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Use of alternative organic fibres in cement composites

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Abstract

Large quantities of lignocellulose wastes are generated worldwide from various sources. While sustainable building materials are becoming very important. Society endeavours to maximize the utilization of wastes in all industrial sectors known as cyclic economy. These by-products and residues, such as waste wood, sugar cane bagasse, coconut fibres, rice straw, rice husk, reeds, corncobs and others, can be utilized to manufacture cement–bonded construction materials. However, the use of lignocellulose fibres, may have retarding effects on cement hydration. The mineralogical analysis and the influence of organic material on cement hydration is measured by using calorimetry and X-Ray diffraction measurements. The objective of the present investigation is to improve understanding of cement hydration reactions affected by different organic compounds present in lignocellulosic fibres. The effect of pure saccharides (fructose, glucose, sucrose, lignin and cellulose) at concentrations of 1 and 0.5 % show a slow down of cement hydration for more than a few hours.

Keywords: Hydration, retarding effect, sugars, natural fibres, waste

1. Introduction

Wood fibres, in various forms, have been mixed with cement to make composite materials for about 100 years [1]. The use of wood and fibre particles has increased rapidly over the past decade, primarily because of the improvement in process technology, and secondly due to economic factors and increased sustainability concerns, such as renewability and recycling of wood and less consumption of cement [2–6]. Nowadays, a large amount of inorganic and organic waste is generated globally [7, 8] with huge potential environmental impact. These waste resources have increasingly been used to develop sustainable construction materials, for instance as fibre-cement composites. Cement can be replaced with rice husk ash and wood with organic waste fibres, like coconut fibres, rice straw, oil palm fibres [9]. However, the development of cement wood composites has been slowed down by a lack of basic understanding of the mechanism involved in the reaction of cement and wood [1]. Previous researchers have shown that not all wood types are compatible with cement because some retard the setting of cement [10, 11] and retardation has also been found to vary [12–14]. Therefore, a number of attempts have been made to accurately assess the compatibility of wood with ordinary Portland cement [15]. Hence this study will focus on the retardation effect of saccharides and natural fibres with ordinary Portland cement.
Wood, as well as alternative organic fibres, can contain many inhibitory saccharides which may hinder or stop the hydration of cement. However, not all have the same inhibitory effect. The quality and quantity of leached saccharides depends on the type of fibres and their growing conditions. The effect of these organic retarders has been widely attributed to a mechanism based on the absorption onto the clinker grains and nucleation poisoning of hydrate surfaces [16]. There are two main properties of saccharides which affect cement hydration. Firstly, their alkaline stability and secondly, their calcium binding capacity. Saccharides have been reported to hydrolyse in the strong alkali environment of cement paste, and their degradation products appear to be more effective retarders than the sugars themselves [17]. Baoguo et al. [18] has shown that sucrose has greater retarding effect than glucose at the same concentration while the research of Sandermann and Brendel [19] has shown no essential effect of fructose on cement hydration in different concentration levels. Juenger and Jennings [20] compared cement with 1wt.% of sucrose addition and cement paste without sugar addition using calorimetry measurements. They have found that an addition of 1wt.% to the cement paste causes the retarding of cement hydration for several months. Bishop and Barron [21] have presented cement hydration inhibition with sucrose and lignosulfonate (product from the production of wood pulp) in concentration of 1% and 1.75%. Both materials stopped the cement hydration, however each material acted through a different reaction mechanism. Sucrose acted through nucleation poisoning/surface adsorption, while lignosulfonates was involved the formation of a semipermeable layer on the cement grains.

As mentioned previously, the use of natural organic fibres as a material used for production of fibre-cement composites is still a limited investigated area. The hydration of cement and fibres is far more complex than the sum of hydration reactions of the individual minerals with saccharides. The study analyses the influence of natural fibres and saccharides in order to demonstrate their influence on cement hydration. It uses XRD and calorimetry measurements, to determine which natural fibres and saccharides are the most dominant factor affecting the hydration of cement. Each of the seven type of natural fibres (bagasse, coconut fibres, hemp, oil palm fibres, rice straw, spruce wood and water hyacinth), have been investigated. This paper aims to analyse the influence on cement hydration mixed with naturel fibres and saccharides and to assess which fibre can be more suitable for fibre-cement composites.

2. Materials and methods

2.1 Materials

In total, fructose, glucose and sucrose will be referred to as sugars, while lignin and cellulose are named polysaccharides. In addition nine types of organic fibres as well as their solution were used in this study. Hemp, coconut, oil palm – empty fruit bunch (EFB), water hyacinth, bagasse and rice straw were provided by Wageningen UR, the Netherlands. Spruce wood was obtained from Knauf Insulation B.V., the Netherlands. Subsequently spruce wood and natural fibres samples were stored in plastic bags in the laboratory until use. The fibres were milled and stored in the desiccator. The milled fibres and saccharides were mixed with ordinary Portland cement (OPC) CEM I 52.5R which is commercially available. Fructose ($C_6H_{12}O_6$, MW:180.16 g/mol) with a purity of 99.0 %, glucose ($C_6H_{12}O_6$, MW:180.16 g/mol) with a purity of 99.5 %, sucrose ($C_{12}H_{22}O_{11}$, MW:342.30 g/mol) with a purity of 99.5 % and lignin, alkali with 4 % sulfur were delivered by Sigma – Aldrich.
2.2 Calorimetry measurements

The hydration tests were done with a TAM Air Isothermal calorimeter at a constant temperature of 20°C. All sugars, lignin, cellulose and fibre extractives samples were mixed with ordinary Portland cement CEM I 52.5R and water. The additional percentage of sugars and lignin mixed with ordinary Portland cement were chosen as follows: 1.0 and 0.5 wt%, the amount of cellulose was only 1.0 wt%. The water/cement ratio was kept constant for all prepared mixes (w/c = 0.5) and the fibres/cement ratio was 0.08.

2.3 XRD measurements of cement/fibres mixtures

In order to determine the mineralogical composition of hydrated products in cement composites containing natural fibres, the X-Ray diffraction analysis was performed a D5000. Cement hydration was stopped at 12 days after mixing with alcohol. The samples were crushed, milled into powder and dried in the oven at 100°C until a constant mass was reached. Subsequently, X-ray diffraction analysis was conducted on the crushed powder samples.

3. Results and discussions

3.1 Saccharides-cement calorimetry measurements

Addition of 1 % saccharides to the cement paste cured at 20°C is shown on Fig. 1, it shows that the fructose, glucose and sucrose have an adverse effect on the cement pastes. They retard the cement hydration for more than 12 days. The addition of 1 % of lignin retards the maximum of hydration for 63 hours. The addition of 1 % of cellulose does not have a retarding effect. This may be caused by the positive effect on the water absorption into cement paste [22] because the cellulose microfibers have modified the pore structure of cement mortars and therefore have strongly increased absorption resistance. Since it has no further influence, it was decided not to investigate cellulose further.

![Figure 1. Effect of 1% sugars, lignin and cellulose on the heat flow of OPC paste at 20°C](image)
The addition of 0.5% saccharides to the cement pastes cured at 20°C shows that the maximum hydration is slowed down (Fig. 2), but less so than with 1% saccharides. It can be seen that fructose delays the hydration for five hours, glucose retards the maximum hydration approximately five days, sucrose shows no hydration in 7 days, while lignin delays the maximum of hydration for 10 hours.

![Figure 2. Effect of 0.5% sugars, lignin and cellulose on the heat flow of OPC paste at 20°C](image)

Figure 2. Effect of 0.5% sugars, lignin and cellulose on the heat flow of OPC paste at 20°C

The highest retarding effect has been observed in cement mixtures with 1% sugars (sucrose, glucose and fructose). The results are similar with results mentioned in literature [20], where sucrose has been shown to delay the hydration for three months. The retardation effect of sugars is different probably due to the different stabilities in the pH environment of cement and that they may be reacted differently with different clinker minerals [23].

### 3.2 Fibre-cement calorimetry measurement

Isothermal calorimetry results of natural fibres without any pre-treatment added to the cement pastes cured at 20°C are shown in Fig. 3. As it was expected the fibres have a huge retarding effect on the cement hydration. The hydration peak of cement with flax and spruce fibres were measured at 72 hours and the measurements have shown slow down hydration of cement caused by fibre addition. The peak for spruce fibres is higher than the peak for flax fibres. The retardation effect of the fibres was high because a fine powder of fibres (<0.18 mm) was used. The powder was used because it can be compared to investigations in literature. Using bigger fibres overall will lead to a more realistic result in regards to board production.
3.3 X-ray diffraction of hydrated product of cement with saccharides

The results of various hydrated mixtures with 1% wt. saccharides are shown in Fig. 4. The hydrated products very similar, only pure cement includes a small amount of ettringite. As it is can be seen in Fig. 4, pure cement and OPC with lignin and cellulose show a similar composition, in OPC mixed with sugars have been observed similar behaviour as in case of mixes with cellulose and lignin, however different compared to the other materials. The peak in position 18° 2θ (Ca(OH)₂) can be seen only in pure cement and cement with lignin and cellulose. The presence of Ca(OH)₂ means the cement is more hydrated than cement with fructose, glucose and sucrose. The XRD measurements showed the following mineralogical composition: Ca(OH)₂, Ca₃SiO₅, Ca₂SiO₄, CaCO₃ and ettringite. The same results are obtained from XRD measurement with 0.5% wt. saccharides.
3.4 X-ray diffraction of hydrated product of cement with natural fibres

Fig. 5 and 6 shows the hydration products of cement with natural fibres. Each measured sample from Fig. 5 shows similar results. The highest peak appeared in the cement mixed with oil palm fibres and the lowest peak was cement with spruce wood. The cement pastes contain \( \text{Ca(OH)}_2 \), \( \text{Ca}_3\text{SiO}_5 \), \( \text{Ca}_2\text{SiO}_4 \) and \( \text{C}_4\text{AF} \). Fig. 6 shows a similar behaviour as it has been found in case of OPC mixed with oil palm fibres and spruce wood. The measured peaks have been identical, besides the peak in position 18° 2θ, where the cement with bagasse, hemp and water hyacinth showed less \( \text{Ca(OH)}_2 \). This means that the cement is less hydrated than cement with oil palm and spruce as can be seen in the calorimetry measurements too. The cement pastes contain \( \text{Ca(OH)}_2 \), \( \text{Ca}_3\text{SiO}_5 \), \( \text{Ca}_2\text{SiO}_4 \) and \( \text{C}_4\text{AF} \).

![Figure 5. X-ray diffraction data for cement with natural fibres (oil palm fibres and spruce wood) mixtures cured at 20°C](image5)

![Figure 6. X-ray diffraction data for cement with natural fibres (bagasse, hemp and water hyacinth) mixtures cured at 20°C](image6)
4. Conclusion and Outlook

The influence of saccharides and natural fibres were investigated in this study. The following conclusions can be made:

- Cement hydration was the most retarded with the addition of 1%wt. of sucrose added to cement paste and the least with 1% cellulose and 0.5% lignin. Sucrose has the biggest influence on cement hydration in all measured concentration, which is in line with results presented elsewhere. Addition of 0.5%wt. saccharides to the cement pastes had lower effects on the cement hydration.

- Calorimetry results of natural fibres were show that cement pastes mixed with flax and spruce fibres had less effect of the cement hydration than another fibres.

- XRD measurements have been used to determine the mineralogical composition the cement pastes with saccharides and fibres. The composition of cement with 1% and 0.5% addition of saccharides is Ca(OH)$_2$, Ca$_3$SiO$_5$, Ca$_2$SiO$_4$ and ettringite has found in pure cement.

- The content of Ca(OH)$_2$ in cement paste measured with XRD can be used as an indication of the degree of hydration in addition to calorimeter measurements. Cement pastes with cellulose, lignin, oil palm and spruce are more hydrated than other samples.

- From those results it could be said which fibre is good option. Flax and spruce fibres had good results than another fibres.

As it was mentioned previously, big influence on the cement hydration had fibres because it was used small particle size. Next step in study will be to use fibres with gibber size, around 10mm. It will be used pre-treatment of fibres to determine better results of the cement hydration with natural fibres.

References


570, 1964.


