INCIDENCE OF THE WATER-SOLUBLE COMPOUNDS CONTAINED INTO LAVENDER AND SUNFLOWER BIOAGGREGATES ON THE HARDENING PROCESS OF MINERAL BINDERS

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Abstract

In building materials, the incorporation of plant aggregates into mineral matrix is an eco-friendly promising solution as these biobased building materials can answer to the new requirements for thermal and hydric performances. Nevertheless, plant aggregates can have deleterious effects on the hardening mechanisms of mineral binders. The aim of this study is to evaluate the incidence of the water-soluble compounds contained into bioaggregates on the hardening process of three mineral binders (CEMI 52.5 cement, metakaolin-based pozzolanic binder, lime-based commercial binder). Two vegetal by-products were selected (lavender straw and sunflower depithed stalk) because of their large availability in France. Distilled lavender straw is the by-product of the steam distillation process applied to lavender plant. Sunflower bark aggregates were obtained from the fractionation of the entire stalk (i.e. bark plus pith). The chemical composition of the two bioaggregates was first studied by the ADF-NDF method of Van Soest and Wine. The cellulose, hemicellulose and lignin contents were thus determined. In addition, the water-soluble components were estimated by measuring the mass loss of the bioaggregates after 1 h in boiling water. Large differences were highlighted concerning the content in water-soluble compounds between the two plant aggregates. Model pastes were then elaborated with a solution obtained by soaking lavender or sunflower powder in demineralized water for 48 h and then filtering. The properties of the pastes were compared with those of neat pastes of the three mineral binders. The setting time (followed by isothermal calorimetry), the hardening mechanisms (followed by X-ray diffraction and thermogravimetric analysis) and the mechanical performance of the different binders were clearly and variously influenced by the lavender and the sunflower extractives at early ages. The level of the impact on the cementitious and pozzolanic binders can be correlated with the content in water-soluble compounds. Concerning the lime-based binder, it was less impacted by the water-soluble species.

Keywords:

Biobased building materials; Mineral binder; Bioaggregate; Lavender; Sunflower; Water-soluble compounds; Hardening

1 INTRODUCTION

The building construction industry has a major impact on sustainable development, mainly in terms of the use of raw material, greenhouse gas emissions and waste production. The civil engineering sector worldwide consumes about 50% of raw materials coming from the soil and produces about 40% of all waste due to human activities [Buyle-Bodin 2002]. Studies on alternative materials are clearly a priority nowadays, to reduce energy consumption and optimize waste management.

Bio-based construction materials could take up this environmental challenge. Bio-based aggregates are renewable; they are mainly produced locally and they constitute an important way to store carbon dioxide. Most of the time, these bio-based aggregates come from local agricultural activities located near the production sites of building materials. As sub-products of these industries, their use helps to decrease the quantity of waste produced.

Many studies have underlined the great interest of the hygrothermal properties of bio-based materials [Cerezo 2005][Chabannes 2015][Evrard 2008][Le Tran 2010]. The thermal conductivity of many materials is clearly reduced by the use of bioaggregates, while moisture exchanges with the air around the material can be very useful to control indoor air humidity and can enhance indoor comfort.

Despite these promising properties, negative chemical interactions can occur between lignocellulosic particles and mineral binders as stressed in the first chapter of the recent state of the art report of the RILEM Technical Committee 236-BBM [Magniont 2017]. At early age, they can disturb the setting and hardening mechanisms of mineral binders, in the hardened state, they can modify the properties of the composite and in the long term, they can influence the durability of the material.

A significant study by Diquelou et al. [Diquelou 2015] explored the chemical interactions between hemp or flax shives and cement matrix. Many other studies confirm the deleterious effects of different lignocellulosic materials on the setting and hardening kinetics and mechanisms of mineral binders [Sedan 2008][Jorge 2004][Na 2014][Govin 2006][Vaickelionis 2006][Irle 1996][Eusebio 1990][Soroushian 2004] [Aggarwal 2008][Bilba 2003][Menezes Brasileiro 2013]. Most of the results reported in these references concern Portland cement hydration and hardening, but the mechanisms involved in the case of lime or pozzolanic binders might be significantly different. One previous study achieved by Ratiarisoa et al. [Ratiarisoa 2016] reported that the setting of a pozzolanic binder (mix of lime and metakaolin) was affected in presence of lavender water-soluble compounds.

Chemical interactions between lignocellulosic particles and mineral binders at early age involve complex mechanisms, which are not yet fully and precisely understood. The deleterious species responsible for the interactions are of two types: water-soluble compounds and products of the alkaline attack generated by the cement or lime-based matrix on the plant aggregates or fibres [Diquelou 2015][Govin 2005].

In order to confirm the hypothesis of typical deleterious interactions between water-soluble compounds contained in the plant aggregates and the binder, this study was conducted on model pastes. The samples were elaborated with the binder and the mixing solution. Three mineral binders were investigated (CEMI 52.5 cement, metakaolin-based pozzolanic binder, lime-based commercial binder). Two vegetal by-products were selected because of their large availability in France: raw lavender and sunflower bark.

The chemical composition of the two bioaggregates was first studied by the ADF-NDF method of Van Soest and Wine.

Model pastes were then elaborated with a solution obtained by soaking lavender or sunflower powder in demineralized water. The properties of the model pastes were compared with those of neat pastes for the three mineral binders. The setting time (followed by isothermal calorimetry), the hardening mechanisms (followed by X-ray diffraction and thermogravimetric analysis) and the mechanical performance of the different pastes were explored.

2 MATERIALS

2.1 Mineral binders

The effect of water-soluble components contained into plant aggregates was studied on three different mineral binders. They were selected because of their distinct mechanisms of hardening (hydraulic setting, pozzolanic reaction and carbonation).

Cement binder (C)

Portland cement is the most widely used hydraulic binder in the world. It has been already used for the design of biobased concretes [Diquelou 2013][Sedan 2007]. The cement used for this study is a CEM I 52.5 N CE PM CP2. Letter C will refer to this binder in the results section.

Pozzolanic binder (M)

The second mineral binder used in this study was designed in previous works [Magniont 2010][Dinh 2014] to be associated with plant aggregates in composites for the design of precast blocks or with fibres for coatings. It associates 30% of aerial lime and 70% of flash calcined metakaolin. The mix was completed by the addition of two admixtures: 3% of potassium sulfate (K_2SO_4) to improve strength at early age and 1.6% of superplasticizer to reduce the mixing water content. Letter M will refer to this binder in the results section.

Lime-based binder (A)

Lime-based binders are the most common binders used in combination with plant aggregates both for research activities [Nguyen 2010][Nozahic 2012] and in the field. The lime-based binder used for this study is composed by 75% of aerial lime, 15% of hydraulic binders and 10% of pozzolanic binders. Letter A will refer to this binder in the results section.

2.2 Plant aggregates

Among the several bio-aggregates that can be considered, the waste derived from food, medicinal or aromatic oils production appears to be an important source. Two plant aggregates were selected for this study because they are both by-products of local agricultural activities and largely available in France.

Sunflower depithed stalk, i.e. sunflower bark(S)

Sunflower culture leads to non-valorized byproducts such as stalks. After a crushing and sorting treatment, two bioaggregates could be used: bark and pith. The part used in this study was the bark only. Sunflower stalks originated from a farm in the south west of France. Letter S (for Sunflower) will refer to this aggregate solution in the results section.

Lavender (L)

Lavender is cultivated since antiquity for its aromatic flowers. The straw collected after the steam-distillation process currently constitutes a non-valorized by-product. For this study, distilled lavender straw was provided by the Lacontal farm located in the Quercy region in the south west of France. Letter L (for Lavender) will refer to this aggregate solution in the results section.

3 METHODS

3.1 Aggregate characterization

Aggregate preparation

In order to optimize chemical extractions and to enhance the effects of water-soluble components on pastes, raw aggregates (lavender straw or sunflower bark) were crushed in powder. Aggregates were treated in two steps. First, a Electra BC P type hammer mill fitted with a 8 mm sieve was used. The obtained sample was then crushed with a FOSS Cyclotec 1093 mill using a 1 mm sieve in the second step.

Chemical composition

Chemical dosage of the plant material was carried out on the vegetal powder using the Van Soest and Wine method [Van Soest 1967][Van Soest 1968]. It is also called ADF-NDF method due to the detergents used for the extraction of the non-water-soluble components (ADF for Acide Detergent Fiber, NDF for Neutral Detergent Fiber). This method leads to the cellulose, hemicelluloses and lignins contents, in percentage of the dry material. In addition, the mineral content was determined according to ISO 749:1977. Lastly, the water-soluble components were estimated by measuring the mass loss of the bioaggregates after 1 h in boiling water.

3.2 Paste characterization

Paste elaboration

To study the effects of water-soluble compounds only (without any aggregate size effect) and to optimize them, all the following tests were performed on model pastes. Three tests were performed for each case. For each binder (C, M and A), three different model pastes were made with demineralized water (W), sunflower solution (S) and lavender solution (L). The following method was used to prepare the plant solutions. First, 500 g of aggregate powder was put in 5 kg of demineralized water and the mix was stirred at 400 rpm during 48 h. Then, the solution obtained was filtered with a 50 µm nylon sheet. The mixing was completed using a mixer complying with NF EN 196-1 with a w/b ratio of 0.4 for the cement and the lime based binders and 0.55 for the pozzolanic based binder. After mixing, the binder paste was cast in 40x40x160 mm^3 moulds complying with NF-EN 196-3 and kept in endogenous conditions in a room at 20°C for 2 days.

Isothermal calorimetry

The setting process of the pastes was studied by isothermal calorimetry using TAM AIR 3116 microcalorimeter. The paste was elaborated manually with components at 20°C outside the calorimeter. Heat flow due to the setting reactions was recorded during 6 days.

Thermogravimetric analysis (TGA)

TGA was performed on dry powdered samples, weighing around 1050–1350 mg each passing through an 80 µm sieve. The thermal analyzer used was a NETZSCH STA 449 F3 Jupiter operating at a heating rate of 10°C/min up to 800 °C. Analyses were performed 6 h and 28 days after mixing.

X-ray diffraction analysis (XRD)

The measuring system was a Bruker D8 Advance diffractometer using Kα (λ=1.542 Å) copper anticathode. The 2-Theta values ranged from 4° to 70° and were recorded at a 0.02° step with an acquisition time of 0.25 s per step. Measurements were performed on crushed samples passing through an 80 µm sieve, of ages 6 h, and 3, 8, 14, and 28 days. Setting and hardening processes were stopped for each time by liquid nitrogen immersion and vacuum lyophilization at 13.3 Pa during 24 h.

Mechanical performance

After 2 days of curing in endogenous conditions, the samples were demolded and sawn in three 40x40x50 mm^3 parts. Cement based and pozzolanic based samples were kept in endogeneous conditions in sealed plastic bags and lime based samples without bags in the laboratory test room at 20°C and uncontrolled RH until the date of the test.

Compressive strength tests were performed on specimens of ages 3, 7, 14, 28 and 49 days. They were conducted with a constant loading rate of 2.4 kN/s on a Controlab compression and flexural testing machine.

4 RESULTS

4.1 Aggregates composition and water-soluble compounds

As mentioned in the introduction, chemical composition of bioaggregates can affect the mechanical behavior and the setting and hardening processes of biobased materials. This study focused on the effects of the water-soluble compounds of the plant material that can affect the binder hydration by diffusion into the mixing water.

Tab. 1: Chemical composition of bioaggregates

% of dry matter	Sunflower bark (S)		Lavender straw (L)	
Cellulose	46.1	±0.2	33.7	±0.7
Hemicelluloses	16.8	±0.1	13.9	±0.1
Lignins	18.6	±0.2	14.7	±0.1
Minerals	4.9	±0.1	7.7	±0.1
Water-solubles	10.6	±0.4	22.7	±0.2

The results of the chemical analysis of sunflower and lavender powders are presented in Tab. 1. Lavender presents a lower proportion of cellulose, hemicelluloses and lignins than sunflower but it contains much more minerals. The most important information given by this analysis is the two times higher rate of water-soluble compounds in lavender (22.7% instead of 10.6%). This difference could lead to stronger interactions when using lavender as bioaggregates in building materials.

Tab. 2: Water-soluble contents in bioaggregates before and after extraction using water as extracting solvent, and in the mixing solutions obtained

	Sunflower bark (S)	Lavender straw (L)
Water-solubles before water extraction (% of dry matter)	10.6 ±0.4	22.7 ±0.2
Water-solubles after water extraction (% of dry matter)	5.2 ±0.1	8.6 ±0.2
Extraction yield in water-solubles (%)	51	62
Concentration in water-solubles in mixing solution (mg/g)	47	131

The same chemical analysis was performed on the sunflower and lavender powders recovered after the water-soluble extraction (i.e. the plant solution preparation). The results are presented in Tab. 2. The latter show a slightly higher extraction rate for lavender (62% instead of 51%). This higher rate, combined to the higher proportion of water-soluble compounds in the raw material, leads to a water-soluble concentration in the final solution used 2.5 higher for lavender compared to sunflower.

These water-soluble compounds (monosaccharides like free sugars, light polysaccharides, amino acids, peptides, phenolic compounds, terpenes or organic acids, etc.) could interact in the setting and hardening processes. The high difference between sunflower and lavender solutions can help to enlighten the effects of water-soluble compounds.

4.2 Effects on setting time

Isothermal calorimetry results are presented in Fig. 1. These results clearly show a major effect of water-soluble compounds contained in the lavender solution on the setting process of each binder.

Tests performed on model pastes with lavender solution show not only a setting delay but also a lower intensity of heat flow for all the binders. Using the binder mixed with mineralized water as the reference, the setting delay on cement and lime based binders is 3 days; on pozzolanic based binder 2 days. The heat flow peak is also reduced to more than half for each case. These results indicate a partial inhibition of the setting due to water-soluble compounds from lavender straw.

(a)

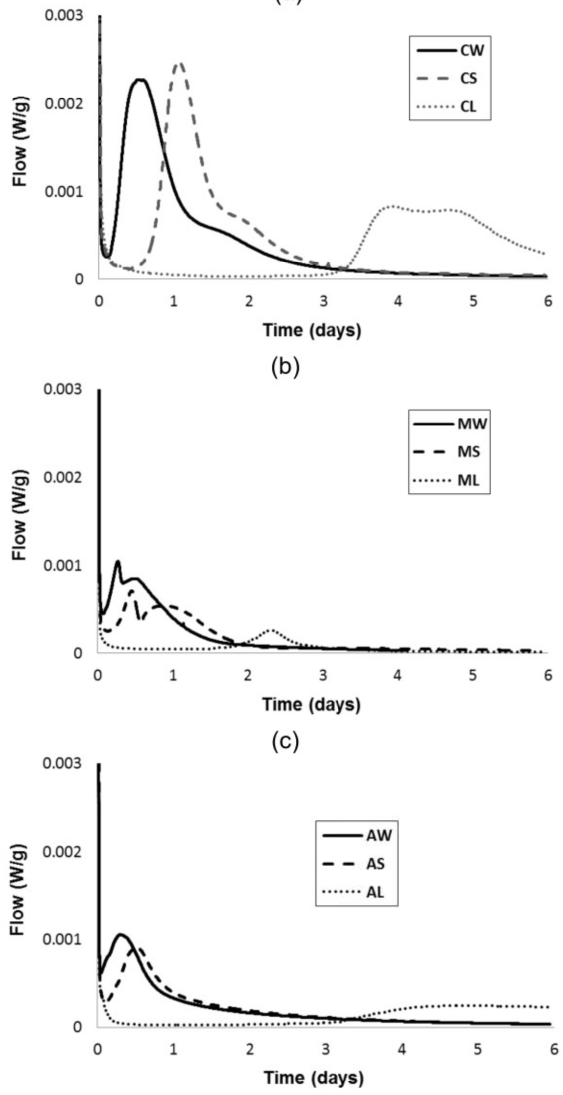

Fig. 1: Isothermal calorimetry results for model pastes (a: cement; b: pozzolanic binder; c: lime-based binder)

Similar results had been reported on Portland cement setting with wood [Na 2014] or hemp [Diquelou 2015]. Terpenes and phenolic compounds are often cited as responsible for setting delay [Na 2014][Semple 2002][Vaickelionis 2006]. In distilled lavender, they are part of the main chemical compounds [Lesage-Meessen 2015]. Other studies enlightened the negative impact of wood pretreatment with high temperature [Govin 2006][Boustingorry 2005] or vapor [Wei 2003]. A similar phenomenon could occur with the distillation of lavender that would explain the high water-soluble rate in the solution and the strong impact on the setting process.

The solution obtained with raw sunflower bark did not lead to effects as strong as lavender. Nevertheless, results evidenced a delay for the three tested binders. However, this delay remains less than half a day in any case and the heat flow peak is only slightly reduced for lime and pozzolanic based binders.

In conclusion, these results confirm the strong correlation between the amount of water-soluble compounds that can be extracted in the mixing water and the intensity of the setting delay and/or the inhibition of the binder.

4.3 Effects on the hardening process

Compressive strength evolution

In order to identify the impact of water-soluble compounds on the hardening process of the selected binders, the compressive strength of the model pastes mixed with sunflower and lavender solution were compared with those of neat paste. All the results are presented in Fig. 2.

The setting delay previously observed with lavender solution clearly influences the mechanical performances of the pastes with cement and pozzolanic based binders. At early age (3 days), the strength drop is significant as no resistance was measured. With the cement based binder, a 25% resistance loss remained from 7 to 28 days and the difference disappeared at 49 days. With the pozzolanic based binder, the model paste resistance was 75% lower with lavender solution at 7 and 14 days, increased to 50% of neat matrix at 28 days and was the same at 49 days.

Model pastes with sunflower solution did not present any significant compressive strength decrease except at early age for the pozzolanic based binder. After 3 days of curing, this deficit is about 40%. This result is in good accordance with the decrease of the heat flow peak observed previously.

No difference was observed on the lime based binder between demineralized water, sunflower solution or lavender solution. This result suggests that the aerial setting due to the carbonation of $Ca(OH)_2$ is not significantly affected by the water-soluble compounds from the plant aggregates. The delay and the large reduction of the amount of heat released observed during the isothermal calorimetry test (see Fig. 1.c) would then be only related to the modification of the hydraulic or pozzolanic part of the setting of this commercial lime-based binder.

These results showed that a high content in water-soluble compounds in mixing water can induce a strong decrease of mechanical performances during the first month for hydraulic and pozzolanic binders.

However, this reduction in the compressive strength disappeared after 49 days.

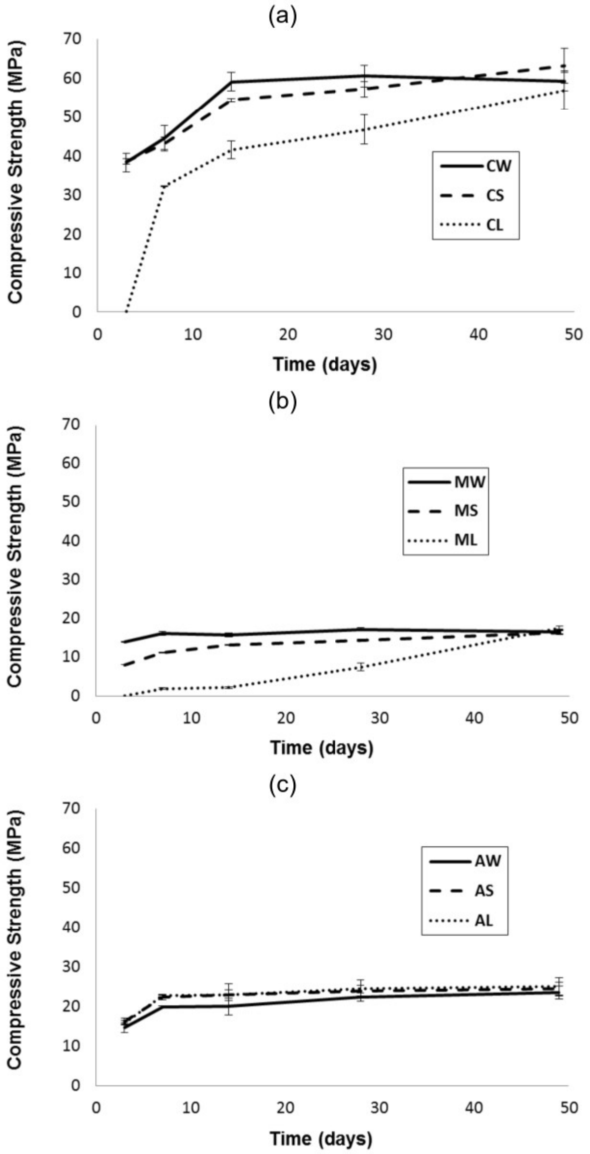

Fig. 2: Compressive strength of model pastes at 3, 7, 14, 28 and 49 day (a: cement; b: pozzolanic binder; c: lime-based binder)

Thermogravimetric Analysis (TGA)

Complementary analyses were carried out using thermogravimetric analysis (TGA). Differential TGA curves after 6 h for neat paste and pastes mixed with solutions are presented in Fig. 3. These results are in good concordance with the results previously reported and show details on the nature of the hydrates impacted by the water-soluble compounds.

At 6 h, for the cement binder (Fig. 3.a), the effects of sunflower and lavender water-soluble compounds were clearly enlightened. The peak ranging from 100°C to 300°C and corresponding to the products of the hydration (C-S-H) is not visible in presence of plant extractives. This is in accordance with the setting delay observed on Fig. 1 (> 12h). Similar observation can be made with the peak corresponding to portlandite (between 450°C and 500°C) which is significantly reduced when water-soluble compounds of sunflower or lavender were added.

For the pozzolanic binder (Fig. 3.b) the addition of lavender induces the strongest impact. After 6 hours, the products of the pozzolanic reaction which degrade between 100°C and 450°C (C-S-H, ettringite, calcium aluminosilicate hydrates and calcium carboaluminate hydrates [Dinh 2014]) are significantly reduced. The amount of residual portlandite (500°C) is also higher in presence of lavender. This result confirms the delay in the development of the pozzolanic reaction. The mix containing sunflower water-soluble compounds presents an intermediate behavior between neat and lavender paste, except for one higher peak around 100°C.

Concerning the lime-based binder (Fig. 3.c), after 6 hours, the main products (C-S-H, C-A-S-H) should result from the reaction of its hydraulic or pozzolanic compounds. The deleterious impact of the compounds of sunflower and lavender on these setting mechanisms are enlightened again, with a more pronounced effect of lavender water-soluble compounds.

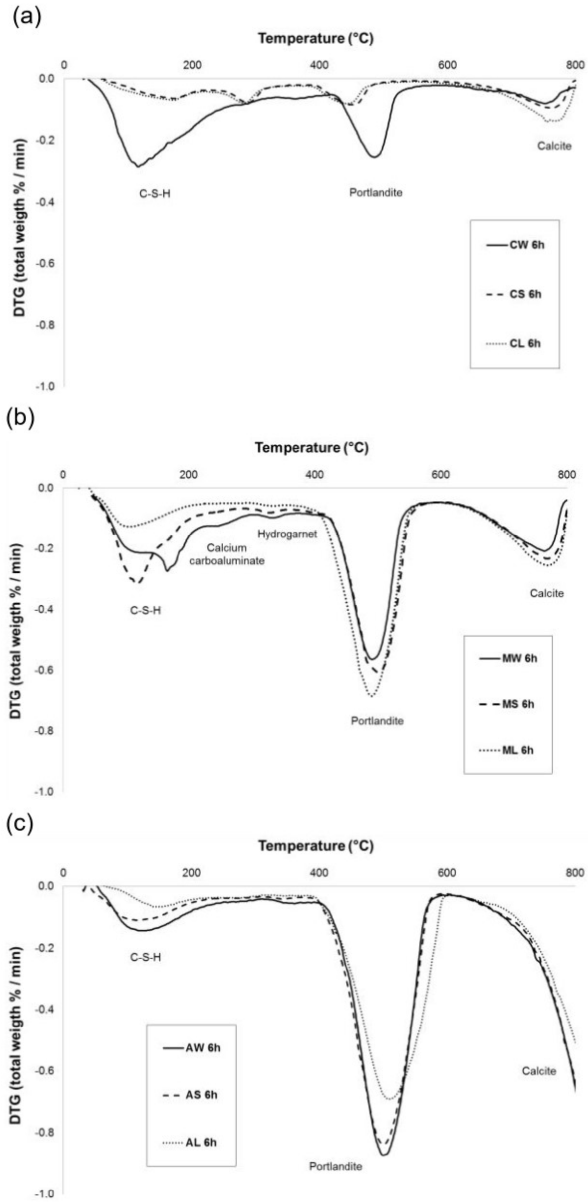

Fig. 3: TGA Analysis of model pastes at 6 hours (a: cement; b: pozzolanic binder; c: lime-based binder)

X Ray Diffraction (XRD)

To complete the results of the thermogravimetric analysis and to explore the mechanisms leading to the setting and hardening delay, XRD was performed on pastes of age 6 hours, 3, 7, 14 and 28 days.

The main observations realized are the following:

- For the cement paste: a clear delay in the appearance of portlandite (at 6 hours, 3 days and 7 days for neat, sunflower and lavender pastes respectively) and in the decrease of anhydrous compounds. Additional compounds such as stratlingite are also produced in presence of plant extractives.
- For the pozzolanic paste: a clear delay in the appearance of stratlingte and calcium carboaluminate hydrates (at 6 hours, 3 days and 28 days for neat, sunflower and lavender pastes respectively) and in the decrease of $Ca(OH)_2$.
- For the lime-based binder, the XRD analysis did not allow to exhibit any significant differences between the three pastes.

5 CONCLUSION

Setting and hardening processes of the cementitious and the pozzolanic binders used were clearly influenced by the addition of lavender and sunflower water-soluble compounds into the mixing solution. The intensity of the impact has been correlated to their concentration. Concerning the lime-based binder, it was less impacted by the water-soluble species.

Lavender straw showed a two times higher rate of water-soluble compounds compared with sunflower stalk. Lavender also presented a slightly higher water extraction rate. These two factors led to a water-soluble concentration in the final mixing solution used for model pastes 2.5 higher for lavender compared to sunflower.

The amount of water-soluble compounds that can be extracted in the mixing water had a strong correlation with the intensity of the setting delay.

A high content of water-soluble compounds in the mixing water induced a strong decrease of mechanical performances during the first month for hydraulic and pozzolanic binders. However, this reduction in the compressive strength disappeared after 49 days.

The amount of hydrates created during the setting of hydraulic and pozzolanic binders is clearly delayed with the lavender solution presenting a high water-soluble concentration.

A raw bioaggregate presenting a high content in water-soluble compounds might be less convenient for building materials based on hydraulic or pozzolanic binders because of a setting delay. This is problematic for applications requiring a short term mechanical strength.

However, it might be emphasized that a lower impact might occur with the intact bioaggregate since the powdering of the aggregates has certainly maximized the effects. Further studies need to explore the relation between the mixing solution effect on model pastes and the raw aggregate real effect on the vegetal concrete performances. Bioaggregates washing and/or pretreatment might be necessary and efficient to extend the plant particles that could be used in biobased building materials.

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