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A New Experimental Setup to Characterize Binder–Vegetal Particle Compatibility in Plant-Based Concrete

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Abstract: The good insulation properties and the low carbon footprint of vegetal concretes make them promising materials whose use tends to grow continuously. To produce optimized building materials, a better understanding of the interfacial transition zone (ITZ) between vegetal particles and cement paste in terms of the reactions involved and the size of the impacted surface was investigated. This research led to the setting of a reliable visual test to observe ITZ, which enables the monitoring of its appearance and development. Different combinations of vegetal particles and cement pastes were tested to compare the formed ITZ: hemp, rapeseed, and bamboo into Portland and Prompt cement. Finally, a clear link was drawn between the sugar concentration and the size of ITZ. Thanks to image analysis, it was shown that ITZ is due to physico-chemical reactions, with the extraction of free saccharide molecules from the vegetal and water suction followed by their release into the cement paste.

Keywords: plant-based concrete; bio-aggregate; cement; composite; hydration; sustainability

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1. Introduction

Vegetal concretes are a mix of vegetal aggregates and a mineral binder. They present lots of advantages, especially in terms of thermal, acoustical, and hygrometric properties. They are a feasible solution to meet the requirements of the new Environmental French regulation RE2020 [1] while maintaining a negative carbon footprint. Indeed, vegetal particles such as hemp and rapeseed are local materials since their cultures are widespread throughout France, and their growing does not require the massive use of fertilizers.

The use of this kind of material is limited due to the great variability of mechanical properties and the formation of a poor interface between vegetal particles and binders. This interface zone is not specific to plant-based concretes; it also exists with regular concrete between the cement paste and aggregates [2]. However, the zone impacted is slightly smaller. In the case of vegetal concrete, a large area called the interfacial transition zone (ITZ) appears around vegetal particles (Figure 1) [3].

Previous research shows that ITZ is composed of non-hydrated products of the binder. Two hypotheses have been formulated to explain this phenomenon [4]. Firstly, the absorption of water from the cement paste via vegetal particles locally creates a non-hydrated zone due to a lake of water (physical hypothesis). Secondly, during mixing, free plant compounds are extracted. In addition, some of the vegetal molecules, such as hemicellulose or lignin, can be degraded due to the alkaline pH value of the mineral binder. These compounds, polysaccharides or polyphenols, diffuse into the cement paste [5] (chemical hypothesis). Polysaccharides are carbohydrate-based compounds that have the potential to modify the properties of cementitious materials. For example, they include hydrogels obtained from calcium and aluminum salts of polysaccharides that are suitable for internal curing of cement pastes, leading to an increase in their hydration degree [6]. The addition of polysaccharides to cement mixtures affects various aspects of the cement hydration process and the mechanical properties of the resulting concrete. Polysaccharides can act as dispersing agents, modifying the rheological properties and potentially improving the workability and flowability of fresh cement paste [7–10]. These studies concern, for example, the use of water-soluble polysaccharide gum to increase the apparent viscosity of cement pastes at low shear rates [7]. Several studies by Peschard et al. [9–11] highlight the setting delay associated with the addition of five different polysaccharides to cement pastes. Depending on the chemical composition of the polysaccharide, the effect is variable and also depends on the type of cement, particularly its C3A content. The origin of retardation could be linked to an adsorption of admixtures on the first hydrates formed, resulting in a less permeable coating.

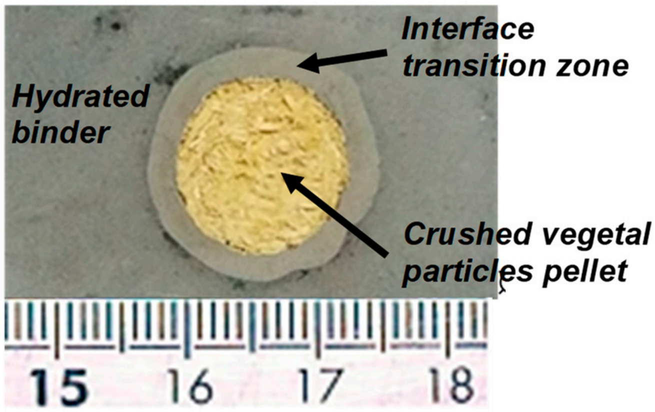

Figure 1. Visualization of ITZ, crushed hemp shiv pellet in Portland cement paste after 24 h.

They can also enhance the adhesion between cement particles and other materials, such as cellulose fibers or bio-based additives, and increase the water-holding capacity of cement, leading to controlled internal water release during cement hydration [11,12]. This has implications for the setting time and strength development of the cementitious materials.

Different types of polysaccharides have been already investigated [13,14], including carboxylates, sulfates, polyacrylamide, polyether polysaccharides, aminosulfonic acid-based superplasticizers, and modified polysaccharides. These studies also examined the effects of additives and chemical treatments on the release of polysaccharides from plant-based materials, such as miscanthus stem fragments, in the presence of cement. These studies show that, in general, polysaccharides, regardless of their chemical base, increase the plastic viscosity, but their effect on the yield stress can vary [15,16]. The chemical modification of starch and cellulose leads to the reduction of their molecular weights and improves the dispersant effect of these compounds [17]. Chemical treatments on vegetal particles, via alkali and silanization, that allow the release of sugars or other free components, result in faster cement hydration [18]. This study shows a relation between the number of sugar-containing molecules able to be extracted from miscanthus particles and the mechanical strength of the vegetal concretes: the lower the number of such molecules, the higher the mechanical strength obtained.

Thus, by understanding the interactions between polysaccharides and cement hydration, it is possible to tailor the composition and formulation of cement mixtures to achieve the desired properties and performance characteristics. Overall, studies highlight the importance of polysaccharides in bio-based concrete technology, providing insights into their effects on cement hydration, rheological properties, and the mechanical performance of cementitious materials.

The conventional method to investigate the interfacial transition zone (ITZ) between aggregates and binders in concrete typically involves a combination of microscopic analysis and physical testing. Microscopic techniques, such as Scanning Electron Microscopy (SEM) coupled with Energy Dispersive X-Ray Spectroscopy (EDX), are used to visualize the microstructure of ITZ and analyze its chemical composition. These methods provide detailed images of the ITZ's morphology and can identify the presence of specific elements or compounds, giving insights into the chemical interactions between the aggregate and binder. Additionally, physical tests, such as microhardness testing and porosity measurements, can assess the mechanical properties and porosity of ITZ, respectively. These conventional techniques, while powerful, can be time-consuming, require specialized equipment and expertise, and often involve the destructive testing of concrete samples, which may not be ideal for in situ analysis or for samples for which preservation is necessary.

Investigating the interfacial transition zone (ITZ) between vegetal aggregates and binders in concrete using conventional methodologies such as Scanning Electron Microscopy (SEM) coupled with Energy Dispersive X-Ray Spectroscopy (EDX), and physical testing presents several limitations. These include reduced sensitivity to organic matter, making it challenging to capture the nuanced interactions and chemical reactions specific to vegetal aggregates, such as the release of substances that affect cement hydration. Additionally, the inherent physical properties of vegetal aggregates, like higher porosity and water absorption, result in a more complex ITZ, which standard testing methods may not accurately assess. The destructive nature of these conventional techniques also prevents longitudinal studies on ITZ evolution, particularly limiting in understanding vegetal concrete's long-term behavior. Furthermore, the complexity and time-consuming aspects of these methods are impractical for routine quality control, especially given the variability of vegetal aggregates. Hence, there is a pressing need for alternative, non-destructive, rapid analysis techniques that are better suited to the unique challenges presented with vegetal aggregates in concrete.

The aim of this paper is to develop a user-friendly and robust test to observe the formation of ITZ to assess the compatibility between different binders and vegetal particles. Based on the test depicted by Diquelou [19], a new protocol is proposed, which allows for a better understanding of the phenomena causing ITZ. Previous research [3] shows that the chemical composition of a shiv is slightly impacted by its origin, the type of harvesting, or the method of defibration. In this study, three different vegetal particles were used: hemp, bamboo, and rapeseed, which were studied by Martinhao [20]. The interaction between vegetal particles and binders was studied using two binders with different compositions: classical Portland cement, CEM I 52.5, and Prompt cement.

2. Method and Equipment

The test of Diquelou [3,6] is an experimental way to observe and study the appearance and development of the ITZ. The principle is to create a halo in the cement paste around the vegetal particle to obtain a visible and quantifiable transition zone. To do this, a crushed vegetal particle pellet, with controlled dimensions and weight, is produced to avoid discrepancies in using an individual shiv (size, weight, and shape). In addition, crushing the plant particles increases their specific surface area, improving the extraction of free compounds, and it overcomes the differences in particle size between the types of plants studied. The pellet is put in contact with the binder; then, the setting of the paste around it is observed. The following protocol resulted from the original one and from some improvements added to reduce the uncertainties linked to the setting of the experiment. Indeed, it was noticed that the repeatability of the experiment must be improved [21]. After the validation of the protocol with robust and reliable results, the following hypotheses had to be tested:

- The impact of the couple binder/vegetal particle of the ITZ size;

- Which of the absorption or the release of vegetal components phenomena is responsible or dominant for the formation of the ITZ.

A total of 74 couples' vegetal particles/binders (between 7 and 21 results by couple) were tested.

To find out how important absorption is, a foam pellet with a high absorption capacity (up to 200 times its weight in water) and a plastic pellet, which cannot absorb any fluid, were placed instead a vegetal shiv pellet. To test the hypothesis concerning the link between vegetal molecules' release and the halo, foam pellets were tested in a sugar solution with a known sucrose concentration. A total of 25 foam pellets (3 to 4 results by sucrose concentration) was tested.

2.1. Improvement of Existing Protocol

In the first attempts, the pellets were placed on top of the cement paste. In this way, they were visible, and it was possible to take pictures during the experiment. But this resulted in irregular halo formation and bad repeatability, mainly due to bleeding water on the surface. To counter this, the pellet was set on the bottom of the mold on a glass plate formwork to be able to film. A special frame was designed to hold a glass plate covered with a transparent film with pellets glued onto it (Figure 2). Cement paste could then be poured over to fill the frame. The difference between a halo obtained with the initial protocol and with the new protocol is visible in Figure 3.

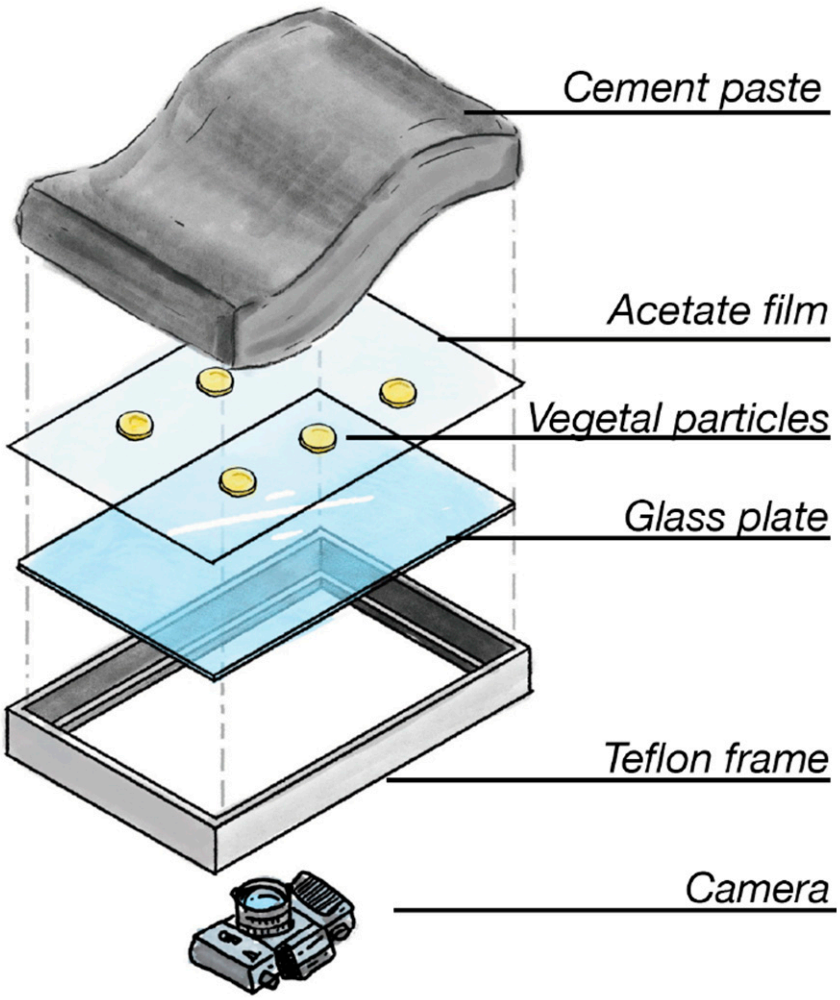

Figure 2. Setting of the experiment.

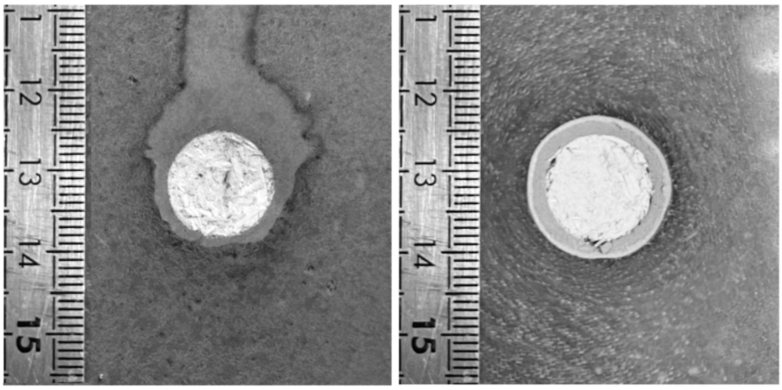

Figure 3. A halo obtained with a pellet on top of the cement paste (**left**) and on the bottom of the mold (**right**). The hemp shiv pellet is 13 mm in diameter.

2.2. Production of Fiber Pellet

The vegetal particles used were dried in an oven at 50 °C for at least 24 h. Vegetal particle pellets were made by crushing 2 g of fibers over 4 min with a planetary mill. A known mass of crushed fibers (between 0.10 and 0.30 g) was then pressed together using a manual hydraulic press under 10 tons for 1 min. Then, the pellets were dried a second time in an oven at 50 °C overnight. The resulting pellets had a diameter of 13 mm and a thickness between 0.8 and 2 mm, depending on the kind of vegetal particles used.

2.3. Production of Foam Pellet Soaked in Sucrose Solution

A foam cylinder of 16 mm in diameter was punched out of a foam board. The cylinder was then sliced to obtain a pellet of about 1 mm in thickness and a diameter of 16 mm. To soak it with a sucrose solution, sugar was dissolved in tap water to obtain a solution at the desired concentration. The foam pellets were then dipped into the solution and allowed to soak in the solution for 10 min. Foam pellets were then placed on a sheet of parchment paper and put in an oven at 50 °C for at least 24 h.

2.4. Preparation of Acetate Film

The pellets were glued onto an acetate film using spray glue. First, a template was placed under the film to mark the position of the pellets in order to prevent two pellets from being too close to each other and interacting. On an A4 size film, up to 12 pellets could be glued. The glue was sprayed on one side of the pellet from about 200 mm. After a few minutes, allowing time for the solvent to evaporate according to the glue supplier's instructions, the pellets were glued onto the film and pressed with a weight of about 800 g for a minute (a bowl filled with 800 mL of water could be used). A prepared film can be seen in Figure 4.

Figure 4. Teflon frame with the acetate film on top of the glass plate. The tape was used to seal the edge of the frame.

2.5. Preparation of Cement Paste

The cement paste was prepared to fill the Teflon frame, whose dimensions were 210 mm × 297 mm × 20 mm. The cement paste was a mix of 950 g of tap water and 1900 g of a binder (water/cement ratio, W/C, = 0.5).

2.6. Setting the Experiment

The acetate film, with the pellets stuck onto it, was fixed to the Teflon mold with adhesive tape to prevent cement paste from running under it. The mold was placed on a holed table so that its bottom was visible from below (Figure 5). The horizontality of the experiment table had to be checked to avoid runoffs. The entire mold was visible to the camera (mvBlueFOX3-2 2071a with a Sony IMX428 sensor and a 3216 × 2208 resolution, a 28 mm lens, and a max aperture of f:1.8). The camera's settings had to ensure a clear image for each pellet without a reflection. Then, the cement paste was poured and leveled by slowly agitating the mold to eliminate air bubbles.

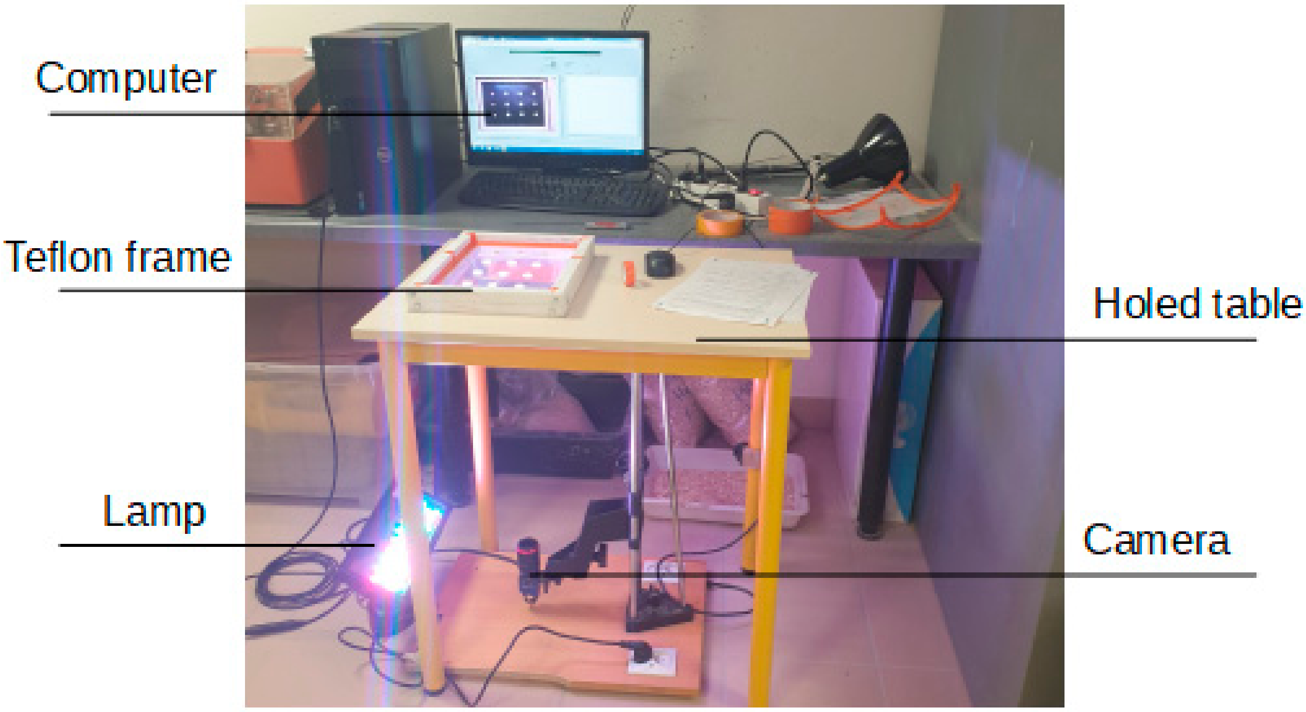

Figure 5. Experimental setup.

2.7. Image Processing and Analysis

The images were captured using a custom LabView program 20.0 [22]. They were corrected and adjusted using Adobe Lightroom 7.0 [23]. The processed images were compiled into a timelapse using MatLab software R2021a [24]. To utilize the result, the area of the halo had to be measured. ImageJ software 1.51 [25] was used to measure the surface of the halo by drawing an ellipse in the software. An example of a processed image is presented in Figure 6.

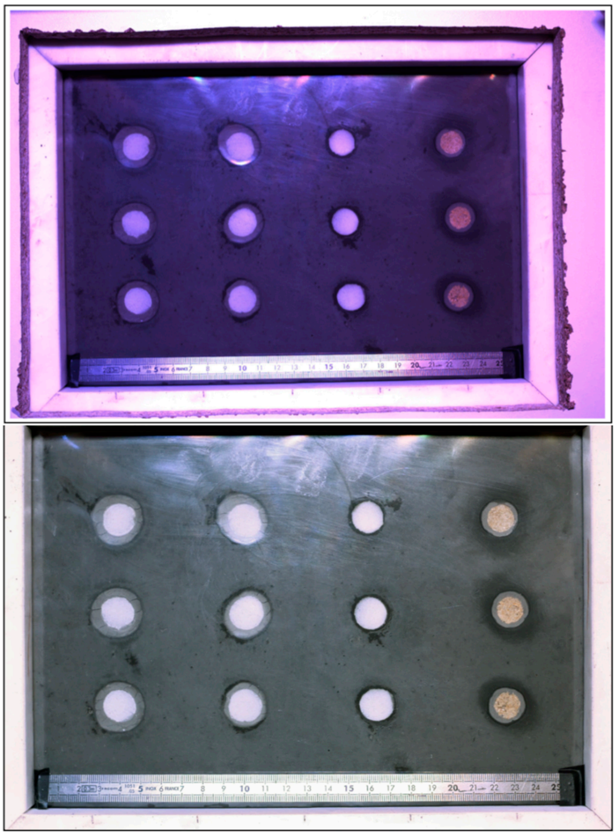

Figure 6. Initial picture (**top**) and image processed through Lightroom and Matlab (**bottom**).

3. Parameters

Once a reliable protocol was set, the parameters were tested. The vegetal particles tested were hemp, rapeseed, and bamboo. Their characteristics are summarized in Table 1. As binders, Portland cement, CEM PERFORMAT 52.5 R, and a calcium aluminate cement (Prompt cement) from the Vicat company were used. Prompt cement sets very quickly (between 90 s and 1 h at 20 °C when using a set retarder), thanks to its composition. It mainly contains belite and calcite [26]. Portland cement is composed of alite, belite, celite, and ferrite. It starts setting after about 2 h at 20 °C [27]. A hemp shiv with Portland cement was considered as a reference value. For the tests with vegetal particle pellets, several parameters were tested: the type of plant, the binder, the mass of the pellet, and the curing conditions with a covered (closed) or uncovered (open) drying surface. For foam pellets, the parameter tested was the sugar concentration of the solution in which the pellets were soaked.

Table 1. Characterization of vegetal particles [7].

	Hemp	Bamboo	Rapeseed
H D50 [mm]	4.7	5.0	6.8
L D50 [mm]	2.8	3.1	3.7
Dust content (%)	1.4	11.5	11.6
Initial rate of absorption (%)	226	38	268
Bulk density before drying, ϱ_{wet} [kg/m^3]	110	305	100
Bulk density after drying, ϱ_{dry} [kg/m^3]	110	304	89

4. Analysis and Discussion

4.1. Compatibility between Vegetal Particles and Binders

An ITZ with lower mechanical property values in comparison to the non-affected paste, as well as better compatibility between the vegetal aggregate and the binder, was considered for a lower surface of the halo. The following results were obtained at a constant pellet mass for all fibers of m = 0.10 g. Figure 7 shows the evolution of the halo surface as a function of the type of vegetal particle and binder used. The experiments showed that hemp is more compatible with the Prompt binder than with the Portland binder. Indeed, the size of the halo was divided by 2 when the Prompt binder was used with hemp. Rapeseed formed a halo in the same order of magnitude in both the Portland and Prompt cements. Among the tested vegetal particles, rapeseed had the best compatibility with the Portland cement. Bamboo seems to have achieved equivalent compatibility with the Prompt and Portland cements. The size of the halo obtained with bamboo particles and the Portland cement was very close (1% variation) compared to that of the hemp shiv in the same binder. The test was repeatable with less than 5% variation in the size of the resulting halo. It can be noticed that the uncertainties with the Prompt cement were more significant than those with the Portland cement. This is partly due to the fact that fewer tests were conducted using the Prompt cement and also because the size of the halo was harder to determine. The edges of the halo were fuzzy; it looked like it had been sprayed onto the surface of the binder (Figure 8).

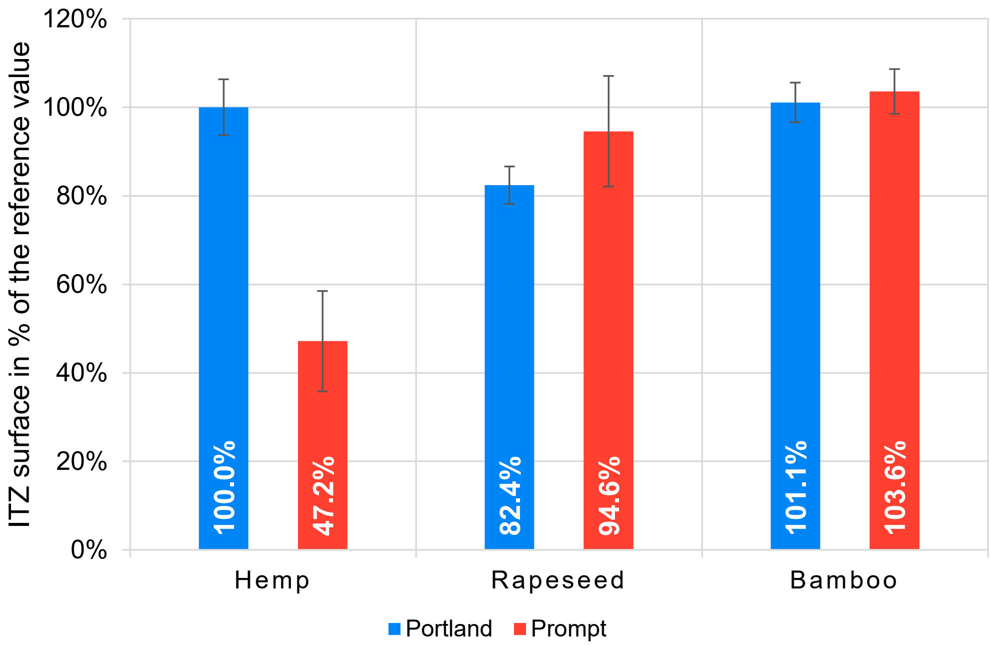

Figure 7. Comparison of mean halo surface between Portland and Prompt binders. Hemp in Portland cement as reference.

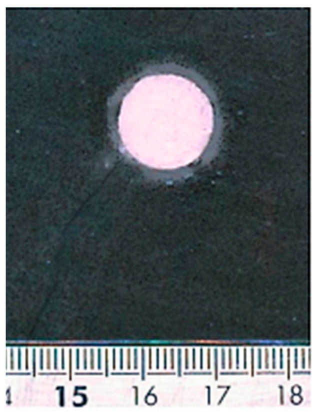

Figure 8. Halo obtained with hemp shiv in Prompt cement.

4.2. Origin of ITZ

To determine the origin of the ITZ formation, the same tests were carried out with plastic and foam pellets. The halos formed are presented in Figures 9 and 10, respectively. These results showed that the influence of absorption was negligible, as no halo was formed with a plastic (Figure 9) or with a foam, which can absorb up to two hundred times its own mass, in water (Figure 10). Consequently, the cause of the halo seems to have been the release of molecules via the vegetal particles. Sugars are known to be retarding agents for the setting of cement paste. By studying the halo formed via foam pellets soaked in different concentrations of sugar (Figure 11), it becomes clear that the size of the halo is linked to the amount of sugar: the more sugar, the bigger the halo. This influence is clearly visible in Figure 11, even with very low sucrose concentrations. The halo reached a maximum size when the sucrose concentration exceeded a threshold value, which was between 10 and 20 g/L (with sucrose soaked into a foam pellet of 16 mm in diameter and approximately 1 mm in thickness). Moreover, tests with different sugars found in the vegetal particles, like xylose, and with various foam porosities had to be investigated. To reduce the size of the halo, one would have to find a way to either reduce the amount of sugar in the fibers or stop the phenomenon of the release of sugars into the cement paste.

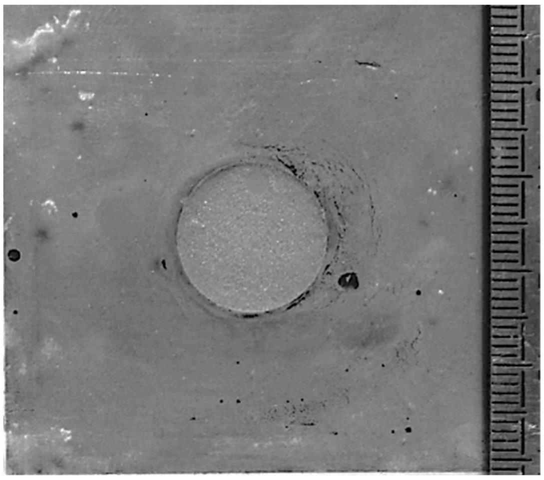

Figure 9. Results with a plastic pellet in Portland cement.

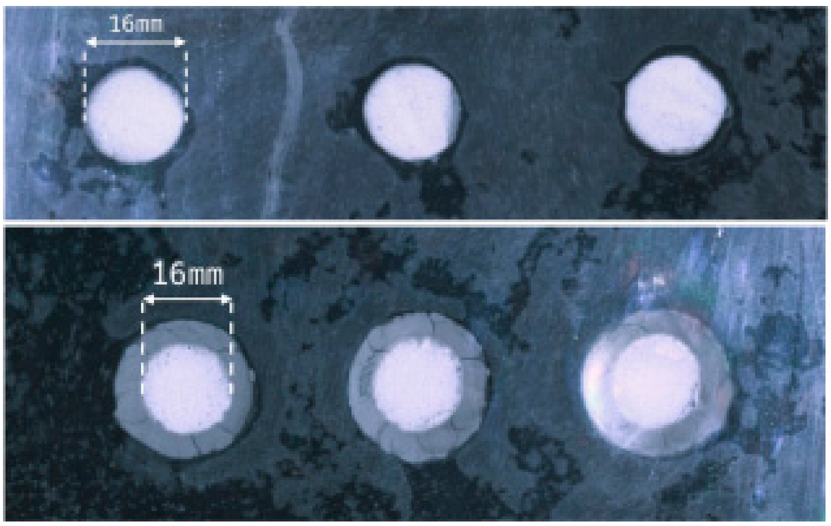

Figure 10. ITZ formed around foam pellet without (**top**) and with sugar (80 g/L) (**bottom**), both in Portland cement.

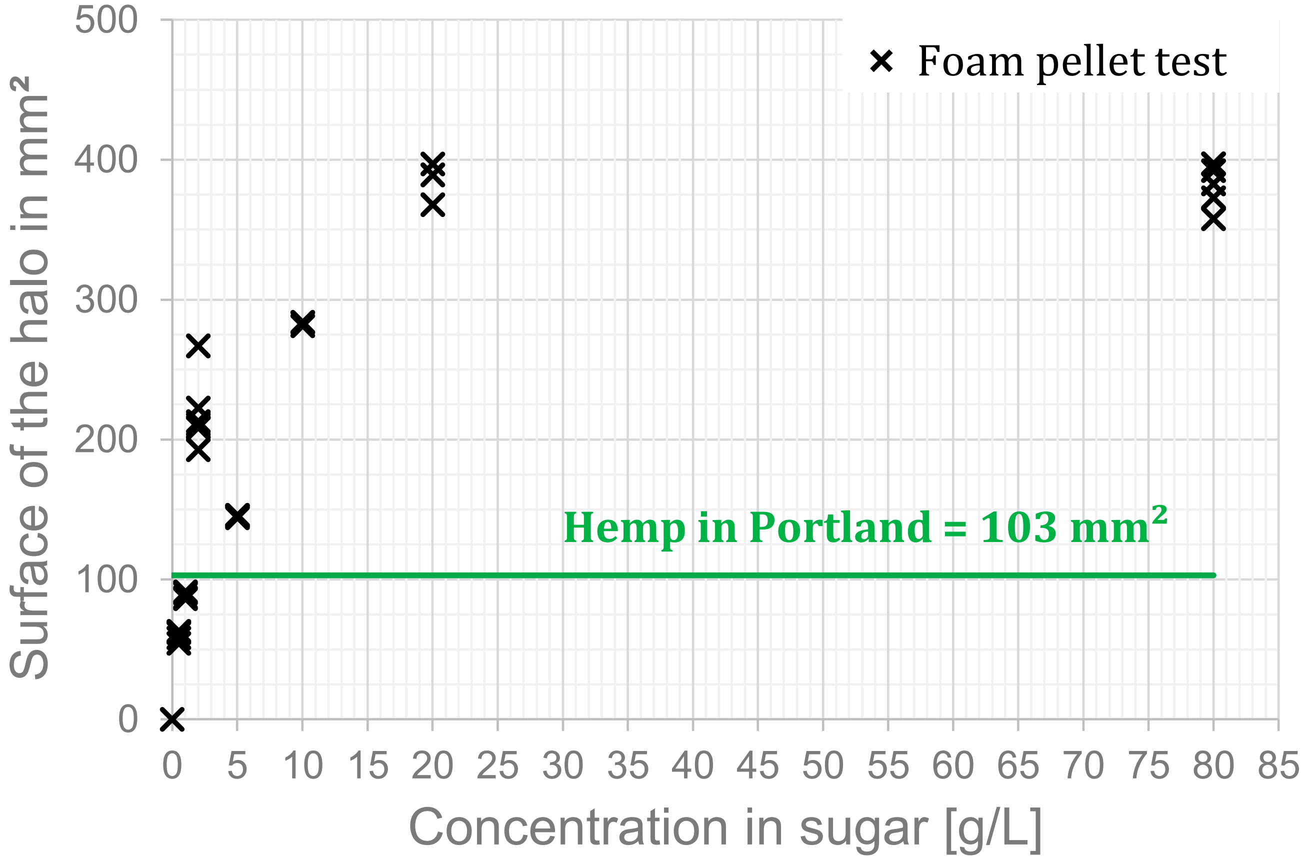

Figure 11. Size of the halo with respect to the sucrose concentration. Foam pellet of 16 mm in diameter in Portland cement. Green line for reference: hemp shiv in Portland.

4.3. Impact of the Mass of Vegetal Particles

To be able to compare vegetal particles with different bulk densities to each other, the following question should be answered: Should one think in terms of mass or volume to produce pellets? Rapeseed and hemp have similar volumes because of their very close bulk densities. However, bamboo is almost three times denser than hemp, and its pellets have a smaller volume (the same diameter but a lower thickness). Bamboo pellets with a different mass, e.g., with a different thickness, were tested. In the case of bamboo particles, the size of the halo depended on the number of fibers contained in a pellet (Figure 12). It is clear that the mass of the pellet has an influence on the obtained halo, no matter what binder is used. This interdependence can be explained by the fact that an increased mass involves a higher quantity of molecules likely to be extracted. It could also be explained by the fact that the contact zone between the vegetal particles and the binder is increased, which could also increase the release of extractable molecules into the binder. Both Portland and Prompt cements show a bigger halo with a heavier pellet, but the distinction between the two binders is not significant. At a mass of 0.10 g, both areas are very similar. At a mass of 0.15 g, the difference between the two cements is more pronounced. In the conditions for the test that were used, the Prompt cement provided better results with a halo 35% smaller than Portland (areas of 121.2 mm^2 in Prompt and 164.5 mm^2 in Portland). At a mass of 0.30 g, the difference was only 7% (Figure 12). Future experiments have to focus on the surface of interaction between the vegetal particles and the binder when using different sizes of pellets. The study of the halo volume, rather than its area, also has to be investigated.

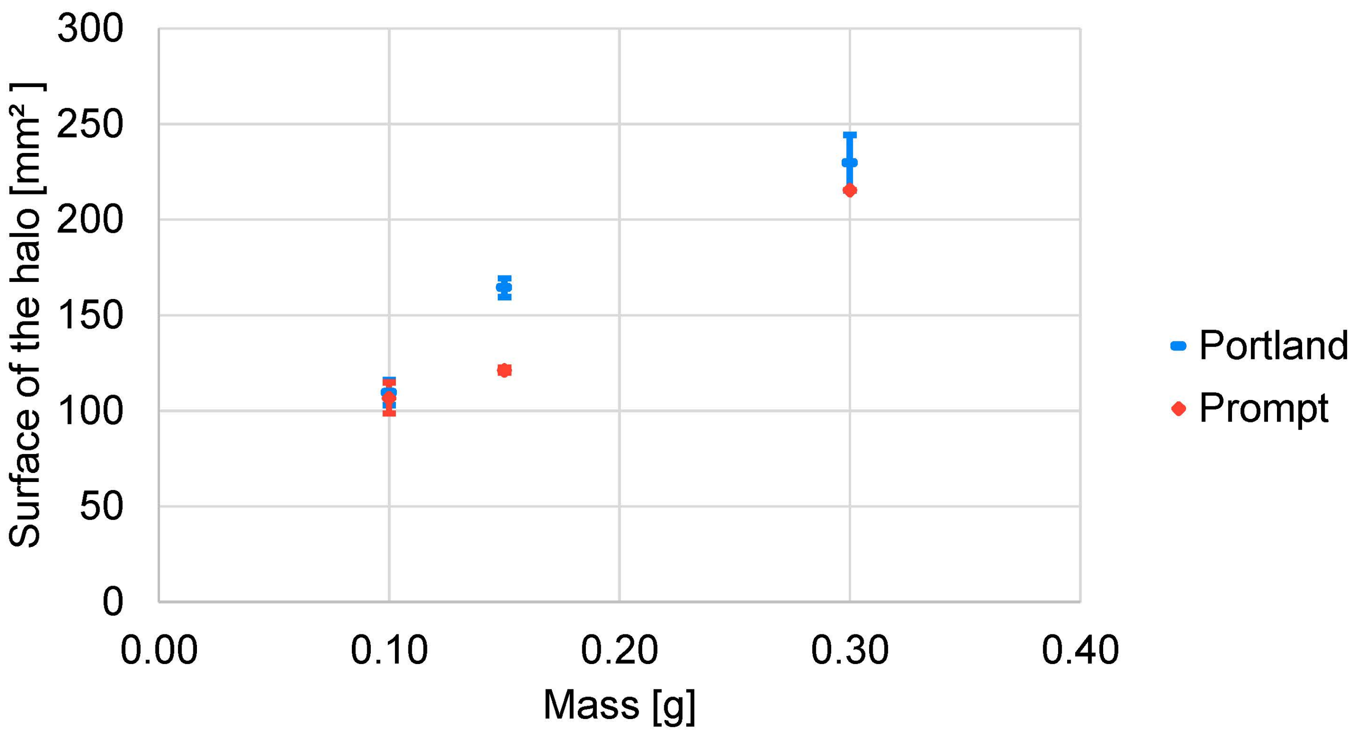

Figure 12. Area of ITZ obtained with bamboo pellets of different masses in both Prompt and Portland cement pastes.

4.4. Color and Kinetics of the Apparition of the Halo

The appearance of the halo created due to the hemp shiv in Portland cement, in Prompt cement, and with open or closed drying surfaces on the top of the mold was studied hour after hour. The goal was to understand how the binder and the curing conditions could impact the kinetics of the reactions (Figure 13). The kinetics were very different according to the binder and the curing conditions (Figure 14). When the halo started to be visible, it almost already achieved its final area (except for Prompt cement with a closed hydration surface, which presented a stage around 8 h before increasing again at 14 h). This means that the diffusion phenomenon had already occurred and was over.

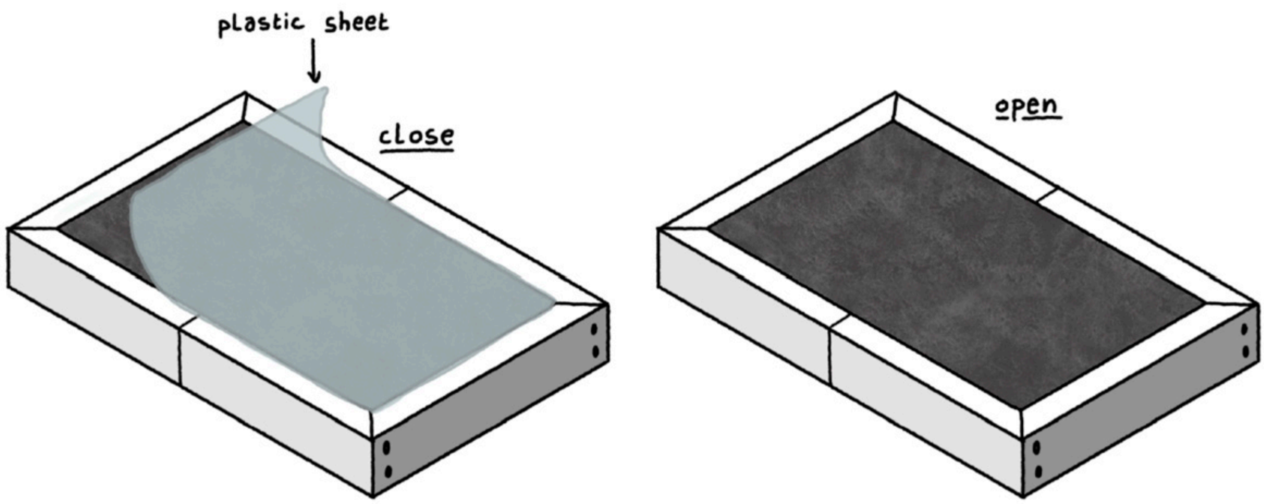

Figure 13. Open (**right**) and closed (**left**) drying surfaces for the top of the mold.

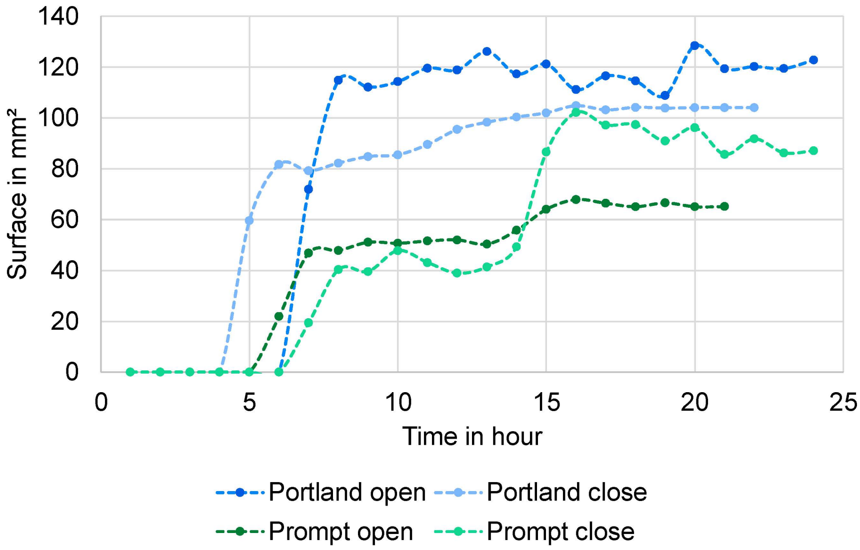

Figure 14. Hemp in Portland and Prompt binders under different drying conditions (top surface open or closed).

Moreover, the color of the halo changed significantly over time: it was first darker, and then it got lighter as the binder set (Figure 15). In the first hours after the beginning of the experiment, water from the cement paste was sucked up into the pellet (until 3 h 30 for the Portland cement and 3 h for the Prompt cement). It was clearly visible because the pellet changed color as it got soaked. After this phase, the water with vegetal components was progressively released back into the cement paste because of the pore pressure. The pellet went back to its original color, meaning that it was dry again, and the halo started appearing.

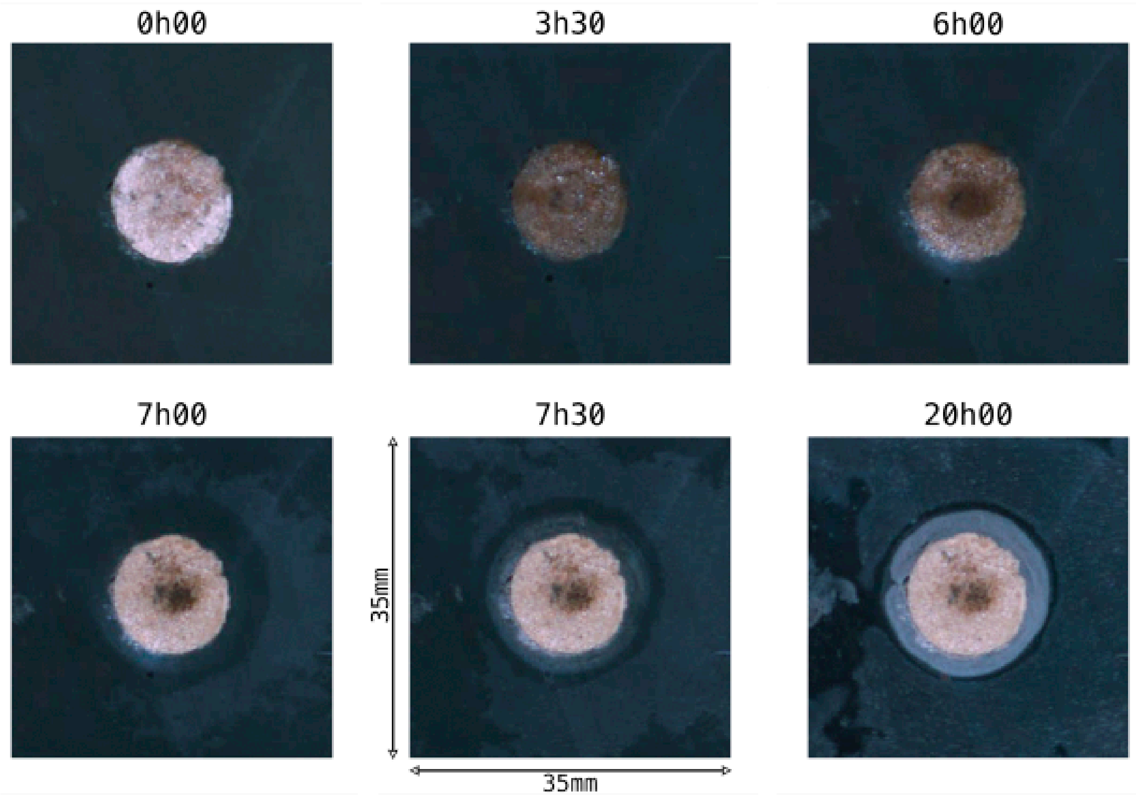

Figure 15. Hemp in Portland cement at different times (mass = 0.10 g; diameter = 13 mm).

Figure 16 shows the kinetics of the halo formation as a function of the vegetal kinds and the hydration conditions. Rapeseed and hemp seem to have similar kinetics in a Portland binder (Figure 16). The halos start appearing at the same time and reach values of the same magnitude. In the case of a closed drying surface (Figure 16B), the halo obtained with bamboo particles starts developing later in time and reaches higher values compared to the open drying surface (Figure 16A). Hemp has different kinetics in Prompt and Portland cement: it starts appearing at 5 h instead of 8 h, and the final size of the halo is divided by 2. Colza and bamboo seem to have the same kind of kinetics in Prompt cement. The sizes of the halo are quite hard to quantify because the limits are less clear than with Portland cement. It must be noticed that, after a few days or even weeks, the size of the halo tends to decrease. This could be explained by the fact that the halo is a zone in which the hydration of the binder is delayed but not completely stopped. In future experiments, it would be interesting to study the halo after some weeks in order to quantify the delay.

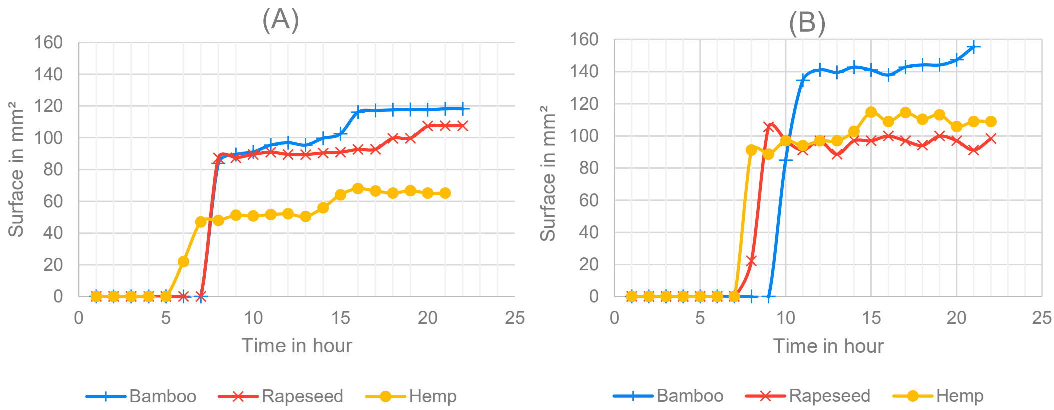

Figure 16. Bamboo, rapeseed, and hemp in Portland cement with (**A**) an open drying surface and (**B**) a closed drying surface.

5. Conclusions and Perspectives

This research successfully developed an innovative, user-friendly test to evaluate the compatibility between various vegetal particles and binders utilized in the production of plant-based concrete. By investigating the formation and characteristics of the interfacial transition zone (ITZ) between vegetal aggregates and cement pastes, we obtained crucial insights into the physical and chemical interactions that influence the mechanical properties of the resulting composite materials.

The key findings of our study are as follows:

- Vegetal particle and binder compatibility: Among the tested combinations, a hemp shiv mixed with Prompt cement exhibited the best compatibility, marked by a significantly smaller ITZ. This compatibility is crucial to optimizing the mechanical properties of the concrete.
- Formation of ITZ: Our experiments determined that the ITZ is primarily formed not due to water absorption from the cement paste via vegetal particles but through the diffusion of vegetal molecules, particularly free saccharide molecules, into the cement paste. This finding shifts the focus towards understanding and controlling the release of these molecules to improve material performance.
- Influence of sugar concentration and pellet mass: The size of ITZ is linked to the sugar concentration in the vegetal particles and the mass of the vegetal particle pellet. Higher sugar concentrations and pellet masses lead to larger ITZs, suggesting that the control of these factors could be vital for material design.
- Experimental protocols: The refinement of experimental protocols led to more consistent and reliable observations of ITZ, highlighting the importance of meticulous experimental design in materials research.

Future research will delve into several key areas to build upon these findings:

- Investigating alternative sugars and porosities: Exploring the effects of different sugars found in vegetal particles, such as xylose, and varying foam porosities could provide deeper insights into the mechanisms of ITZ formation.
- Surface interaction studies: Further experiments should focus on the interaction surface between vegetal particles and binders, considering different sizes of pellets to understand the role of physical contact in ITZ formation.
- Long-term analysis of ITZ: Studying the ITZ over longer periods will be crucial to assessing the delayed hydration processes and the eventual reduction in the ITZ size, which could impact the long-term mechanical properties of concrete.
- Mechanical testing: Compressive tests with cubic samples of vegetal concrete are needed to directly correlate ITZ characteristics with the mechanical strength of the materials, validating the effectiveness of the developed test in predicting material performance.

In conclusion, our study provided a significant step forward in the understanding and development of plant-based concretes, highlighting the potential of these materials in sustainable construction. The insights gained lay the groundwork for future innovations in the field, driving towards the optimization of vegetal concrete for practical applications.

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