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Comprehensive Characterization of Agricultural By-Products for Bio-Aggregate Based Concrete

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Abstract. The valorization of available agricultural by-products is important for the development of bio-aggregate based concretes as eco-friendly solutions for building materials. However, their diversity requires to assess their potential of use in vegetal concretes. This study aims to propose simple and relevant multi-physical characterization methods for plant aggregates. Basic and complementary characterizations were carried out on hemp shiv as a reference plant aggregate, and nine by-products available in the South-West part of France, *i.e.*, oleaginous flax shiv, sunflower pith and bark, coriander straw, wheat straw, wheat chaff, corn shuck, miscanthus stem and vine shoot. The basic characterizations performed were those recommended by the TC-RILEM 236 BBM, *i.e.*, particle size distribution, bulk density, water absorption and thermal conductivity. Complementary characterizations have also been proposed, taking into account the possible environment of the binder and the vegetal concrete manufacturing method. The additional tests developed or adapted from previous research assess the following properties: the content of water-soluble compounds at pH 7 and 12, the dry density of plant aggregates compacted in wet state, the real water absorption after compaction and the compression behavior of these compacted aggregates. This complete characterization highlights the distinct behavior of the different agrosources and allows to correlate these characteristics to the use properties of hardened composites.

1 Introduction

The building sector is the world leader in energy consumption (36%) and carbon dioxide emissions (39%). Therefore, a change in current construction practices must be undertaken in order to reduce its environmental impact. The use of local agricultural resources as a source of new building materials could be an answer to this problem since they are renewable, biodegradable and constitute a carbon sink. Over the past couple of decades in France, most of the research incorporating agricultural by-products with mineral binder to form insulating vegetal concretes has been carried out with hemp shiv. However, this by-product represents only less than 0.02% of the total crop by-products available in the metropolitan France, and it is almost absent in the South-West (Laborel-Préneron et al., 2018). Nine by-products available in this region were identified as alternative bio-aggregates for construction. However, their diversity requires adequate multi-physical characterizations in order to assess their potential as plant aggregates for vegetal concretes.

Currently, no standard governs the characterization of plant aggregates as constituent of building material. However, some characterization methods like the particle size distribution, bulk density, water absorption and thermal conductivity have been recommended by TC RILEM 236-BBM (Amziane et al., 2017). These methods have been implemented with hemp shiv. Nevertheless, these characterizations are not sufficient to qualify the aggregates for use with a mineral binder. Indeed, the literature has shown the existence of deleterious chemical interactions between the water-solubles inside plant aggregates and reactive mineral binders, thus causing a perturbation of the

setting at early age, the hardening mechanism which impacts the mechanical performance of the composite and its durability (Bourdot et al., 2019; Diquélou et al., 2015). In their studies so far, the amounts of water-soluble compounds have been determined in a neutral medium. However, most of the binders used like cement, lime, and pozzolanic binders generate an alkaline medium. Hence, it is necessary to determine the content of water-solubles extracted under alkaline conditions. In addition, the bulk density does not represent the behavior of aggregates, which have been wetted and then compacted in the composite. So, it is necessary to set up a characterization method making to get closer to the behavior of the aggregates in the composite by wetting and compacting. It could also give the real water demand of each aggregate inside the formulation, due to the water need competition with the hydration of the binder affecting the mechanical performance of the composite (Arnaud and Gourlay, 2012). Few authors have studied the mechanical behavior of plant aggregates without binders (Arnaud and Gourlay, 2012; Cérézo, 2005; Tronet et al., 2014). Cérézo (2005) concluded that for a hemp concrete with a low binder dosage, the mechanical compression behavior of the composite is influenced by the highly compressible behavior of hemp particles alone. The behavior of the composite could then be predicted by a compression test on samples of aggregates alone.

In the present study, relevant multi-physical characterization methods are applied on ten plant aggregates in order to compare their various behaviors, which could influence the use properties of the composites and serve as parameters for the manufacture of the latter. This conference article only deals with the characterization of aggregates, but the correlation with the mechanical and thermal properties of composites has been realized and will be the subject of a journal article.

2 Materials and Methods

2.1 Materials

Ten agricultural by-products were chosen for this study. Apart from hemp shiv, which was chosen as the reference plant aggregate, the other by-products were chosen for their availability in the South-West part of France and low cost, and they were subjected to the same transformation process in order to obtain the studied plant aggregates. This process was a series of grinding, sieving and mechanical separation. After a drying step, by-products were ground using a hammer mill fitted with a 32 mm diameter grid. Then, the ground by-products were sieved through a 1 mm sieve to remove fine particles. In the case of sunflower stem, a tilted conveyor belt and a blowing system were used to separate the pith particles (soft internal part of the stem) from the bark one (rigid and ligneous external part). Finally, they were screened on a 12 mm square mesh screen to obtain the plant aggregates used in this study (Fig. 1). According to their origin, the ten plant aggregates can be classified into four categories:

- Fiber plant by-products: hemp shiv (HS) and flax shiv (FS);
- Oilseed stems or straw: sunflower pith (SP), sunflower bark (SB) and coriander straw (CSt);
- Cereal plant by-products: wheat straw (WS), wheat chaff (WC) and corn shuck (CSh);
- Other by-products: miscanthus stem (MS) and vine shoot (VS);



Fig. 1: Plant aggregates: (a) HS, (b) FS, (c) SP, (d) SB, (e) CSt, (f) WS, (g) WC, (h) CSh, (i) MS, (j) VS

2.2 Basic physical characterizations

The basic physical characterizations were carried out according to the recommendations of the RILEM TC 236-BBM (Amziane et al., 2017).

2.3 Content of water-soluble compounds at pH 7 and 12

The test was conducted with a Foss Fibertec FT 122 hot extractor on aggregate powders according to the method of Van Soest and Wine (1968), using a neutral solution of distilled water (pH 7), and a sodium hydroxide solution having a pH value equal to 12. The mass loss of the analyzed sample was measured after one hour in the boiling solution.

2.4 Dry density of aggregates compacted in wet state, and real water absorption after compaction

The main steps of the protocol to determine these properties are shown in Fig. 2. First, the aggregates were immersed in water for 30 sec, drained by shaking 50 times, and mixed manually to ensure good homogeneity (Fig. 2, steps 1 and 2). Then, as for the manufacture of a concrete specimen with immediate demolding, the wet aggregates were introduced and compacted in three layers of equal thickness in a cubic mold ($15 \times 15 \times 15 \text{ cm}^3$) (Fig. 2, step 3). The load applied for compaction was adjusted until the sample became cohesive after immediate demolding. The sample was then demolded and dried in an oven at 60°C until the weight was constant (weight variation of less than 0.1% between two weighings, 24 h apart) (Fig. 2, step 4). For each plant aggregates, three samples were manufactured. The dry density of aggregates compacted in wet state, ρ_{comp} ($\text{kg}\cdot\text{m}^{-3}$), and the real water absorption after compaction, w_{real} (%), are calculated from equations (1) and (2), respectively.

$$\rho_{comp} = \frac{m_d}{V_d} \quad (1) \quad w_{real} = \frac{m_{wet} - m_d}{m_d} \cdot 100 \quad (2)$$

Where m_d and V_d are, respectively, the mass (kg) and volume (m^3) of the dry sample, and m_{wet} the mass of the wet compacted sample (kg).



Fig. 2: Experimental protocol to measure wet compacted dry density and real water absorption

2.5 Compressive strength and Young's modulus

The mechanical characterization was carried out by performing a monotonic compression test on the previously manufactured cubic samples of dry plant aggregates compacted in wet state without binder. The compression test was performed on a Zwick/Roell Z100 press equipped with two circular plates (13 cm in diameter) and a 5 kN force transducer. In order to properly distribute the forces on the surfaces of the analyzed sample, a rigid plywood was placed on the bottom plate and a thin rigid metal plate above the sample. The compression test was controlled in displacement with a speed of 3 mm/min up to 20% deformation. In order to compare the results on plant concretes with those in the literature, the compressive strength (σ) recorded was the stress at 1.5% deformation ($\epsilon=1.5\%$) (Bourdot et al., 2019; Nguyen et al., 2010). The Young's modulus (or elastic modulus) was calculated by the Hook's law on the linear part of the curve at its very beginning (*i.e.*, between 0% and 0.5% deformation).

3 Results and Discussion

3.1 Physical properties

Circularity is a shape parameter determined from image analysis, and its value ranges from 0 (infinitely elongated) to 1 (perfect sphere). The results of this parameter on the studied plant aggregates allowed to classify them into three categories (Fig. 3). The first type, which includes CSt, WS, WC, CSh and VS, is characterized by low circularity, of which more than 60% of the projected areas of the aggregates have circularities between 0 and 0.25. The second type, containing HS, FS, SB and MS, has an average circularity with more than 60% of the projected areas having circularities between 0.20 to 0.50. The last type, formed by SP only, is the most circular with more than 60% of the projected areas having circularities between 0.40 and 0.65. Although shape and size are parameters that can be changed from the transformation process, these results show the tendency of straws, chaffs, husks and shoots to be more elongated than the shiv, bark and stem, which are moderately elongated. For their part, the pith particles are more circular.

In addition to the shape, the size of the aggregates is also important especially for the mechanical properties of the composite. Most of the authors have concluded that the mechanical performance of the composites is reduced when the size of the aggregates increases for the same type of aggregate, but no trend was clear between aggregates of different types (Stevulova et al., 2013). The evolution of the results of the mechanical parameters of each aggregate as a function of the major axis in Fig. 4 could provide more clarification on the effect of size on the mechanical properties of the composites. In general, it is observed that for aggregates having a comparable nature, the more the major axis increases, the more the compressive strength or the elastic modulus of the aggregates decreases. In fact, CSh, WC and WS are formed by flexible particles that are easily compressible, and the decreasing tendency is observed between them. It is the same for the family composed of HS, FS, SB, VS, MS and CSt, which are formed by more consistent particles. Between these two families of aggregates, for the same size of aggregate, the mechanical performance of the aggregates of the first family is always lower than those of the second one. The size of the aggregates linked to their nature could then be correlated with the mechanical properties of the composite. The high mechanical performance of sunflower pith is due to the binding properties of the pectins released on the surface of the aggregates in their wet state, resulting in cohesion between the particles in their dry state.

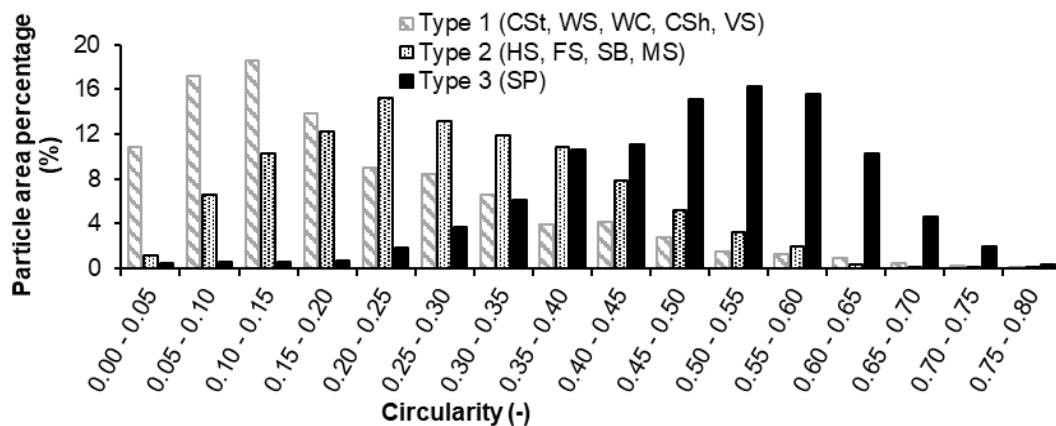


Fig. 3: Circularity of types 1 to 3 plant aggregates

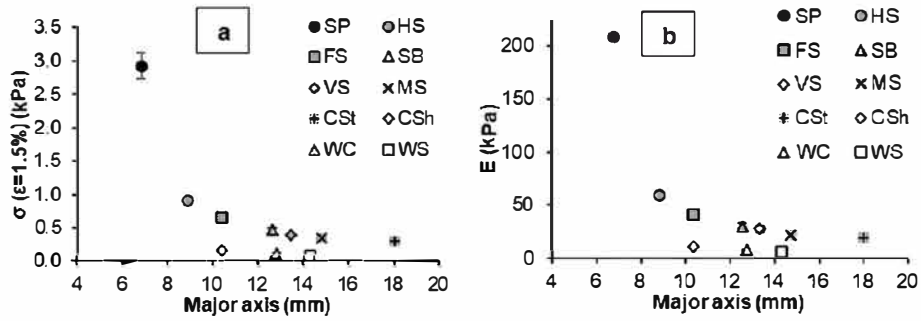


Fig. 4: (a) Compressive strength, and (b) Young's modulus of plant aggregates versus their major axis

The comparison between the bulk density and the density of dried plant aggregates compacted in wet state allows to observe two distinct behaviors of aggregates subjected to compaction in the wet state and then dried (Fig. 5a). The first behavior, for VS, SB, FS, HS and CSt, is a swelling of the aggregates resulting in a dry density of plant aggregates compacted in wet state much lower compared to the bulk density. In fact, wet aggregates swell so that the amount of particles that can be introduced into the mold is less than a loose aggregate, despite the compaction. The second behavior is that of CSh, WS and SP, whose wet compacted dry density is higher than the bulk density. This can be explained by rearrangement and flattening of porous aggregates, due to their compaction in the wet state. When the density remains the same in bulk and under compaction, as in the case of MS and WC, a neutralization between the swelling of the aggregates and their rearrangement occurs. These results show the variability of the volume behavior of the aggregates between the bulk arrangement and the dried particles after having been compacted in wet state, which could be linked to the porosity, rigidity and water absorption of the aggregates. Given that the density of dried aggregates compacted in wet state clearly reflects their behavior as in the manufacture of the composite, it would be a good formulation parameter in order to determine the adequate quantities of aggregates according to its behavior.

The result of the thermal conductivity of the aggregates as a function of the density (Fig. 5b) shows a linear correlation between these two parameters regardless of the nature of the aggregates. The low thermal conductivity of aggregates (all less than $0.080 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$) also shows their performance as bulk thermal insulators. The thermal conductivity of the order of $0.033 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ for SP is even lower than those of bulk insulators already in the market, *i.e.*, cellulose wadding or wood wool, which are in the $0.038\text{-}0.045 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ range.

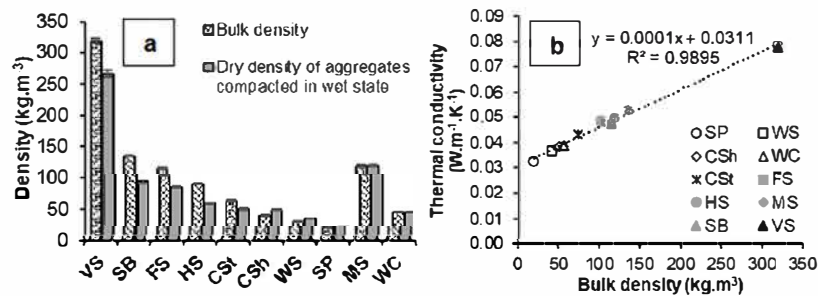


Fig. 5: (a) Comparison of bulk density and dry density of plant aggregates compacted in wet state, and (b) plant aggregates thermal conductivity as a function of their bulk density

Usually, to assess the absorption capacity of aggregates, the IRA (Initial Rate of Absorption), which corresponds to the absorption rate at 1 min, is used. With the determination of the real water absorption after compaction, it can be seen in Fig. 6a that the real quantities of water absorbed significantly differ from the tendency of IRA, and they are much greater for all aggregates. This parameter then allows to adjust the quantity of water necessary for the manufacture of the composites. However, the representation in mass percentage is not relevant for the comparison of the aggregates

in the composite given that they do not have the same mass dosages. A representation of the amount of water per volume of dried aggregates compacted in wet state, more closely reflects the amount of water of the aggregates inside the composite (Fig. 6b). Indeed, if the difference in water absorptions is considerable between SP and the other aggregates for a mass representation, a volume one shows a clear reduction of this difference. The order of the aggregates has also changed, due to the volume variation.

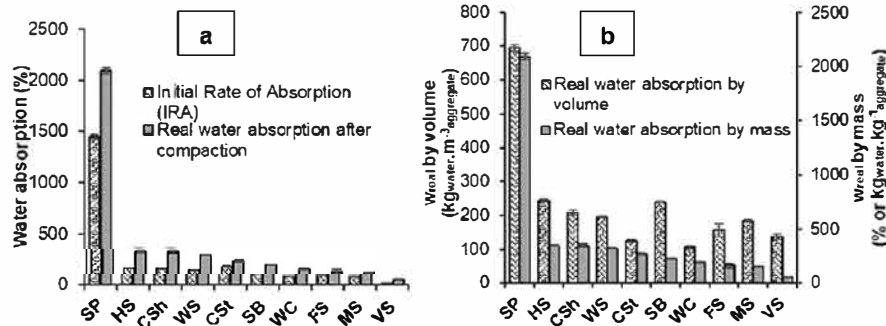


Fig. 6: (a) Comparison of IRA and real water absorption, and (b) volume and mass representations of real water absorption (i.e., mass of water absorbed in proportion of the aggregate volume and mass, respectively)

3.2 Chemical properties

The results of the water-solubles inside the aggregates at pH 7 and 12 show a large difference with an increase ranging from 33% for VS up to 126% for HS (Fig. 7a). This difference depends on the content of components soluble at pH 12 but insoluble at pH 7, in particular hemicelluloses. In addition, the impact of pH on the water soluble extracts also varies from one aggregate to another. Indeed, if the water-solubles of CSh (12%) and WC (16%) are respectively lower than those of WS (14%) and VS (17%) at pH 7, they become higher at pH 12 (CSh (23%), WC (25%), WS (19%) and VS (22%)). These results show that it is judicious to determine the water-solubles at pH 12 for a better understanding of their impact on the composites. As for water absorption, a volume representation of the water-solubles extracted per cubic meter of wet compacted dried aggregates shows more reality in the composite since the aggregates are not comparable in mass because of their different dosages (Fig. 7b). If for a mass representation SP seems to have the highest amount of water-solubles at pH 12, and therefore possibly a detrimental effect for the composite, a volume representation shows its low amount in water-solubles, which is of the same order of magnitude as HS. On the other hand, water-solubles in SV become considerable because of its high density. These results could be correlated with the mechanical properties of the composites.

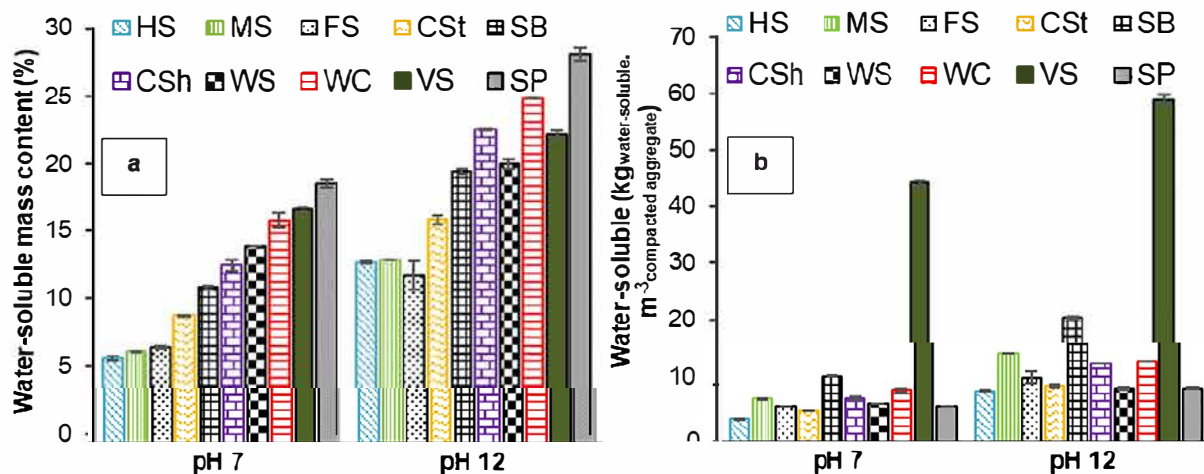


Fig. 7: (a) Mass and (b) volume representation of water-solubles in plant aggregates at pH 7 and 12

3.3 Mechanical properties

Compression of aggregates shows two types of behavior (Fig. 8a). The first one is characterized by the existence of a peak which can be explained by the achievement of a breaking stress maintaining the cohesion between the aggregates by entanglement (HS, VS and MS) or with the pectins (SP). The second behavior shows a stiffening of the samples (FS, SB, CSt, CSh, WC and WS). In fact, as the compressive stress increases, the aggregates rearrange and deform without breaking, reducing their inter- and intra-particle porosities. The low rigidity of the CSh, WC and WS families is also evidenced compared to the other aggregates. These behaviors of aggregates alone could greatly affect the mechanical behavior of their composites manufactured with a commercial formulated lime binder at constant dosage of 258 kg.m^{-3} , as it is shown in Fig. 8b. The cubic specimens ($15 \times 15 \times 15 \text{ cm}^3$) of the composites are tested in cyclic compression, and their floating Young's modulus was determined according to the method of Niyigena et al. (2015). For most particles, a clear correlation can be evidenced between the Young's modulus of the composite and that of the particles arrangement. Mixtures including vine shoots deviate from this trend, due to their high water-soluble content. Considering sunflower pith aggregates, as previously explained, the high degree of rigidity of the particles arrangement could be attributed to their binding by their own pectins.

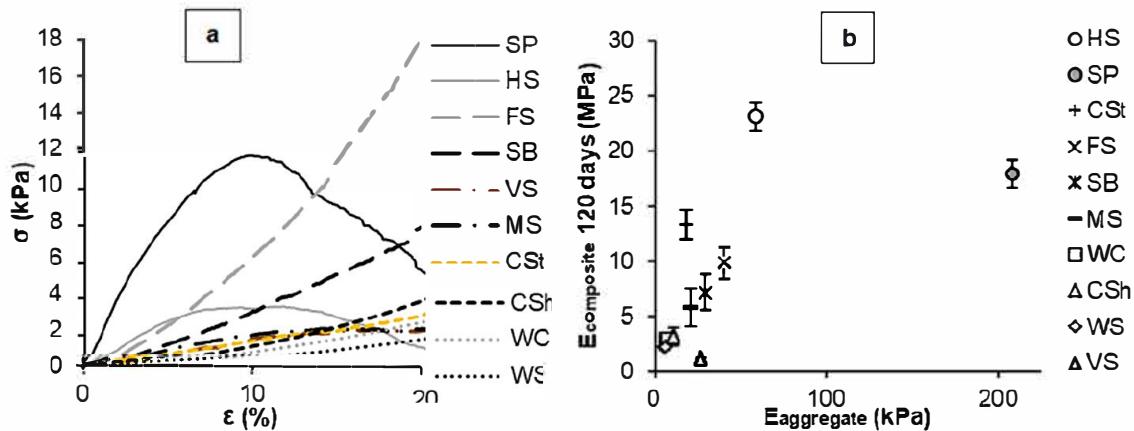


Fig. 8: (a) Compression behavior of dried plant aggregates compacted in wet state, and (b) Young's modulus of composites at 120 days versus Young's modulus of aggregates

4 Conclusion

In this study, multi-physical characterizations are proposed and applied on ten agricultural by-products in order to verify their relevance for an application with mineral binder. It was concluded that the size and form parameters, the dry density compacted in wet state, the real water absorption after compaction, the content in water-solubles at pH 12 and the mechanical parameters allow to compare the aggregates according to their nature, and to qualify them for their application in vegetal concretes. Nevertheless, the high porosities of the aggregates also require the development of another method to determine them. With all these parameters linked to the aggregates, future work would be to find a formulation method applicable to any type of aggregate, taking into account these properties.

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