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Effect of Soluble Components From Plant Aggregates on the Setting of the Lime-Based Binder

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Abstract: Plant aggregate-based building materials are a viable solution for reducing greenhouse gas emissions and providing good thermal and acoustic performances. In this study, the investigated lightweight mortars require a hydraulic reaction. Laboratory and on-site empirical observations about plant aggregate-based materials indicate a delay in setting time and a decrease in the mechanical performances of concretes based on plant aggregates. The natural origin of plant-aggregates causes a lot of variability in their properties. Related studies have shown that the incompatibility between plant aggregates and cement is mainly caused by the dissolved and decomposed components of plant aggregates in alkaline environments. Currently, no research on the specific impact of each component on the setting time of lime-based binder exists. In this work, the impact of some soluble compounds (e.g., sucrose, glucose, fructose, pectin) on the setting reaction of the lime matrix was studied using Vicat test, thermogravimetric analysis (TGA) and FT-IR spectroscopy. Experimental results showed that carbohydrates such as glucose and sucrose not only affect the setting time but also have a long-term impact on the carbonation of lime. The measure of the setting time by Vicat test appears as a possible indicator to predict the compatibility between the matrix and plant aggregates.

Keywords: Plant aggregates; setting time; lime-based binder; carbohydrates; hydration

1 Introduction

In France, the building sector is responsible for 45% of energy consumption and produces about 50% of waste. The development of new materials with lower consumption of fossil energy during materials' use and production is a topical subject. In this regard, the building sector is working on plant aggregate-based materials (e.g., hemp concrete). Plant aggregates are sustainable and environmentally friendly additives in conventional and modern building materials (e.g., cement, lime, earth matrix) [1-4] and bring new properties to materials like thermal insulation [5], sound absorption. Plant aggregates are obtained from the transformation processing of crops such as flax, hemp, sunflower, wood, bamboo, bagasse, etc. Cement is the most commonly used binder, but pozzolanic materials and slaked lime are often added to improve the performance of the binder and reduce the amount of cement [6,7]. However, some of the plant aggregates have a compatibility problem with the binder (e.g., a low cohesion between aggregates and the binder), which leads to a decrease in mechanical performances and a delay in the setting time [8-10]. Investigation has shown that compatibility is difficult to predict as there are many factors affecting the setting and development of plant aggregate-based mortars, for example, origin [11], binder properties [12], thermal and chemical treatment methods [13], harvest time [14] and field retting [15,16]. One possible assumption is that the various factors listed above affect the setting of the binder by altering the soluble components of plant aggregates in an alkaline environment [11].

The composition of plant aggregates is determined by the representation of all components (organic substances such as cellulose, hemicelluloses, lignin, pectins, extractives, waxes and inorganic components such as ash [17]) and it depends on plant variety, localization in plant and growth and process conditions. Bilba et al. [13] found that the lignin admixture (1% of the weight of cement) had a retarding effect on the setting of the mortar, while the same amount of cellulose and hemicelluloses had no apparent retarding effect. However, investigation has shown that the content of cellulose, hemicelluloses and lignin do not seem to be a key factor in the cement hydration retardation. However, the soluble components (e.g., monosaccharides, organic acid) from plant aggregates have deleterious effects on setting and hardening [11,18]. Diquélou et al. [11] found a high quantity of carbohydrates in the water extracts of different hemp shives and a delay of the setting time with plant-aggregate based mortars. It was proved that saccharides have a significant impact on the cement hydration. For example, if 0.2 wt% sucrose is added, the setting time of cement can be extended by several days [18]. Besides, the pozzolanic reaction is like hydration and can be delayed or even halted by plant leachate [19].

The mechanism by which saccharides delay the cement setting time has not been well identified, as there are always exceptions difficult to interpret. The most widely accepted view is that carbohydrates can block the C-S-H¹ nucleation sites by adsorbing on the surface of cement particles (such as C₃S, C₃A), thereby hindering cement hydration [20]. The reason for the sorption is that sugar molecules chelate with the metal cations (e.g., Ca²⁺, Al³⁺) on the surface of cement particles. A common characteristic of most organic retarders is that they contain a HO-C-C=O group; other retarders without the HO-C-C=O group can be converted into saccharinic acids containing this group in dilute alkaline solutions [21,22]. The oxygen atoms of this particular group can approach each other, which is the requisite condition for chelation [23,24].

Unlike pure cement, commercial binders (which are mixtures of cement, pozzolanic ash, lime and some additives) appear to be less sensitive to hydration retarders [25]. The hydraulic binder (e.g., Portland cement) is normally indispensable because it determines the early age properties such as setting time and strength development; the pozzolans can reduce the use of cement [19]. It has been proved that increasing the lime content can improve the mechanical performance of the cement-based hemp concrete [12]. In this study, the effects of four typical organic components on a commercial lime-based binder were studied using Vicat test, thermogravimetric analysis (TGA) and FT-IR spectroscopy. The result of this work can help improve the development of lightweight concretes based on plant aggregates.

2 Materials and Methods

2.1 Raw Materials

In this work, four different organic components were added in binder mixtures and their impact on the setting of the binder was studied. The four components are carbohydrates. Glucose and fructose are monosaccharides, sucrose is a disaccharide and pectin is a polysaccharide. Glucose is the most important monosaccharide because cellulose is a linear chain of several hundred to many thousands of $\beta(1\rightarrow4)$ linked glucose units. Fructose is also a monosaccharide, found in many plants (fruits and vegetables), where it is often bonded to glucose to form the disaccharide sucrose. Both are hexose sugars, i.e., have six carbons in their structure. The difference between glucose and fructose lies in the position of anomeric carbon. The anomeric carbon is the one containing the carbonyl group, which reacts to form the cyclised structure. In glucose, the anomeric carbon is the first carbon, therefore glucose is an aldohexose. In fructose, the anomeric is the second carbon, so fructose is a ketohexose. In sucrose, components glucose and fructose are linked via a glycosidic linkage $\beta(1\rightarrow2)$. Pectin is a general name for complex heteropolysaccharides contained in the primary cell walls of plants. It consists mainly of a chain of (esterified or not) D-galacturonic acid residues linked together via a glycidic $\alpha(1\rightarrow4)$ bond. These 4 carbohydrates were chosen because they are potentially present in plants.

¹ Cement chemistry notations: C = CaO, S = SiO₂, A = Al₂O₃, H = H₂O.

C-S-H: Calcium silicate hydrate; C₃S: Tricalcium silicate; C₃A: Tricalcium aluminate

D-Fructose (purity of 99%), glucose (purity of 99.5%), sucrose (purity of 99.5%) and pectin (made from citrus peel) were from Sigma-Aldrich Company. The binder used in this study is Tradical PF70 of Lhoist, an air lime pre-formulated binder, which is widely used for the manufacture of hemp concrete blocks and insulation in Europe. Tradical PF70 is composed of 75 wt% of hydraulic lime², 15 wt% of hydraulic binder and 10 wt% of pozzolanic material. Chemical composition of Tradical PF70 was obtained X-Ray Diffraction (XRD): X-Ray Diffraction (XRD) was used to characterize the composition of binder used. Results of XRD analysis of tradical PF70 are presented in table below: Portlandite: 43.15%; C3S: 23.00%; C2S: 9.15%; C4AF: 0.85%; C3A: 0.06%; Calcite: 6.34%; Quartz: 14.90%.

2.2 Sample Preparation and Experimental Methods

2.2.1 Mixing Procedure

In this work, 13 mixtures were tested. The aim was to study the effects of four carbohydrates at different concentrations (1%, 0.5% and 0.2% w/w) on the setting time of the binder. The detail of the mixtures is given in Tab. 1. Mineral binder was poured into mixer. The organic compound was solubilized in the water before being added to the mineral binder. After preliminary tests, the best conditions for a good workability were a ratio of water/binder of 0.6 and an agitation time of 3 minutes.

Table 1: Details of the tested mixtures

Symbol		Binder (g)	Admixture (g)	Water (g)
R	Reference (100% binder)	600	0	360
S _{1%}	1% sucrose	594	6	360
F _{1%}	1% fructose	594	6	360
G _{1%}	1% glucose	594	6	360
P _{1%}	1% pectin	594	6	360
S _{0.5%}	0.5% sucrose	597	3	360
F _{0.5%}	0.5% fructose	597	3	360
G _{0.5%}	0.5% glucose	597	3	360
P _{0.5%}	0.5% pectin	597	3	360
S _{0.2%}	0.2% sucrose	598.8	1.2	360
F _{0.2%}	0.2% fructose	598.8	1.2	360
G _{0.2%}	0.2% glucose	598.8	1.2	360
P _{0.2%}	0.2% pectin	598.8	1.2	360

2.2.2 Determination of Setting Time by Vicat Test

The setting times of mortars were determined by the Vicat test according to NF EN 196-3 [26] using a manual Vicat apparatus at room temperature. Vicat mold (cylinder 40 mm in height) was filled with 300 g of fresh paste. The Vicat test consisted of measuring the needle penetration depth into the paste in order to calculate the degree of solidification at the current time. When the paste was totally fluid, the penetration was 40 mm. When the hardening of paste was progressing, the penetration depth decreased until the needle was completely unable to insert (i.e., penetration depth was 0mm). Three penetration measurements per mixture were performed every 30 minutes and the experimental results were the average of values of the three repetitions.

² The hydraulic lime is mainly composed of CaCO₃, Ca(OH)₂ and CaO, it contains also silicates and aluminates.

2.2.3 Determination $\text{Ca}(\text{OH})_2$ and CaCO_3 Proportions by TGA Analysis

Samples of 13 mixtures were collected manually at 9 sampling times (1, 3, 7 hours and 1, 3, 7, 14, 28, 90 days) and kept in 3 sealed plastic tubes. Since the TGA test takes a long time (i.e., 2 hours for each sample) it was not possible to test all the samples at each sampling time. The sealed plastic tubes were soaked in liquid nitrogen (-196°C) for 30 seconds and then stored in a -80°C freezer. This process was proved to be able to inhibit even stop the hydration and carbonation reaction of the sample. The TGA result for each mixture is the average of three samples retained.

Samples were carried out on a TGA 295 F1 Libra thermogravimetric analyser (Netzch). The samples (50 mg) were heated from 25°C to 800°C , with heating rate of $10^\circ\text{C}\cdot\text{min}^{-1}$, under argon atmosphere. TGA experiments were used to measure the $\text{Ca}(\text{OH})_2$ and CaCO_3 contents of the first four samples of Tab. 1 (i.e., R, S1%, F1%, G1%, P1%), and the calculation method was presented by Diquelou et al. [11]. Fig. 1 gives two examples of TGA results. Three losses of mass are noticed. Mass loss from 25°C to 380°C is mainly due to the loss of water from the pore and some hydrate products (e.g., C-S-H). Mass loss from 380 to 510°C and from 510 to 800°C are respectively due to the decomposition of $\text{Ca}(\text{OH})_2$ and CaCO_3 .

According to Fig. 1, when the sampling time is 28 days, the carbonation level of the sample supplemented with 1% sucrose is significantly lower than that of the control group.

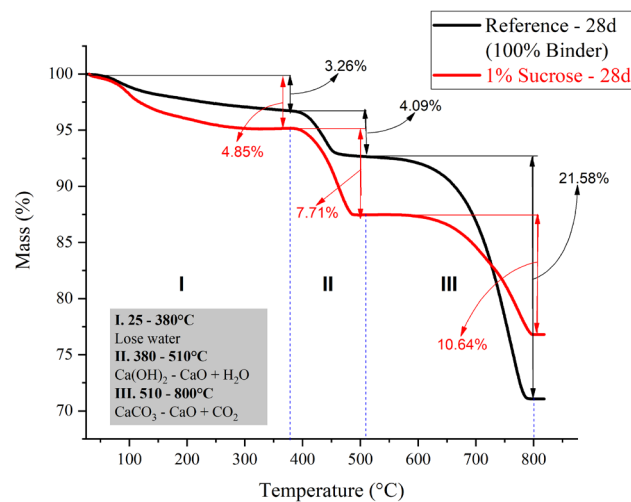


Figure 1: The thermogravimetric curves of Reference-28d and S_{1%}-28d

The dry masses of $\text{Ca}(\text{OH})_2$ and CaCO_3 (wt%) were respectively calculated according to Eq. (1) and Eq. (2), where LCO_2 represents the mass loss of CO_2 between 510 and 800°C , LH_2O and $\text{LH}_2\text{O}'$ are respectively the mass loss of water occurring between 25 - 380°C and 380 - 510°C [9]. MW is the abbreviation of molecular weight. For example, $\text{MW}(\text{CaCO}_3)$ and $\text{MW}(\text{Ca}(\text{OH})_2)$ respectively represent the molecular weight of CaCO_3 and $\text{Ca}(\text{OH})_2$, i.e., $100.087 \text{ g}\cdot\text{mol}^{-1}$ and $74.093 \text{ g}\cdot\text{mol}^{-1}$.

$$\text{Ca}(\text{OH})_2(\text{wt}\%) = \text{LH}_2\text{O}'(\text{wt}\%) \times \frac{\text{MW}(\text{Ca}(\text{OH})_2)}{\text{MW}(\text{H}_2\text{O})} \times \frac{1}{1 - \text{LH}_2\text{O}(\text{wt}\%)} \quad (1)$$

$$\text{CaCO}_3(\text{wt}\%) = \text{LCO}_2(\text{wt}\%) \times \frac{\text{MW}(\text{CaCO}_3)}{\text{MW}(\text{CO}_2)} \times \frac{1}{1 - \text{LH}_2\text{O}(\text{wt}\%)} \quad (2)$$

The Vicat test and thermogravimetric analysis can evaluate the effect of different admixtures on the long-term carbonation reaction of the binder. To explain the mechanism of phenomenon, analysis of FT-IR spectrometry, scanning electron microscope (SEM) and physico-chemical properties were performed.

2.2.4 Physico-Chemical Properties

The chemical reaction between the four admixtures and the solution of binder leaching (“limewater”) was investigated. This experiment can simulate the chemical reaction in the aqueous phase (pore solution) of the mortar at the initial stage of the reaction. In order to obtain the mortar leaching solution, the water-binder ratio was increased to 4 and 2 respectively, as shown in Fig. 2.

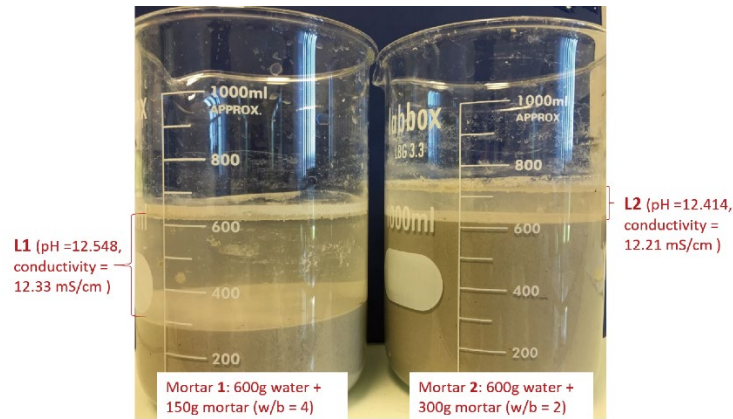


Figure 2: Preparation of the solution of mortar leaching

After thorough mixing, the mixture was allowed to stand for 10 minutes, the upper leaching solution (i.e., L1 and L2 in Fig. 2) was filtered out with a 10 μm pore size filter paper. pH and ionic conductivity of the solution were measured with a multi-functional pH meter. Although mortar 2 had two times higher binder content than mortar 1, their leaching solution L2 and L1 had almost the same pH and conductivity, which indicated that both L1 and L2 were saturated solutions of the binder. So, the solution used for the next analysis was L1 (larger amount).

Main ionic components in the binder leaching solution were Ca^{2+} and OH^- and both Ca^{2+} concentration and pH affect cement hydration [23]. The pH and ionic conductivity of the mixed solution were measured to investigate the ionic reaction between the commercial admixture and the binder leaching solution. The details and results were presented in Fig. 3 and Tab. 2.

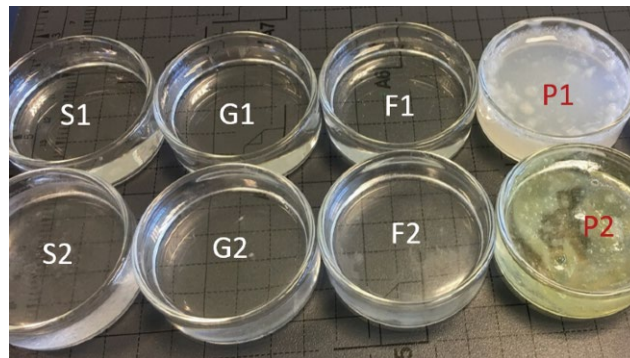


Figure 3: Dissolving admixtures in water (S1, G1, F1, P1) and binder leaching solution (S2, G2, F2, P2)

Table 2: The pH and ionic conductivity (IC) of the solution in Fig. 3 and a control group (NaOH solution, S3, G3, F3, P3)

Mixture solution	pH	IC (mS/cm)
Water	6.946	0.003
S1 (60g water + 1g sucrose)	7.561	0.556
G1 (60g water + 1g glucose)	7.506	0.547
F1 (60g water + 1g fructose)	7.588	0.581
P1 (60g water + 1g pectin)	3.942	1.191
<hr/>		
Leaching solution L1	12.548	12.33
S2 (60g L1 + 1g sucrose)	12.366	7.55
G2 (60g L1 + 1g glucose)	11.951	4.90
F2 (60g L1 + 1g fructose)	11.837	4.05
P2 (60g L1 + 1g pectin)	10.100	2.41
<hr/>		
NaOH solution	12.480	1.118
S3 (60g NaOH solution + 1g sucrose)	12.346	0.828
G3 (60g NaOH solution + 1g glucose)	11.967	0.572
F3 (60g NaOH solution + 1g fructose)	11.851	0.544
P3 (60g NaOH solution + 1g pectin)	10.679	0.445

Fig. 3 showed that addition of pectin formed a green gel in the solution of mortar leaching (i.e., sample P2 in Fig. 3). Interaction between anionic groups (COO^-) of pectins and divalent metal ions (e.g., Ca^{2+} , Fe^{2+} , Zn^{2+}) can explain by this gelation phenomenon [27,28]. In addition, pectin macromolecules can form covalent bonds (i.e., $-\text{C}-\text{O}-\text{Si}-$ bond) with silica and form pectin-silica gels [29].

After alkaline treatment and etherification, fructose can chelate with calcium ions to form fructose calcium [30]. This is also a method of industrial separation of fructose and glucose. The chelation of pectin and fructose molecules with calcium ions produces precipitates or chelates that may increase the nucleation seed of the cement hydration reaction, thus accelerating coagulation [20].

2.2.5 FT-IR Spectrometry

The samples showed in Fig. 3 were analyzed by Fourier Transform Infrared Spectrometry (FT-IR) in transmission mode with a Nicolet iS10 FT-IR spectrometer. The transmission spectra were recorded with a resolution of 2 cm^{-1} and + at a wavenumber range of $4000\text{-}650 \text{ cm}^{-1}$. The amount of admixture used in previous section (Fig. 3 and Tab. 2) (i.e., liquid / admixture = 60/1) was too small to be detected by Infrared spectrometry, therefore the admixture content was increased to 10g (i.e., liquid/admixture = 60/10) and part of the results were in Fig. 7.

3 Results and Discussion

The results of Vicat test are given in Fig. 4. Changes in needle penetration over time can be used to evaluate the setting time and degree of solidification.

The maximum needle penetration was 40 mm. The setting time depends on the sort of added carbohydrate and its concentration. In order to compare the 12 different mixtures, the setting time was estimated for each mixture and the values can be transcribed according to the concentration of each added carbohydrate, as shown in Fig. 5.

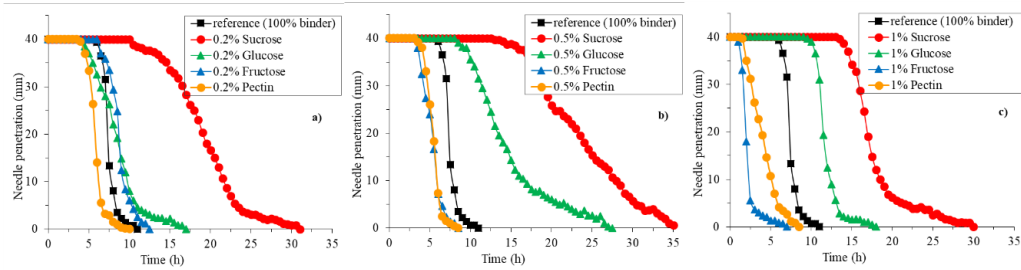


Figure 4: Vicat test results with the admixture concentration (a): 0.2%, (b): 0.5%, (c): 1%

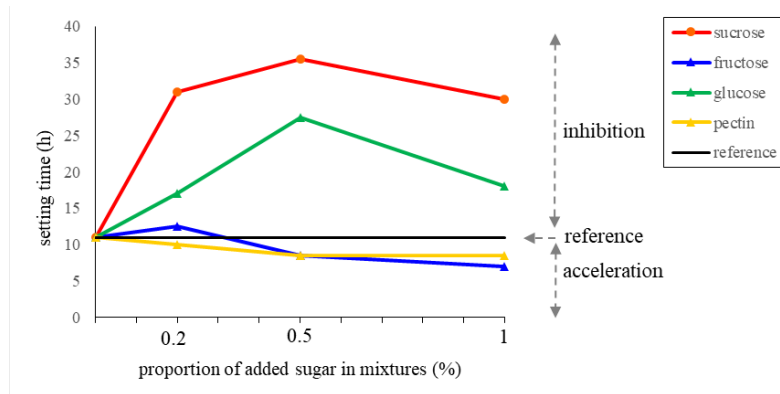


Figure 5: Setting time depending on the proportion of added carbohydrate in the mixture

The setting time for reference, i.e., mixture without added carbohydrate, is about 11 h. All added carbohydrates affect setting time. When the value of setting time is inferior to 11 h, the admixture can be considered as an accelerator, and when the value of setting time is superior to 11 h, the admixture can be considered as an inhibitor. Sucrose and glucose have a stronger retardation effect than the other two admixtures. The maximum of inhibition is found when the proportion of sucrose or glucose is 0.5%. Despite this, lime matrix appears to be less affected by the setting retarder: It was reported that sucrose and glucose could delay the cement setting for several days when adding 0.2 wt% [18,31]. Diquelou et al. [12] pointed out that the addition of lime can increase the compressive strength of hemp concrete and reduce the incompatibility between hemp and the cement because calcium hydroxide and calcium carbonate particles can provide more nucleation sites [32].

Fructose and pectin accelerate the solidification of the lime-based binder. In particular, the addition of 1% fructose or pectin leads to a significant reduction in initial setting (Figs. 4 and 5). The setting times were reduced by 36 and 22%, respectively. However, no existing study that clearly interprets the mechanism how some organic admixtures (e.g., fructose, pectin) accelerate the setting of the Ordinary Portland Cement (OPC) or lime-based binder. Kavas et al. [33] found that pectin can accelerate the cement hydration because pectin could decrease the concentration of Ca^{2+} in the pore solution accelerating the dissolution of calcium from C_3S and forming hydration products. However, this assumption has not been experimentally proven. On the other hand, in the lime-cement matrix, the concentration of dissolved Ca^{2+} is maximum, even though fructose and pectin can react with Ca^{2+} , it is difficult to reduce the amount of Ca^{2+} substantially in the pore solution. Depending on its concentration, fructose can be either an inhibitor or an accelerator.

Figs. 6(a) and 6(b) respectively indicated that, for different mixtures, the content of CaCO_3 and $\text{Ca}(\text{OH})_2$ changed over time with a different rate, through TGA analysis. The long-term (3 d, 7 d, 14 d, 28 d, 90 d) content change of CaCO_3 and $\text{Ca}(\text{OH})_2$ was negatively correlated, because the long-term solidification of the binder was mainly caused by the carbonation reaction, i.e., carbonation of $\text{Ca}(\text{OH})_2$ to CaCO_3 . However, the short-term (1 h, 3 h, 7 h, 1 d) content change of $\text{Ca}(\text{OH})_2$ was relatively complicated, the

main reason is that the content of calcium hydroxides is affected by both carbonation reaction, cement hydration and pozzolanic reaction.

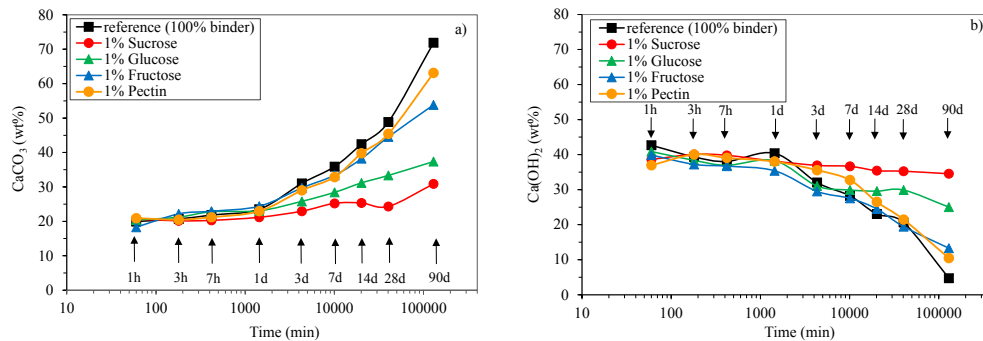


Figure 6: Thermogravimetric analysis results. (a): changes in CaCO₃ content over time (b): changes in Ca(OH)₂ content over time

No significant correlation between CaCO₃ content and initial setting can be noted. Solidification was almost completed (R, F1%, P1%) or still not started (S1%, G1%) during the first 7 hours, whereas the CaCO₃ content was similar in each case. The setting time of the binder may not be determined by the rate of lime carbonation. This phenomenon agrees with the assumption that the early age property of lime-based binder is mainly provided by the hydraulic binder (e.g., Portland cement, pozzolanic materials) [19].

Fig. 6 showed that the carbonation reaction is a slow and long-lasting reaction. From the third day (3d), the CaCO₃ content of S1% and G1% was significantly lower than that of other samples, while the Ca(OH)₂ content was higher than other samples. This indicated that sucrose and glucose not only hinder the cement hydration but also have a strong inhibitory effect on the carbonation of calcium hydroxide. This result proved that sugar molecules (i.e., sucrose and glucose in the present study) could be sorbed on calcium hydroxide (Ca(OH)₂) and cement particles (e.g., C-S-H, C₃S, C₂S), thereby hindering the subsequent reaction [23]. Although fructose and pectin have no inhibitory effect on the initial setting, they have a negative impact on long-term carbonation. After 90 days, the CaCO₃ contents for samples with 1% of fructose or pectin were significantly lower than the control group. The high-molecular-weight polymers (e.g., pectin in this study) tend to agglomerate binder particles and cations thus form larger polymer particles, while small molecules (i.g., fructose, glucose, sucrose) can easily be adsorbed on binder particles, thus prohibiting the hydration of cement [34,35]. Tab. 2 showed that all tested admixtures decrease the conductivity of the mortar leachate. One possible explanation is that saccharides neutralize leaching solution by the trapping of cations like Ca²⁺ and Al³⁺ [36]. In the aqueous phase of lime and cement matrix, cations are derived from the dissolution of the binder particles (e.g., Ca(OH)₂, C₃S, C₃A, etc.).

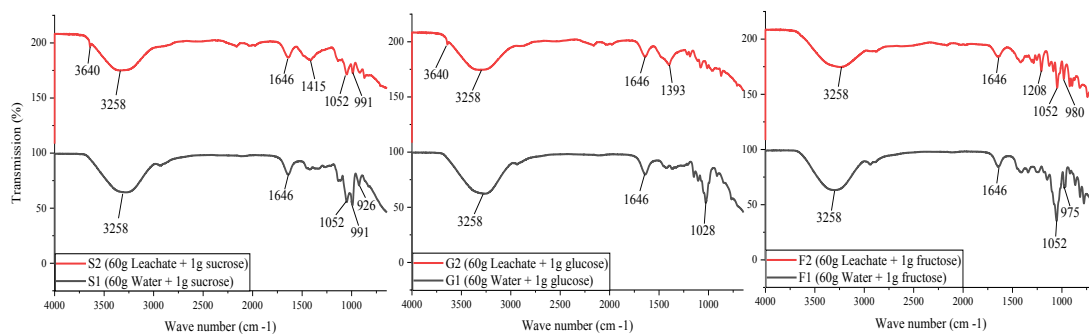


Figure 7: The infrared spectrum of part of samples described in Fig. 3 and Tab. 2. (a) S1, S2; (b) G1, G2; (c) F1, F2

Fig. 7 is the result of the FT-IR analysis for part of the samples showed in Fig. 3. The most significant FT-IR bands were $1052/1028\text{ cm}^{-1}$ (-C-O stretching vibrations), 1415 cm^{-1} (C-O-H in plane bending in carbohydrates), 1393 cm^{-1} (-CH₃ out-of-plane bending vibrations), $991/926\text{ cm}^{-1}$ (C=C-H out of plane bending vibrations), and 1646 cm^{-1} (-C=C stretching vibrations) [37]. The 1646 cm^{-1} band peak of the sucrose, glucose, and fructose in water and in the chelating solution were essentially unchanged, indicating that their -C=C- bond was not destroyed. It was observed that glucose's 1028 cm^{-1} band peak almost disappeared, indicating that its C-O-C bond was destroyed, i.e., the glucose molecule changed from a ring to a chain.

Smith et al. [38] thought that glucose became calcium gluconate under alkaline conditions. The -C-O bond became COO⁻ and formed a complex with Ca²⁺ which condenses on the surface of the cement particle. However, we can't still explain why the same concentration of sucrose inhibits cement hydration more strongly than glucose, and why the fructose accelerates cement hydration.

4 Conclusion

In this study, effects of organic components on lime-based binder were observed. These effects are different to those observed with cement-based binder. Concerning the setting time, the lime-based binder is less affected by admixtures than cement, but the long-term carbonation reaction can be irreversibly hindered by sucrose and glucose. For an optimal use and development of mortars lightened by plant aggregates, plants with high sucrose content (e.g., sugarcane bagasse) must be avoided. Since the carbonation reaction is indispensable for strength development and requires a long time to be tested, finding a rapid method to predict the compatibility between plant-aggregates and binder matrix is necessary. The setting time (Vicat test) is a possible indicator to predict the compatibility between the binder matrix and plant aggregates. When the value of setting time is inferior to that of the pure binder, the admixture can be considered as an accelerator, otherwise the admixture can be considered as an inhibitor. In this work, the setting delay corresponds well with the carbonation halting. The setting accelerators (i.e., fructose and pectin) can also affect the carbonation reaction, but the inhibition is not as strong as with sucrose and glucose. One hypothesis is that these admixtures can adsorb on the surface of Ca(OH)₂ particles and block the nucleation site or prevent Ca(OH)₂ from reacting with CO₂ and H₂O. This hypothesis was studied using infrared spectrometry, conductivity analysis. This research can help improve the formulation of bio-based mortars used in the building sector and promote further research to find more appropriate plant aggregates and treatment methods.

Further research aims to study the mechanical properties of plant-aggregate-based mortars in order to evaluate the impact of added carbohydrates on flexural and compressive strengths. Otherwise, this work can be extended with the study of other polymers (lignin, hemicellulose, cellulose) as additives and with the study of the influence of their molar masses.

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