption of C-S-H gel formation [18].

After bagasse, oil palm leachate has the highest total sugar content with 2.65 mg/l, which explains the long cement retardation. In contrast, water hyacinth leachate contains relatively low amounts of sugar (0.73 mg/l) but causes similar retardation.

Spruce wood leachate contains the lowest amount of sugars since the producers of WWCB use very simple methods to remove sugar from the wood. Logs with a length of around 50 cm are stored outside for 6–12 months to reduce sugar. This characteristic explains why spruce wood is commonly used as reinforcement and filler for board production.

In general, there is a good correlation between the total amount of monomeric sugars in the leachate and the total heat released during the first 3.5 days of cement hydration, which shows the degree of reaction in that time (Fig. 8a).

Hemicellulose is described in the literature as the main source of monomeric sugars [43]. However, by comparing the effect of leachates of fibre on the cement hydration (Fig. 7a) and the chemical composition of fibre (Table 1), a high hemicellulose fraction does not correlate well with a high cement retardation. It means that rather than the simple fibre composition, the nature of the leachate, i.e. its chemical composition and concentration are a better predictor.

If monomeric sugars can cause a significant delay to the cement hydration due to their degradation products, the pH of the cement during the hydration phase is also a critical factor and may be affected by the numerous carboxylic acids leached by cellulosic fibres [42,44]. In order to evaluate the effect of the acid on the cement hydration, the overall pH of the leachates was measured along with, two types of uronic acid (Fig. 4) here, namely the galacturonic acid (GAA) and glucuronic acid (GLA), which are reported as the two main leached acids from natural fibre [45]. GAA is derived from galactose and GLA is derived from glucose [46]. Table 4 also depicts the concentration of leached GLA and GAA from the selected natural fibres. A very low amount of GAA has

Table 4
Monomeric sugars concentration in the leachates measures by HPAEC after H₂SO₄ hydrolysis and uronic acids concentration in the leachates measures by CG after methanolysis.

Sample	[mg/ml]							
	Arabinose	Galactose	Glucose	Xylose	Mannose	Total sugars	GAA	GLA
Bagasse	0.24 ± 0.02	0.39 ± 0.03	7.4 ± 0.33	0.61 ± 0.03	0.56 ± 0.02	8.65 ± 0.43	0.07	0.74 ± 0.01
Coconut	0.21 ± 0.02	0.08 ± 0.01	0.04 ± 0.01	0.56 ± 0.05	0.02 ± 0.01	0.91 ± 0.1	0.02	0.07 ± 0.01
Hemp	0.11 ± 0.01	0.28 ± 0.03	0.07 ± 0.01	0.09 ± 0.01	0.08 ± 0.01	0.63 ± 0.07	0.02	0.07 ± 0.01
Oil palm	0.65 ± 0.01	0.62 ± 0.01	0.75 ± 0.01	0.36 ± 0.01	0.27 ± 0.01	2.65 ± 0.05	0.02	0.39 ± 0.01
Spruce	0.03 ± 0.01	0.04 ± 0.01	0.03 ± 0.01	0.12 ± 0.01	0.07 ± 0.01	0.29 ± 0.05	0	0.01 ± 0.01
Water hyacinth	0.08 ± 0.01	0.29 ± 0.02	0.24 ± 0.02	0.03 ± 0.01	0.09 ± 0.01	0.73 ± 0.07	0	0.13 ± 0.01

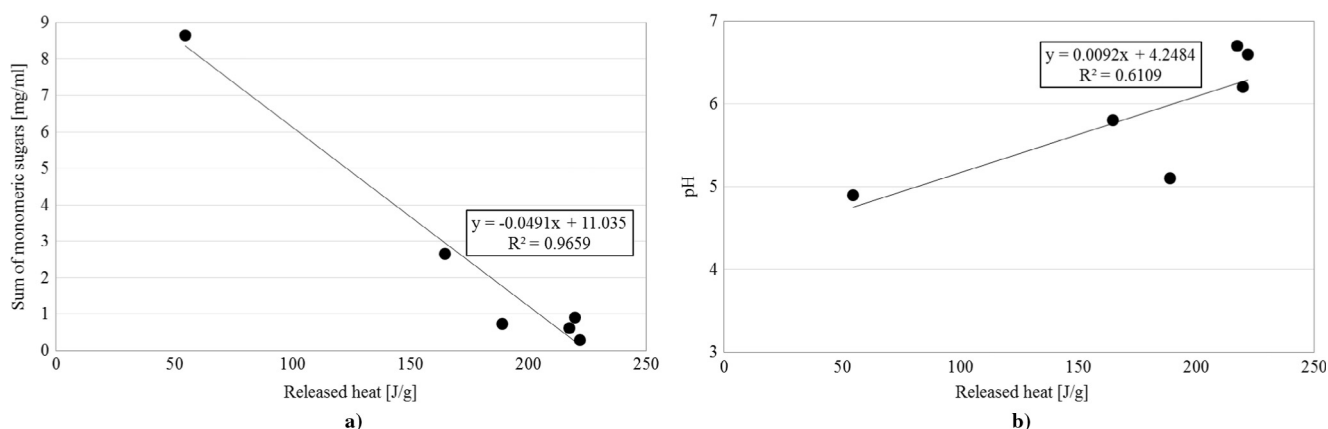


Fig. 8. A comparison of the a) total sugar content and b) pH with the released heat after 3.5 days.

been measured in all leachates from 0 mg/ml for the spruce and water hyacinth to 0.07 mg/ml for the bagasse.

The highest GLA concentration was measured in the leachate of bagasse (0.74 mg/ml) whereas the lowest was measured in the leachate of spruce (0.1 mg/ml). Leachates of coir and hemp have the same GLA concentration (0.07 mg/ml) and water hyacinth has a slightly higher GLA (0.13 mg/ml). Table 3 shows the pH of all leachate solutions. Bagasse pH has the lowest pH (4.9), followed by leachates of water hyacinth and oil palm (5.1 and 5.8 respectively). With the exception of water hyacinth, all pH values correspond well with the amount of GLA measured, as well as the total heat released during the first 3.5 days of cement hydration (Fig. 8b). The low pH of the water hyacinth leachate is likely caused by organic acids other than GLA and GAA, for example, levulinic and shikimic acids [47], and cause the strong retardation in absence of high sugar concentrations in the leachate.

A comparison of the sugar leachates (Fig. 8a) and the acid concentration (Fig. 8b) with the normalised heat after 3.5 days shows a good correlation except for water hyacinth. Indeed, water hyacinth is known for its important water uptake which can significantly affect the retardation of cement hydration as it has been shown in several studies [48,49]. The correlation between total sugar content and released heat and can be used to predict the retarding behaviour of different fibres. On the other hand, the pH of the leachate (Fig. 8b) can also be used as a fast indicator of the cement-fibre compatibility although the correlation is less clear.

This suggests that a low pH is a useful indicator for a strong retarding effect. Cement paste has a pH of around 12–13 and acidic solutions could slow down the cement hydration [50] and cause a decrease of crystallinity, strength and hydration of cement [27].

4. Conclusion

The influence of pure saccharides and the leachates of six natural fibres on the hydration of OPC have been investigated. The following conclusions can be drawn:

- Sucrose had the highest impact on the cement hydration, while lignin and cellulose had very little impact. At lower concentrations, fructose and lignin showed similar behaviour. The fibre composition, particularly a high hemicellulose fraction, does not appear to have a correlation with the cement hydration retardation.
- Bagasse leachate caused the longest cement hydration retardation due to the high sugar content, followed by oil palm and water hyacinth leachate. Spruce leachate had very little retardation influence due to the low sugar content.
- The total monomeric sugar content of the investigated fibre leachates corresponds well with the total heat released during cement hydration as measured with calorimetry. This shows that the reaction of natural fibres leachates with OPC is a good way to evaluate the cement/fibre compatibility. Compared to mixing natural fibres directly with OPC, it overcomes the influence of the fibre size and the water adsorption of the fibres on the reaction.
- The pH of the leachate is well correlated with the content of GLA and GAA and the total released heat during hydration. Thus pH can be a good indicator in order to characterise fibre/cement compatibility in addition to other methods such as chemical analysis of the fibres and their leachate.
- In general, using isothermal calorimetry to study fibre leachates mixed with cement is an easy and cost efficient way to investigate the compatibility of different fibres for a cement fibre composite. Using a leachate prepared as described in this study allows a direct comparison without having to take other factors

such as fibre size or water adsorption into account. This can be helpful because both properties influence the hydration behaviour of cement and can vary widely between different fibres.

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