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## **EVALUATION OF THE POTENTIAL OF CORN AND SUNFLOWER BARK PARTICLES AS BIO-AGGREGATES FOR LIGHTWEIGHT CONCRETE**

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### **Abstract**

Biomaterials are an alternative to non-renewable materials. Hemp shives allowed developing an industrial sector favorable to sustainable development. Hence, hemp concrete is currently used as filling material in a timber frame (cast onsite, sprayed or precast). Nevertheless, hemp shiv, the reference biobased aggregate, has a limited availability. It is thus necessary to consider other bio-aggregates largely and locally available. Sunflower and corn stalks were selected for their large availability in south-west of France. The objective of this paper is to evaluate the potential of these two agricultural by-products as alternative bio-aggregates for vegetal lightweight concrete through the determination of aggregate properties and the study of chemical interactions between plant particles water-soluble compounds and a pozzolanic binder. The results revealed a strong deleterious effect of corn water-soluble compounds on the setting and the hardening mechanisms of the pozzolanic binder. A 24 hours long setting delay was not only observed but an almost complete inhibition of the hardening was also highlighted by the mineralogical analysis and the evolution of the mechanical performances. Sunflower particles could be a good candidate for hemp shives substitution, since their characteristics are similar and their impact on the binder setting and hardening is moderate.

### **1. Introduction**

The building sector is responsible for major environmental impacts (consumption of non-renewable raw materials, emission of greenhouse gases and waste production). As a consequence, the design and characterization of innovative eco-friendly building materials has become a priority. The incorporation of bio-based raw materials could be a response to this

environmental challenge since they are renewable, are mainly by-products of local crops, and are carbon neutral. Over the last fifteen years, these environmental benefits have contributed to the development of a specific building material called hemp concrete. This composite combines a mineral binder and a plant aggregate: hemp shiv, i.e. the ligneous by-product resulting from the mechanical extraction of technical fibers from hemp stalk. Nevertheless, although hemp has been considered as the reference agro-resource for bio-aggregates based building materials, its availability is limited. Therefore, it is necessary to consider other bio-aggregates largely and locally available.

The present study aims to evaluate the potential of two agricultural by-products (corn and sunflower bark particles) as alternative bio-aggregates for vegetal lightweight concrete. Sunflower and corn stalks were selected for their large availability in south-west of France. Corn is the second most cultivated cereal in France, generating large quantities of by-products: 5.2 Mt of stalks in 2015. Sunflower is grown for its seeds to produce vegetal oil with about 614,000 ha cultivated nationwide. 230,000 tons of by-products were produced each year due to this agricultural crop. In comparison, only 17,000 t/year of hemp by-products are available [1].

The first part of the study focused on the plant particles characterization. According to the recommendations of RILEM TC 236-BBM [2], bulk density, particles size distribution and water absorption capacity were determined on hemp shives and on bark particles extracted from corn and sunflower stalks. In a second part, the impact of the water-soluble components extracted from the different bio-aggregates on the setting and hardening mechanisms of the lime-metakaolin binder was investigated.

## 2. Materials and methods

### 2.1 Bio-aggregates

Two bio-aggregates are evaluated : corn and sunflower bark particles compared to hemp shives. The samples are presented in Fig.1.

Alternative aggregates result from the combination of a preliminary stage of grinding of the entire stalk and a second phase of separation of the pith from the bark using a tilted conveyor belt and a blowing system. The bark particles under 1 mm were then eliminated by sieving.

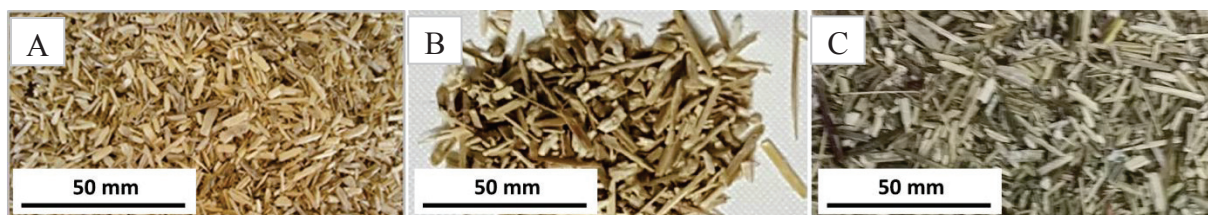


Figure 1: Photographs of the three studied bio-aggregates: hemp shives (A), corn (B) and sunflower (C) bark particles

### 2.2 Mineral binder

The binder is composed of 70 %wt of metakaolin and 30 %wt of aerial lime. Metakaolin, a pozzolanic admixture, is mainly composed of quartz, silicon and aluminium oxides with an

amorphous silicoaluminate mineralogical form. The aerial lime is around 92%  $\text{Ca}(\text{OH})_2$ . Potassium sulfate and a superplasticizer glycerol carbonate (GC) were added.

### 2.3 Aggregate characterization

The physical characterization of plant particles (bulk density, water absorption and particle size distribution) was realized according to RILEM Technical-Committee 236 – BBM (Bio Based Building Materials) recommendations [2].

### 2.4 Experimental methods on binder pastes

Four different mixtures were tested and differed by the nature of the mixing solution: a control paste was made with demineralized water, and the other three pastes were mixed with the filtrate of bio-aggregates. The filtrates were obtained after immersion of crushed particles (under 1 mm) in demineralized water for 48h with a water to particles ratio 15. The distilled water or filtrate to binder ratio was 0.55. The potassium sulfate to binder ratio was 0.03 and the superplasticizer to binder ratio was 0.016.

After mixing, the binder paste was cast in  $40 \times 40 \times 160 \text{ mm}^3$  moulds complying with NF-EN 196-3 [3]. The samples were then demolded at 3 days, cut in three samples and continuously cured in a room at  $20^\circ\text{C}$  and  $\text{RH} > 95\%$  until the date of the test. The pastes are noted Hemp\_P, Corn\_P, and Sunflower\_P. Compressive strength tests were conducted after 3, 14 and 150 days with a constant loading speed of 2.4 kN/s according to NF-EN 196-1 [4].

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

The mineralogical evolution was carried out by X-ray diffraction and thermogravimetric analyses. The measuring system was a Bruker D8 Advance diffractometer using  $\text{K}\alpha$  ( $\lambda=1.542 \text{ \AA}$ ) copper anticathode. The 2-Theta values ranged from  $4^\circ$  to  $70^\circ$  and were recorded at a  $0.02^\circ$  step with an acquisition time of 0.25 s per step. Thermogravimetric analyses were performed on a thermal analyser NETZSCH STA 449 F3 Jupiter® operating at a heating rate of  $10^\circ\text{C}/\text{min}$  up to  $1000^\circ\text{C}$ . For each age, the hydration is stopped by immersion in liquid nitrogen and freeze-drying.

## 3. Results and discussion

### 3.1 Bio-aggregates properties

The bulk densities of bio-aggregates are  $154.2 \pm 0.6 \text{ kg/m}^3$ ,  $120.2 \pm 1.0 \text{ kg/m}^3$ ,  $168.2 \pm 4.5 \text{ kg/m}^3$ , for hemp, corn and sunflower particles, respectively.

The fitted curves and histograms of particle size distribution are presented in Fig. 2.

Fig. 2 reveals that hemp shives present a very narrow particle size distribution in comparison with corn or sunflower bark particles. This could be corrected by a more adequate calibration process for these alternative aggregates in order to eliminate the longer particles. Their circularity is also lower than for hemp particles in particular for corn particles. This result is consistent with bulk density measurement. Corn particles are more elongated and consequently induce a larger interparticular porosity responsible for a lower bulk density.

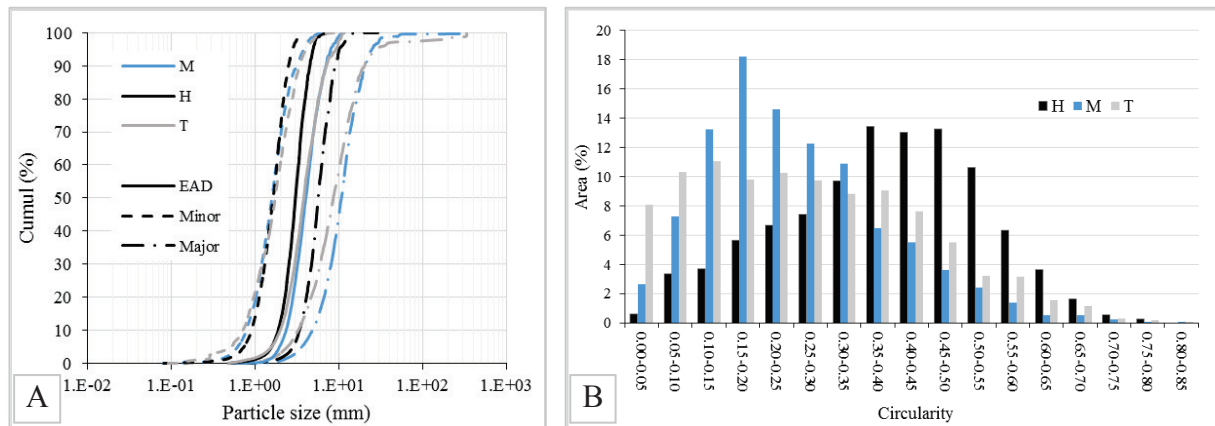


Figure 2: Particle size distribution (A) and circularity (B) of hemp, corn and sunflower particles

The kinetics of water absorption of the three plant particles have been assessed and are plotted in Fig. 3. The figure presents the evolution of the water content of plant particles with the time of immersion and as a logarithmic function of time. The three bio-aggregates present high water absorption capacity, from 250 to 300 %wt after 48h immersion. Fig. 3B evidences distinct initial rate of absorption (IRA) between the particles. After 1 minute, hemp and sunflower aggregates already retain more than 150 %wt while corn particles only absorb 100 %wt. This could be a benefit for corn particles at the time of mixing with the mineral binder.

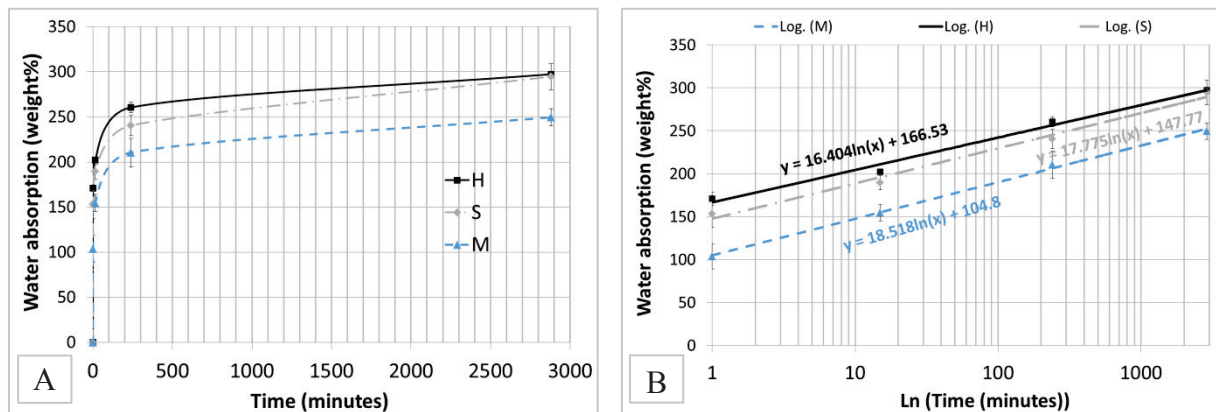


Figure 3: Water absorption curves as a function of time: linear (A) and logarithmic (B) scale

### 3.2 Interactions between bio aggregates extractives and pozzolanic binder

#### 3.2.1 Composite setting study

The isothermal calorimetry tests results are presented in Fig. 4. The heat flow curves highlight the impact of bio-aggregate extractives on the setting mechanisms of the pozzolanic matrix. Two phases are noted. The first ① corresponds to the exothermic reaction between potassium sulphate and calcium ions forming ettringite. The second ② is attributed to the pozzolanic reaction. Regarding hemp and sunflower pastes, a slight delay is observed. In addition, the heat flow intensity related to the reaction is reduced. However, the setting reaction takes place. The corn extractives induce the highest impact by a delay of 27 hours and a significant reduction of the heat flow intensity related to the reaction implying partial inhibition of the setting and hardening mechanisms of the pozzolanic binder. This effect was previously reported in the literature during the hydration of Portland cement in the presence of wood, hemp or lavender.



### 3.2.2 Mechanical performance of pastes

The compressive strengths of pastes are presented in Fig. 5. The results show that the filtrates of bio-aggregates can strongly influence binder pastes strengths. The effect of extractives can be observed from 3 days with a compressive strength value for control paste (CP) of 15.2 MPa while for the other pastes made with extractives the values are lower. Hemp and sunflower filtrates moderately impact the 3 days strength (11.7 Mpa). On the contrary, the model pastes with corn filtrate only reach 1.0°MPa. This decrease noticed at 3 days is still observed after 150 days of curing. At that age, the reduction of compressive strength in comparison with control paste is about 27, 14 and 88 % for hemp, sunflower and corn based pastes, respectively.

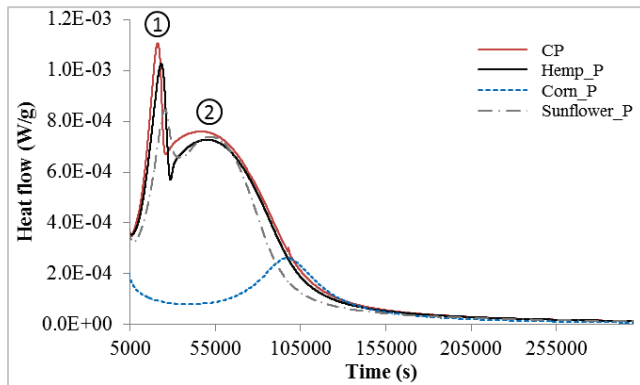


Figure 4: Heat flow by isothermal calorimetry of model pastes.

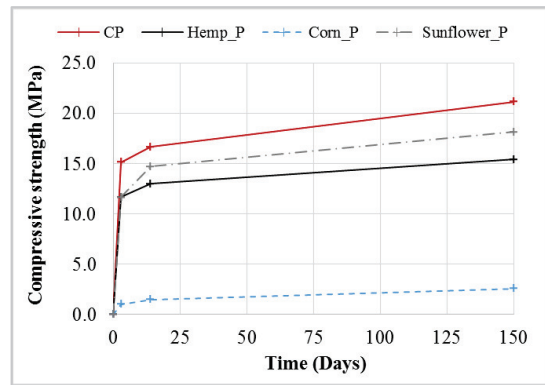


Figure 5: Compressive strength of model pastes at 3, 14 and 150 days.

### 3.2.3 Mineralogical evolution

The short term effect of bio-aggregate extractives was studied by XRD and TGA in order to explore the hardening mechanisms of model pastes. Differential TGA curves realized 24 hours after mixing are plotted in Fig. 6A.

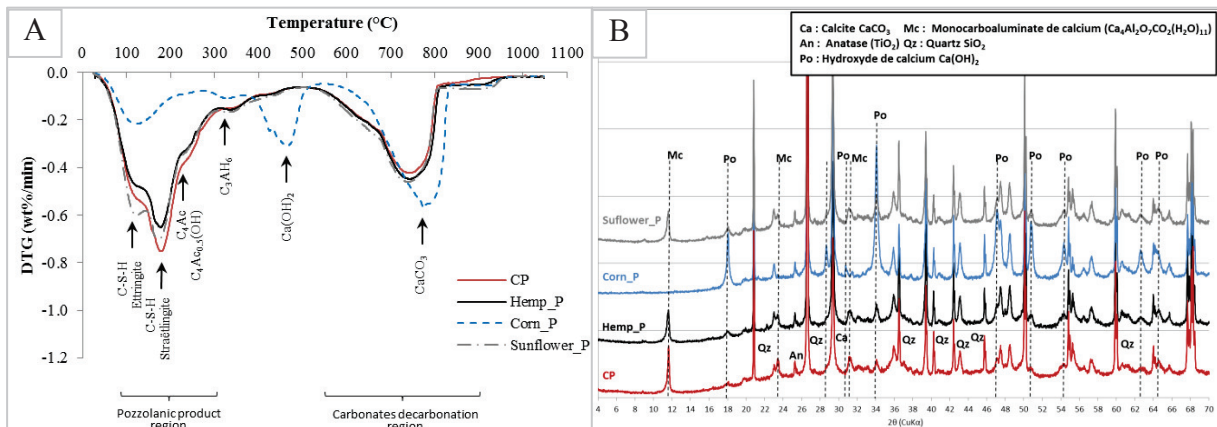


Figure 6: Differential TGA curves (A) and XRD analyses (B) of model pastes after 24h.

Extractives have a significant impact on the pozzolanic reaction development by reducing the amount of reaction products. The difference between the pastes based on hemp and sunflower extractives, and the control paste is mainly related to the peaks in a temperature range of 100-250°C. These peaks are attributed to the dehydration of C-S-H, ettringite and straetlingite.

The small peak at 250°C is attributed to calcium carboaluminates phases: calcium aluminium oxide carbonate hydrate,  $\text{Ca}_4\text{Al}_2\text{O}_6\text{CO}_3(\text{H}_2\text{O})_{11}$  (noted C<sub>4</sub>Ac) and calcium aluminium oxide hemi-carbonate hydroxide hydrate,  $(\text{Ca}_4\text{Al}_2\text{O}_6(\text{CO}_3)_{0.5}(\text{OH})(\text{H}_2\text{O}))_{11.5}$  (noted C<sub>4</sub>Ac<sub>0.5</sub>(OH)) [5]. No significant delay is observed in the consumption of portlandite (430°C) in these pastes. On the contrary, in the paste based on corn extractives, a large amount of residual portlandite is observed after 24 hours. A deficit in the formation of C-S-H and straelingite phases is also evidenced. These phenomena may be responsible for the decrease in mechanical performances. Finally, the increased presence of carbonates could be related to the release of carbon dioxide induced by the alkaline degradation of corn particles, which was responsible for the carbonation of portlandite [6]. These results are consistent with XRD analyses (Fig. 6B) that show similar crystallised compounds between control, hemp and sunflower pastes while corn paste is distinguished by the absence of calcium carboaluminates and the presence of portlandite.

#### 4. Conclusion

This study aims to evaluate the potential of bark particles of sunflower and corn as bio-aggregates for lightweight concrete. It has been shown that:

- Particle size distribution of sunflower and corn particles could be corrected through a calibration process, notably based on the elimination of the longer particles.
- Corn particles present a limited water absorption capacity. This could be valorized in association with a clay matrix for example. On the contrary, the deleterious effect of corn extractives on the setting and hardening mechanisms of the pozzolanic binder would largely reduce the mechanical performances of vegetal concrete designed with these two components.
- Sunflower bark particles appear to be a good candidate for hemp shives substitution since their characteristics are close and their impact on the setting mechanisms of the pozzolanic binder negligible.

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