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Abstract

The use of bio-based concretes or renders is rapidly developing. These sustainable materials present many advantages such as their acoustical and hygrothermal properties. These bio-sourced composites are made of vegetable aggregates, associated with a hydraulic binder. One of the most common aggregates is hemp shiv, which is an agricultural waste. However, hemp shiv is not available in big quantities nor in all geographical areas, so it seems interesting to introduce other bio-sourced aggregates in order to increase the availability. Sunflower and maize pith are unexploited agricultural by-products which can have a great potential as insulation materials. To use these aggregates, it is essential to assess first their interactions with different binders. In the present paper, the impact of density and type of aggregate (either hemp shiv or pith) on several key physical parameters was evaluated. These parameters were the uniaxial compressive strength test, the thermal conductivity, the sorption isotherms and the water vapor permeability, as well as the moisture buffering value (MBV). The use of pith shows promising results in terms of thermal and hygroscopic performances, the results also indicate that specific precautions should be taken to preserve the porosity of the soft pith material to optimize the hygrothermal performances.

Keywords: bio-based composites, hygrothermal properties, hydraulic binder, hemp shiv, sunflower and maize pith

1. Introduction

The building sector represents 20% of the final energy consumption in the world [1], 26.1% of the energy consumption in the EU and 27.9% in France [2]. The urge to reduce those emissions is becoming more and more a political issue, the recent conference of the parties on climate change organized by the United Nations reflects those preoccupations.

The building sector is currently facing major challenges, due to a growing concern on the energy consumption of buildings and its impact on the current climate crisis. A 2017 report by the UN Environment and International Energy Agency [3], reports the building sector being responsible for 36 % of the global final energy use. While the first challenge remains the reduction of energy consumption of the buildings during their use, the second challenge will be to reduce the embodied energy, in the building materials themselves and energy consumed during the construction phase.

One promising option to reduce embodied carbon in building materials is to favor locally sourced and renewable materials [4]. Agricultural by-products become more and more used, some already have established uses, such as hemp lime or the use of straw bales [5][6]. Many other agricultural by-products can potentially be used, research is ongoing worldwide to implement plant aggregates in the formulation of light weight concretes or mortars [7][8]. Generally, such materials present good qualities in terms of thermal insulation, even if large differences exist in function of density and formulation. For example, [9] measured thermal conductivity for bulk hemp shiv with a density of 130 kg/m^3 and obtained 54.5 mW/(m.K), as well as for sunflower pith with a density of 20 kg/m^3, obtaining 50.9 mW/(m.K). These values are quite close to ones of synthetic insulation materials like expanded polystyrene or glass wool, which commonly vary between 30 mW/(m.K) and 40 mW/(m.K). Once mixed with a binder, those values do however increase, [10] found thermal conductivity values oscillating between 117 and 138 mW/(m.K) for hemp composites of densities between 510 and

630 kg/m^3. The same range of values was measured by [11][12][13] for hemp composites made with lime and metakaolin and by [14] for maize and sunflower stalk based composites. In all these cases, the sample densities were ranging between 500 and 600 kg/m^3. [15] studied the impact on thermal conductivity of the composition of two corn stalk and magnesium phosphate cement composites. They noticed that samples with a higher corn stalk content had a lower thermal conductivity. [16] found this same result on hemp composites, detailing however that the relation between the amount of hemp shiv and thermal conductivity is not linear. Furthermore, [15] observed that for a same corn stalk content, large stalks presented a lower thermal conductivity than small ones, which was explained by a lower density and a higher porosity. Many parameters have an impact on thermal properties of bio-based composites such as water content, density and the formulation. It has been highlighted in the past that density has the biggest influence because a larger air volume in the material results in a low thermal conductivity [10][15][16].

In addition to their promising thermal performances, bio-based composites are known to exhibit good to excellent hygroscopic potential [9][17]. It traduces their ability to be used as a passive humidity regulation system thanks to their high capacity of adsorption (and desorption) of water vapor molecules from (to) the surrounding atmosphere. Due to the latent heat of adsorption and desorption processes, these materials might also have the potential to be used as passive temperature regulation system [11], even if the real impact of this effect on building energy consumption still needs to be better assessed.

Other important assets of bio-based composites are their high vapor permeancy and their light weight, which make them suitable to be used to rehabilitate the national heritage, typically built in stone, adobe, rammed-earth, etc. [14][16][18]. Indeed, they do not constrict vapor exchanges between walls and their environment and they do not overcharge the existing structure.

On the other side, bio-based composites do not have mechanical performances as good as classic building materials [10][17][19][20]. That is why they are most of the time rather used as filling and/or insulation materials, for which a high loading capacity is not mandatory. Anyway, it is commonly agreed that compressive strength and deformation modulus are good indicators of binder-aggregate compatibility. The several studies led on these parameters lead to the conclusion that there is no good or bad aggregate or binder, but good or bad combinations of them [21]. For example, [22] show that a first composite made of corn stalk and ordinary Portland cement threw a 1.5 MPa compressive strength value, while a second composite made of corn stalk and magnesium phosphate cement presented a compressive strength more than two times higher (3.83 MPa). Another sound example is given by [14]. In this study compressive strength at 60 days was measured on composites made of lime and sunflower (LS) with a density of 540 kg/m^3, metakaolin and sunflower (MS) with a density of 510 kg/m^3 and finally, metakaolin and maize bark (MM) with a density of 530 kg/m^3. These composites developed a mechanical resistance of 0.21 MPa, 0.34 MPa and over 0.35 MPa respectively.

Most of the time, the aggregate used in the formulation of bio-based composites is the hemp shiv. In France, for example, the soles professional rules that exist for this kind of materials are dedicated to hemp composite [5]. Nonetheless, it is essential to give alternatives to hemp [8][14][23] so as to have access to varied resources regarding geographical origin with the goal of reducing transport carbon print. For that purpose, a large variety of aggregates can be found, such as flax, straw, wood, coconut, miscanthus, corn or sunflower, etc. [18][19][24][25][26].

Based on this observation, this paper focuses on sunflower and maize pith, which, like hemp shiv, are considered as agricultural by-products. Both sunflower and maize grow in most European countries and they already have been studied by some authors [14][23][27][28][29][30][31].

The binders used are lime based and contain cement and admixtures in certain cases. The goal is to determine the impact of the use of pith instead of hemp on the performance of the composite. For that purpose, two ranges of densities (low densities around 150 kg/m^3 and medium densities around 500 kg/m^3) and three binders are considered. Let us underline that it has already been determined in a previous paper [32] that the geographical origin of the sunflower and maize piths does not impact the performance of the composites. Their performance was assessed through their mechanical, thermal, sorption and vapor transport properties. Thermal conductivity was studied through two different methods, the hot wire method and a novel device conceived for more accurate measurement. Finally, the hygrothermal potentials of the composites were evaluated through the moisture buffering test, and the consistency between results was analyzed using the MBV analytical expression provided by [33].

Since hemp dominates the bio-based construction field and the regulations about vegetal composites are based on it, the aim of this paper is to determine whether maize and sunflower pith aggregates can be used at the same level, not to replace hemp but to provide more choice. In addition, the impact of the aggregate, of the binder and of the interaction of both will be assessed. We will as well determine whether used binders, which have originally been conceived for hemp shiv, are appropriate for maize and sunflower pith or not.

2. Materials

2.1 Plant aggregates

As part of this experimental campaign, three types of plant aggregates were studied: hemp shiv (H), sunflower pith (S) and maize pith (M) (cf. **Fig 1**). The hemp shiv aggregates were received from an agriculture cooperative based in the north of France. Loose hemp shiv aggregates had a density of 100 kg/m^3, an average width of 4.1 mm and an average length of 7.6 mm. The sunflower pith comes from an agriculture cooperative based in the south-east of France. Loose density of this sunflower pith was around 14 kg/m^3, an average diameter of the aggregates was around 4.2 mm. The maize pith comes from an agriculture cooperative based in west France with a more oceanic climate. The maize pith aggregates had a loose density of 18 kg/m^3, an average width of 2.2 mm and an average length of 9.6 mm. The density of the aggregates has been determined following [34]'s protocol, whereas the average aggregate dimensions have been calculated through a sifting granulometric curve for pith and through a 2D image granulometric analysis developed by [35] for hemp shiv.

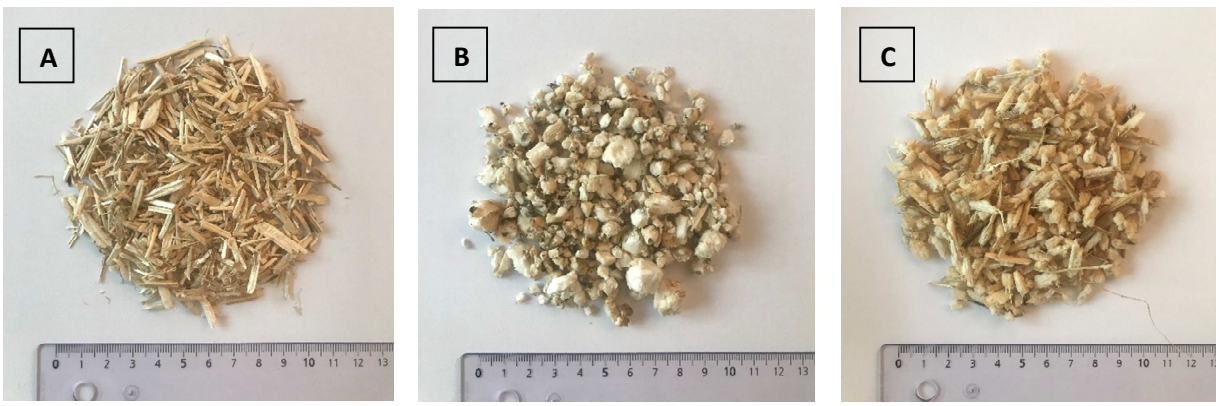

Fig. 1. Aggregates used in this study (A) Hemp shiv, (B) Sunflower pith and (C) Maize pith

2.2 Binders

Three different types of binders were used in this study. The first one named C1 is a calcic lime based binder. The second one named C2, is a formulated lime which is composed of hydraulic lime, calcareous charges, hydrophobic and rheological admixtures. The last one, named HB, is a cement and lime mixture with further additives, this formulation was developed by an industrial partner and whose exact composition is not available.

2.3 Bio-based composites

The samples were manufactured in the same conditions (50%RH and 23°C) and the main proportion of the mixtures of all kinds of formulations used are summarized in **Table 1**. Samples were sprayed in cylindrical formworks with a 16 cm diameter and a 32 cm height in a single layer except for C2-H*, which was manufactured in 6 layers of about 5 cm each. Each layer was compacted at 0.05 MPa following [5]. It is recalled that H, S and M are the aggregates explained in §2.1, more specifically hemp shiv, sunflower pith and maize pith respectively, while C1, C2 and HB are the binders explained in §2.2. C2-H* corresponds to a formulation that was compacted.

Table 1 Summary of the formulation and mass ratio.

Type of binder	**C1**	**C2**				**HB**		
Type of aggregates	H	H	H*	S	H+S	H	S	M
Designation	C1-H	C2-H	C2-H*	C2-S	C2-H+S	HB-H	HB-S	HB-M
Hemp shiv/binder (-)**	0.33	0.33	0.33	0	0.17	0.33	0	0
Pith/binder (-)**	0	0	0	0.10	0.06	0	0.20	0.16
Water/binder (-)**	0.81	0.88	0.88	0.80	1.07	0.88	2.92	2.92
Mass proportion of hemp shiv (-)	0.15	0.15	0.15	0	0.07	0.15	0	0
Mass proportion of pith (-)	0	0	0	0.05	0.03	0	0.05	0.04

* corresponds to a formulation which has been densified using a tamper tool.

** correspond to mass ratio between constituents

A pycnometer was used in order to determine the physical properties such as the skeletal porosity, dry and skeletal densities of the different formulations according to the standard NF EN 1097-7 2008**.** Results are shown in the **Table 2**.

Table 2 Summary of the physical characteristics.

Formulation	**C1-H**	**C2-H**	**C2-H***	**C2-S**	**C2-H+S**	**HB-H**	**HB-S**	**HB-M**
Porosity	0.76±0.02	0.79±0.01	0.70±0.02	0.77±0.01	0.70±0.02	0.78±0.02	0.94±0.01	0.92±0.01
Dry density (kg/m^3)	470 ±5	440 ±6	600 ±10	550 ±5	590 ±6	410 ±8	150 ±4	160 ±5

Skeletal density (kg/m³)	1960	2090	2000	2390	1970	1860	2500	2000

The formulations C2-H*, C2-H+S and C2-S aimed at testing the impact of the aggregate on the performance. Indeed, they were mixed with different aggregates but with the same binder, and their dry densities are in the same range. The formulation C2-H, which is similar to C2-H*, was made to test the impact of a moderate variation of density.

The impact of the binder will be assessed through the comparative analysis of the results of the C1-H, C2-H and HB-H formulations.

Finally, two light formulations, namely HB-S and HB-M were made in order to test the ability of pith aggregate to be used in light composites with high thermal performances. **Fig. 2A and 2B** show a HB-S and a C1-H specimen respectively.

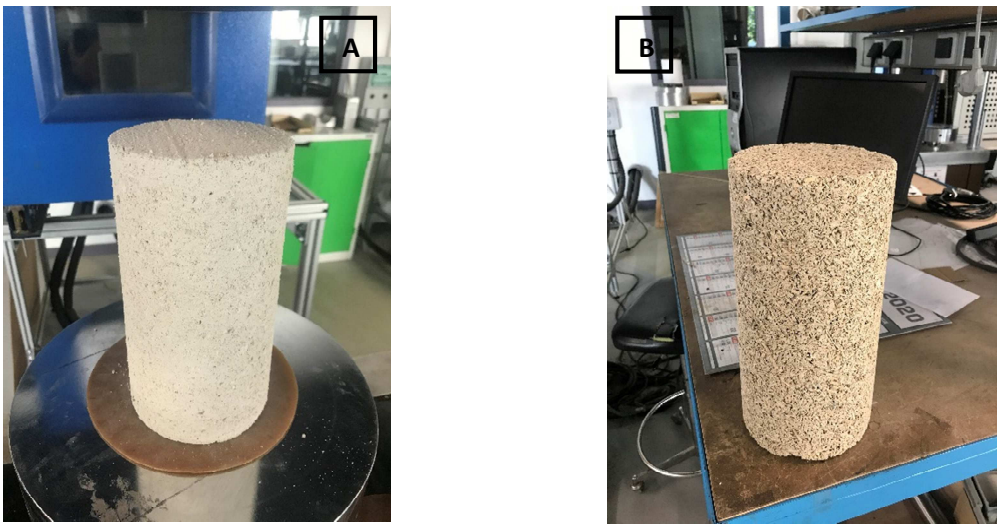

Fig. 2. A 16*32 cm HB-S specimen (A) and a 16*32 cm C1-H specimen (B)

2.4 Methods

2.4.1. Uniaxial compression test

To study the mechanical properties of materials, non-cyclic uniaxial compression tests were performed as done by [18][36][37][38] in accordance with French professional rules “Building with hemp”. Both compressive strength (Rc) and global initial deformation modulus were evaluated. For each value, three uniaxial compression tests have been performed.

Compressive strength increases gradually during the hydration process. [18] studied a series of composites made of a mixture of hydraulic and air lime, hemp and water in which each composite had a different binder percentage and she observed a common pattern. All the composites presented three phases of strength development, the first between 0 and 3 months, in which compressive strength doubled its value; the second one between 3 months and 1 year, when compressive strength increased by 10 to 50% depending on the formulation, and a third one after one year, in which Rc increased by 10% every year.

To study this strong temporal evolution during the first 3 months, uniaxial compression tests were made at two curing times (28 and 60 days). Some tests were performed after a curing of 90 days, but the results were not presented here since they were found to be similar to those at 60 days.

Mechanical strength and deformation modulus were determined on the “dense formulations” HB-H, C2-H+S and C2-S and the “light formulations” HB-M and HB-S. The tests were carried out on cylindrical samples of 16*32 cm using an INSTRON press controlled at 3 mm/min displacement.

Before the compression tests, samples were stored at 23 ± 2°C and 50%RH, then dried in an oven at 50 ± 1°C for 96 hours following the [5] recommendations. The tests were performed in triplicate, and the average reading was reported. Density of each sample was measured before testing.

2.4.2. Thermal conductivity

Two techniques were used in order to measure the thermal conductivity.

The hot wire apparatus was used for all the formulations. For that purpose, cylindrical specimens 16*32 cm of composite were cut after 21 days of manufacture in small cylindrical samples (16*6 cm), which allowed us to obtain three samples per formulation. They were conserved in a room at about 30°C (±5°C) and 60%RH (±5%RH) and were tested after 100 days of curing. In these same conditions, the thermal conductivity measurement of the plant aggregates was carried out using plastic boxes filled with hemp shiv (H) and sunflower pith (S), following the procedure presented in [9]. Each formulation was tested using the three different samples, the results being the arithmetic mean value of the three tests.

The hot wire method is however frequently pointed out due to some of its limitations. Namely that the area covered by the probe which emits heat is fairly representative, and that the samples have irregularities on the surface, which makes the heat transfer not unidirectional.

To avoid these issues and to check accuracy of the hot wire measurements, a novel measuring device has been developed. This device measures the thermal conductivity of a representative sample (35*35*7 cm). The principle is similar to the hot plate’s, which consists in creating a unidirectional heat flow crossing the thickness of our sample by imposing a thermal gradient between two chambers, a cold one and a hot one. The chambers (A and B) are made of wood and configured as seen in the **Fig. 3**, the warm atmosphere being monitored by a PID controller (Proportional Integral Derivative) and the heat being generated using a light bulb. The cold atmosphere is generated by a cryostat. Both the PID and the cryostat are connected to a computer. In order to homogenize, the atmosphere is ventilated in the two compartments.

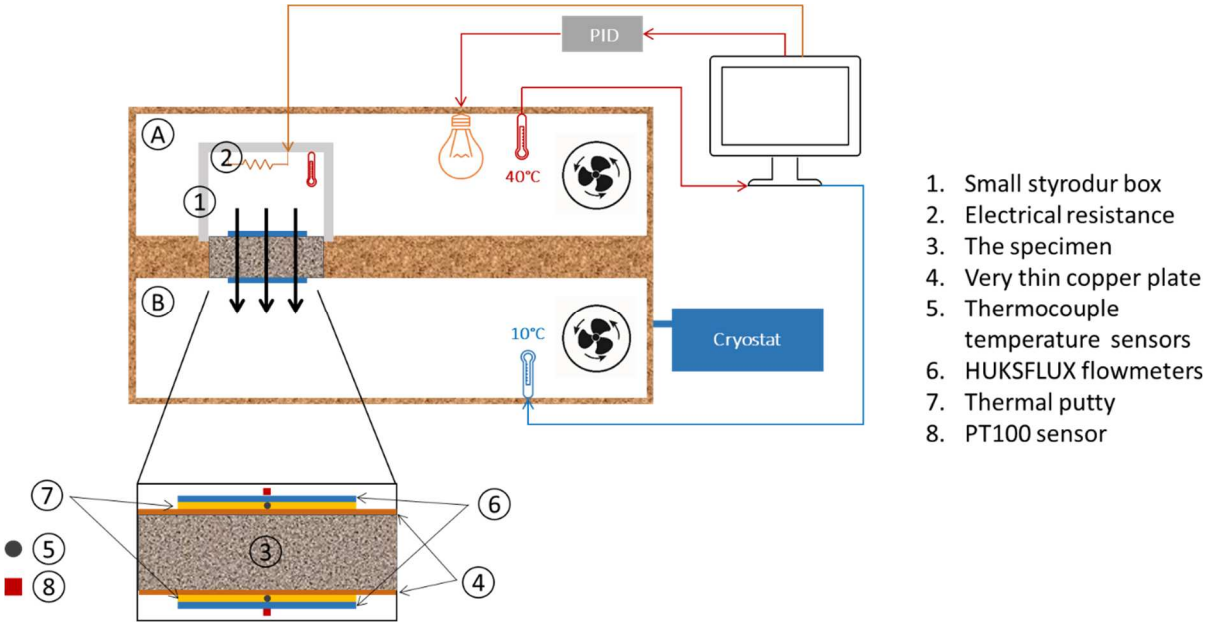

Fig. 3. Thermal conductivity measuring device.

A small styrodur box (1) has been manufactured and placed above the sample in such a way that there is no heat loss to the outside environment. Without it, the hot environment around the sample would suffer heat leaks and would not be stable. This box is heated using an electrical resistance (2) controlled by a computer. A temperature sensor has been placed in each compartment. The sample (3) is placed in the cavity that connects both chambers. Once the sample is placed, we stick on both sides a very thin copper plate (4), which has a very high thermal conductivity, in order to ensure a flat surface that guarantees a unidirectional vertical thermal field. On the latter, thermocouple temperature sensors (5) are taped with aluminum tape to keep track of the temperature during the test. We then glue the HUKSFLUX flowmeters (6) using thermal putty (7) to ensure good thermal conductivity and on top of that, we add another PT100 sensor (8) to measure the value of the surface temperature. The device has been calibrated using polystyrene, whose thermal conductivity is well known. This test was performed on three formulations, HB-S, C1-H and HB-H, and for each of them, three samples were studied. The results in § 3.2 are the arithmetic average for each formulation. In the sake of comparability, samples were stored at the same conditions as hot wire's, 30°C (±5°C) and 60%RH (±5%RH), and tested after 100 days of curing. This age was chosen because the variation of mass was under 1% between two measures performed 24 hours apart. For the test, the temperature difference was stablished at 30°C, with 40°C in the hot atmosphere (both outside and inside the styrodur box to ensure a stable temperature) and 10°C in the cold one. This values can be configured by the user. Humidity conditions have no impact since the sample is completely covered by the copper plates on both faces and surrounded by aluminum scotch.

This device has been dimensioned to perform tests as long as the user requires, but in this experimental campaign, the tests took approximately 30 minutes, the time to achieve the heat flux steady state through the sample.

2.4.3. Sorption isotherms

Two methods were used for the determination of the sorption curves: "desiccator method" and "DVS method". The equivalence between these two methods has already been discussed in [39]. It requires that the protocols used to measure the dry masses in each method lead to the same dry density, which was here successfully checked.

The "desiccator method" was performed on three representative samples of formulations HB-H, HB-S, HB-M, C2-H and C1-H according to the standard EN ISO 12571 (CEN 2000). The samples were cubic and had 4 cm in edge. This representative volume was determined from the apparent density variation against volume [40]. The first step was to dry all samples in an oven at 50°C in order to start the test at dry mass. Since the samples contained organic material, the maximum temperature that could be used was 60°C, because the fibers in the material may suffer from degradation at higher temperatures. To ensure the reproducibility of dry mass measurements and the consistency between desiccator and DVS methods, the relative humidity within the oven was checked to be lower than 5%. The samples were placed in six different levels of RH (23, 43, 59, 75 and 85%) using saline solutions. For this test, the temperature was maintained at 23±2°C. A portable sensor (Rotronic HygroLog HL-NT) was used for constant control of the RH and the temperature in the boxes. The starting point of the sorption curve corresponds to the dry mass of the samples. After finding the initial point of the curve, the samples are then placed at relative humidity values aforementioned following an adsorption stage and then a desorption stage.
The Dynamic Vapor System (DVS Intrinsic SMS) method was used to draw the sorption curves of the aggregates H, M and S and of the formulations HB-M, HB-S, C2-S, C2-H+S and C2-H*. For both methods, each formulation was tested using three samples and the given results are the arithmetic average of the three samples of each formulation. HB-M and HB-S were tested using both methods in order to check if results were close. Since they were, the values shown in §3.3 for these two formulations are only those obtained through the DVS.

2.4.4. Water vapor permeability (Wet Cup method)

The water vapor permeability of all formulations was measured using the Wet Cup method according to the standard EN ISO-12572 (CEN 2001). It consists in generating a vapor pressure gradient by adjusting the relative humidity to 50% in the climatic chamber and 85% in the cup. The space between the saline solution and the bottom of the sample is called the air layer and was 20mm for all samples. The set-up of the cups was performed according to the procedure followed by [41]. Therefore, a thin layer of silicone was applied to seal the samples on Plexiglas cups. A vapor-tight aluminum tape was used to seal the sides of the sample with the side of the cup. The use of aluminum tape is justified by its properties: it is impervious and does not absorb moisture itself [42]. Environmental conditions (50% RH and 23°C) were constantly monitored by the climatic chamber. For each formulation, samples of 3 thicknesses (2 cm, 4 cm and 6 cm) were tested in order to take into account the surface resistance effect, as it was recommended by [43]. The result for the vapor permeability of each formulation is the arithmetic average of the value for each thickness. As explained by McGregor, the water vapor permeability value for each sample takes into account the effect of the sample's thickness.

2.4.5. Dynamic moisture exchange behavior (Moisture Buffering Value)

In order to quantify the hygroscopic potential of a porous medium, a common method consists in performing a dynamic adsorption-desorption test commonly called MBV. The protocol for this test was initially defined as part of the NordTest project. This test indicates the amount of moisture absorbed or released by a material when exposed to cycles of repeated relative humidity changes between 33% and 75% at 23°C, with time steps of 8 hours to high relative humidity and 16 hours durations at low relative humidity (other relative humidity cycles can be chosen, but in our case we have maintained reference cycle values) [33]. The tested sample is sealed on all sides using aluminum tape, except for the upper side, and the Practical Moisture Buffering Value is calculated as the mass variation per unit area (denoted A) according to the formula:

$$MBV_{\text{pratical}} = \frac{\Delta m}{A * \Delta RH} \quad (1)$$

with Δm (in g) the mass variation during the cycle, ΔRH the variation of relative humidity (in %) and $MBV_{pratical}$ the Practical Moisture Buffering Value expressed in $g/(m^2.\%RH)$.

Cylindrical specimens 16*32 cm were cut beforehand to make 3 identical samples of size 16*6cm for each type of insulation, which makes us a total of 27 samples. The result for each formulation is the arithmetic mean of the three samples. The mass monitoring was carried out with an interval of 1 hour then 2 hours until reaching the maximum of the cycle and then a last measurement 24h after the beginning of the cycle. The MBV was calculated when the regime was stabilized, i.e. when the mass difference between the beginning and the end of a cycle was less than 5%.

3. Results

3.1. Uniaxial compression test

The results of the uniaxial compression test are reported in the **Table 3**. The apparent global deformation modulus (E) was estimated on the linear part of the axial stress-axial strain curve. The compression resistance Rc is considered to be the limit between the elastic and elasto-plastic behavior of the axial stress – axial strain curve, which happened at 3% ± 0.5 strain in all cases. The apparent global stiffness modulus (E) is estimated as the slope of the linear part of the curve. As bio-based composites typically present a heterogeneous deformation, strain has been determined as the variation of the total thickness of the sample divided by the initial thickness using a position sensor incorporated in the press.

Table 3 Average of global stiffness modulus of the formulations.

Type of samples	HB-H	HB-S	HB-M	C2-H+S	C2-S
Density (kg/m³)	410 ±8	150 ±4	160 ±5	590 ±6	550 ±5
Rc_{28} (MPa)	0.53 ±0.03	0.10 ±0.01	0.11 ±0.02	0.58 ±0.03	0.50 ±0.01
E_{28} (MPa)	46 ±2.1	-	-	52.6 ±2.7	50.5 ±1.2
Rc_{60} (MPa)	0.66 ±0.02	0.27 ±0.01	0.24 ±0.03	0.78 ±0.02	0.64 ±0.02
E_{60} (MPa)	68.5 ±1.8	10.0 ±0.8	7.5 ±1	73.8 ±2.4	64.5 ±1.6

Results of HB-S and HB-M appeared to be clearly different from the others (namely HB-H, C2-H+S and C2-S). Compressive strength values at 28 days of 0.10-0.11 MPa were found for HB-S and HB-M, and of 0.5-0.58 MPa for the other three. At 60 days, compressive strength increased up to 0.24-0.27 MPa for HB-S and HB-M, while the three others reached 0.64-0.78 MPa. The deformation modulus also appears to be strongly lower for HB-S and HB-M formulations, as well as the ratio between the deformation modulus and the compressive strength. For HB-H, C2-H+S and C2-S formulations, $E_{60}/Rc_{60} \approx 100$ while $E_{60}/Rc_{60} \approx 35$ for HB-S and HB-M. These significant differences in behavior can be explained by their dissimilar density (150-160 kg/m³ for HB-S and HB-M while the others range from 410 to 590 kg/m³).

To go further in the analysis, the compressive strength values obtained at 28 days in this study were compared with the ones reported in literature for hemp, corn stalk and pith composites. Results are reported in **Fig. 4**.

At first, it can be seen that our values are in the range of ones reported in literature. In this graph, the strong impact of the dry density on the compressive strength, whatever binder and aggregate

natures appears to be quite obvious. However, a closer look seems to indicate that, for a given density, compressive strength is slightly lower for pith-based composites than for hemp-based composites. This tendency can be also observed in the results of this study from the comparison between C2-S+H, C2-S and HB-H formulations. A possible explanation might be the higher porosity of pith (see **Table 2**), which results in a low compressive strength of the aggregates. It is however quite complicated to conclude on that point, since it is not possible to keep constant both dry density and particle/binder ratio, and these two parameters are known to significantly impact both compressive strength and deformation modulus. Thus, a precise assessment of the impact of aggregate nature on the mechanical performance would have required a more extensive campaign of uniaxial compression test, which was clearly out of the scope of this paper.

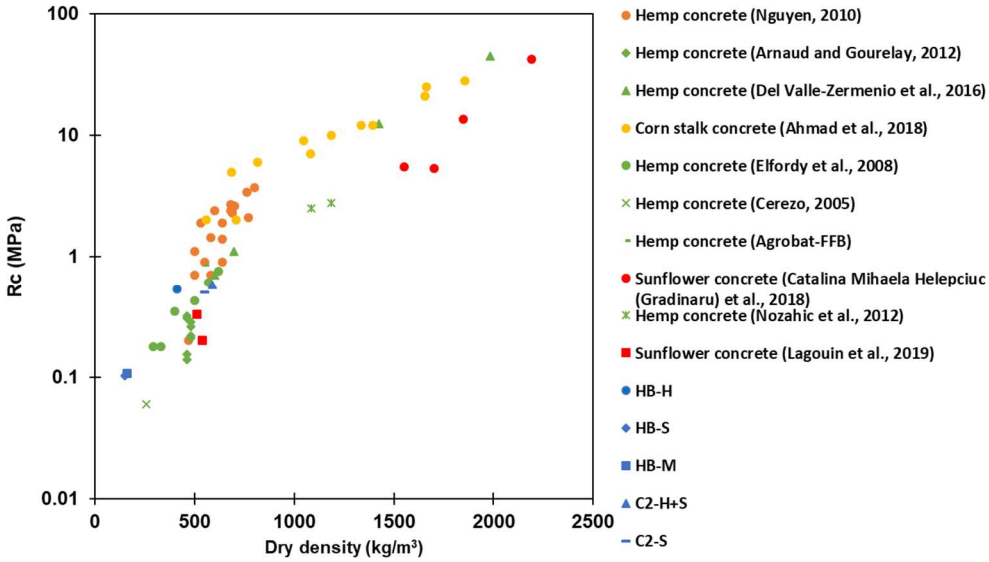

Fig. 4. Compressive strength vs dry density at 28 days of the formulations tested in this study and from several studies from literature

Anyway, we can conclude that all the formulations studied in this paper led to sufficiently cohesive materials in order to be used for building insulation purposes. In the framework of the French hemp construction guide "Building with hemp" specifications [5], HB-S and HB-M can be used only for roof applications while HB-H, C2-H+S and C2-S can be used as a plaster, a wall, floor or roof. The other main information is that the use of pith instead of hemp does not prejudicially drop the mechanical performance of the composite, even if it might slightly reduce it.

3.2. Thermal conductivity

The results of thermal conductivity are shown in the **Table 4**. $\lambda_{hot\ wire}$ represents the thermal conductivity measured with the hot wire apparatus, while $\lambda_{new\ device}$ is the one measured with the home-made device presented in the §2.4.2.

Table 4 Summary of the thermal parameters of the tested formations.

Type of samples	Density (kg/m³)	$\lambda_{hot\ wire}$ (mW/(m.K))	$\lambda_{new\ device}$ (mW/(m.K))

C2-S	550 ±5	190 ±7	-
C2-H	440 ±6	135 ±10	-
C2-H*	600 ±10	150 ±10	-
C2-H+S	590 ±6	140 ±8	-
HB-S	150 ±4	65 ±2	66 ±1
HB-M	160 ±5	75 ±3	-
C1-H	470 ±5	155 ±10	147 ±2
HB-H	410 ±8	90 ±10	75 ±2
Hemp shiv (H)	100 ±5	70 ±6	-
Sunflower pith (S)	14 ±4	50 ±1	-

Values obtained with the two methods are not strongly different but they show some inaccuracies. For HB-S it can be observed that both values are almost identical. However, results are not as good for C1-H and for HB-H, for which hot wire apparatus tends to higher values. The main difference between the specimens is that samples containing sunflower pith present a flat surface whereas hemp samples show strong irregularities on the surface. One reason of this trend could be that the size of the material heterogeneities (size of aggregates and pores) is too large for hemp-based formulations compared to the zone scanned by the hot wire. It leads to underestimate the thermal insulation contribution caused by the porosity of the material. However, the tendencies obtained with the hot wire were found to be correct and this method can be used for comparison purposes.

The first analysis that can be made is that the general tendency is a decrease of the thermal conductivity with density. This is consistent with observations made in previous studies, describing this tendency [14].

Thermal conductivity measurements were also made on loose aggregates. A thermal conductivity of 0.07 W/(m.K) was found for Hemp shiv (H), while 0.05 W/(m.K) was found for sunflower pith (S). These results appeared to be a bit higher than expected, but the significantly lower thermal conductivity of sunflower pith when compared to hemp shiv was quite expected.

This tendency is however reversed once the aggregates are mixed with the binder. For example, the thermal conductivities of both C2-H and C2-H* are lower than C2-S's one, even if they were formulated with the same binder and if their densities are in the same range (440 kg/m^3 for C2-H, 600 kg/m^3 for C2-H* and 550 kg/m^3 for C2-S). The same conclusion can arise from the comparison between HB-S and HB-H. While it exhibits a significantly higher dry density (410 kg/m^3 vs 150 kg/m^3), the thermal conductivity of HB-H appears to be only slightly higher than those of HB-S (0.075 W/(m.K) vs 0.066 W/(m.K)). Therefore, thermal conductivity of the composite is found to be impacted by the type of aggregate. But this impact is not trivial. It can notably depend on the coating rate of the particle by the binder, as well as on the physical and/or chemical interactions between them. It will thus not be possible to predict easily the thermal conductivity of the composite from the knowledge of the thermal conductivities of its constituents. Let us underline that this result is in contradiction with [9], which found no significant influence of the nature of the aggregates on the thermal conductivity of bio-based composites. Additional studies are thus required in order to draw a definitive conclusion on that point.

Another analysis that can be made is the impact of the binder, for a given density and type of aggregate. No clear consensus exits on that point. While [9][44] mentioned that binder and, more specifically, the thermal conductivity of the binder, probably have little influence on the thermal conductivity of the composite, [45] found the opposite. In the present study, the comparison between C1-H, C2-H and HB-H, which have practically the same dry density, ranging from 410 to 470

kg/m^3, but which showed λ values oscillating between 0.09 and 0.155 W/(m.K), rather tends to the conclusion that the binder nature has a significant impact.

To conclude, the aforementioned results suggest that density is the parameter that influences the most the thermal conductivity. However, the aggregate type was found to have a certain impact, as well as the binder type.

3.3. Sorption isotherms

Adsorption-desorption isotherms are presented in **Fig. 5 and Fig. 6.** They are of type II according to IUPAC classification [46]**.** It is consistent with which is commonly observed in literature for bio-based materials [47][48] and for composites [49][50].

The comparison of the sorption curves can be made either with the water content expressed in kg/m^3 (denoted by w) or expressed in kg/kg (denoted by u). They are both linked together through the dry density of the sample (denoted by ρ_d):

$$w = \rho_d\, u = \rho_d \left(\frac{m - m_d}{m_d}\right) \qquad (2)$$

where m is the current mass of the sample and m_d its dry mass.

These two means of comparison do not exactly provide the same information. u is a specific value. It is useful to have the intrinsic curve of a material or a component. For example, to characterize bio-based granulates, it is better to use u than w. On the other side, the water content in kg/m^3 represents the actual water quantity stored in the material volume. This latter should rather be used in order to compare the hygroscopic potential of final products, since it is this latter which is involved in the mass conservation equations [51]. Let us underline that most of the comparisons in literature have been made with water content in kg/kg, which may induce to some ambiguities on the real water storage capacity of materials.

The specific adsorption-desorption curves on the aggregates are shown in **Fig 5B.** The values in kg/m^3 reported in the **Fig 5A** correspond to samples of loose aggregates of same apparent density to those used to estimate the thermal conductivity, vapor diffusivity and MBV of aggregates. Results are in range of what it is generally reported in literature even if moisture content at high hygrometry for sunflower pith was greater than that found by [14].

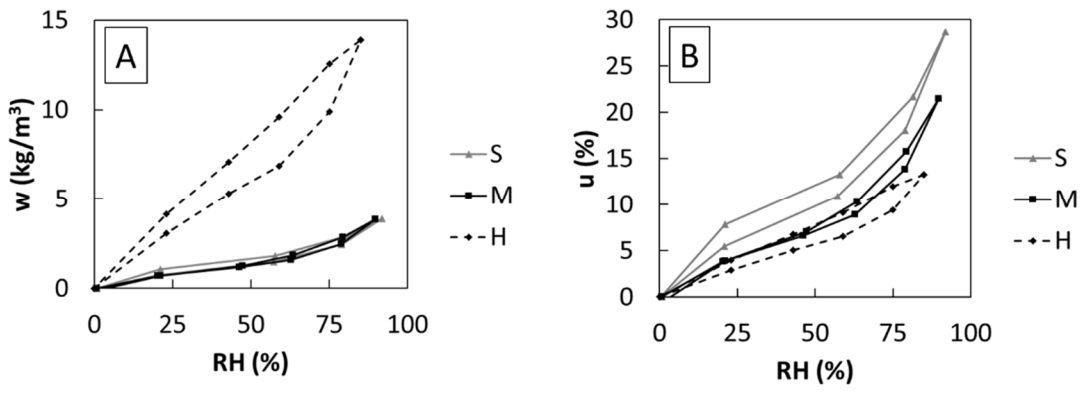

Fig. 5. Adsorption/desorption curves for the three bio-based aggregates with the water content in (A): kg/m^3, and (B): kg/kg.

In **Fig 5**, maize and sunflower aggregates exhibited a very similar behavior, with almost no hysteresis. On the other side, hemp shiv showed a lower specific water content and a more important, although limited, hysteresis. Due to its larger density, the **Fig. 5A** indicates that a volume of loose hemp shiv has the ability to store about three times more water than the same volume of loose sunflower or maize pith sample.

Fig. 6 shows the adsorption and desorption curves of bio-based composites. The order of magnitude obtained for moisture amounts at high hygrometry of composite are in the range of what is classically observed for this kind of materials. For example, [14] found for sunflower and maize composites a practically identical moisture capacity than the one obtained for HB-S and HB-M, as well as the same change in slope at 75% RH. Concerning hemp-based composites, C2-H*, C2-H, C1-H and HB-H give similar behaviors than the ones reported by [52] for sprayed hemp-lime composite, and by [53] and [54] for hemp-lime composite. In particular, the strong slope increase in the adsorption curve at 80% of RH and the significant hysteresis which can be observed in all the hemp-based formulations were also found by numerous authors [48][55][56][57][58].

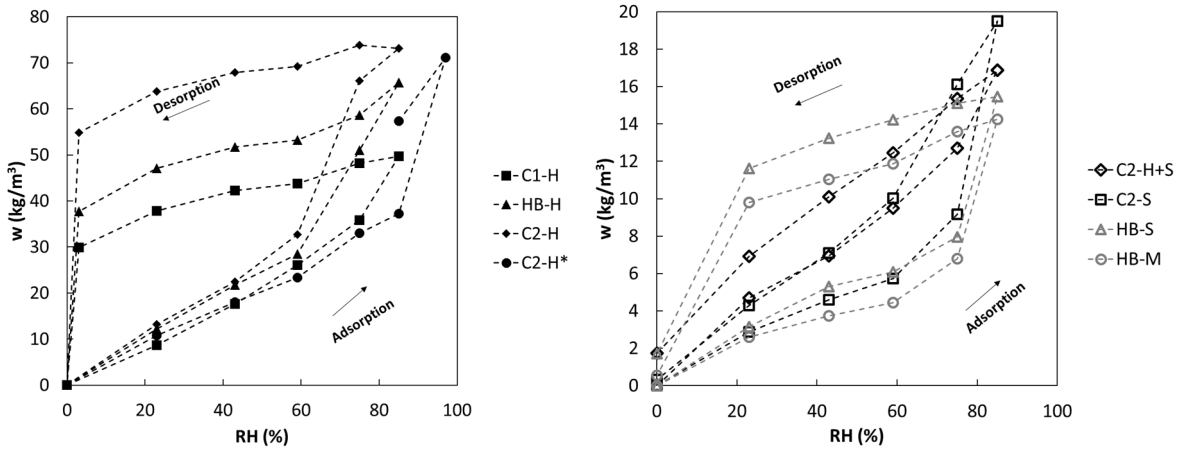

Fig. 6. Adsorption/desorption curves for the eight formulations. Left: hemp-based formulations. Right: Pith-based formulations and hemp+pith-based formulation.

The first remark that can be done on the sorption curves of composites is that the tendencies observed on the loose aggregates are maintained. Sunflower and maize piths-based formulations almost give the same results, with a water content at 80%RH between 13 kg/m^3 and 20 kg/m^3. On the other side, all of hemp-based formulations have a stronger water content at 80%RH, that oscillates between 37 kg/m^3 and 70 kg/m^3, and show strong hysteresis. These observations are all the more surprising that a lower value of water content was found for all the pith-based formulations, whatever their density. This first analysis suggests that the sorption curves are more impacted by the aggregate than by the binder.

Within each group (either pith-based or hemp-based formulations), the influence of the binder can however be appreciated, even if it is secondary compared to aggregates' one. Indeed, C binder allows a bigger water intake than HB binder, which at the same time allows a bigger water intake than C1 binder.

All these results indicate dissimilarities between the microstructures of the formulations. In particular, [59] suggest that hysteresis in lime composites is due to a double sorption mechanism. That is, a fast process that takes place at the surface of the solid and a slow process that consists in the integration of the water molecules into the solid matrix. This phenomenon is linked to cellulose based materials. The noticeable difference in the hysteretic behavior of hemp-based and pith-based composites may be a result of a difference in water exchanges dynamics at the micro-scale.

Realistic hygrometry cycles in buildings do not generally exceed 75% of RH and do not fall below 33% of RH. Therefore, it is reasonable to focus the study between these two values. Moreover, it seems more interesting to compare materials using one single parameter. Moisture capacity, denoted by ζ, is the parameter that expresses the capacity of the material to store the water molecules within itself. It is calculated as the slope of the adsorption ($\zeta_{adsorption}$) or desorption ($\zeta_{desoprtion}$) isotherms between 33 and 75% of RH. **Table 5** summarizes the moisture capacity of all isotherm curves.

In the bibliography aforementioned, which only includes adsorption curves, moisture capacity values go from 10.44 kg/m^3 [53] to 57.29 kg/m^3 [48], with [56] between both and throwing a value of 26.03 kg/m^3. Moisture adsorption capacity of HB-M, HB-S and C2-H* are in line with these results. Yet, C1-H, HB-H and C2-H present a very high capacity of water vapor storage during adsorption comparing to other existing materials.

A difference between the moisture capacity of adsorption and desorption can be noticed. For hemp-bases formulations $\zeta_{desoprtion}$ values are significantly lower than $\zeta_{adsorption}$ ones. The same result was found by [47]. For pith-based formulations, the difference between $\zeta_{desoprtion}$ and $\zeta_{adsorption}$ is within the range of uncertainty, and they can thus be considered as equal.

In view of these results we can notice that if strong differences exist on $\zeta_{adsorption}$ (between 136 kg/m^3 and 8 kg/m^3), $\zeta_{desorption}$ remains within a more limited range of values (between 22 kg/m^3 and 5 kg/m^3). The distinction that was observed on the sorption curves in function of the aggregate nature is clearly visible on $\zeta_{adsorption}$, but it is no more visible if the comparison is made on $\zeta_{desorption}$. For this latter, the distinction is rather between low density formulations, which have $\zeta_{desorption}$ values bellow 10 kg/m^3 and high density formulations which rather shows values between 16 kg/m^3 and 22 kg/m^3 (excepting values of C2-H).

Table 5 Summary of the hygric properties.

Type of samples	$\zeta_{adsorption}$ (kg/m^3)	$\zeta_{desorption}$ (kg/m^3)	μ (-)	$MBV_{practical}$ (g/m^2/%RH)	ζ_{ideal} (kg/m^3)	$\rho^*_{aggregate}$ (kg/m^3)	$\zeta^*_{aggregate}$ (kg/m^3)
C2-S	12 ±NA	15 ±NA	7 ±1.4	1.21 ±NA	6.7	0.05	23
C2-H	136 ±NA	8 ±NA	3.4 ±0.1	1.98±0.09	8.9	-	-
C2-H*	47 ±NA	20 ±NA	3.9 ±0.2	1.63 ±NA	6.7	0.15	10
C2-H+S	15 ±3	16 ±0.4	3.9 ±0.6	1.71 ±NA	7.4	**H:** 0.08 ; **S:** 0.03	15
HB-S	8 ±0.4	5 ±0.05	4.7±0.3	1.25 ±0.06	5.0	0.05	16
HB-M	10 ±0.5	7 ±0.5	5.1 ±0.6	1.35 ±0.03	5.7	0.04	13
C1-H	57 ±NA	18 ±NA	4.2 ±0.7	1.56 ±0.05	6.6	0.12	5
HB-H	92 ±NA	22 ±NA	4.0 ±0.3	1.60 ±0.07	6.7	0.11	5
Hemp shiv (H)	14 ±0.4	17 ±0.2	1.2 ±0.002	2.90 ±0.02	6.6	-	-
Sunflower pith (S)	3 ±0.2	3 ±0.1	1.0 ±0.006	2.60 ±0.03	4.4	-	-

3.4. Water vapor permeability

Wet cup test results are presented in the **Table 5** in terms of μ value, which is the resistance to diffusion of vapor within the material. μ value of loose aggregates was found to be close to 1. It means that, once the permanent state is reached, these latter have almost no impact on the vapor diffusion.

Composites present values between 3.4 and 4.9, except for C2-S, whose value is 7. These values are much lower than those observed by [10][12][48][55] and [57], which range from 5 to 12 for hemp, rape straw and flax composites. This difference with the literature is probably attributable to experimental conditions, which could have a significant impact on the values obtained. With the cup method, [60] studied the influence of different test parameters such as thickness and area of the specimen, thickness of the air gap at the lower surface of the specimen, air velocity, cup height, and hygrometry inside and outside the cup. Especially for hygroscopic materials such as bio-aggregate based building materials, the water vapor permeability is strongly linked to the local relative humidity. Thus, measurements of water vapor permeability, which are performed at different levels of relative humidity (dry-cup and wet-cup), may result in different values for one saline solution and the same material. The increase of the water vapor permeability with relative humidity was proved by [52] and [55]: the water vapor permeability diffusion coefficient increased by a factor of seven depending on the hygrometry. This may have been due to contribution of liquid water transport through the sample, which becomes noticeable at higher humidity values.

It is commonly assumed that, other parameters being equal, μ should increase with density (and thus decrease with porosity), which is consistent with the results obtained with the formulation C2-H and C2-H*. An increase of density from 1440 kg/m^3 to 1600 kg/m^3 led to an increase of μ from 3.4 to 3.9. This tendency is however no more observed when the binder-aggregate couple is changed.
More precisely, pith-based formulations show globally higher values of μ. The one of the formulation C2-S is twice that of C2-H* and C2-S+H, while their binder and their density are approximatively the same. In the same line, the μ-values of formulations HB-S and HB-M are in the same range as those of the hemp-based formulations, while their density is strongly lower. Actually, as it was already mentioned for the thermal conductivity measurements, morphology of hemp-based and pith-based composites are clearly different. As it is sketched in the pictures of the **Fig 7**, while open connected pores are obviously visible at the surface of the former, the latter exhibit a flatter surface with almost no asperity. Even if no direct link can be established between this visual observation and connectivity of samples' porous network, it clearly indicates a difference in microstructure between these two formulations.

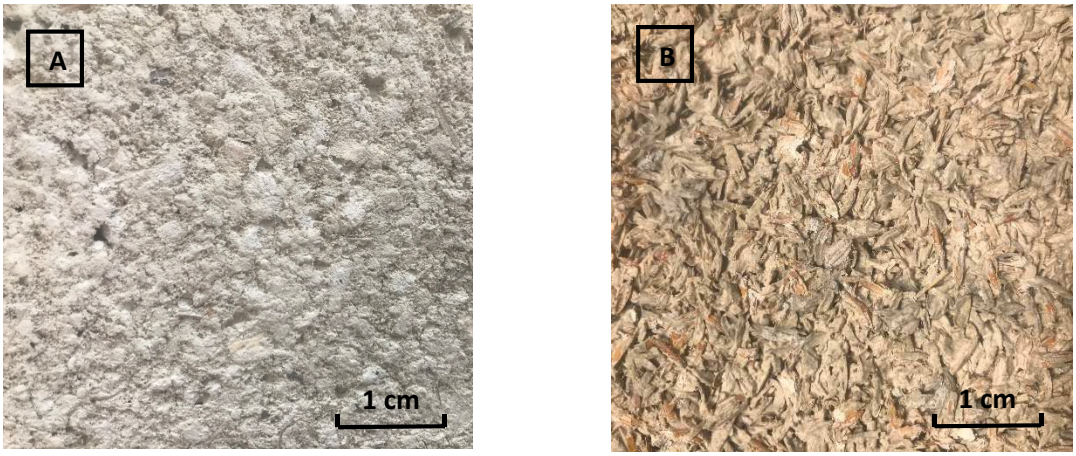

Fig. 7. Pictures of the surface texture of (A) pith-based formulations and (B) hemp-based formulations.

4. Discussion on the hygroscopic potential of pith-based composites

The moisture buffering value of aggregates and composites has been estimated following the NordTest protocol [33]. Results are show in **Table 5**. An example of the mass tracking curve, from which $MBV_{practical}$ is determined, of HB-S and HB-M is given in **Fig. 8**.

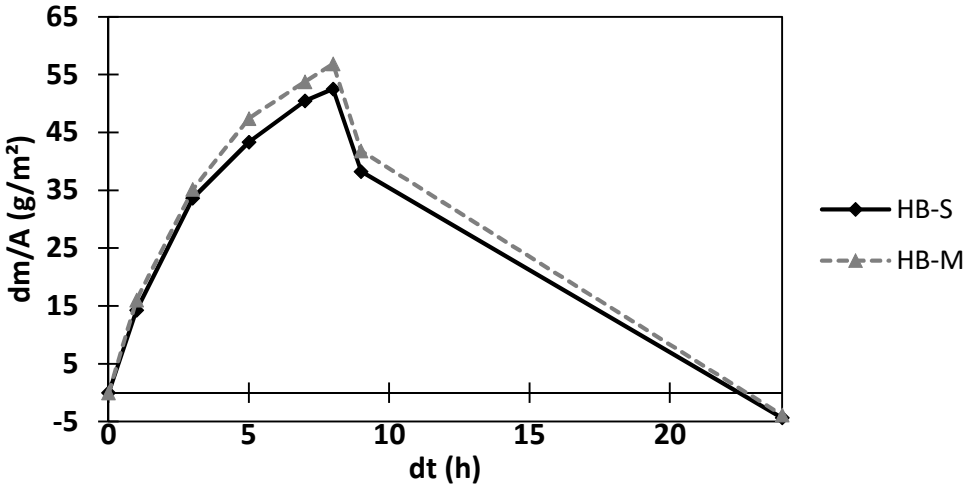

Fig. 8. Mass tracking of HB-S and HB-M during the MBV test.

The first remark that can be made is that the results can be divided in three groups. Loose aggregates have a moisture buffering value (MBV) higher than 2 g/m^2/%RH, which makes them materials with an excellent hygroscopic potential according to the classification proposed by [33]. The hemp-based and hemp+pith-based formulations have values ranging between 1.56 g/m^2/%RH and 1.71 g/m^2/%RH. Finally, the MBV of the pith-based formulations is rather between 1.21 g/m^2/%RH and 1.35 g/m^2/%RH. All these formulations can be classified as materials with a good hygroscopic potential.

This hierarchy on the hygroscopic potential is quite expected with regard to the results of both sorption and vapor diffusion properties of the tested materials. However, in view of the significant dissimilarities found on the sorption curves, a wider range of MBV may have been anticipated.

Actually, the storage capacity ζ is known to significantly influence the hygroscopic potential of materials [61]. However, as it is sketched in the results of the section 3.4, high uncertainty exists on this value since more than one order of magnitude of difference can be observed in some samples between the storage capacity in adsorption and in desorption.

[33] have developed an analytical solution in order to predict the MBV value in function of ζ and μ. The calculated value is called the MBV_{ideal}, while we recall that the measured one is denoted by $MBV_{practical}$. Another way to use this analytical solution can be to calculate what would be the storage capacity that would effectively lead to an MBV_{ideal} equal to the $MBV_{practical}$. This storage capacity will be noted ζ_{ideal} throughout the rest of this paper. At 23°C, the vapor pressure at saturation and the free vapor diffusion coefficient in air are respectively equal to 2814 Pa and 1.97 10^{-10} kg/(m.s.Pa). By application of the MBV_{ideal}'s formula with MBV_{ideal}= $MBV_{practical}$ and cycles duration of 24 hours, it leads to:

$$\zeta_{\text{ideal}} \approx 0.65\,\mu\left(MBV_{practical}\right)^2 \qquad (3)$$

In this relation $MBV_{practical}$ must be expressed in g/(m².%RH), while ζ_{ideal} is in kg/m³.

The obtained values are reported in the **Table 5**. The first remark that can be made is that for most of the formulations, both $\zeta_{desorption}$ and $\zeta_{adsorption}$ values (cf. **Table 4**) are above ζ_{ideal} ones. However while $\zeta_{desorption}$ remains in the same order of magnitude than ζ_{ideal}, $\zeta_{adsorption}$ values appear to be strongly higher. The same kind of results were found by [47] on hemp composite. It led to the recommendation to rather use cycles of relative humidity (or at least the desorption curves) than the adsorption curves to predict the hygroscopic potential of hemp composites. One reason of that could be that not all the water within the material is mobilized during the cycles of relative humidity. Thus, if the adsorption curve starts on a strongly dried material, some of the water that will be captured within the material microstructure will not be released during the desorption stage, unless it is subjected to severe drying conditions, that would not classically happen.

Interestingly, as it is sketched in the **Fig. 9,** $\zeta_{desorption}$ and ζ_{ideal} appear to be almost the same for loose sunflower pith and lighter formulations (HB-S and HB-M), while more important differences are observed for loose hemp shiv and denser formulations. It tends to indicate, that static characteristics (namely $\zeta_{desorption}$ and μ) are not sufficient to accurately reproduce dynamic hygroscopic behavior of hemp shiv and dense formulations of this study. This difference between static and dynamic hygroscopic characteristics has already been highlighted by [62]**.** To avoid this problem, the efficiency of the aggregates within composites will be studied through the ζ_{ideal} values, since they are the ones which are linked to the dynamic behavior of the material.

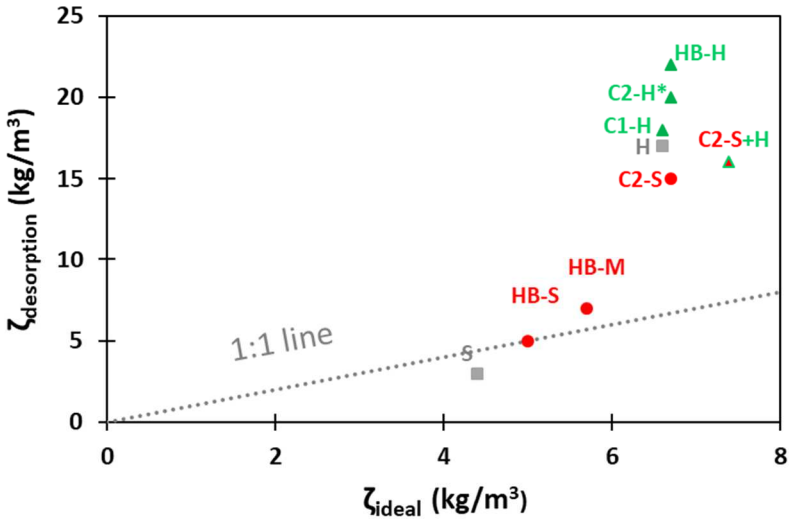

Fig. 9. Adsorption coefficient estimated from the desorption curve in function of the one calculated from $MBV_{practical}$ through the eq. (3).

Mass proportions of the aggregates in mixtures, denoted by $p_{aggregate}$, are indicated in the **Table 1.** From this value, it is possible to know the mass of aggregate per unit of composite samples volume, denoted by $\boldsymbol{\rho^*_{aggregate}}$, through the relation:

$$\boldsymbol{\rho^*_{aggregate}} = \frac{m_{wet}}{V}\, p_{aggregate} \qquad (4)$$

where m_{wet} is the mass of the composite sample just after mixing, while V is its volume. Values of $\boldsymbol{\rho^*_{aggregate}}$ obtained for the different formulations are reported in the **Table 5.** For hemp-based formulations, $\boldsymbol{\rho^*_{aggregate}}$ remains quite close to the loose density of hemp shiv (between 110 kg/m³

and 150 kg/m^3 for the former and 100 kg/m^3 for the latter). It indicates that the binder has filled the gaps between hemp particles without significantly modifying their geometrical arrangement. On the opposite, $\boldsymbol{\rho^*_{aggregate}}$ of pith-based formulations are about four times higher than loose density of sunflower pith (between 40 kg/m^3 and 50 kg/m^3 for the former and 14 for the latter). In consequence, this probably indicates a compaction of the pith particles during manufacturing of the samples, the pith is a rather soft material that can be easily compressed, compared to the harder hemp shiv.

The ideal storage capacities of the aggregates (either hemp shiv or sunflower pith) which are calculated with the eq. (5) and whose values are reported in the **Table 5,** will be noted $\zeta^{ideal}_{aggregate}$. They can be understood as the dynamic storage capacity of loose aggregate samples. For a given aggregate microstructure, it should vary linearly with apparent density. In consequence, if aggregates are not subjected to any microstructure modification nor interaction with the binder, their contribution to the dynamic storage capacity of the composite will be equal to:

$$\zeta^{*}_{aggregate} = \frac{\boldsymbol{\rho^*_{aggregate}}}{\rho_{aggregate}} \zeta^{ideal}_{aggregate} \qquad (5)$$

$\zeta^{*}_{aggregate}$ has been calculated for all the formulations and the results are shown in the **Table 5**. For hemp-based formulations, $\zeta^{*}_{aggregate}$ is very close to the dynamic storage capacity of the composite (that is the $\boldsymbol{\zeta_{ideal}}$ of the composite). It denotes that hemp shiv aggregates play the main role in the dynamic hygroscopic behavior of these formulations and that the binder does not alter significantly their hygroscopic potential. On the opposite, $\zeta^{*}_{aggregate}$ values are clearly higher than $\boldsymbol{\zeta_{ideal}}$ ones for pith-based formulations. This observation is another proof of the strong interaction between the binder and the pith, that alters their hygroscopic potential. It is consistent with the differences in behavior which were observed for pith-based formulations for both thermal conductivity and vapor diffusivity. All these results converge to the fact that when they are mixed with the binder, sunflower and maize pith aggregates undergo quite important microstructural transformations, which limit their contribution to both thermal insulation and moisture buffering potential.

In consequence, one challenge for the optimized use of this kind of aggregate might be the development of dedicated binders which would limit these interactions while keeping acceptable mechanical strength.

Anyway, despite this observation, mechanical, thermal and hygroscopic performance of the tested pith-based composites remain quite close to hemp-composite ones. In addition, this kind of granulate was found to allow the fabrication of low-density insulation composite with a quite competitive thermal conductivity and hygroscopic performances. Thus, even if more researches need to be conducted in order to optimize their use, pith aggregates appear to be particularly promising.

5. Conclusion

Bio-based building materials, namely vegetal composites, are very promising for modern construction since they are eco-responsible due to their lower embodied energy and to the possibility of local production. The current challenge is to determine the formulations that present interesting mechanical and hygrothermal qualities while respecting environmental and economic criteria. Numerous pieces of research work have studied hemp composites in the last few years, but other aggregates such as sunflower and maize pith are little known. The interest of studying new aggregates is to propose greater geographical availability of resources and this way reduce the carbon print linked to transportation.

In this paper, vegetal composites made with three kinds of binder mixed with hemp shiv, sunflower pith, are/or maze pith are studied. Even if these bio-based composites cannot be used in load-bearing members of the building, some of them qualify to be used as plaster, as a wall, as floor or as a roof according to French hemp construction guide (HB-H, C2-H+S and C2-S) whereas others qualify as roof material (HB-S and HB-M). This proves that pith composites can have the same applications as hemp composites. The mechanical resistance of vegetal composites has been found to be proportional to density, as well as thermal conductivity. Moreover, it has been observed that thermal conductivity is also influenced more moderately by the type of aggregates and the type of binder.

In this study, some composites show remarkable hygrothermal characteristics, particularly those based on hemp shiv (C2-H, C1-H and HB-H). Thus we can conclude that the type of aggregate has a considerable impact on these properties. Hysteresis is present for all the formulations as it is commonly observed in bio-based materials. The study of the MBV is more accurate when calculated from the cycles of RH rather than from the sorption curves, given that the cycles represent more closely the dynamic behavior.

For all the aforementioned properties, it has been noticed that in most cases, the behavior of the materials cannot be properly explained by the aggregate type or the binder type. On the contrary, interactions between binder and aggregate has been observed, namely chemical interactions, a transformation of microstructure and the immersion and isolation of aggregate particles. The latter two seem to be stronger underlined for pith composites.

To conclude, pith composites studied in this paper met mechanical and hygrothermal requirements expected for insulating bio-based materials. However, possibly due to the interactions between the binder and pith particles, the hygrothermal potential of these latter within the composite seemed to be lowered. This result suggests that it should be interesting to search for dedicated binders for pith aggregates that enhance the hygroscopic and mechanical properties of the composite.

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