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ORIGINAL PAPER

Characterization of Barley Straw, Hemp Shiv and Corn Cob as Resources for Bioaggregate Based Building Materials

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Abstract Expanding the use of low-environmental impact materials in the field of building materials is a major aim in a context of sustainable development. These alternative materials should be non-polluting, eventually recycled, and locally available. Bioresources are already used in some building materials but few studies have investigated their relevance in such applications. The aim of this paper is to evaluate the suitability of three kinds of vegetal aggregates: barley straw, hemp shiv and corn cob. The availability of these bioresources, extracted from a French database, is discussed, as are their physical properties and chemical compositions. Their microstructure is described with SEM images and their particle size distributions are provided through image analysis. Sorption–desorption isotherms are measured by a Dynamic Vapour Sorption system. Bulk density, thermal conductivity and water absorption are also quantified. The results highlight a tubular structure for the three different aggregates, with low bulk density and thermal conductivity (0.044, 0.051 and 0.096 W m^{-1} K^{-1} respectively for straw, hemp shiv and corn cob) and high water absorption, especially for barley straw and hemp shiv (414 and 380% vs. 123% for corn cob). Their hygric regulation capacity is also sufficiently good, with a water sorption of between 20 and 26% at 95% of relative humidity. These plant aggregates could therefore be used as additions in an earth matrix, or a hydraulic, pozzolanic, air lime or gypsum binder, or just as loose-fill insulation material. However, future research should focus on their resistance to fire and bacterial growth to validate this approach.

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Introduction

The impacts of buildings on environment, and especially on energy consumption and CO_2 emissions have now become priority issues. Energy use in buildings generates about 40% of the EU's total final energy consumption and 36% of its CO_2 emissions [1]. Innovation or changes are thus necessary to decrease buildings' environmental impact and improve their energy efficiency. Currently, in France, a huge proportion of non-renewable materials is used in the construction industry and large amounts of waste are produced (around 50 million tons per year, while municipal solid waste is around 30 million tons per year [2]).

On the way to a sustainable future, eco-friendly building materials could be part of the solution. These materials would allow consumption and pollution to be reduced during the production process and also during their whole service life and their end-of-life. In that context, bio-based building materials present the advantage of using plant resources that have absorbed CO_2 through photosynthesis and can thus reduce the material's environmental impact by sequestering CO_2 for at least the life-time of the construction [3]. Bio-sourced materials and the building sector have been identified by the French Ministry of Ecology, Sustainable Development and Energy (Commissioner-General for Sustainable Development) as one of the 18 "green" sectors with a high potential for economic development in the future. In order to produce these low carbon materials, renewable resources, such as by-products from agriculture or forests, are needed. An example that is being increasingly studied is a bio-based earth material. Unfired earth bricks are fibred with distinct renewable resources such as kenaf fibre [4], straw [5], wood chips [6] or wool fibre [7]. Plant concretes, which combine bioaggregates with a pozzolanic, lime-based or cement matrix, are also a good alternative. Numerous studies on hemp concrete currently exist [8–11] and there are also some concerning sunflower [12] or lavender [13] concrete. Although bio-resources are renewable, they also need to be cultivated at locations close to where they are implemented so as to avoid unnecessary transportation and its related environmental impacts. For that reason, the present paper focuses on estimating the potential availability of such resources in the case of France, where by-products and the availability of agricultural land are increasingly being studied, especially with a view to the use of bio-fuels [14–16]. However, these studies provide information about quantities of available, still-unused by-products which could be employed as building materials. A similar study has already been carried out by Palumbo et al. [17] for insulating materials. Their study focused on the main resources available in Europe and especially in Spain, i.e. cereals and sunflower. Biomass is more and more in demand. In the past, it was already used but population growth engendered an increase in food needs. Studies are now being conducted to avoid biomass usage conflicts between priority sectors and others. The former concern human consumption, including animal feed and litter, whereas the latter are related to industry (biomaterials, bio-based chemistry) and energy, which are considered as lower priority by France Agrimer [18]. This study will focus only on plant-sourced biomass.

Plant particle characteristics, which are very specific to the raw material, are studied and compared in the present paper. These characteristics have to be taken into account in the development and the characterization of further composite materials. Three plant aggregates were studied in this work because they were readily available and presented important morphological differences: barley straw, hemp shiv and corn cob. Straw is currently one of the most commonly studied plant aggregates, and is the subject of one-third of the 50 references reviewed by Laborel-Préneron et al. [19]. This resource, very common in the plant world, is also present in our cultural heritage [20, 21]. It can come from wheat [22–24], barley [22, 25], oats [26] or other cereals. Hemp shiv constitutes a resource that has received considerable attention in France, which is the greatest producer of hemp in Europe with more than 50% of the total European production [27]. This plant aggregate is especially studied for use in hemp concrete [28, 29] but also as a bio-composite material with earth [30, 31]. Corn cob is an original resource that has been studied only once with an earth matrix, but not crushed [32]. It was studied by Verdier et al. in a pozzolanic matrix [33], and corn pith, which is softer, was studied by Palumbo as an insulating material in an alginate matrix [34].

The use of bio-based aggregates in building materials is becoming increasingly widespread. It is worth noting that various plant aggregates are available in the world and could be used for building construction. However at the present time, no international standardized method exists for characterizing such materials, as it already exists for mineral aggregates. The new standard could define testing protocols for the characterization of bio-aggregates and also the restrictions applying to each application in building materials. This lack of recognized procedures led the RILEM BBM Technical Committee to work on recommendations concerning protocols for bio-aggregate characterization, mainly on hemp shiv [35].

Applying these recommendations to other plant aggregates is a way to check the validity of the method. The objective of this study is thus to characterize and observe the differences of three available agro-resources by following the RILEM recommendations in terms of bulk density, thermal conductivity, water absorption and particle size analysis. Complementary characteristics proposed by other authors are also studied: availability in France, microscopic description, chemical composition and sorption–desorption capacity. This whole methodology could allow the differences between the characteristics of these three plant particles to be highlighted and their potential for future applications in building materials to be assessed.

Materials and Methods

Materials

Barley Straw

Straw is an agricultural by-product and is the part of cereal stems rejected during the harvest. Barley is harvested once or twice a year. It is the third most cultivated cereal in France with 10 million tons per year [18]. The straw studied here (Fig. 1), already chopped, was supplied by the CalyClay company (Drôme, France), which is specialized in services for straw and earth construction.

Hemp Shiv

France was the first producer of hemp in 2013 [36]. Hemp shiv is the by-product of the hemp defibration process and corresponds to the lignin-rich part of the stem (Fig. 1). It was provided by the Agrofibre company in Cazères (Haute-Garonne, France).

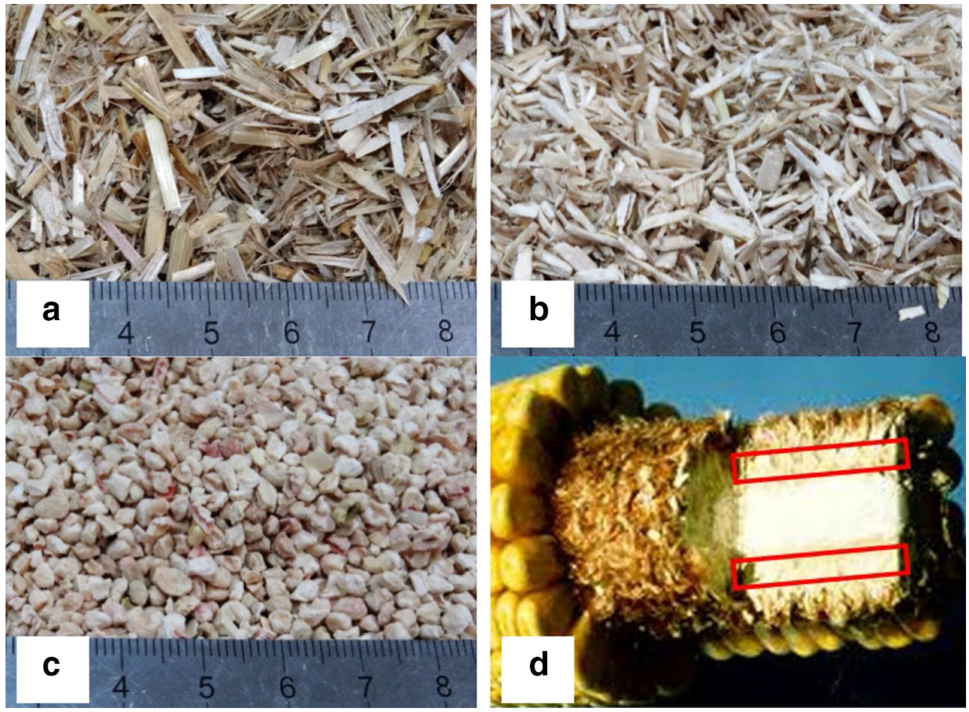

Fig. 1 Raw materials **a** Barley straw, **b** Hemp shiv, **c** Corn cob and **d** Part of corn studied (in *red*)

Corn Cob

Maize is the second most cultivated cereal in France, with around 15 million tons per year [18]. Corn cob is the central part of the ear of maize, cleared of grain and crushed. The "woody" part (in red in Fig. 1), which is also the hardest part, was studied here. This corn cob, already calibrated, was provided by the Eurocob company in Maubourguet (Hautes-Pyrénées, France).

Availability of Agricultural and Forestry By-Products

The availability of resources was estimated for the case of metropolitan France and specifically for biomass from agriculture, the agri-food sector, industrial crops and forestry residues. Most data providing both the yearly total and the available production of crop by-products come from a national authority, France Agrimer [18], which monitors products and biomass from agriculture and the sea. The data presented in this study are from 2015 and some values were completed by data from 2013 to 2014 in Agreste [37], provided by the French *Ministère de l'Agriculture, de l'Agroalimentaire et de la Forêt*. In these two references, data were collected from various economic bodies such as Ademe (*Agence de l'Environnement et de la Maîtrise de l'Energie*), IGN (*Institut National de l'Information Géographique et Forestière*) and the FAO (*Food and Agriculture Organization of the United Nations*). Nevertheless, the collected information should be interpreted cautiously, as a resource may not have been fully counted.

Some data, specifically for industrial crops, were expressed only as quantity produced, and were therefore converted into quantity available by applying the unused biomass factor found in Jölli and Giljum [38]. Furthermore, data corresponding to forestry biomass were given by volume per year (Mm^3/y). In order to be compared with the other resources, volume was converted into mass assuming a density of 0.88 t/m^3, which is an average for various wood species from an FCBA memento [39]. No recent data were available concerning fruit production, except for those for wine and cider production. Agri-food industry by-products will be thus slightly undervalued.

Twenty-four distinct resources were documented in these two references (France Agrimer and Agreste), such as soft wheat, sunflower and beetroot. It was decided to group them into 5 families: cereals, oilseed crops, industrial crops, agri-food industry residues and wood residues.

The method for determining the available by-products (Ba) was based on the following Eq. 1, greatly inspired by the work of Palumbo et al. [17]

$$Ba = Bt - Bn - Bu \quad (1)$$

where Bt is the total by-products (Mt/y), Bn is the non-harvestable by-products (Mt/y) (necessary to maintain soil fertility or inaccessible) and Bu corresponds to by-products allocated for other uses (Mt/y) such as litter, animal feed or energy (only in the case of wood). All quantities of by-products were expressed by mass (dry basis) over one year and they were determined from areas and farm yield. This equation is recapitulated by Fig. 2.

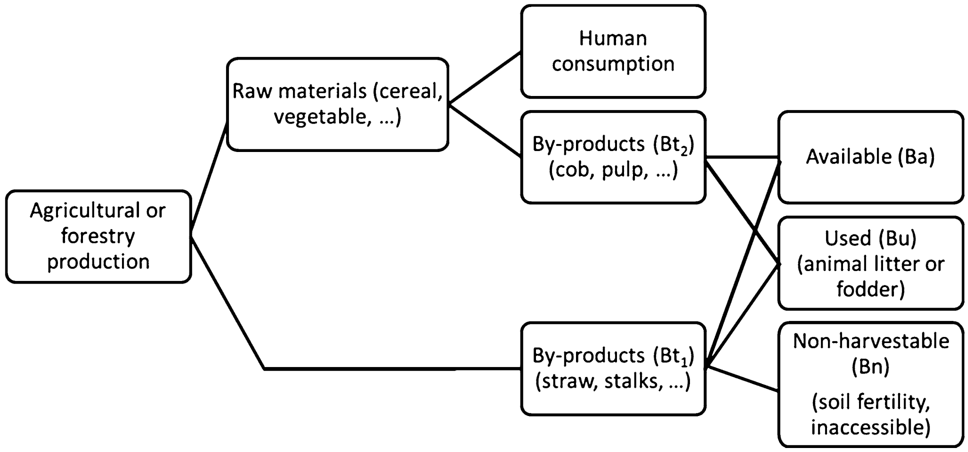

Fig. 2 Recapitulative scheme of utilization of by-products

For example, for oleaginous plants, it was considered that the minimum return to the soil to maintain agronomic potential was 50% of the straw produced [18].

Physical Properties of Plant Aggregates

Most of the physical plant aggregate characterization tests were based on a current work of the RILEM TC 236-BBM because there is no standardized method for this kind of material. To evaluate the validity of the results and analyse the dispersion of the results, these protocols recommend calculating a coefficient of variation. This coefficient corresponds to the ratio between the standard deviation and the mean value. The mean value is considered to be representative if the coefficient is lower than 5%. The methods are explained in the following sections.

Microscopic Description (SEM images)

Porous structure and morphology were analysed visually with a JEOL—JSM-6380 LV Scanning Electron Microscope (SEM). The particles were glued onto a metallic support and then covered with a thin layer of evaporated gold before observation. These microstructural investigations were carried out with a 15 kV accelerating voltage in high vacuum mode.

Particle Size Analysis

A particle size analysis was performed by image analysis using ImageJ software. This increasingly used method [40–42] is particularly interesting in the case of non-spherical particles. Size distribution and morphology (width and length) were determined using this method whereas traditional mechanical sieving analysis would have given only the width. However, this method is only achievable for small quantities of particles.

First, the particles were sieved at 500 µm to remove dust. The plant aggregates were then homogenized before being distributed into small boxes. The particles were scanned on a black background in order to obtain better contrast for the ImageJ analysis (Fig. 3). All the particles were then grouped into a single table to plot representative curves. In total, more than 7000 particles were analysed for each type of plant aggregate which corresponds to a mass of 6.7 g of straw, 13.6 g of hemp shiv or 71.0 g of corn cob. This is more than the mass recommended by the RILEM TC 236-BBM, of between 3 and 6 g, or the minimum number of

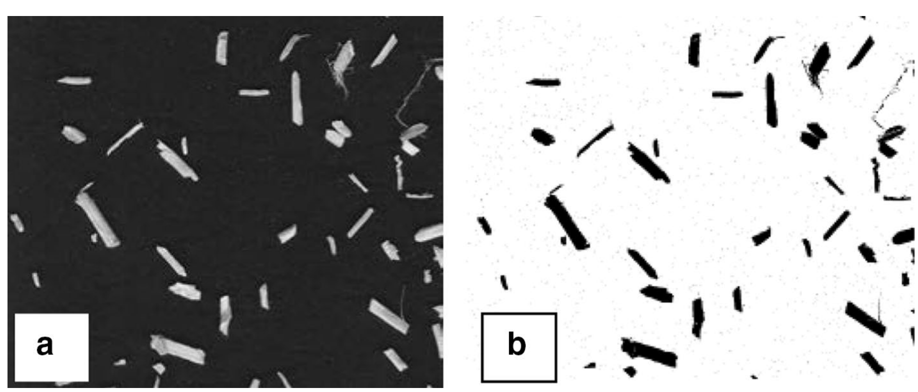

Fig. 3 Particle image processing of straw: **a** Scan in shades of grey, **b** Image processing

1000 particles suggested by [43]. Sampling quality is a key point for the representativeness of the results.

This analysis gave the geometrical parameters of the particles: the major and minor axis lengths (Major and Minor respectively), and the Equivalent Area Diameter (EAD), based on a particle of circular cross section and calculated with Eq. 2:

$$EAD = \sqrt{\frac{4 * A}{\pi}} \quad (2)$$

with A the cross sectional area of the particle (m²). It also gave the aspect ratio (AR) which is the length to width ratio, or Major to Minor ratio. If the value is close to 1, the particle is almost circular or square; the more AR differs from 1, the more elongated is the particle.

Bulk Density

Three specimens of each plant aggregate were dried at 60 °C until the weight became constant (weight variation of less than 0.1% between two weighings 24 h apart). Each specimen was put in a cylindrical mould 12 cm in diameter and 24 cm high. The mould dimensions were chosen in accordance with the RILEM work. The RILEM TC observed that the mould size used to measure bulk density had very little effect on the density as long as the height was at least twice the diameter and the diameter was at least 10 cm (big enough compared to the particle size). The quantity of material was selected by a quartering procedure and adjusted to be half the volume of the mould. The mould was then upended 10 times before the final level was marked with a cardboard disk. The volume occupied by the particles was measured from the weight of the corresponding volume of water and the bulk density (ρ_b, kg m^{-3}) was calculated using Eq. 3:

$$\rho_b = \frac{m_d}{m_w} \times \rho_w \quad (3)$$

where m_d is the mass of dry particles (kg), m_w is the mass of water (kg) and ρ_w is the density of water (1000 kg m^{-3}).

The bulk density of a given type of plant aggregate was taken as the average value measured on three samples.

Thermal Conductivity

Three specimens of each plant aggregate were dried at 60 °C until the weight became constant (weight variation less than 0.1% between two weighings 24 h apart). The particles were put in a PVC box of dimensions 15×15×5 cm^3, thermal conductivity 0.11934 W m^{-1} K^{-1} and thickness 1.6 mm. According to the recommendations of the RILEM TC 236-BBM, the dry density of the sample for thermal conductivity measurement was checked and adjusted (by shaking) to the same value measured during the bulk density test. For this last test, the procedure recommends upending the cylindrical mold ten times. The measurements were made with a hot plate apparatus (λ-meter EP 500) and were performed at 25 °C. The specimen was located between the two plates of the apparatus, one hot and the other one cold, with a temperature difference of ΔT = 10 K. A steady state was assumed to have been reached when the change in conductivity was less than 1% in 60 min. The apparent thermal conductivity of the plant aggregates within the PVC box (λ_{app} in W m^{-1} K^{-1}) was calculated at the steady state with the following equation:

$$\lambda_{\text{app}} = \frac{Q.e_t}{\Delta T.S} \quad (4)$$

where Q is the heat input (W), e_t the total thickness (m) and S the cross section of the specimen (m²). Knowing the dimensions and thermal conductivity of the PVC box, the thermal conductivities of the plant aggregates were then deduced. A transfer by conductivity through the plant aggregates and the box was assumed.

The specimens were weighed at the end of the test to measure the water uptake during the measurement.

Water Absorption

Water absorption during, 15 min, 4 and 48 h was measured on 3 specimens of each of the 3 plant aggregates. Each sample was dried at 60 °C until the weight became constant (weight variation less than 0.1% between two weighings 24h apart). Specimens were put into permeable nets for which the water absorption was negligible. Their mass, of 25 g for straw, 20 g for hemp shiv and 50 g for corn cob, depended on the available volume of the nets. It is assumed that representativeness was ensured by careful mixing and quartering the particles. After water immersion, the samples were drained for 1 min in their nets with a salad spinner and then weighed to determine their water absorption (%) according to the following equation:

$$w(t) = \frac{m(t) - m_0}{m_0} \times 100 \quad (5)$$

where m(t) is the wet mass after spinning (kg) and m_0 is the dry mass (kg).

Chemical Characterization

Before the tests, samples were crushed to a grain size of less than 1 mm and dried at 105 °C for at least 12 h. The main chemical compounds were measured by the Eurofins company using the Van Soest method, according to standard NF V18-122 [44]. This test provides 3 results: NDF (Neutral Detergent Fibre), corresponding to the total fibre;

ADF (Acid Detergent Fibre), which contains mainly cellulose and lignin; and ADL (Acid Detergent Lignin), corresponding to the lignin. Measurement uncertainties were 10% for NDF and ADF and 15% for ADL. Cellulose and hemicellulose were thus calculated by the subtractions ADF-ADL and NDF-ADF, respectively.

The following two tests were carried out in triplicate in the laboratory. To determine the proportion of water-soluble components, around 1 g of dried material (m_i, kg) and 100 mL of distilled water were introduced into a flask and boiled for 1 h in a heating system with return flow. The mixture was then separated by filtration on a sintered-glass filter. The flask and the filter containing the sample were dried at 105 °C and weighed to obtain the mass of aggregates after boiling (m_f, kg) by deducting the tare weights t_1 (flask) and t_2 (filter). The water-soluble content (WS) was determined according to Eq. 6:

$$WS\ (\%) = \frac{m_i - m_f}{m_i} \times 100 \tag{6}$$

To determine the mineral matter content, around 1 g of dried material (m_i) was heated at 550 °C for 7 h in a crucible of tare weight t. After cooling in a desiccator, the ash was weighed in the crucible (m_f). Mineral matter content (MM) was calculated with Eq. 7:

$$MM\ (\%) = \frac{m_f - t}{m_i} \times 100 \tag{7}$$

The coefficients of variation were between 10 and 18% and between 2 and 8% for extractive and ash contents respectively.

Sorption–Desorption Isotherms

The sorption–desorption property is necessary to model the buffering effect of a material and has great influence on its resistance to the proliferation of micro-organisms [34]. The sorption–desorption isotherms of the 3 plant aggregates were evaluated by the Dynamic Vapour Sorption (DVS) method. Temperature and relative humidity were regulated by the device (Surface Measurement Systems, London, UK). The uncertainties of the device are ±0.01 mg for the micro-balance, ± 0.2 °C for the PT100 thermometer and ±0.5% for the dew point sensor. The specimen, suspended by a micro-balance, was weighed every 60 s. Two specimens of each plant aggregate were tested and were assumed to be representative of these very hygroscopic materials [45–47]. The mass of the samples tested was very low, between 13 and 65 mg, due to the volume available in the sample holder. The samples were composed of 10 to 20 plant particles. However, Bui et al. [47] have shown that a sample of 20 mg of cut straw shows quite a good representativeness on DVS measurements. Before testing, the specimen was dried for 2 h at 50 °C (using dry N_2 gas) in the DVS device. The test was carried out at 23 °C, which is the temperature specified in the standard for the saturated salt solution method, NF EN ISO 12571 [48]. Relative humidity was regulated in successive stages from 0 to 95% by steps of 10%, except for the last stage, which was equal to 5%. For each step, the specimen was considered to have reached moisture balance if $dm/dt < 5.10^{-5}$ % min^{-1} over a ten-minute period [49] or in a maximum time interval of 360 min (twice this time for the last three steps). Figure 4 shows an example of straw sorption–desorption behaviour with this programme. It can be seen that, at high relative humidity, the change of step was triggered by the time criterion, which resulted in a slight underestimation of the moisture content for the last three steps.

Results and Discussion

Availability of By-Products

Figure 5 represents the quantities of by-product produced (Bt) and available (Ba) for the 5 families mentioned in the Material and Methods section. Production is greatest for the cereal straw family with 85 Mt/y, while the most available by-product is wood residue with 33 Mt/y. Nevertheless, the availability of cereal straw is 7 Mt/y.

The families of the three plant particles of the present study are framed in orange: barley straw, hemp shiv and corn cob, belonging respectively to cereal straws, industrial crops and agri-food industry by-products.

The quantities of by-products produced are represented in Fig. 6 for the 3 families (agricultural crops, agri-food industry and industrial crops). Production is presented rather than availability, because available resources are not reported by France Agrimer [18] or others for the agri-food industry and industrial by-products. The majority of agri-food industry by-products are used for animal fodder.

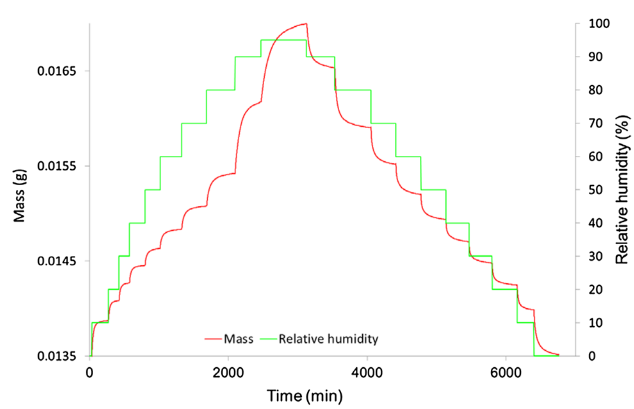

Fig. 4 Typical variation of mass and relative humidity for the DVS (here sorption–desorption of barley straw)

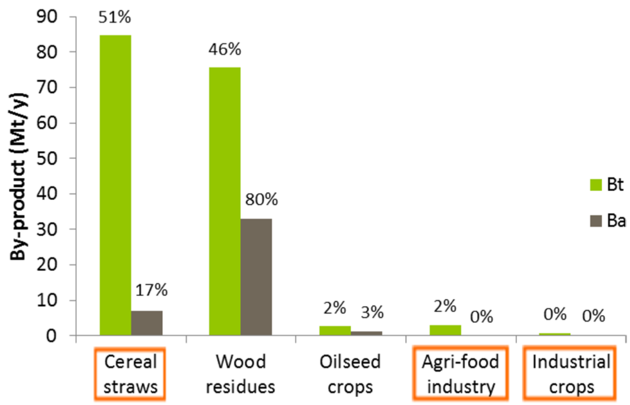

Fig. 5 Production and availability of different by-product families

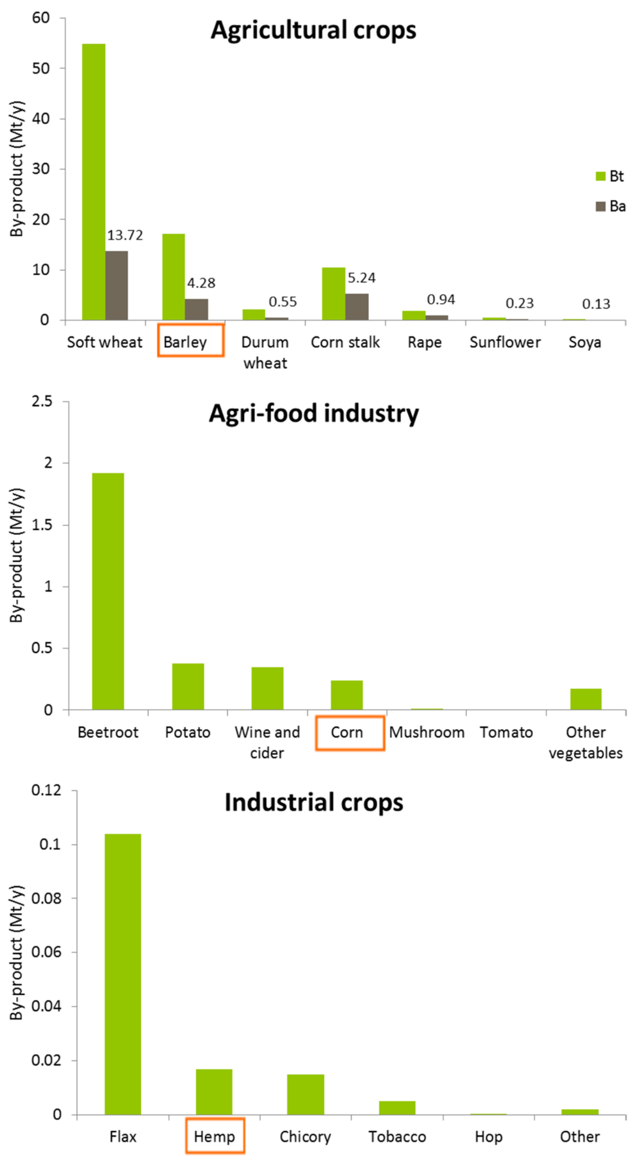

Fig. 6 Quantities of by-products from agricultural crops, agri-food industry and industrial crops

Cereal straw and stalks and oleaginous crops are included in agricultural crops. Barley straw makes up the second highest quantity produced in this category and the third most available crop by-product, with 4.3 Mt/y, after soft wheat straw and maize stalk. Much more cereal straw is produced than oleaginous straw (rape, sunflower and soya).

Corn cob is a by-product of the agri-food industry. It is included in corn by-product, along with leaf and damaged grain. The quantity of corn by-product produced is around 0.24 Mt/y, which makes it the fourth category produced in this family.

Hemp is the second most-produced industrial crop by-product after flax, with around 17,000 tons. This value is much lower than those of the other plant aggregates selected, but it is a plant by-product that has already been the subject of a number of studies for building materials. France is the biggest European hemp producer [27], and some professional rules exist [50]. This has led to the development of construction or rehabilitation of buildings with hemp concrete in the past 10 years.

In France, various types of by-products are available, in different proportions. Cereal straws are the greatest resource available. It is thus interesting to study barley straw in order to develop new bio-composites promoting this kind of resource. Maize and hemp do not provide the greatest quantities of by-products of their respective families. However, they both have huge potential: maize is the cereal most produced in the world [36] and France is the biggest hemp producer in Europe [27]. Forthcoming studies should focus on bioresources with widespread availability, such as wood residues, which represent 80% of the by-products available; flax, which yields around five times as much by-product as hemp; or beetroot residue, which constitutes the largest by-product by weight from agro-industry.

Physical Properties

Microscopic Description

The morphology and porous structure of the plant particles are illustrated by the SEM images of Fig. 7.

These SEM images clearly show a tubular microstructure for the three materials. However, some differences can be observed. The pores of the straw, from 2 to 100 µm, are multi-scale and the cell walls are very thin (maximum thickness of 2 µm). Hemp shiv pores range from 5 to 40 µm and the particles present regular cell walls 4 µm thick on average. Concerning corn cob, the diameter of the pores is between 20 and 80 µm with a thick cell wall of up to 45 µm. The fact that the parenchyma is thicker for corn cob than for hemp shiv or straw indicates a lower macro-porosity and thus a higher density of the plant particle. According to Pinto et al. [51], this

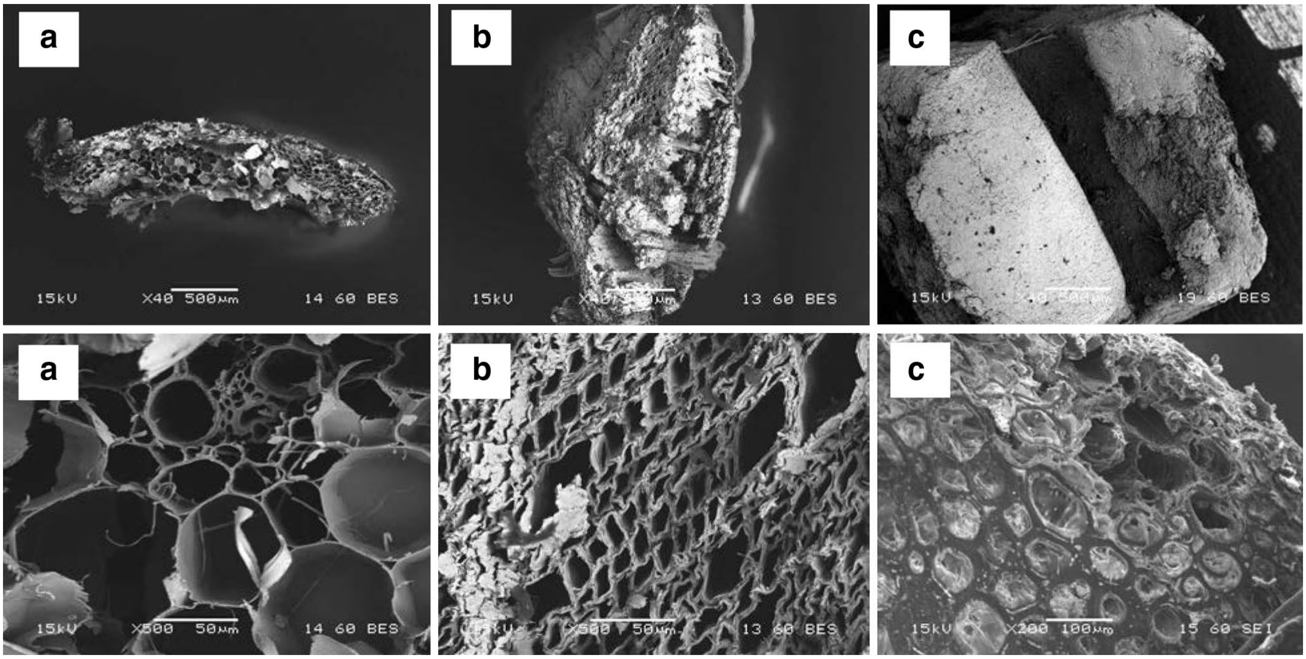

Fig. 7 SEM images of: **a** straw, **b** hemp shiv and **c** corn cob at low (first *line*) and high (second *line*) magnification factors

could lead to a strength capacity comparable to that of soft woods.

Particle size Analysis

The particle size distribution of plant aggregates seems to have an influence on the mechanical performance of bio-based building materials. For example, Millogo et al. have shown that the compressive strength of an earth-based composite with short pieces of straw (3 cm) is higher than that of similar composite with longer pieces (6 cm) [4]. Conversely, in the case of hemp concrete, it has been shown that the coarser the hemp shiv is, the higher is the mechanical performance at 28 days [11, 52]. Danso et al. [53] have studied the effect of fibre aspect ratio on mechanical strength in an earth matrix. Compressive and tensile strengths improved for coconut, oil palm and bagasse fibres when the major axes increased.

In this study, the morphological characteristics of straw, hemp shiv and corn cob were compared. Dust content, determined by sieving at 500 µm, was about 7.2, 2.1 and 0.3% respectively for the straw, hemp shiv and corn cob. The grading curves are presented in Fig. 8. Figure 10 Averages and standard deviations of the major and minor axes, EAD or aspect ratio were calculated from equations 8 and 9 [40] and are presented in Table 1.

$$E_{am}(x) = \frac{\sum A_i(x_i)}{\sum A_i} \tag{8}$$

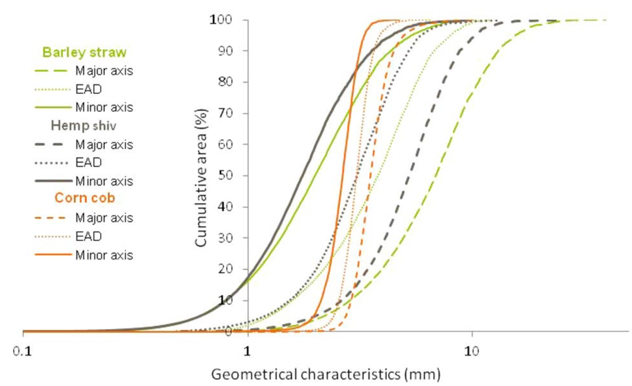

Fig. 8 Grading curves of the plant aggregates as a function of the cumulative area

Table 1 Arithmetic means and dispersions of plant aggregate dimensions

Dimension	Barley straw	Hemp shiv	Corn cob
Major (mm)	7.6 ± 4.4	5.6 ± 2.6	3.6 ± 0.8
Minor (mm)	2.3 ± 1.5	2.0 ± 1.2	2.6 ± 0.4
EAD (mm)	4.0 ± 2.1	3.3 ± 1.6	3.1 ± 0.5
AR	4.1 ± 3.7	3.3 ± 2.0	1.4 ± 0.4

$$Sd_{am}(x) = \sqrt{\frac{\sum A_i (x_i - E_{am}(x))^2}{\sum A_i}} \tag{9}$$

where $E_{am}(x)$ is the arithmetic mean of the dimension x (Major, Minor, EAD or AR), A_i is the projected area of each particle detected (mm²), x_i is the dimension of each particle detected and $Sd_{am}(x)$ is the associated standard deviation

The standard deviations associated with the mean dimensions of barley straw and hemp shiv are higher than for the corn cob. This is due to the marked heterogeneity of these particles, which are obtained by mechanical grinding without any specific calibration process. In contrast, the standard deviation associated with the mean values of the corn cob morphological parameters is quite low since this aggregate is made up of a single granular fraction obtained after an industrial process of calibration

It can be observed in Fig. 8 that the corn cob spindle (between the major and minor axes) is the smallest and the steepest. This means that the major and minor axes are of similar length and the size of the particles is fairly regular. This result is understandable because corn cob is a calibrated material (by the Eurocob company). Grading curves are quite similar between straw and hemp shiv. The size of straw particles is just slightly larger than the size of hemp shiv particles.

Proportions of AR in cumulative area are represented in Fig. 9.

It can be noted that the AR of corn cob is very close to 1, meaning that corn cob particles present a nearly circular cross section. Straw and hemp shiv are more elongated (especially straw) with more variation among particles.

In Fig. 10, the reciprocal of the AR is represented as a function of the average minor axis, in order to be compared with some values found in the literature. The minor axis of this study was considered to be equivalent to the diameter of the literature. These values from the literature, quoted in a review by Laborel-Préneron et al. [19], were classified by type of particle: fibres, aggregates and straw. Their AR was lower than that of the aggregates studied here, although their minor axes were of the same order of magnitude as in the particles of this study. For the straw, this can be

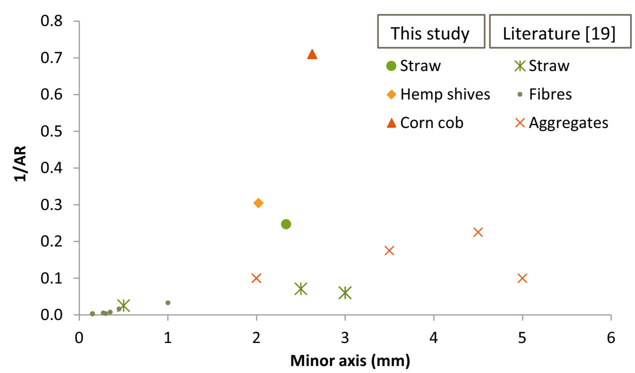

Fig. 10 Reciprocal of the aspect ratio as a function of average minor axis

Fig. 9 Relative area distribution as a function of aspect ratio

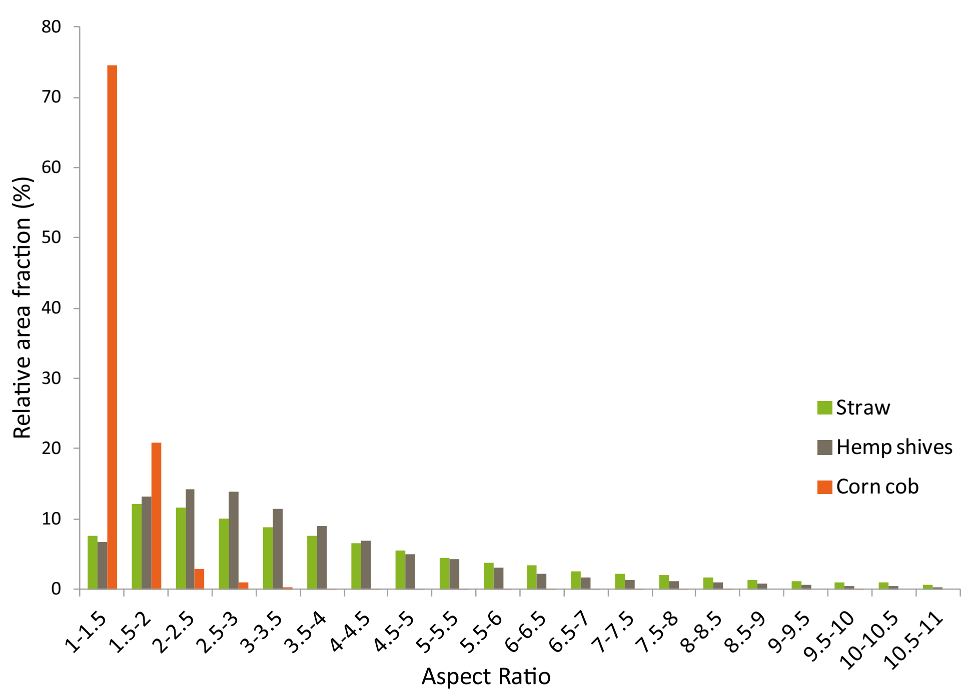

Table 2 Bulk densities (kg m^{-3}) of the plant aggregates studied

Plant aggregate	Barley straw	Hemp shiv	Corn cob
This study	57 ± 1	153 ± 2	497 ± 14
Literature	47 [55] 116 [56]	110.9 [9] 148.3 [57] 130 [29]	450 [58] 495 [57]

Table 3 Thermal conductivity (W m^{-1} K^{-1}) of the plant aggregates studied

Plant aggregate	Barley straw	Hemp shiv	Corn cob
This study	0.044 ± 0.001	0.051 ± 0.002	0.096 ± 0.001
Literature	0.035–0.054 [22]	0.058 [9] 0.055 [28] 0.057 [42]	0.099 [57]

explained by the shortness of our particles even though the diameter was similar to those in the literature. Hemp shiv was more crushed and finer than in the literature and corn cob was almost spherical, which is not very common for plant aggregates.

Optimizing the particle size distribution could be an interesting lever to improve the compactness of the particle arrangement of bio-based building materials, as is common for basic concretes. The selection of specific particle fractions or the combination of particles with different morphologies could lead to an enhancement of their mechanical performances.

Bulk Density and Thermal Conductivity

Using insulating materials in buildings is a way to save energy. Adding some vegetal particles into existing building materials such as earth or concrete is a means of reducing their density and thus improving their thermal insulation [6, 54]. Such particles can also be applied in buildings as loose-fill insulation. Bulk density (Table 2) and thermal conductivity (Table 3) are presented together, because thermal conductivity is highly dependent on the density [19].

Table2 presents the average bulk density of each plant aggregate with the standard deviation and some values from the literature. The values from this study are close to the values from literature, except for one value for barley straw. This difference may be due the length of the straw or the method used to measure it. Barley straw has the lowest bulk density, followed by hemp shiv and then corn cob, which has the highest. The coefficients of variation are 2.0, 1.6 and 2.8% respectively for straw, hemp shiv and corn cob. This value is lower than 5% which suggests good representativeness of the different samples.

Table 3 shows that the thermal conductivity values measured were similar to those found in the literature. The coefficients of variation are 1.4, 3.9 and 1.1% respectively for straw, hemp shiv and corn cob. This value is lower than 5% which suggests good representativeness of the different samples. Moreover, the water uptake during the test was lower than 0.2% for each specimen. The moisture content, influencing thermal conductivity, can thus be neglected.

Figure 11 compares the thermal conductivity of various plant aggregates from this study and the literature in function of their bulk densities. The relation plotted is linear: the lower the bulk density is, the lower is the thermal conductivity. Thermal conductivity depends mostly on the bulk density. However, there are small variations even within a given particle type. They can be explained by the difference in moisture content or in the testing method. The type of vegetal does not seem to influence the thermal conductivity significantly, but more results would be needed to confirm that hypothesis.

The barley straw and hemp shiv characterized in this study present bulk densities and thermal conductivities similar to those of loose-fill insulation materials such as cellulose wadding or glass fibre (quoted in Goodhew and Griffiths [59]). Maskell et al. [60] characterized other natural loose-fill insulation materials: wool, hemp fibre and wood fibre.

Such lightweight aggregates could be used in great quantities in an earth matrix as was done by Labat et al. [5] with an earth clay material. The density was 241 kg m^{-3} for a thermal conductivity of 0.071 W m^{-1} K^{-1}, much lower than for a traditional earth material, for which the corresponding values are between 1940 and 2007 kg m^{-3} and 0.47 and 0.59 W m^{-1} K^{-1} [49]. As far as hemp concrete is concerned, it presents a thermal conductivity between 0.06 and 0.19 W m^{-1} K^{-1} for a dry density between 200 and 840 kg m^{-3} [29].

These materials, especially barley straw and hemp shiv, could thus be used either as lightweight aggregates to improve the thermal insulation properties of an earth or mineral matrix, or as loose-fill insulation materials. A material is considered to be a thermal insulator when the thermal conductivity is lower than 0.065 W m^{-1} K^{-1} [61]. However, the thermal conductivity found for these agro-resources was measured for particles in the dry state and it is known that thermal conductivity increases with relative humidity [62]. The round robin tests of RILEM TC 236 BBM [35] revealed an increase of the thermal conductivity by 12.9% between the dry state and a relative humidity of 50%. If that increase is applied to the values measured for the dried plant aggregates, thermal conductivities become 0.049 W m^{-1} K^{-1} for straw, 0.058 W m^{-1} K^{-1} for hemp shiv and 0.109 W m^{-1} K^{-1} for corn cob. In the case of straw and hemp shiv, these

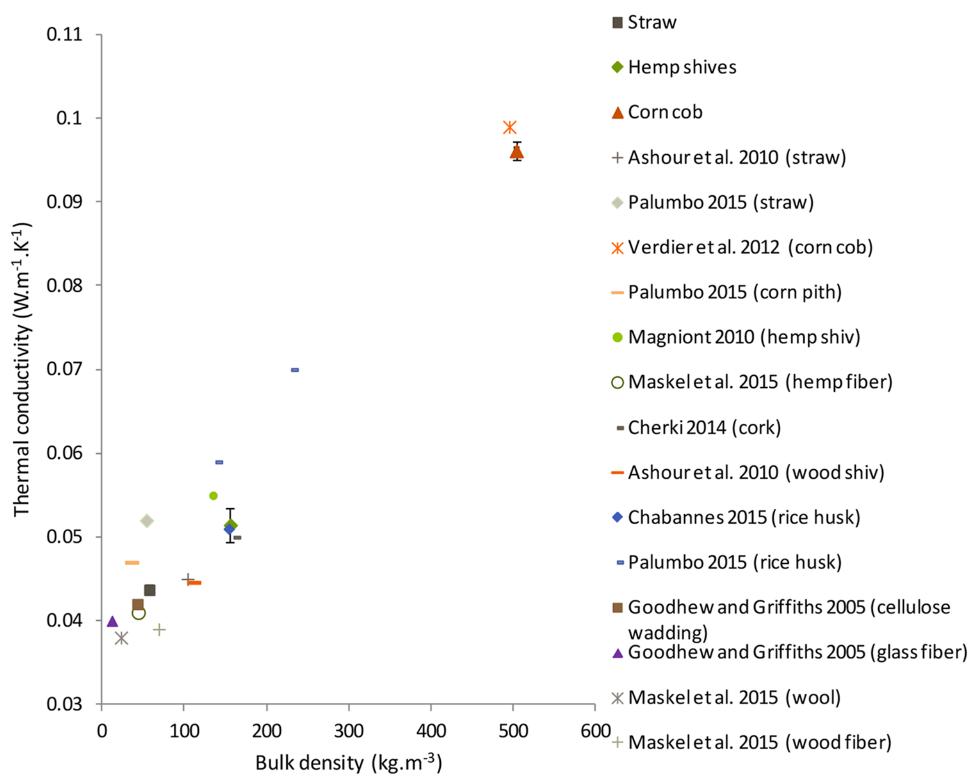

Fig. 11 Comparison of thermal conductivity values measured experimentally for straw, hemp shiv and corn cob and values found in the literature

Table 4 Absorption capacity (%) after 48 h of immersion

Plant aggregate	Barley straw	Hemp shiv	Corn cob
This study	414 ± 4	380 ± 11	123 ± 2
Literature	500–600 [25] 400 [55]	280 [30] 301 [9] 406 [42]	90 [58] ~ 150 [57]

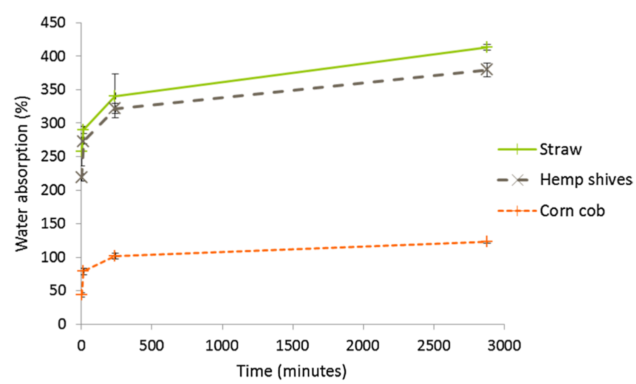

Fig. 12 Water absorption as a function of time

estimated values are still lower than the value expected for an insulation material.

Water Absorption

Within a hydraulic matrix, plant aggregate water absorption is an important formulation parameter as competition can occur between the particle absorption and the matrix hydration [9], leading to potential problems. Consequently, the amount of mixing water has to be largely overestimated. This implies a very long drying time, hardly compatible with the current rate of building. Water absorption was evaluated for up to 48 h for the three kinds of aggregates (Table 4).

Maximum absorption was 414% for straw followed by 380% for hemp shiv and 123% for corn cob in the present work. Calculating the coefficients of variation gave 1.1% for barley straw, 2.8% for hemp shiv and 1.3% for corn cob. These values are lower than 5%, which indicates good representativeness. These retention capacities seemed to be consistent with the literature, which presents nevertheless a high range of water absorption. This high variability could be due to the diversity of the plant particle itself or to the test method used. Only the value from [9] was obtained with the same protocol. Absorbent paper was used in references [30, 57] and the method used was not specified in the other cases. The low water absorption of corn cob could be interesting because, according to Bouhicha et al. [25], a high retention capacity is not good for adhesion to a soil matrix. During the manufacturing process, the swelling of the particles engendered by water absorption in the first 24 h pushes the soil away. Then, when the composite material dries, shrinkage creates voids around the particles at the interface with the soil [63].

The kinetics of this absorption is presented in Fig. 12. It is very fast for these plant aggregates: absorption by straw and hemp shiv at 1 min is close to 60% of the final absorption (48 h) and 36% for corn cob. These curves can thus be considered in two parts: the first one, with fast kinetics, represents an absorption by capillarity forces in the pores filled by free water [12, 55]. The second one corresponds to a diffusion mechanism based on Fick's law in the micropores and water bonding through openings (20–40 nm) in the plant cell walls [64].

Water absorption occurs through the multi-scale porosity of the particles, especially for stems such as straw and hemp shiv, which transport the sap in the plant. This water content seems to be linked with the internal porosity of the material [41]. On the SEM pictures (Fig. 7), high internal porosity can be seen for straw and hemp shiv. Corn cob is less porous and the water absorption is calculated from the particle mass, so the volume of water absorbed might not be very different from that found for straw and hemp shiv.

This absorption can be represented as a function of logarithm of time (Fig. 13), where the curves of the equation:

$$W = IRA + K_1 \times Log(t) \quad (10)$$

proposed by Nozahic and Amziane [12], are linear. The Initial Rate of Absorption (IRA, %) is the absorption relative to an immersion of 1 min, representing the adsorption of external water on the surface of the particles [65]. K_1 represents the diffusion rate in the cells (% (log min)$^{-1}$), the second part of the curve. Both values, IRA and K_1, are recapitulated in Table 5, with R^2, the correlation coefficient between the experimental values and the straight line.

K_1 and IRA, to a lesser extent, of barley straw and hemp shiv are very close to each other. However, the logarithmic representation of barley straw water absorption is less linear (R^2 of 0.96). The coefficient K_1 of corn cob is lower than the other two, meaning that the water diffusion rate in the cells is quite low.

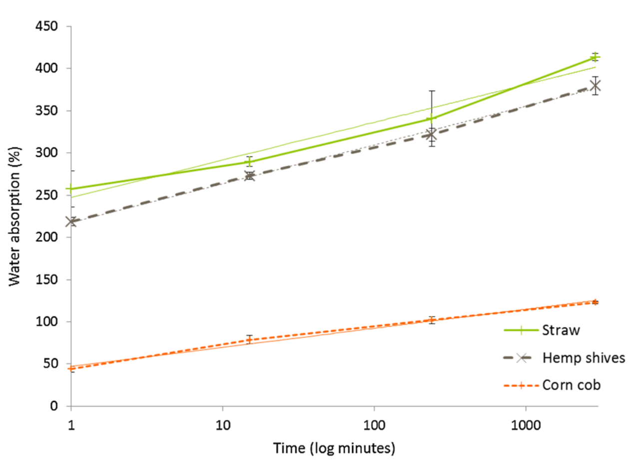

Fig. 13 Water absorption as a function of logarithm of time

Table 5 Water absorption parameters

Plant aggregate	Barley straw	Hemp shiv	Corn cob
IRA[a]	247	218	48
K_1[b]	19.4	19.9	9.8
R^2[c]	0.9632	0.9972	0.9874

[a] IRA is the Initial Rate of Absorption

[b] K_1 represents the diffusion rate in the cells

[c] R^2 is the correlation coefficient

As previously commented, due to the high water absorption of barley straw and hemp shiv, the amount of mixing water required should be increased if these particles are added to a hydraulic binder [66]. This can lead to a problematic increase of the drying time after demoulding [9]. However, different treatments could be applied to reduce the water absorption and drying time. Some treatments can also increase the bonding between the plant particles and the matrix. Nozahic and Amziane [12] studied three different treatments to improve the adhesion of sunflower aggregates to a mineral matrix: pre-wetting by an alkaline solution, a linseed oil coating, and a paraffin wax coating, which showed the best efficiency. Other plant aggregate treatments were studied in the case of an earth matrix, such as acetylation, rosin-alcohol coating or thermal immersion in boiling water, and are summarized in Laborel-Préneron et al. [19].

Chemical Characterization

The chemical characterization of plant aggregates is important in the case of bio-composites with hydraulic binder as chemical composition can influence their properties or those of the composites, such as setting time or hydration mechanisms.

Lignocellulosic raw materials are composed of three main components: cellulose, hemicellulose and lignin. There are also other chemical species present such as pectin, extractives and ash. Cellulose, a polymer containing various alcoholic hydroxyl groups, can significantly affect the mechanical performance of the fibres [67]. Hemicellulose is a highly hydrophilic component, easily hydrolysed by acids and soluble in dilute alkali solutions. This could thus influence the water absorption of the plant particle and affect its durability in an alkaline mineral matrix, such as cement or lime, and the bonding mechanisms at the interface in these composite materials [68]. Lignin is a polymer able to protect the stem of the plant from chemical or physical aggressions, notably from most microbial attacks.

The nature and the amount of lignin thus affect the durability and the biodegradability of the distinct vegetal materials [67]. A variety of functions is attributed to pectins, including mechanical properties, cell–cell adhesion, wall porosity and binding of ions [69]. An affinity between pectin and cations exists and could affect the setting mechanisms of mineral binders (Portland cement, lime, etc.) used in bioaggregate-based composites.

These interactions can take place at different times. At early age, they can disturb the setting and hardening mechanisms of mineral binders. Hemp shiv [70], hemp fibres [71], wood particles [72–75], cereal straw [76, 77] (cited in [78]), arhar stalks [79], sugar cane bagasse [80] and coir particles [81], among others, have been shown to negatively impact the setting and early hardening of cement paste. Wood particles can also be associated with a plaster matrix (calcium sulfate hemihydrate). Boustingorry et al. [82] emphasized that poplar and forest pine extracts clearly delayed the hydration of hemihydrate. Finally, the setting of a pozzolanic binder (mix of lime and metakaolin) has been shown to be affected by the presence of lavender stalks [13]. In the hardened state, they can modify the properties of the composite. Poor cohesion has been observed between plant aggregates and the mineral matrix, associated with a powdering of the binder [70]. In the long term, they can influence the durability of the material through mineralization of the plant aggregates by cement hydration products [83]. This engenders a loss of ductility of the fibres, which is a disadvantage for plant fibre reinforced concrete, which has to work in flexion. However, it might be an advantage for bioaggregate-based composites, leading to a continuous enhancement of compressive strength [84].

The chemical composition of the three plant aggregates is presented in Table 6 and compared with some other values from the literature. Values are expressed in percentage of dry mass of the plant material. The column "Extractives" refers to the water-soluble content (this study), the sum of pectin, wax, fat and protein, or the content obtained with Soxhlet extraction (literature).

Among the three types of particles studied, barley straw showed the highest amount of extractives (14.4%) and ash (12.3%). The extractive contents of hemp shiv and corn cob were 5.2 and 6.9%, respectively. The ash content of these two aggregates was also lower, at 2.1 and 1.4%. It is important to take the extractives content into account because it is the main cause of interactions with the hydraulic binder.

These chemical compositions are represented in a ternary diagram showing lignin, cellulose and hemicellulose (Fig. 14).The values are normalized to 100%, as in [68]. The comparison of the composition of particles is facilitated by this presentation. The plant aggregates used in this study are circled in red.

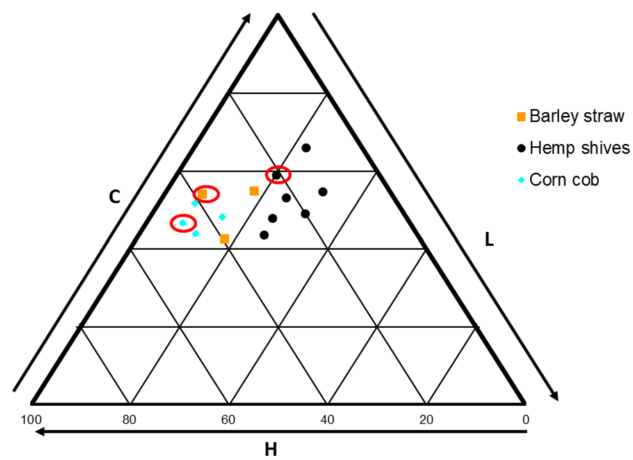

Fig. 14 Chemical composition of the 3 plant aggregates

Table 6 Chemical composition of the 3 plant aggregates

Aggregate	Reference	Lignin (%)	Cellulose (%)	Hemicellulose (%)	Extractives (%)	Ash (%)
Barley straw	This study	5.5	37.7	26.7	14.4	12.3
	[85]	15.8	37.6	34.9	–	–
	[86]	15	46	23	–	–
Hemp shiv	This study	17.2	50.3	17.9	5.9	2.1
	[87]	18	52	9	–	2
	[88]	22.1	37.7	26.8	8.9	–
	[70]	21.8–23.3	45.6–49.2	17.8–21	5.1–6.2	2.6–3.7
	[89, 90]	28	48	12	10	2
	[91]	28	44	18	10	2
	[92]	23	44	25	4	1.2
Corn cob	This study	6.6	41.4	40.7	6.9	1.4
	[58]	6.8	47.1	37.3	–	1.2
	[93]	6.7–13.9	32.5–45.6	39.8	–	–
	[68]	14.7	48.1	37.2	7	–

All the bioaggregates were rich in cellulose, which several authors link to the mechanical performance of the aggregate [67, 94, 95]. Corn cob was the aggregate presenting the highest hemicellulose content, a component easily dissolved by alkaline attack. It is thus the least usable in an alkaline mineral matrix. The lignin content was higher for hemp shiv. This aggregate could thus be the most durable of the three types.

Values for a type of aggregate are quite scattered. These variations may be due to the measurement method used but may also be connected with differences in the maturity of the stems, the season of harvest, the variety or, in the case of hemp, the retting process [96].

Sorption–Desorption Isotherms (DVS)

Figure 15 presents the sorption–desorption isotherms of the 3 plant aggregates.

The shape of the sorption curves is similar for all the particles and corresponds to a sigmoidal isotherm, Type II according to the IUPAC (International Union of Pure and Applied Chemistry) classification. This result is very common for cellulosic and lignocellulosic materials [34]. Although the results are similar for the three aggregate types, straw has a slightly higher sorption value at high relative humidity (but below 90% of relative humidity), followed by hemp shiv and corn cob.

Hysteresis (Fig. 16) represents the gap between the sorption and desorption isotherms (moisture content in desorption is higher than in sorption). This phenomenon is commonly explained by capillary condensation, the ink-bottle effect and the contact angle difference between adsorption and desorption [97, 98]. This phenomenon also seems to be influenced by the lignin content of the natural fibres [98]. The hysteresis values, ranging from 0.1 to 3.8%, increase with relative humidity except for the last 1 or 2 steps. They are comparable for the three plant particles.

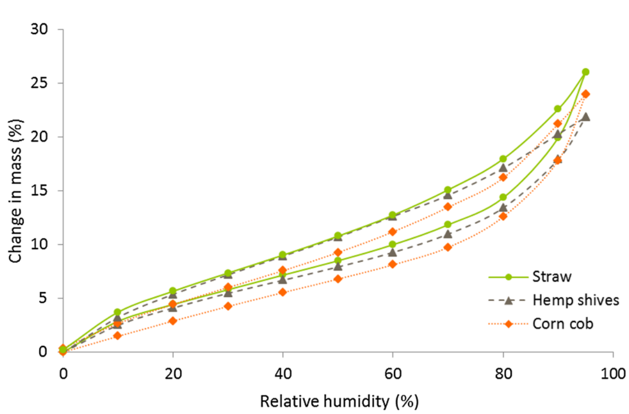

Fig. 15 DVS water vapour sorption–desorption isotherm of the 3 plant aggregates at 23 °C

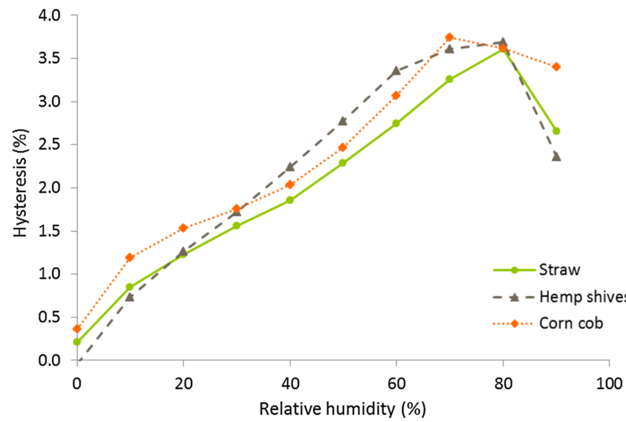

Fig. 16 Hysteresis calculated from the sorption and desorption isotherms of the 3 plant aggregates at 23 °C

These plant aggregates, whose sorption values reach 20 to 26% at 95% of relative humidity, could be used as aggregates in a matrix to increase the sorption capacity of the material. A high sorption capacity could induce good moisture buffering if the kinetics of sorption–desorption proved to be fast. This would improve the balance of the indoor air humidity, making it more comfortable for the occupant [99]. Complementary tests to study the kinetics of the sorption and desorption of each materials would be necessary to conclude on the subject of moisture buffering. This improvement has already been studied by Ashour et al. [100] with straw added to an earth plaster. The sorption capacity improved from 1.7% for earth alone to 6.5% with the addition of 75% of straw by volume.

At 80% RH, the sorption capacity of a lime plaster studied by Černý et al. 2006 [101] was lower than 1% and those of pozzolanic lime plasters were up to 4%. The sorption capacity of the plant aggregates of this study was between 12 and 14%. Their addition to that kind of mineral matrix could increase the sorption capacity of the composite material.

Conclusion

In this paper, three potential resources for bio-based building materials were characterized: barley straw, hemp shiv and corn cob. Their availability in France, physical properties, chemical composition and hygric properties were investigated. This overall methodology is mostly adapted from the RILEM method for characterizing bio-based aggregates. Although the round robin test was performed for hemp shiv, it can be adapted to other plant particles: the mass of the samples should be modified according to the bulk density of the particle type. However, image analysis may be less useful in the case of spherical particles, such as corn cob, which can be characterized by the faster sieving method, as is done for mineral aggregate. The study of the availability of plants was deliberately restricted to France, as the use of local resources allows the environmental impact to be limited. Nevertheless, this kind of investigation should be carried out to evaluate the potential each time an agro-resource is considered for the development of a new bio-based material.

The main results are summarized below:

- Barley straw is a resource having good availability, with more than 4 million tons available each year. Corn cob and hemp shiv also present an interesting potential with 240,000 and 17,000 tons of by-products available each year.
- The microstructure of these three aggregates is composed of tubular pores, but with a different cell wall thickness for each type.
- Bulk density, highly dependent on these microstructures, is around 60,150 and 500 kg m^{-3} respectively for the straw, hemp shiv and corn cob.
- Thermal conductivity is, like bulk density, the lowest for straw, followed by hemp shiv and then corn cob.
- The morphology of the particles is quite similar for straw and hemp shiv (the straw being slightly more elongated) whereas corn cob aggregate is more spherical. That property could lead to different qualities of adhesion in case of use in a matrix.
- Water absorption is very high for straw and hemp shiv (414 and 380% respectively after 48h of immersion) but it is much lower for corn cob, with only 123% of water absorption.
- The chemical composition of the bioaggregates is rich in cellulose. Corn cob is rich in hemicellulose, hemp shiv has the highest lignin content (17%) and straw presents the highest extractive and ash content (around 25%).
- Sorption–desorption isotherms are similar for the three types of particles.

Some conclusions can thus be drawn for each particle type. Barley straw is the most available agro-resource of this study and the best thermal insulator thanks to its low bulk density. A major drawback is its high water absorption which would be prejudicial in case of introduction of this straw into a pozzolanic or hydraulic matrix. It would induce the need for a surplus of water leading to a longer drying time. However, there are some possibilities for treating the particles to reduce this absorption and avoid its negative effects on adhesion or setting time. Straw also presents the highest aspect ratio, which seems to be a positive factor in terms of mechanical strength in an earth matrix [53]. Hemp shiv appears to be the most suitable plant aggregate for use within a hydraulic matrix thanks to its lowest hemicellulose and extractive contents. The thermal conductivity of both straw and hemp shiv is compatible with their use as loose-fill insulation. As far as corn cob is concerned, its use might be interesting because of its low water absorption, which would avoid the earth to be expanded. However, its high hemicellulose content, sensitive to alkaline attack, limits its use in a pozzolanic or hydraulic matrix for example.

All three plant aggregates could be used in an earth matrix, where there is no risk of interaction. The high vapour sorption capacity of these lignocellulosic materials could be useful to improve the capacity of earth or mineral matrices to store moisture. Concerning morphological parameters, it has been shown that mechanical strength is higher when coarser particles are incorporated [52]. However, complementary studies will be necessary to correlate morphological parameters or size distribution of the plant particles with the performances of the composites. These bioaggregates show promise for the development of bio-based building materials if they are used in an appropriate matrix. More research is needed in order to study other parameters such as mechanical strength, fire resistance or microbial growth resistance. Other available resources should also be investigated to develop new building materials, with beetroot for instance.

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