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COMPATIBILITY OF PLANTS WITH A MINERAL BINDER

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Abstract

Hemp concretes are the most widely used biobased concretes in France. However, their growth is still limited by the lack of knowledge and high variability of the performances of biobased concretes, especially for their mechanical properties. These results are related to interactions between the mineral binder and plant compounds that modify the hydration of the cement. In this work, the interactions between cement and eight types of hemp shiv, and a flax shiv are studied by isothermal calorimetry. The setting delays observed in the presence of plants are interpreted by analyzing the molecules extracted from these plants in water. A link can be observed between the setting delay and the coloration of the extract solutions or their concentration in reducing sugars and in polyphenols.

These results constitute a basis in the objective to define an indicator enabling to predict the compatibility between plants and mineral binders.

Keywords:

Plant based concretes, cement, vegetal, compatibility, sugar, extraction, polyphenols, hydration

1 INTRODUCTION

Biobased materials, produced from biomass, are being increasingly used on a massive scale in the construction sector. From an environmental point of view, they allow the storage of CO₂ during their entire lifetime thanks to the mechanism of photosynthesis. In addition, their high porosity and chemical composition give them high hygrothermal and acoustic insulation properties. Therefore, these materials are key alternatives to traditional construction materials for the energy-efficiency renovation of existing buildings. Up to now, biobased construction materials are mostly used for their insulating properties than for structural purposes. For example, mortars and concretes, which are made of plant aggregates, a mineral binder, and water, are used in walls, floors, ceilings or as plasters (Amziane and Collet, 2017). Hemp concretes are the most widely used biobased concretes in France, but thanks to the variety of locally available bioresources and binders that can be associated, it is theoretically possible to formulate a very wide range of products.

Although construction rules exist for these materials (Collectif SEBTP, 2012), their growth is still limited by the lack of knowledge and high variability of the performances of biobased concretes, especially for their mechanical properties (Delannoy et al., 2018; Niyigena *et al.*, 2018). Besides, even if it is not the main performance expected, they still require sufficient mechanical resistance to be unmolded, transported onsite, and handled. Several parameters, such as setting delays of the binder or potential degradation of plant particles have been identified to explain their mechanical weakness. Moreover, on existing buildings, problems of powdering of the materials in the core have been observed even when using vegetal-mineral couples validated by the construction rules.

A correlation can be done between the biochemical nature of raw plant materials, including the plant cell wall quality (types, and relative sugars, phenolics, and lignin contents) and the analysis of the leachates, and the mechanical properties of biobased concretes (Bourdot et al., 2019; Delannoy et al., 2020).

Existing studies explain the impact of plants on the setting of mineral binders with two main mechanisms:

- The absorption of organic molecules on anhydrous cement grains, leading to the formation of a thin low permeability layer surrounding them. Therefore, the limitation of the diffusion of water induces a lack or a total inhibition of the hydration of the binder (Na et al., 2014; Doudart de la Grée et al., 2017). This mechanism involves sugars. The impact of their nature and amount has been particularly investigated (Boix et al., 2020).
- The capacity of some organic compounds to chelate Ca²⁺ ions (Milestone, 1979), resulting in the formation of Ca²⁺ complex which blocks the nucleation sites and prevent the cement hydrates growth (Bourdot et al., 2019). This mechanism is correlated with presence of specific plant polyphenols, especially flavonoïds (Miller and Moslemi, 1991).

In a previous study (Delannoy *et al.*, 2020), three hemp concretes having the same formulation, but containing three hemp shiv of different origins, had very different compressive strengths from each other. The characterization of the compounds extracted from the plants showed the impact of the quantity of sugars on the setting time of the cement. Other parameters, such as the concentration of polyphenols, can also modify the hydration kinetics of the binder.

In this work, the same methodology is used to generalize these first results obtained by characterizing the interactions between a CEM I type cement and eight hemp shiv from different origins. Another variety of plant material, flax shiv, is also included in this study. The influence of the presence of vegetal on the hydration of the binder is first characterized by isothermal calorimetry in order to measure the setting delay of the cement. In a second step, the extractible compounds of plants in water are also analyzed: the total amount of extractible compounds, reducing sugars and monosaccharides, as well as polyphenols.

The final objective of this project is to define an indicator to predict the compatibility between a mineral binder and vegetal particles in order to generalize the production of plant-based concrete based on local resources.

2 MATERIALS AND TECHNIQUES

2.1 Materials

The mineral binder is a Portland cement CEM I.

Eight hemp shiv from different origins in France were selected (referred as to S1 to S8), as well as one type of flax shiv (referred as to F1). They are reduced to powder using a bladed crusher, sieved to a particle size of 500 μ m and then dried at 40°C until constant mass.

The aspect of the obtained powders is shown in Fig. 1, where the different coloration of the plants is visible.

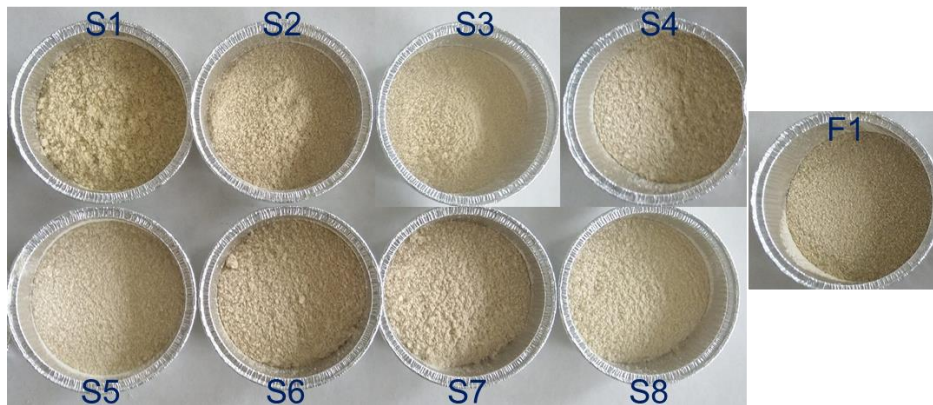


Fig. 1: Vegetal powders

2.2 Techniques

The binder hydration is measured by isothermal calorimetry at 25°C (TA Instruments, TAM AIR 8, Reference sample of Fontainebleau sand). The measurements are made on cement paste and cement pastes containing shiv powders (Vegetal to cement ratio: 2%, Water to cement ratio: 0.5).

A spectrophotometer (Ci4200, X-Rite) was used to characterize the color of the solutions of extractives. The analyses were carried out on 10 mL of solution placed in an optical glass cuvette 31.6 mm in diameter. The results were pinpointed in the CIELAB colour space by the coordinates L^* (luminosity) and the chromaticity coordinates: the $-a^*/+a^*$ axis from green to red and the $-b^*/+b^*$ axis from blue to yellow.

The extraction of vegetal compounds are carried out in distilled water at 20°C during one hour with a water to vegetal ratio of 100. The characterization of the extraction solutions (total extractible compounds, reducing sugars, monosaccharides and polyphenol contents) is detailed in a previous work (Delannoy *et al.*, 2020).

3 INFLUENCE OF PLANT ON CEMENT HYDRATION KINETICS

To study the influence of plants on cement setting kinetics, isothermal calorimetry measurements were performed with a plant to cement ratio of 2%. The results obtained are presented in Fig. 2 and the delays measured with respect to cement alone are also reported in this figure.

It can be observed that the presence of vegetal powders has a very variable impact on the setting of the cement: a delay ranging from one to six hours, as well as a decrease in the value of the maximum of the heat flux linked to the hydration reaction. Flax powders have the least delay in the setting of the material.

These results confirm the conclusions already obtained in previous studies on the impact of plants on the setting of mineral binders (Bourdote *et al.*, 2019; Delannoy *et al.*, 2020). Moreover, for the same variety of plants, the impact on the setting of the binder is also variable and impossible to predict. The characterization of compounds extracted from plants is necessary to understand the origin of this strong variability of the obtained results.

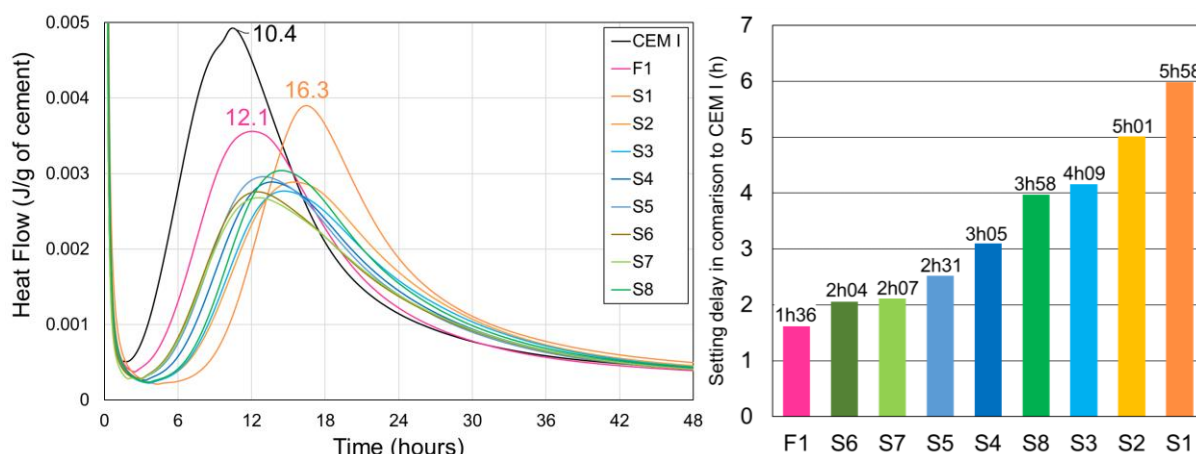


Fig. 2: Heat flow for cement pastes containing vegetal powders and setting delay measured in comparison to CEM I

4 EXTRACTION OF VEGETAL POWDERS IN WATER

4.1 Coloration of extraction solutions

The extraction solutions are obtained after one hour of immersion of the powders in distilled water with a mass concentration of 1%. After filtration, the color of the solutions is characterized by colorimetry. The results obtained are shown in Fig. 3 as a function of the setting delay measured for the corresponding plant powder. In the following graphs, the less colored solutions (S6, S7 and S8) are represented by green dots, the intermediate solutions (S3, S4 and S5) by blue dots and the darkest solutions (S1 and S2) by orange dots. The flax shiv are represented by a magenta dot.

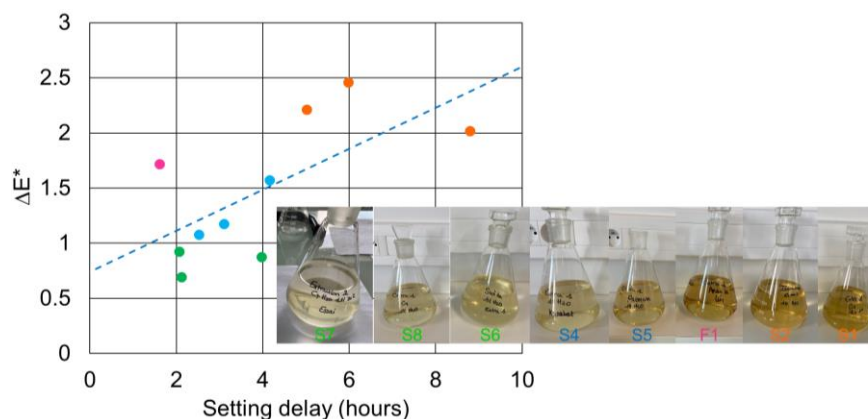


Fig. 3: Color of the extraction solutions according to the measured setting delay for the corresponding powders

In Fig. 3, it can be seen that, globally, the darker the extraction solutions of the hemp shiv powders are, the greater the delay in setting time. The extraction solution corresponding to the flax shiv is darker and does not correspond to the same colorimetric range.

The coloring of the solutions is related to their chemical composition, and in particular to their concentration in chromophore molecules such as polyphenols, basic constituents of plants. In the next part of this study, the total amount of extracted compounds is measured, as well as their concentration in reducing sugars and total monosaccharides, compounds already identified as cement setting retarders, and in polyphenols.

4.2 Quantification of extractable compounds

By weighing the dried powders before and after immersion in water, the total amount of extracted compounds can be determined and the results obtained are presented in Fig. 4 as a function of the setting delay measured by isothermal calorimetry. A link can be made between setting delay and overall quantity of extracted compounds. The latter are therefore the cause of the modification of the cement hydration kinetics.

Using the protocols presented by Delannoy *et al.*, 2020 for the dosage of reducing sugars and total monosaccharides (Fig. 4), it is possible to also observe a relationship between the amount of extracted reducing sugars and the setting delay. On the other hand, the amount of total monosaccharide is stable for all plant powders tested in this study. It is therefore the reducing sugars that seem to have an impact on the setting of the cement (Thomas and Birchall, 1983).

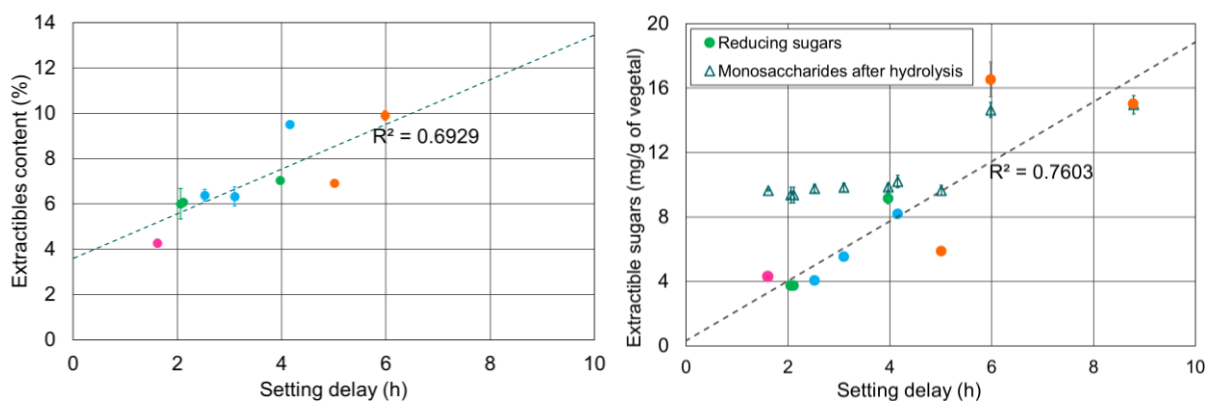


Fig. 4: Quantification of total leachate and of sugars contents extracted from vegetal powders as a function of cement setting delay

Comparing the results obtained for hemp powders and those resulting from flax shiv, it is observed that the latter cause the lowest setting delay and also contain the lowest amounts of total extractable compounds and reducing sugars. On the other hand, the color of the extracted solution is darker than that of the hemp shiv, which have the lowest retardation. Consequently, it seems impossible to directly compare plants of different species.

4.3 Presence of polyphenols

The presence of polyphenols in the extracted solutions is analysed by UV-visible spectroscopy between 200 and 500 nm (Fig. 5). The spectra of the different solutions have the same shape, characterized by a peak between 270 and 295 nm and a shoulder around 310 nm. We can conclude that the molecules extracted from plants are of the same nature, and the absorbance values are linked to their concentrations.

Furthermore, in Fig. 5, higher absorbances can be observed between 400 and 500 nm for the darker solutions. This wavelength range corresponds to the yellow coloration of the solutions.

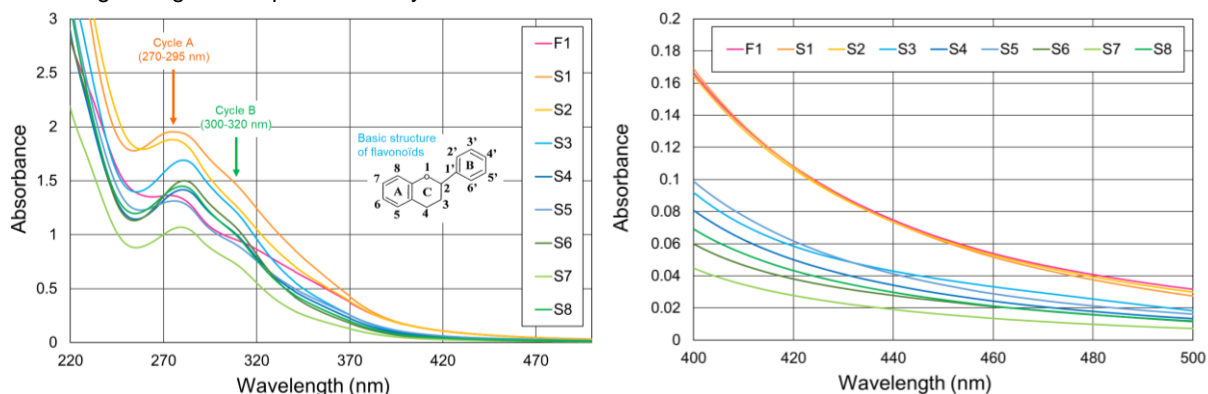


Fig. 5: UV-visible spectra for extraction solutions in water, and zoom between 400 and 500 nm

In the spectral zone corresponding to UV light absorption (190–400 nm), the absorption band at 280 nm and the shoulder observed at 310 nm shows the presence of extracted phenolic compounds (Diquélou et al., 2015), associated, for example, with lignin, with flavonoids, coumarins or tannins.

Moreover, by plotting the value of the maximum of the absorbance peak at 280 nm as a function of the setting delay, a link between these two parameters can also be observed. Polyphenols, at the origin of the coloration of the extraction solutions, have therefore also an impact on the hydration kinetics of the cement. However, it is not possible, by UV-Visible spectrometry, to identify the exact nature of these compounds.

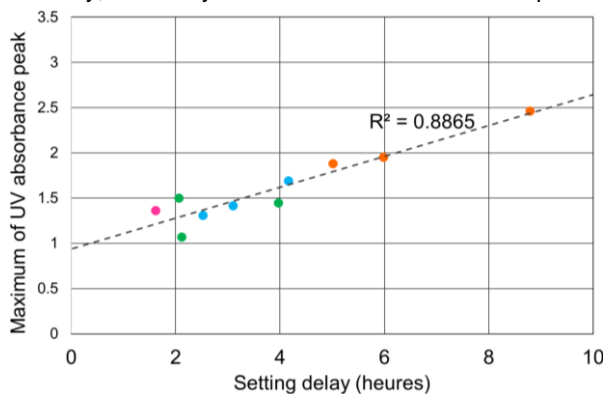


Fig. 6: Value of the maximum absorbance peak of UV-visible spectra between 270 and 300 nm as a function of the setting delay

5 CONCLUSION

The objective of this study was to characterize the compounds from plants extractible in water and which could have an impact on the hydration kinetics of cements. Eight types of hemp shiv and a flax shiv were studied and their influence on the setting of the binder was demonstrated with setting delays varying from one to five hours compared to non-modified cement. To explain these results, the extractible compounds of the different plants were characterized (total amount of leachates, concentration of reducing sugars, total monosaccharides and polyphenols). Thanks to these experiments, a link between the setting delay, the total quantity of leachates, reducing sugars and polyphenols could be demonstrated. The coloration of the extraction solution is also related to the setting delay. Comparing the results obtained for hemp and flax shiv, it can be concluded that it is not possible to directly compare the results obtained for plants of different species.

In parallel, the same work was carried out for plant compounds extracted in a basic solution, more representative of the high pH of the fresh cement during the manufacturing of the materials. Larger quantities of extracted compounds, whatever their nature, are then observed, but without modification of the conclusions obtained after extraction in water.

The chemical nature of the extracted molecules was not characterized in this study. These analyses are currently in progress by liquid chromatography, by widening the range of analyzed plants (miscanthus, sunflower, corn).

The final objective of this project is to define an indicator to predict the compatibility between a mineral binder and vegetal particles in order to generalize the production of plant-based concrete based on local resources.

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