



INFLUENCE OF ORIGIN AND YEAR OF HARVEST ON THE PERFORMANCE OF PITH MORTARS

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Abstract

The plant aggregates used for the production of bio-based mortars and concretes, present a natural variability due to their geographical origin, climatic conditions and the year of harvest. This variability might be a problem for the promotion of such materials if it significantly impacts the performance of the final product. To investigate such influence, five pith aggregates of the same kind but from different regions in France were used to prepare bio-based mortars. The impact on the mechanical performance have been evaluated through unconfined compression test. Hot wire thermal conductivity, moisture adsorption/desorption isotherm, moisture permeability and Moisture Buffer Value (MBV) tests were used to evaluate the hygrothermal behaviour. Results show no major difference in the mechanical resistance of the pith mortar, in fact the drying procedure showed to have a stronger influence. Only slender differences were observed for hygrothermal properties.

Keywords:

Bio-based mortar; Hygrothermal; Pith; Compression test

1 INTRODUCTION

The use of bio-based materials is an increasingly adopted solution in the field of construction. Indeed, as any other field (transport, aeronautics, etc.), this sector must deal with the issue of greenhouse gas emissions, as well as the scarcity of raw materials, the problems of recycling and sustainable development. At present, the use of plant-based products as a raw material is a solution that is booming in many industries. In the field of construction, aggregates of mineral origin are substituted by aggregates of plant origin. We can cite as an example, sunflower grinds or wood, and even more widely the use of hemp.

Due to the low density and high porosity of annual plant stems, the combination of lignocellulosic aggregates and a cementitious binder creates a building material with properties that differ from those of conventional concrete. It has a lower density, lower thermal conductivity and higher permeability [Nozahic & Amziane, 2012].

Globally, the use of renewable, recyclable and sustainable plant aggregates can be a way to drastically reduce environmental impacts of the building sector [Collet, 2004, El Hajj, 2010, Van Schoubroeck et al., 2018]. In order to valorise these materials for construction, repeatability of performance is essential. This point gave rise to many research works [Collet 2004, Cerezo 2005, O'Dowd & Quinn, 2005, Arnaud et al., 2006, Arnaud 2000, Evrard 2008, Tran Le et al., 2015, Marceau et al., 2017]. They lead to the conclusion that both mechanical and hygrothermal performance of

bio-based concretes and mortars are significantly influenced by the dosage in binder and by the nature of the bio-aggregate, even if no clear relation still exists in order to link its characteristics and bio-based mortar performances. For example, in some studies, finer particle where found to increases both mechanical strength and thermal conductivity [Cigasova et al., 2013]. This can be attributed to narrower particle settlements [Arnaud et al., 2013]. On the other hand, others have found that a coarser particle quality gives better mechanical properties, attributed to a greater overlap of particles [Ceyte 2008]. One of the possible reasons for the disagreement of these conclusions may be the small fluctuations in the reported properties compared to the relatively large natural variation.

One other source of variability of bio-based mortar may be the influence of the geographical origin and the harvesting period of plant aggregates.

This study was motivated by the lack of available data on that point, in particular concerning one promising type of bio-based material which is the pith mortar.

For that purpose, 5 varieties of pith were analysed. These piths differ in their geographical origin and also in the harvest period, but have similar specifications in term of their main physical properties which are the apparent density and the particle size distribution. Indeed, the impact of these two last points on compressive strength and thermal conductivity have already been established [Williams et al., 2018; Magniont et al., 2012].

As a starting point, the choice is made to investigate the mechanical properties in order to check if the tested materials are compatible with the specifications of the French hemp building guide of best practice. Thereafter, the impact of pith origin on hygrothermal properties, which are water vapour permeability, sorption capacity, moisture buffering value and thermal conductivity, were determined. The interpretation of the results can contribute to a better insight of the behaviour and the formulation of bio-based insulating mortars.

2 MATERIALS AND METHODS

2.1 Materials

Aggregates

As part of this experimental campaign, two types of bio-sourced aggregates were studied: sunflower and maize pith. Five different types of pith were selected; the D2016 and D2017 are sunflower piths from the same region in the south east of France but which differ in the year of harvest. Two further sunflower piths referenced as O and G originate from the west of France with more oceanic climates. The maize pith is referred as M in this document. O and M were from the harvest of 2016 and G from 2014.

A high fibre content leads to a reduction in number of contact points between the pith particles, with a decrease in adhesive properties and a loss of mechanical strength. It is, therefore, preferable to use a pith as pure as possible [Marechal & Rigal, 1999]. Even if the aggregates used are different in their extraction mode and preparation, they all meet the same specifications in term of bulk density and particle size distribution. A variability of the size of the grains can be observed in the marrow sac but in our case of study this point is not taken into account, because the properties of all the aggregates used were checked before carrying out the mixtures.

Bio-based concretes

The same binder formulation was used for the manufacturing of each insulating mortar. The specimens were prepared using a hydraulic binder only with water and the aggregates. In order to determine the physical characteristics such as the skeletal porosity, dry and skeletal densities of the five insulating mortars, the pycnometer was used according to standard NF EN 1097-7 2008 and the results are summarized in the table 1.

Tab. 1: Summary of the physical characteristics.

Characteristic	D2017	M	O	D2016	G
Porosity	0.92	0.92	0.93	0.94	0.92
Dry density (g/cm ³)	0.18	0.16	0.16	0.15	0.18
Skeletal density (g/cm ³)	2.35	2.04	2.37	2.41	2.21

The values in table 1 show the very low apparent density of the mortars, while the density of the skeleton obtained is in line with that usually observed for bio-sourced concrete. So it is found a much greater porosity than that measured on hemp mortars, for example [Aït Oumeziane et al., 2014].

2.2 Methods

Uniaxial compression test

The mechanical resistance was studied on cylindrical specimens (16x32cm). Three series of tests were carried out, each series contained 5 types of concretes in 3 copies which makes a total of 15 test pieces per series. Two deadlines of crushing were chosen 60 and 90 Days.

For 60 days, two series have been tested with two different drying modes:

- Drying 1: demoulding at 28 days, drying at 50°C to constant mass and storage at 30(±5)°C and 60(±5) % RH until tested.

- Drying 2: demoulding at 28 days and kept at 30(±5)°C and 60(±5) % RH until tested. For 90 days, only type 2 drying has been done.



Fig. 1: Simple compressive strength Test.

The simple compression tests were carried out according to the protocol defined in the professional rules 'Building with hemp'. In addition to the compressive strength (R_c), the global stiffness modulus was evaluated for all samples.

Thermal conductivity (hot wire method)

The thermal conductivity was measured using the hot wire apparatus. Cylindrical specimens 16*32cm were cut after 21 days of manufacture in small cylindrical samples (16*6cm), which allowed us to obtain 3 samples per mortar mixture. These were stored in a room at about 30(±5)°C and 60(±5) % RH for air drying and were tested after 100 days of curing.

Sorption Isotherms (dynamic vapour sorption method)

The sorption tests are conducted in compliance with standard NF EN ISO standard 12572: 2000. It was made possible to draw the representative sorption curve for a given temperature, the variation of the water content of the material as a function of the relative humidity of the ambient air. This is defined by formula (1):

$$\omega = \frac{m - m_0}{m_0} \quad (1)$$

With m_0 the mass of the sample in the dry state using a dry air flow of less than 5% RH. 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 humidities of 23%;

49%; 59%, 75%; 85% following an adsorption stage and then a desorption stage.

In this experimental campaign, the automated DVS system was used, for which a specific sample holder was realized to maximize the quantity of pith during the measurement.

Water vapour permeability (wet cup method)

The water vapour permeability was measured in this study according to the standard ISO-12572, 2001 using the Wet Cup method. It consists of generating a vapour pressure gradient by adjusting the relative humidity to 50% in the climatic chamber and 85% in the cup. The relative humidity level in the cup is maintained using saturated saline solution of potassium chloride and the space between the saline solution and the bottom of the sample called the air layer was 20mm for all samples in accordance with ISO 12572 (15±5mm). The water vapour permeability and the water vapour resistance can be determined from the rate of water vapour transmission through the sample. The design of the cups was performed according to the procedure followed by [McGregor et al., 2014]. Therefore, a thin layer of silicone was applied to seal the samples on Plexiglas cups. A vapour-tight aluminium tape was used to seal the sides of the sample with the side of the cup. The use of aluminium tape is justified by its properties: it is impervious and does not absorb moisture itself [Svennberg K., 2006]. Environmental conditions (50% RH and 23°C) were constantly monitored by the climatic chamber.

Dynamic moisture exchange behaviour (Moisture Buffering Value)

In order to quantify the hygroscopic potential of a porous medium a common method exists. It 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) [Rode et al., 2005]. The tested sample is isolated on all sides by aluminium scotch, except the upper side, and the moisture buffering value (or MBV) is calculated as the mass variation per unit area (denoted A) according to the formula (2):

$$MBV_{practical} = \frac{\Delta m}{A \cdot \Delta RH} \quad (2)$$

With Δm (g) the mass variation during the cycle and $MBV_{practical}$ ($g/(m^2 \cdot \%RH)$).

Cylindrical specimens 16*32cm were cut beforehand to make 3 identical samples of size 16*6cm for each type of insulation, which makes us a total of 15 test pieces. The mass monitoring was carried out with an interval of 1h then 2h until reaching the max of the cycle and then a last measurement after 24h of the beginning of the cycle. The MBV is calculated when the regime is stabilized, i.e. when the mass difference between the beginning and the end of a cycle is less than 5%.

3 RESULTS AND DISCUSSION

3.1 Results

Uniaxial compressive strength

The results of the uniaxial compression test are reported in the table 2a and 2b. For each value three uniaxial compression tests have been performed. The apparent global stiffness modulus (E) is estimated on the linear part of the axial stress - axial strain curve.

Compression tests at 28 days were done to estimate the evolution of the mechanical strength during the hydration stage of the binder.

Tab. 2a: Summary of average compression strength.

Rc (kPa)	D2016	D2017	M	G	O	
Normal Cure	28 Days	95.5	-	-	-	
	60 Days	225.4	270.7	237.9	241.4	279.4
	90 Days	292.5	260.2	226.3	227.9	285.3
Dry Cure	60 Days	163.5	225	205	173.8	252.5

Tab. 2b: Summary of average of stiffness modulus (E).

E (MPa)	D2016	D2017	M	G	O	
Normal Cure	28 Days	-	-	-	-	
	60 Days	10.4	8.5	9.3	9.6	8.6
	90 Days	15.9	7.9	7.5	9.6	8.8
Dry Cure	60 Days	9.5	7.9	9.6	6.9	10.8

Thermal conductivity

The results of the thermal conductivity are shown in the table below represent the average of the 3 measured values.

Tab. 3: Thermal conductivity of studied materials.

Designation	D2016	D2017	M	G	O
λ (mW/m·K)	65	83	75	74	63.7

It thus appears that all the thermal conductivities of the pith mortars measured during this study are between 0.063 and 0.083 W/m·K. It is important to recall that measurements were made using the hot wire method. This technic is known to overestimate the thermal conductivity of bio-based materials. One reason of this trend could be that the size of the material

heterogeneities (size of aggregates and pores) is too large compared to the zone scanned by the hot wire. It leads to an underestimation of the thermal insulation contribution caused by the porosity of the material.

Anyway, it is assumed that the hot wire method can be used for comparison purpose.

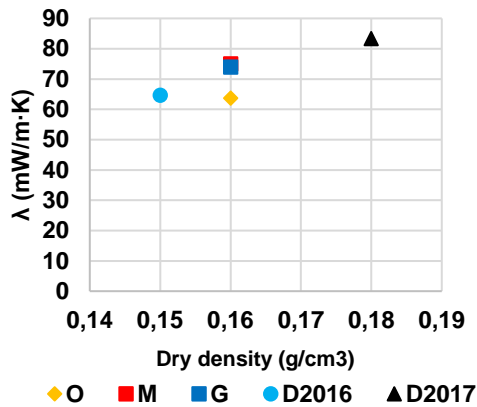


Fig. 2: Variation of thermal conductivity function of dry density of the 5 mortars.

As it is sketched in Figure 2, whatever the aggregate used, thermal conductivity values remain quite close and seem to follow the same variation with dry density.

Sorption Isotherms

The adsorption-desorption curve was plotted for the 5 mortars. The figure 3 shows the results.

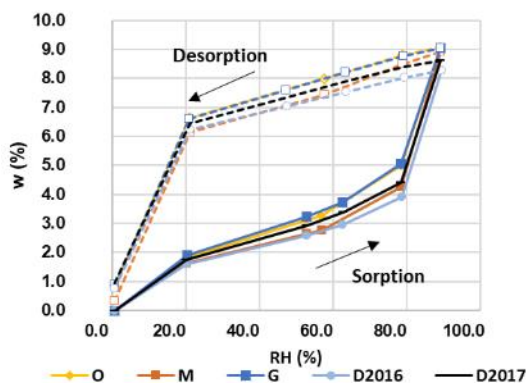


Fig. 3: Adsorption curves (solid lines) and desorption (dotted lines) for the 5 mortars.

All the adsorption-desorption curves obtained using DVS had a similar form even though there were slight differences in the amounts of water absorbed by the five concretes.

Hysteresis can be observed between the adsorption and desorption isotherms. This difference between the curves of adsorption and desorption has already been studied in hemp concrete [Andy et al., 2012; Collet et al., 2012; Evrard et al., 2005], in wood [Merakeb et al., 2008], in sago starch films [Bajpai et al., 2011], in concrete [Baroghel-Bouny, 2008] and in bentonite [Mihoubi & Bellagi, 2006]. The evolution of the adsorption and desorption value from the DVS method versus the relative humidity of the 5 concretes is shown in Figure 4.

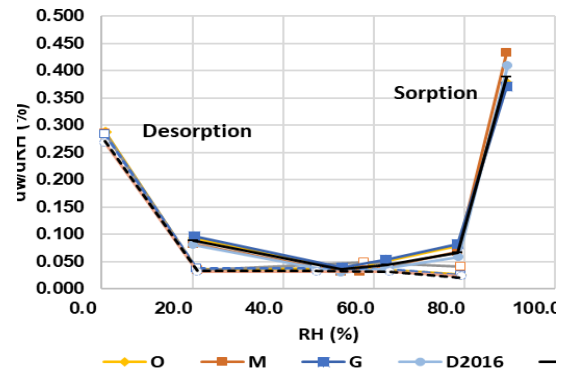


Fig. 4: derived from the adsorption curves (solid lines) and desorption (dotted lines) for the concretes.

It appears that the adsorption slope depends on the relative humidity. Knowing that realistic hygrometry cycles in buildings do not generally exceed 85% RH and do not fall below 23% RH, an average slope can be determined in this range for experimental results. These results are presented in table 4.

Tab. 4: Average storage capacity between 20% RH and 60% RH.

Designation	G	D2016	O	D2017
ξ (-)	0.043	0.032	0.040	0.038

Water vapour permeability

From Fick's law of diffusion, the water vapour permeability is deduced, according to EN ISO-12572. The gross value obtained was corrected following the protocol proposed by [McGregor et al., 2017] to account for the effect of the surfaces vapour resistances.

Figure 5 shows the mass variations of the total set consisting of plastic cup, saline and maize sample as a function of time. Similar behaviour of the three tested samples belonging to the same formulation is observed. All samples reached a steady state after a single day. During this steady state stage, with a homogenous vapour pressure gradient, a linear relation between the vapour flux and pressure gradients allows to calculate the apparent water vapour permeability of the material.

MAIZE (M)

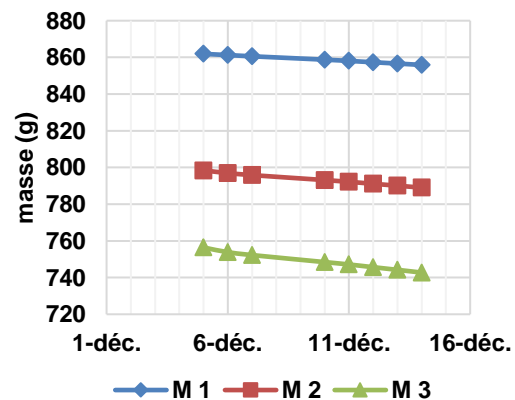


Fig. 5: Mass variation of the Wet Cups for maize samples.

Once the slope of the regression line is determined from the results of the wet cups, the water vapour permeability δp (kg/s·m·Pa) of each sample can be deduced using the approach described in [McGregor et al. 2017]. The vapour resistance factor (μ) can be determined as it is often used rather than the water vapour permeability. It is the ratio between the water vapour permeability of the air (δa) and the material (δp):

$$\mu = \frac{\delta a}{\delta p} \tag{3}$$

The results are shown in the table 5.

Tab. 5: Water vapour diffusion resistance factor, μ value.

Designation	G	D2016	O	D2017	M
μ	5.5	4.9	5.9	4.7	4.8

Moisture buffering value

The figure below shows the mass variations recorded for the last cycle of the moisture buffering test with cycles of 8h at 75% RH and 16h at 33% RH. These variations were normalized by the exposed surface.

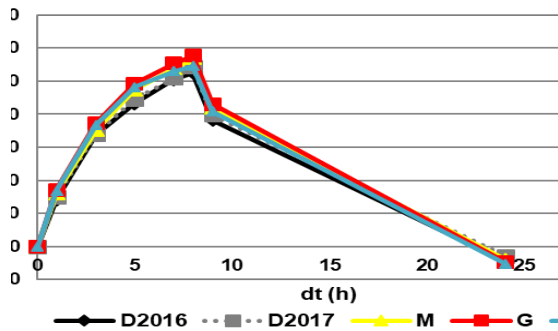


Fig. 6: Mass evolution of the samples during the MBV Cycle.

Using the practical MBV definition given on NordTest the water buffer value is measured for each insulator and the results are summarized in Figure 7.

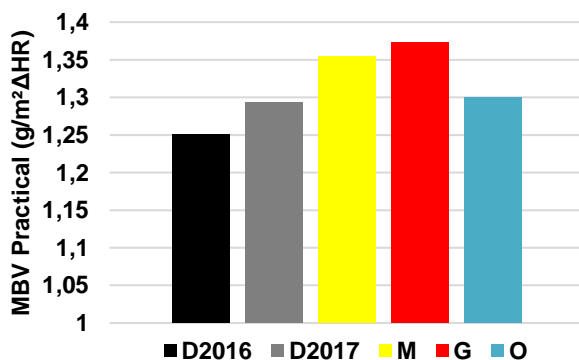


Fig. 7: Average MBV obtained for different pith based mortars.

The moisture capacity, ξ , can be determined experimentally using the pseudo-linear section of the sorption isotherm which is in the middle range of the isotherm [Hall H. & Allinson D., 2009]. This latter

indicates the moisture content adsorbed by the material to reach equilibrium with the vapour pressure of the surrounding environment.

From this point, and to verify the validity of the corrected vapour permeability and the practical MBV obtained previously, we decided to use them in order to estimate the moisture buffering value by an indirect analytical calculation. Thereafter, our analytical result will be compared with the experimental one determined in this study.

The analytical solution for the MBV test has been developed in [Rode et al., 2005]. It is noted MBV ideal. It provides the estimation of the moisture buffer value using equation (4), where t_p is the period corresponding to moisture absorption (in hours) and b_m is the moisture effusivity (kg/m³):

$$MBV_{Ideal} \approx 0.00568 P_{v,sat} b_m \sqrt{t_p} \tag{4}$$

The moisture effusivity constitutes one theoretical possibility to express the rate of moisture absorbed by a material when it is subjected to a sudden increase in surface humidity, and it's linked to the water vapour permeability and the moisture capacity of the material as follow:

$$b_m = \sqrt{\frac{\delta p \cdot \xi}{P_{v,sat}}} \tag{5}$$

Using the results of the DVS we get ξ , which represents the slope of the stagnant phase of the sorption curve. We thus obtain:

Tab. 6: MBV measured and calculated for the 5 studied formulations.

Aggregate	D2016	D2017	M	O	G
MBV measured (g/m ² /%RH)	1.25	1.29	1.35	1.37	1.3
MBV _{ideal} calculated (g/m ² /%RH)	1.14	1.37	1.18	1.31	1.24

3.2 Discussion

Regarding the first part of the study, the mechanical results, the harvesting period doesn't have any effect on the strength while the geographical origin has a low impact on the global stiffness modulus. As first glance, it can be seen that the material studied is compatible with the specifications of the French hemp building guide of best practice to use only on the roof. Whatever the formulation, it is obvious that the value of compressive strength increases significantly between 28 days and 60 days in all cases, then slightly between 60 days and 90 days. This behaviour is similar to hemp concrete one's, as concluded in [Gross et al., 2014]. In addition, it can be seen that the drying mode has an influence on the mechanical properties with a drop in strength of between 10% and 30% for the samples having been dried at 50°C. The global stiffness moduli are also influenced by the drying method: a slight decrease in stiffness modulus is observed, except for maize (M) and O, for samples that have been dried at 50°C. It can be concluded that the conditioning protocol plays an important role in the measured performance of the material. Hence it appears that it is necessary to define a curing protocol adapted to pith mortars, which

would be consistent with on-site's conditions. According to [Zadeh & Bobko, 2014], the curing at elevated temperature will intensify the exchange of water between bio-based aggregates and binder matrix, which will deteriorate the concrete bond associated with the formation of a more porous transition zone and a worse interlock between the paste and the aggregate.

Concerning thermal conductivity, the obtained values are almost the same but a variation with the dry density of the samples is observed. This variation seems however independent of the origin and year of harvest of the aggregates. Note that the tests were performed at $T = 27(\pm 1^\circ\text{C})$ and $60(\pm 0.1\%)$ RH. These conditions are closer to the real conditions of construction than if the material was completely dry. According to [Mazhoud et al., 2017], the thermal conductivity changes with the temperature of the environment and also with the water content of the material. Therefore, it would be interesting to pursue the experimentation to confirm this premise on the studied materials. To compare with other materials studied in the literature, lime-hemp concrete presents low thermal conductivity as well and is very sensitive to the presence of water vapour, its thermal conductivity being proportional to the relative humidity. The thermal conductivity values are $0.105 \text{ W/m}\cdot\text{K}$ for dry sample (15% RH) and $0.116 \text{ W/m}\cdot\text{K}$ when the relative humidity reaches 65% for the same material that has the density ranging from 830 to 880 kg/m^3 [De Bruijn & Johansson, 2013]. The lower values of thermal conductivities obtained in our study can be explained by the lower dry density of our samples (between 150 and 180 kg/m^3).

As for the sorption isotherms, the hysteresis observed in Figure 3 on the bio-based materials for a relative humidity of 80% is between 3.5 and 4%. This quite important hysteresis loop, which is commonly observed for bio-based materials [e.g. Fabbri & McGregor, 2017], can be notably explained by the independent domain approach developed by [Derluyn et al., 2012] and which assumes that relative humidity at which adsorption and desorption processes occur on the sorption sites within the material depend on their geometry and their chemical composition.

It is interesting to note that the five mortars had very similar hysteretic behaviour. Also, Table 4 shows that the storage capacity doesn't depend on the aggregate type.

As can be seen in Table 5, water vapour resistances of D2016 and D2017 samples are almost identical despite the difference in the harvest year, which is also comparable to maize value, while G and O have a little more resistance.

From the results for Moisture Buffering Value (MBV), it can be noted that the variability of the aggregates has no significant influence on the value of the water buffer, even if different harvesting methods are used with different campaigns as well as different types and sources of aggregates. The average of the buffer values of the pith varieties is $1.248 \text{ g/m}^2\cdot\Delta\text{RH}$ and the standard deviation does not exceed $0.0736 \text{ g/m}^2\cdot\Delta\text{RH}$, which represents around 6% of the average. This result makes sense given that the MBV ideal is calculated from the storage capacity (ξ), which varied scarcely according to the sorption isotherms results. To compare with other common building materials, the classification proposed by [Rode et al., 2005], which defines five ranges from negligible to excellent for practical Moisture Buffer Values, was used. This classification of the materials

studied (obtained with samples of the same thickness of 7 cm) in comparison with other materials tested in the NordTest project, shows that our materials have a "good" moisture buffer performance.

4 CONCLUSION

The objective of this study was mainly to investigate the impact of geographical origin and year of harvest of plant aggregates on the mechanical and hygrothermal properties used for light-weight insulating mortars.

The exploitation of the results obtained makes it possible to conclude that the variability of the aggregates has a limited impact on the mechanical and hygroscopic performance of the insulating concrete at material scale.

At the end, additional tests still need to be realized in order to reach complete assessment of the impact of aggregates origin on the bio based concrete.

Finally, in order to properly predict the behaviour of our material with respect to its durability, a multi-scale numerical simulation will be made on the basis of the results obtained in this experimental study to assess the impact of the proposed materials on energy building consumption as well as hygrothermal comfort of occupants.

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