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Impact of phase change materials on lightened earth hygroscopic, thermal and mechanical properties

Farjallah Alassaad¹, Karim Touati¹, Daniel Levacher², Nassim Sebaibi¹

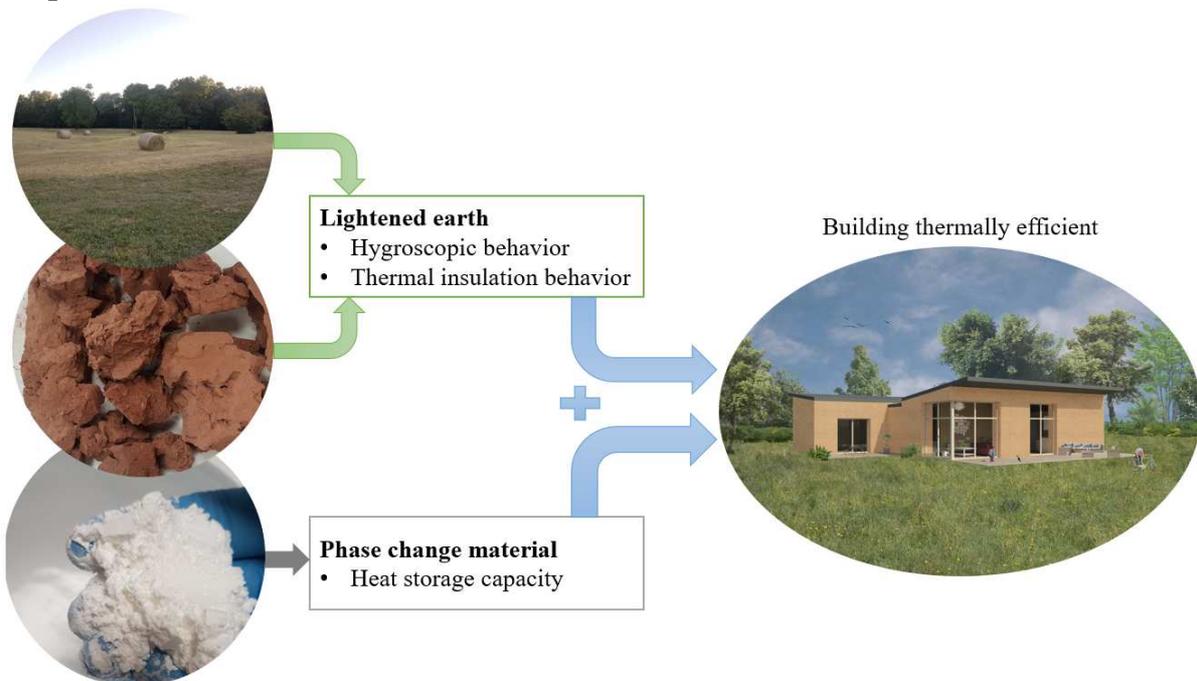
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Highlights

- Introduction of phase change materials (PCM) into lightened earth.
- Improvement of lightened earth thermal properties with PCMs.
- Degradation of lightened earth hygroscopic properties with PCMs.
- Addition of PCM has no significant effect on lightened earth mechanical strength.

Graphical abstract



Abstract

Climate change is leading construction engineers and building stakeholders to improve materials thermal insulation and heat storage capacity. The development of energy efficient building materials with a low environmental impact is a real challenge. In this sense, earth-based materials are known worldwide and have been used since thousands of years. These materials present many advantages but their deployment on a large scale, while respecting the operative standards, is still limited.

Considering lightened earth, present work aims to improve their thermal properties by using phase change materials (PCM). The incorporation of PCM in construction materials has aroused great interest due to their capacity to store thermal energy.

In present study, mechanical, hygrometric and thermal experimental studies were carried out on soil-fiber-PCM mixtures. The PCM incorporation showed an interesting improvement of the lightened earth thermal properties and a deterioration of the hygrometric properties while keeping their mechanical properties relatively unchanged.

Keywords: building insulation, heat storage, light earth, phase change materials, thermal properties.

Nomenclature

d_a	air layer thickness	[m]
e	sample thickness	[m]
g	vapor flux density	[kg.m ⁻² .s ⁻¹]
p_0	accessible porosity	[%]
t	time	[s]
A	specimen exposed area	[m ²]
C_p	specific heat capacity	[J/(kg.K)]
F_s	soil mass fraction	[-]
F_f	fiber mass fraction	[-]
F_{PCM}	PCM mass fraction	[-]
G	water vapor flow	[kg.s ⁻¹]
LL	soil liquidity limit	[%]
MBV	Methylene Blue Value	[g/100g]
M_{oil}	mass of saturated specimen in oil	[kg]
M_{air}	mass of saturated specimen in air	[kg]
M_{dry}	mass of dry specimen	[kg]
PL	soil plasticity limit	[%]
PI	plasticity index	[%]
RH	relative humidity	[%]
T	temperature	[°C]
W	water vapor permeance	[kg.m ⁻² .s ⁻¹ .Pa ⁻¹]
W_c	corrected water vapor permeance	[kg.m ⁻² .s ⁻¹ .Pa ⁻¹]
Z_a	air layer water vapor resistance	[m ² .s.Pa/kg]
Z	water vapor resistance	[m ² .s.Pa/kg]
ΔP_v	difference in water vapor partial pressure	[Pa]
δ	sample water vapor permeability	[kg.m ⁻¹ .s ⁻¹ .Pa ⁻¹]
δ_a	water vapor permeability of air	[kg.m ⁻¹ .s ⁻¹ .Pa ⁻¹]
\emptyset	diameter of the cylindrical samples	[cm]
λ	thermal conductivity	[W/(m.K)]
μ	water vapor resistance factor	[-]

1. Introduction

Face to global warming and its consequences, governmental authorities and NGOs have adopted regulations to reduce energy consumption and consequently CO₂ emissions. It is known that construction field is one of the major sectors emitting greenhouse gases. It should be noted that building engineering accounts for 40 % of the world's energy consumption, a large part of that consumption being devoted to heating [1]. Specifically in France, the tertiary sector, which includes the residential house is responsible for around 45 % of total energy consumption and 21 % of CO₂ emissions. Heating accounts for about 60 % of the total energy consumption in housing [2].

As part of the global energy transition, several research paths are explored to reduce the energy consumption and carbon footprint of buildings without degrading the occupants' comfort. Among the investigated solutions, heat storage represents a very promising technology which is capable to improve the energy efficiency of buildings and their technical installations (heating, air conditioning and domestic hot water). Moreover, heat storage is also

investigated to compensate for the intermittency of renewable energies (thermal and hybrid solar systems, wind farming, etc.). Depending on the targeted application, latent heat storage, sensible heat storage or thermochemical storage will be selected.

For housing, some researchers try to improve the thermal performance of the walls without disturbing the building envelope by using active heat storage system [3] or adding insulation layers [4]. Active systems are those where the fluid circulation in the components is driven by a mechanical system (fan, pump, etc.) with the aim of replacing free convection by forced convection. This active action allows the capacity to store and/or withdraw energy depending on demand.

Otherwise, many research investigations are performed to improve the walls themselves. New innovative solutions begun to break through in the world of improving building walls, mostly made of concrete. These consist in adding to the cement matrix components that can improve the thermal behavior as the increase of the voids in the concrete by foam [5] or the addition or substitution of light aggregates [6–8]. The porosity provided by the foam or lightweight aggregate decreases the thermal conductivity of the concrete according to their addition (dosage) into the mixture.

In order to comply with thermal and environmental regulations a lot of investigations have been undertaken, at housing full scale and especially their walls, in order to find an innovative solution limiting energy consumption. Long known earth building techniques (rammed earth, cob, lightened earth, etc.), have been revisited for a better understanding of their thermal, hygroscopic and mechanical behavior [9–14].

Building with earth-based materials represents a real alternative. Build with these kinds of materials can bring many benefits, mainly the renewability of the main components and the low carbon footprint. Earth is a natural resource that is often widely available. Almost all mineral soils that contain clay can be used for construction [15]. Usually, these latter do not require any treatment or transformation and are enough locally abundant allowing the reduction of emissions due to transportation.

Nowadays, the interesting properties of earth *i.e. raw soil*, mainly consist in its capacity to regulate the building hygrometry and having a high thermal mass, at low economic and environmental cost. As a raw material, without additives or cooking, earth is an excellent humidity regulator due to its absorption and evaporation capacity. It captures or releases humidity from the air according to the ambient humidity, thus promoting a healthy indoor environment [16–21]. This natural humidity regulation is a quality that does not found with conventional building materials such as concrete.

Consequently, earth construction investigation had a renewed interest these last years with many research projects acted and in progress. Hereafter, some of them dealing with earth construction (light earth, rammed earth, cob, etc.) are reported with their main objectives:

- ECO-TERRA is a research project that aims to acquire scientific and technical knowledge necessary to develop light earth construction materials for efficient green buildings. To enhance resources value from different territories, studies were carried out to understand the links between constituents variability and physical properties of earth-based mixtures [9].
- PRIMATERRE research project concerns “*the renovating and building sustainably: the challenge of raw materials*”. It mainly focuses on proposing means of measuring and guaranteeing the hygrothermal, mechanical and seismic performances of earth materials. The case of rammed earth was particularly studied. Development of recommendation guides and educational courses is also targeted [22].
- CobBauge is a European research project focusing on developing, testing and establishing an innovative low carbon technology using local soil and vegetal fibers. This technology, based on mixtures of soil in a plastic state and plant fibers, aims the development of a structural and insulating material. Formulation studies have been carried out to determine optimal earth / fiber mixtures. These optimal mixtures are adopted to produce two levels building that meets the French and UK standards. In this project, walls are made up of two layers, namely a structural (cob) and a thermal layer (light earth). Two experimental buildings are under construction, in France (Manche department) and in England (Devon), to assess the site behavior of buildings (housings) using the developed optimal earth / fiber mixtures [14,23].

In order to improve earth-based materials thermal properties, many studies have been implemented. As example, the impact of density on the hygrothermal properties of earthen-based materials has attracted a particular interest [9,12,17,18,24,25].

More, these recent years new method targeting improvement of the thermal behavior of buildings (or housings) has appeared. It consists of incorporating phase change materials (PCM) into the construction walls. The PCM principle in the building engineering can be clearly explained considering a front wall in which PCMs are used. When the outside temperature exceeds the PCMs melting temperature range, this latter absorb heat by changing from a solid to a liquid state, thus delaying the heat flow inside the building (or housing). Then, the stored heat is released when temperature drops below the PCM solidification temperature range. This provides more homogeneous indoor temperatures, which tend to be close to the melting point of the PCMs. This technology has the interesting feature of shifting the building heating and cooling load from peak to off-peak periods of electricity consumption [26]. Furthermore, most of these materials are based on non-renewable resources with a high environmental footprint [27]. For building applications, PCMs are introduced mainly under microcapsules form. They are constituted in a paraffinic core and a polymeric shell [28]. Paraffin is considered as an inherently biodegradable material [29] but the polymeric shell is a non-biodegradable substance. Nevertheless, readily biodegradable and biobased microencapsulated PCMs start reaching the market [28,29]. Also, recent research is focusing on these aspects [30–34]. Accordingly, biobased PCMs cores [31,32] and shells [30,31,35,36] are being developed.

Several studies have examined the use of many PCM types adapted to the building engineering. Types concerned various aspects *i.e. free, micro or macro encapsulated*. The incorporation of PCMs in mortar or concrete gave promising results by reducing their thermal conductivity and increasing their thermal mass [37,38]. However, the incorporation of PCMs into structural concrete/mortar degrades its mechanical performance [37,39–43]. In spite of the numerous studies carried out in this field, performances of these materials are not completely controlled.

Present study aims to combine the suitable hygrometric and thermal conductivity of lightened earth, with the PCMs appropriate heat storage capacity. Indeed, when compared to other earth-based materials, lightened earth presents a low density. Incorporation of PCM into this latter could improve its thermal buffering capacity. First, microencapsulated PCMs will be introduced into soil-fiber mixtures. Mixtures containing 0 %, 2 %, 5 %, 10 % and 20 % of PCM will be formulated. Then, mechanical, thermal (thermal conductivity and specific heat capacity) and hygroscopic behaviors (sorption/desorption capacity, water vapor permeability) of the soil-fiber mixtures will be analyzed for the different PCMs dosages.

2. Materials

2.1 Earth material or soil-fiber mixture

The chosen soil-fiber mixture was investigated in CobBauge research project for which different formulations were studied. Various soil and fiber types *i.e. combination of 12 soils and 6 fiber types (wheat straw, hemp straw, hemp seed, flax straw, flax shives and reed)*, were mixed, tested and discussed. In present study, the soil-fiber mixture that gave the best thermal properties was selected [44]. So, the mixture composed of silty-clay soil and reed was chosen.

The soil classification is made using conventional geotechnical characterization and regarding applicable standards. The grain size distribution was conducted differently according to the fine (laser granulometry with LS 13 320 from Beckman Coulter) and coarse particles (>80 μm) following the standard XP P94-041 [45]. In addition, soil plasticity is determined through the Atterberg limits in accordance to the standard NF P94-051 [46]. The clay activity is determined via the methylene blue value (MBV) according to the standard NF P94-068 [47]. Atterberg limits allow the determination of the soil liquidity limit (LL), plasticity limit (PL) and plasticity index (PI). The methylene blue value and granulometry allow the soil classification as sandy, silty or clayey.

The characterization test results are gathered in Table 1 and shown in Figure 1.

Table 1: Atterberg limits and methylene blue values of the crushed soil.

Parameter	LL [%]	PL [%]	PI [%]	MBV [g/100g]
Soil	57.8	42.5	15.3	5.64

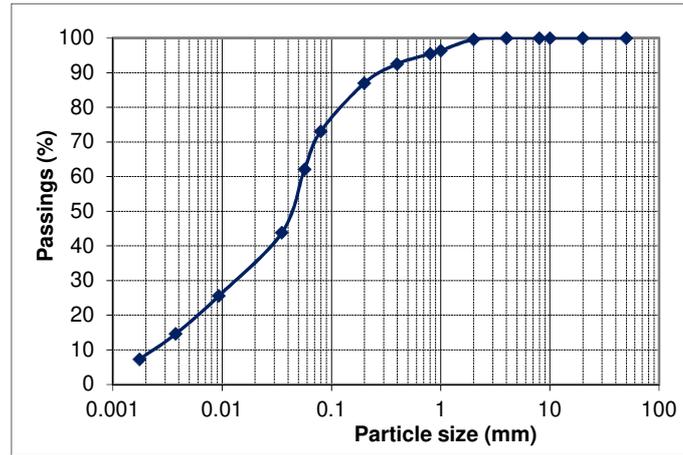


Figure 1: Soil particle size distribution after crushing.

To be able to adjust the water content in mixtures, the reed fibers water absorption capacity is performed according to RILEM protocol [48]. The raw fibers were cut but left in their natural raw shape, they were not crushed. Reed fibers length is ranged from 4 to 6 cm.

Then, soil, fiber and PCM Nextek 24D absolute density was determined by using a Helium pycnometer (Accupyc II 1340) and given in the Table 2.

Table 2: Absolute density measured by helium pycnometer.

Material	Absolute Density [kg/m ³]
Soil	2630
Fiber	1020
Nextek 24D	845

2.2 Phase Change Materials (PCMs)

The selection of the PCM to be incorporated in soil-fiber mixture was based on several criteria, namely: the phase change latent heat, chemical properties and especially the melting temperature. The selected melting temperature must take into consideration the expected thermal comfort and the typical climatic conditions of the building (or housing) location [49]. The average temperature in France varies between 0 and 30 °C during the year [50]. Thus to satisfy the climate conditions encountered in France, the initial selection is limited to PCM having a melting temperature ranging from 5 to 25 °C. Microencapsulated PCMs are preferable compared to bulk PCMs, as they prevent leakage during solid-liquid phase transitions and also offer a larger heat transfer surface per unit volume [51]. Then, to ensure the human comfortable environment, the PCM phase change temperature should be ranged approximately between 16 and 25 °C [52].

According to the above-mentioned criteria and the commercially available PCMs, Nextek 24D demonstrated favorable thermal properties. Nextek 24D is composed by a paraffinic core and a polymeric shell. The Nextek 24D is encapsulated in patented robust capsules that offer great thermal stability and high dispersibility [28]. So, in present study, Nextek 24D was adopted for the soil-fiber mixture series tested and analyzed. The characteristics given by providers are checked before starting any mixture. The verified properties are: density (helium pycnometer), particle size distribution (laser particle size), phase change latent heat and specific heat capacity (Differential Scanning Calorimeter DSC at 10K/min). The different results are given in Table 3.

Table 3: Physical properties of the studied PCM.

PCM	Particle size [μm]	Bulk Density [kg/m ³]	Melting temperature [°C]		Latent heat [J/g]	Specific heat capacity [J/(kg.K)]	
Nextek 24D	0-80	≈ 300-400	22,4*	24 ± 2**	168*	170**	12000*

* measured in the laboratory, ** Product data sheet [28]

2.3 Samples preparation

First, the soil is crushed in order to obtain particles having a maximum diameter of 2 mm. Then, it is dried in a ventilated oven at 40 °C for 2 weeks. The fibers are hand cut at 5 ± 1 cm length. Then, they were covered and stored at room temperature, *i.e.* 20 °C.

The different soil-fiber-PCM mixtures weight ratios are: water/soil ratio is equal to 1 and fiber/soil ratio is equal to 0.25; PCM/(soil+fiber) vary from 2 % to 20 %, see Table 4. Ratios used correspond to desired soil-fiber formulation.

Table 4: Lightened earth formulation (in wt%).
Fiber and water percentages are function of soil amount. The PCM percentage is function of (soil+fiber) dry mass.
SF_i: S = Soil, F = Fiber, and i = PCM content.

Formulation	Soil	Fiber	Water	Nextek 24D
SF0	100	25	100	0
SF2	100	25	100	2
SF5	100	25	100	5
SF10	100	25	100	10
SF20	100	25	100	20

The sample preparation method is the same for all the different mixtures. In order to ensure a homogeneous distribution, soil and PCM with their corresponding mass are first mixed manually at dry state within a mixing tank. So, the necessary water is added and the mixing is allowed to continue until the soil mixture reaches a liquid state. The fibers are then added gradually. The mixing procedure is carried out within a mixer (PROVITEQ Concrete Mixer 65 L) for 120 s. At the end, the soil-fiber mixture is bagged and stored at room temperature *i.e.* 20 °C. The next day the mixture is manually compacted with the help of a wooden cleat in previously oiled molds. Knowing molds volume, mixtures appropriate weights were calculated, in order to obtain a same density. To get a cylindrical sample for compressive testing of approximately 700 kg/m³ dry density, 1.46 kg of dry mixture is needed. Considering the water in the formulation, the necessary mass to be filled in the mold is 2.628 kg. It should be noted that fiber length, samples dimension and compaction direction promoted a preferred orientation of the fibers over the sample width, see Figure 2. Then, the molds are left during one more day at room temperature and then placed in an oven at 40 °C. After two days of drying in the oven, the cylindrical (diameter Ø11 cm x height 22 cm) molds are completely removed. The prismatic molds (22 cm x 22 cm x 4 cm) are partially removed (every two days, two sides at a time) while keeping the samples at 40 °C. The drying process takes almost 3 weeks. The previously dried samples are put at a room conditions of 20 °C and 50 % RH. Characterization of the prepared samples begin once equilibrium is reached (when difference in mass between two daily weightings is less than 0.1 %). This equilibrium is reached usually after 1 week at room conditions. Table 5 summarizes the different tests performed, met standards, size and number of the tested samples.

Table 5: Standard, number and size of samples for testing.

Test	Standard	Sample dimensions [cm]	Number of samples
Moisture sorption isotherm	ISO 12571 [53]	3x3x3	3
Water vapor permeability	ISO 12572 [54]	Ø15x6	3
Thermal conductivity	ISO 8301 [55]	22x22x4	3
Compressive strength	EN 1015-11 [56]	Ø11x22	3

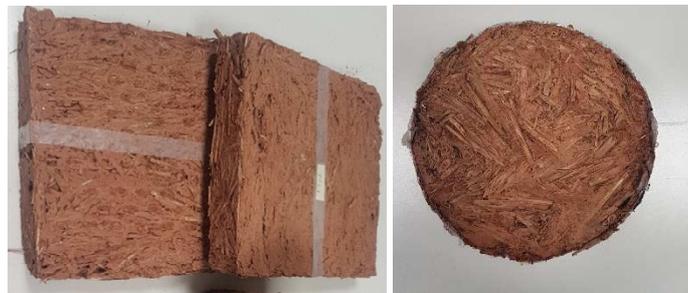


Figure 2: Samples used in thermal (22×22×4 cm³) and water vapor permeability (Ø15 cm x 6 cm) testing.

3. Experimental methods

3.1 Hygroscopic behavior

It is well known that lightened earth, as all the hygroscopic materials, has a real potential in improving thermal and indoor humidity comfort. More, these materials significantly impact the buildings (or housings) indoor air quality and energy consumption. In this section, the lightened earth hygroscopic properties are presented with the description of methodologies used to study these latter.

To investigate solids interaction with vapor, the Dynamic Vapor Sorption (DVS) technique is usually used. In present study, sorption isotherms of the raw materials (soil, fiber, PCM) and the mixtures are studied according to the standard ISO 12571 [53]. To do so, the ProUmid SPSx-1 μ sorption/desorption system was used. This equipment allows a precise measurement of the samples mass and sorption kinetics through a precision balance and a careful control of temperature and humidity. Before adsorption analysis begins, the samples are first dried to reach a constant weight (weight change is less than 0.1 %). After that, the samples were exposed to an atmosphere relative humidity ranging from 10 % to 90 % in 5 steps, while maintaining the test temperature at 23 °C.

Then, to study the ability of a material to allow a vapor to pass through, the water vapor permeability should be known. This parameter represents the ratio of the quantity of water vapor passing through a material per unit: of thickness, of time, and of vapor pressure difference existing on each side of the material. In present study, the water vapor permeability of the different samples was measured according to the standard ISO 12572 [54]. This is achieved by following the mass of a sample submitted to a moisture gradient. The *dry cup* method requires to create a moisture gradient from inside of the cup (≈ 0 % RH) to outside (≈ 50 % RH) through the sample.

Measurements of water vapor permeability using the dry cup method provide information on the material behavior when moisture transfer is dominated by vapor diffusion. The flow of vapor through the material is obtained from weighing the sample cup assembly. At steady state, the water vapor flow (G) through the sample is given by the slope of the regression line of the sample cup assembly mass vs time. This is achieved after eliminating the test initial non-linear phase.

Thus, flux density (or transmission rate) of the vapor (g) is calculated by the formula in equation (1), where A is the area of the exposed specimen.

$$g = \frac{G}{A} \quad (1)$$

In Equation (2), it can be seen that water vapor resistance (Z) can be calculated from water vapor permeance (W). This latter depends, among others, on the difference water vapor partial pressure between sample sides (ΔP_v).

$$Z = \frac{1}{W} = \frac{A \times \Delta P_v}{G} \quad (2)$$

Then, from Equation (3), water vapor resistance of the air layer present in the measuring cup (Z_a) is achieved by knowing the air layer thickness (d_a) and air water vapor permeability at atmospheric pressure (δ_a) which is equal to $2 \times 10^{-10} \text{ kg} \cdot \text{m}^{-1} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$.

$$Z_a = \frac{d_a}{\delta_a} \quad (3)$$

The corrected water vapor permeance W_c is obtained using equation (4).

$$W_c = \frac{1}{Z - Z_a} \quad (4)$$

Then, the sample water vapor permeability (δ) can be determined using equation (5). e is the sample thickness.

$$\delta = W_c \times e \quad (5)$$

Finally, sample water vapor resistance factor (μ) is found by the following equation (6).

$$\mu = \frac{\delta_a}{\delta} \quad (6)$$

Moisture levels necessary to perform the tests are generated by silica gel ($\approx 0\%$ RH), magnesium nitrate ($\approx 50\%$ RH). All samples were upstream conditioned at a temperature of $23\text{ }^\circ\text{C}$ and a relative humidity of 50% .

The determination of moisture sorption isotherms and water vapor permeability allow evaluation of the PCM impact on soil-fiber mixtures hygroscopic behavior.

3.2 Thermal behavior

To use a given material as thermal insulator and/or heat storer in buildings (or housings) envelope, some thermal properties should be known. Sometimes, these properties must satisfy threshold values that can be defined in standards. In present study, we are concerned in two thermal properties: thermal conductivity and specific heat capacity. Methodologies used to obtain these parameters are described hereafter.

Thermal conductivity is a physical property that characterizes the ability of a material to conduct heat from a hot to a cold point. The suitable methodology to determine this property depends on the nature, the shape and the size of the samples to investigate. For building materials, thermal conductivity measurements are usually performed with a heat flow meter HFM 436 Lambda. This technique consists in creating a temperature gradient between two plates through the investigated material. By means of two heat flow sensors in the plates, heat flow entering and leaving the material is measured. When the equilibrium state is reached and the heat flow is constant, then the material thermal conductivity is calculated using Fourier's law [57], assuming that the sample dimensions are known. In present study, thermal measurements were carried out on $22 \times 22 \times 4\text{ cm}^3$ prismatic samples at different mean temperatures: 14 , 24 and $34\text{ }^\circ\text{C}$. The temperature difference between the two sides of the samples is fixed at a value of $10\text{ }^\circ\text{C}$ for each measurement.

Specific heat capacity (C_p) is another physical property that characterizes the ability of a material to store heat. The measured parameter represents the quantity of heat required to raise the temperature of one kilogram of a material by one Kelvin. To determine this parameter, Differential Scanning Calorimetry (DSC) technique is usually used. This technique is especially relevant when investigations are run on small samples (weighing up to few grams).

In present work, C_p measurements are performed with a DSC calorimeter according to the standard ISO 11357-4 [58]. During tests, temperature was increased from $-20\text{ }^\circ\text{C}$ to $60\text{ }^\circ\text{C}$ continuously. Heating rate was $10\text{ }^\circ\text{C}\cdot\text{min}^{-1}$. This rate can affect the C_p absolute values, but it remains relevant in the case of comparative study.

3.3 Mechanical behavior

To analyze the effect of PCM incorporation on the mechanical strength of the soil-fiber mixtures, unconfined compressive strength (UCS) tests have been performed. The test recommendations stipulated in the standard EN 1015-11 [56] were followed. The UCS strength were obtained using INSTRON SCHENCK press machine with a capacity of 100 kN .

3.4 Porosity measurement

Global porosity is an important physical parameter in the characterization of a material. It is measured by immersing small samples in a non-wetting oil, in this work disaromatized petroleum is used. The tests are carried out according to the standard NF ISO 5017 [59].

The samples are previously saturated under vacuum in a desiccator for at least 24 hours which allows the liquid to replace the air in the pores without interacting with the sample.

Later, they are weighed under oil and then weighed in air. The samples were dried in the oven at $105 \pm 5\text{ }^\circ\text{C}$ until a constant mass is reached, i.e. until two successive weighing, before and after a stay of 24 hours in the oven, do not differ by more than 0.05% , considered as the dry mass. This makes it possible to obtain the volume of the voids initially filled with oil, by means of a mass difference between the saturated and the dry states. Hence the accessible porosity p_0 is given by:

$$p_0 = \frac{M_{air} - M_{dry}}{M_{air} - M_{oil}} \quad (7)$$

where:

- M_{dry} is the mass of dry specimen.
- M_{air} is the mass of saturated specimen in air.
- M_{oil} is the mass of saturated specimen in oil.

4. Results and discussion

4.1 Moisture sorption isotherm

The moisture sorption isotherms reported in Figure 3 show, for each investigated material, evolution of moisture content versus ambient air relative humidity at a constant temperature of 23 °C. According to Brunauer's proposal [60], the sorption curves correspond to the type III and show hysteretic loops. Such kind of curves is typical of porous materials, as the investigated soil-fiber mixtures.

It is observed that the amount of absorbed water vapor from the ambience is inversely proportional to the percentage of PCM incorporated. For a better analysis of this decrease in the mixture's sorption/desorption values, the raw materials (soil, reed fiber and PCM) were characterized, see Figure 4. The results demonstrate that mixtures sorption/desorption behavior is largely governed by the presence of reed fibers. As PCM is a hydrophobic material, its addition into the mixtures implies a decrease in the amount of absorbed moisture in soil-fiber mixtures.

Soil-fiber mixtures slightly lose their hygroscopic performances with the addition of PCM. Nevertheless, these remains enough high compared to those of a standard cement mortar. The mixtures sorption/desorption capacity is 4-5 times larger than the one of standard cement mortar, see Figure 3. This mortar is prepared in the laboratory conforming to the standard NF EN 196-1 [61]. In addition, for a sorption/desorption cycle, the soil-fiber-PCM mixtures retrieve their initial hygric state whilst a cement mortar retains a certain amount of moisture within it.

For all soil-fiber mixtures at each relative humidity, moisture content varies significantly within 24 hours of the change in relative humidity and then the phenomenon slows down to equilibrium. A better reactivity is observed both in mixtures sorption and desorption paths compared to standard cement mortar, see Figure 5. The rapid response of the earth-based material to a change in relative humidity is perhaps one of the strongest properties for moisture regulation in the interior of a building.

From Figure 3, it can be noted that the area of the zone located between sorption and desorption curves, hysteresis decreases slightly with PCM addition. The decrease in the mass difference value between different dosages is due to the component's nature, while hysteresis is probably due to the porosity of the mixtures. To verify this assumption, the mixtures overall porosity is measured by immersing small samples in a non-wetting oil. The results presented in Table 6 confirm the porous nature of the mixtures and point a decrease in porosity with addition of PCM. Looking its granulometry, we suppose that added PCM embeds and fills the mixtures pores.

Table 6: Porosity measured on the different soil-fiber-PCM mixtures.

Formulation	SF0	SF2	SF5	SF10	SF20
Porosity	58 %	57 %	56 %	52 %	50 %

Furthermore, these porosity results can be correlated with those reported on the sorption/desorption curves. Indeed, the lower porosity acts to reduce areas of the hysteresis seen on Figure 3 and consequently leads to a reduction of the mixtures hygroscopicity.

When a material sorption ability is high that means it will prevent surface condensation pathologies (mildew). Consequently, that will increase moisture content inside the material leading to a degradation of its thermal insulation properties. In this context, knowing the material water vapor permeability becomes essential.

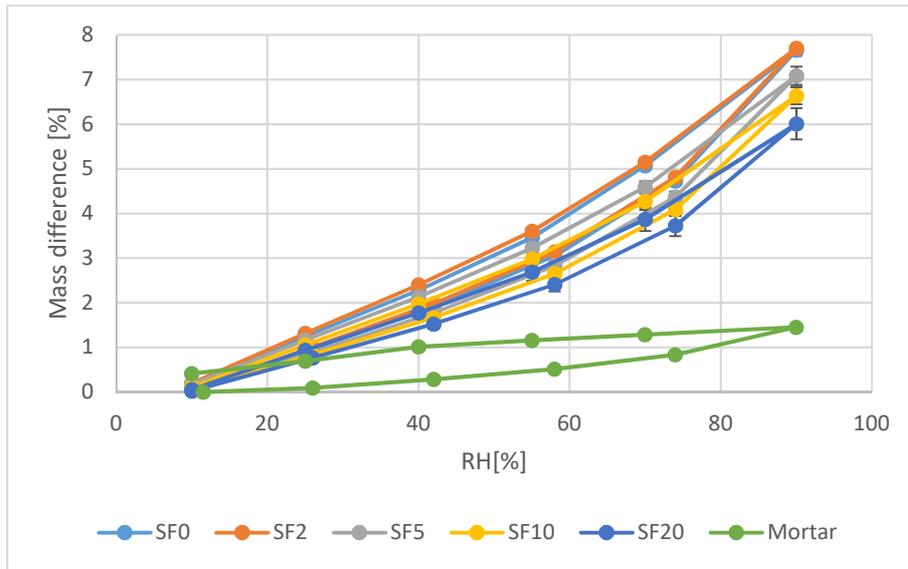


Figure 3: Moisture sorption isotherm of the different soil-fiber-PCM mixtures and a standard cement mortar.

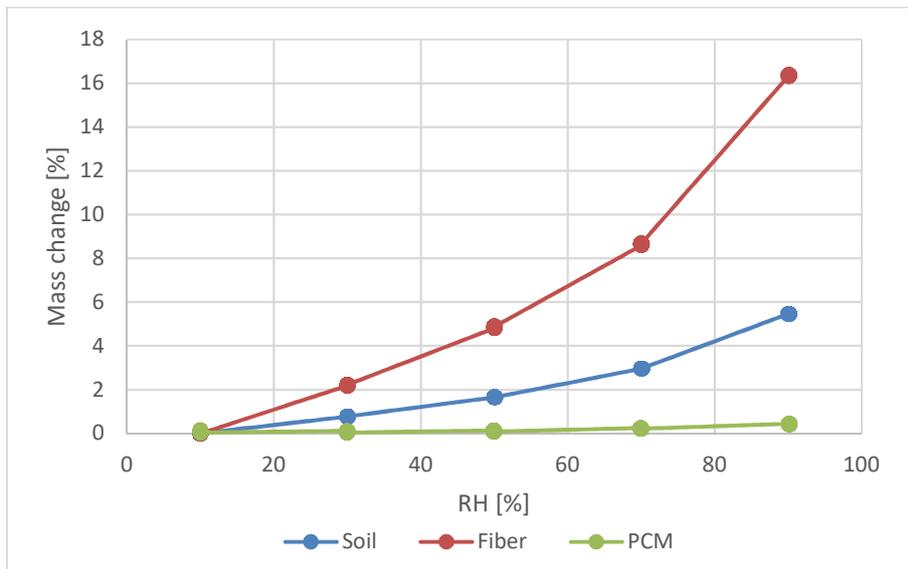


Figure 4: Moisture sorption isotherm of soil, fiber and PCM.

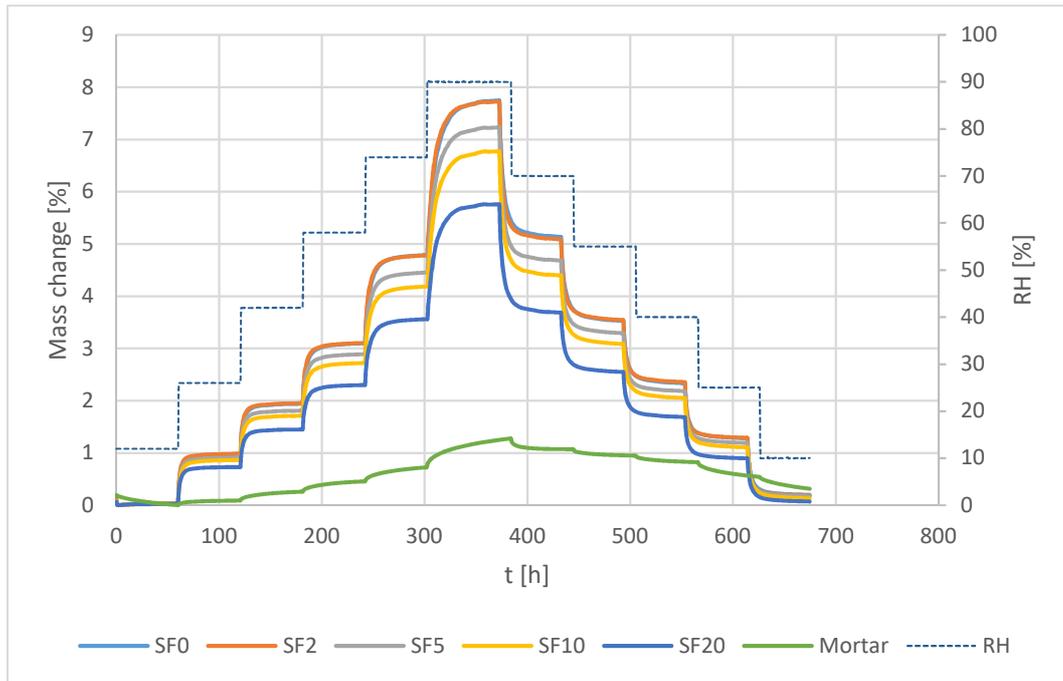


Figure 5: Dynamic sorption curves of the different soil-fiber-PCM mixtures and a standard cement mortar.

4.2 Water vapor permeability

Results of the measurements performed on the different studied mixtures are reported in Table 7. From this table, first we can notice that soil-fiber mixtures present a water vapor permeability 7-8 times larger than the one of a standard cement mortar [62]. The value of the water vapor resistance factor of the formulation without PCM (SF0) is relatively close to the one reported by Phung ($\mu \approx 9$ for 3 % reed) [12]. Afterwards, these results show that PCM addition decreases water vapor permeability of the soil-fiber mixtures. The diminution is more pronounced as the PCM rate is high. As for the sorption/desorption test, the diminution of the water vapor permeability with PCM content is due the decrease of porosity and also the hydrophobic nature of the PCM. The addition of PCM has the same effect on the ability of the mixtures to fix or to let pass moisture through it. Even though the decreases are in the range of few percent, the hygroscopic character of such materials is still conserved. Whether sorption or water vapor permeability, the soil-fiber-PCM mixtures remain more hygroscopic than structural cob [63] and much more hygroscopic than cement mortar.

Table 7: Water vapor permeability measured on the different soil-fiber-PCM mixtures.

	SF0	SF2	SF5	SF10	SF20	Mortar [62]
Water vapor permeability $\times 10^{-12}$ [kg.m ⁻¹ .s ⁻¹ .Pa ⁻¹]	25.631	24.997	23.468	22.227	21.305	3.160
Water vapor resistance factor μ [-]	7.101	7.217	7.788	8.284	8.673	62.000
Decrease in permeability [%]	0	2.5	8.4	13.3	16.9	-

4.3 Thermal conductivity

The thermal conductivity (λ) of soil-fiber-PCM mixtures was measured at three different temperatures (14 °C, 24 °C and 34 °C). Temperatures are chosen so as to investigate thermal conductivity in the PCM phase change zone (24 °C) and outside the PCM phase change zone (14 °C and 34 °C). At a temperature of 14 °C, the PCM is in a solid state and at a temperature of 34 °C, in a liquid state. Taking a look at Table 8 and Figure 6, first, it can be seen that thermal conductivity increases with temperature for the sample without PCM and for the samples containing PCMs when temperature is outside the phase change zone. This evolution was expected as it is well known that thermal conductivity of insulating materials rises with temperature [64].

Then, it can be observed that the addition of PCMs decreases thermal conductivity of soil-fiber mixtures on the whole temperature scale. However, this decrease is not always proportional with the incorporated PCM amount. When the PCM is completely solid or liquid (at 14 °C or 34 °C), thermal conductivity tends to stabilize and the decrease in λ becomes negligible beyond 5 % of PCM. Otherwise, in the transition zone (24 °C), thermal conductivity decreases continuously until the quantity of PCM approach 10 %. Beyond this PCM content, the diminution of λ slows down.

Thus, this overall decrease in thermal conductivity can be due to the PCM low thermal conductivity compared the one of lightened earth *i.e.* soil-fiber mixture, and probably to the disorder brought by the PCM incorporation in this mixture. In addition, the diminution is more significant in the phase change zone. At 24 °C, SF20 thermal conductivity is approximately 14 % less than the one of SF0. This diminution can be due to disorder increase inside the microcapsules (coexistence of solid and liquid phases).

More PCM quantity is high more the decrease in thermal conductivity is significant until a kind of saturation at approximately 5 % or 10 % PCM. This indicates that a formulation may have an optimum of PCM amount to add. The value of this optimum should be defined regarding the material's different properties and nature. As illustration, Dakhli *et al.* [39] reported an optimum value of approximately 20 % PCM for a standard cement mortar. Finally, it can be confirmed that addition of Nextek 24D PCM improves the insulating properties of soil-fiber mixture depending on its incorporation level and temperature.

Table 8: Thermal conductivity of lightened earth with 2 %, 5 %, 10 % and 20 % of microencapsulated PCM at three different temperatures 14, 24 and 34 °C.

Formulation		λ [W/(m.K)]				
		SF0	SF2	SF5	SF10	SF20
T [°C]	14	0.151 ± 0.007	0.149 ± 0.005	0.144 ± 0.010	0.144 ± 0.004	0.143 ± 0.005
	24	0.156 ± 0.008	0.144 ± 0.005	0.142 ± 0.007	0.135 ± 0.003	0.134 ± 0.004
	34	0.159 ± 0.007	0.153 ± 0.005	0.151 ± 0.008	0.151 ± 0.003	0.151 ± 0.003

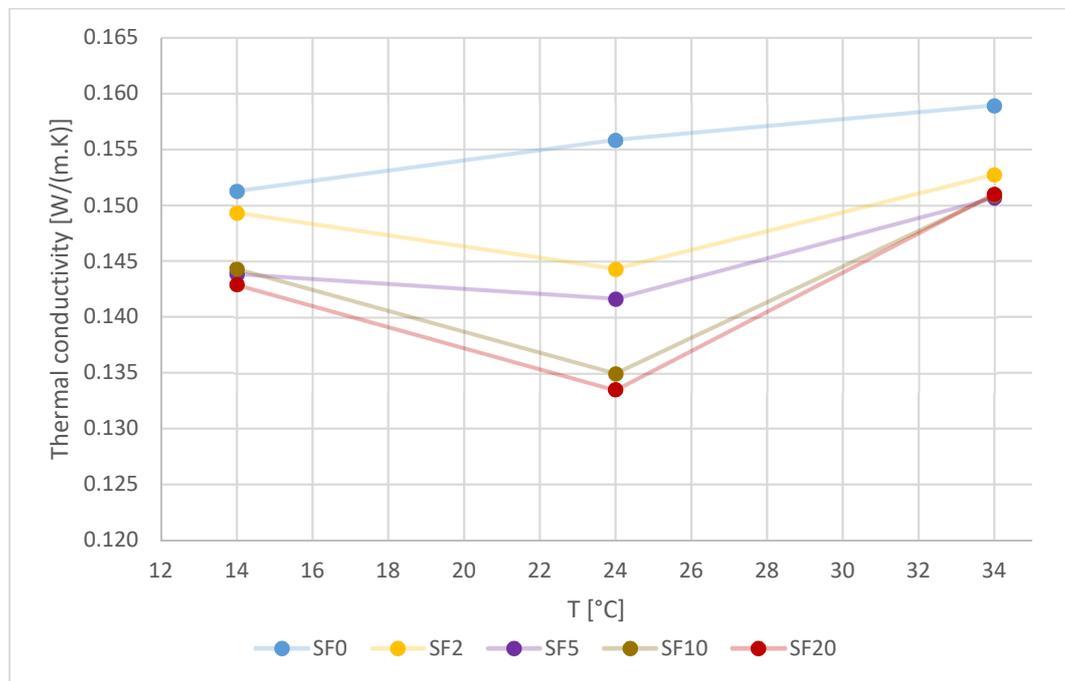


Figure 6: Evolution of thermal conductivity of the soil-fiber mixtures as function of temperature and PCM content.

4.4 Specific heat capacity

As this study deals with multicomponent mixtures, the specific heat capacity can be calculated using a mixing law. To do so, Cp of the different constitutive components in a raw state is determined by the DSC technique. To get the mixtures specific heat capacity, Equation (8) is applied at each measuring temperature [9].

$$Cp = F_s \cdot Cp_s + F_f \cdot Cp_f + F_{PCM} \cdot Cp_{PCM} \quad (8)$$

In this equation, Cp and F are respectively dry specific heat capacity and mass fraction of the component in the mixture. Indexes s , f and PCM represent soil, fiber and phase change material. The obtained results are presented in Figure 7. The constitutive materials mass fractions are calculated at the laboratory ambient environment ($\approx 50\%$ RH and $20\text{ }^\circ\text{C}$).

Figure 7 represents the evolution of mixtures calculated specific heat capacity as function of temperature and PCM content. From a first sight on the different curves, it can be noticed that Cp increases with temperature. The increase slope becomes steeper when temperature exceeds $20\text{ }^\circ\text{C}$, this is particularly visible on the mixture without PCM.

Regarding the PCM effect, from Figure 7 it can be assumed that, in general, addition of Nextek 24D increases Cp value of the soil-fiber mixtures. Cp value increases linearly with PCM content. Two separate parts can be identified: Outside and within the phase change zone. Before phase change, specific heat capacity is 4 %, 11 %, 21 %, and 38 % larger than the one of the control sample for PCM content of 2 %, 5 %, 10 % and 20 %, respectively. After phase change, Cp evolution presents the same allure than before the transition zone. In general, the specific heat capacity increases as we approach the phase change temperature. At the pic value, mixtures Cp values are 45 %, 108 %, 207 %, and 380 % larger than the one of the control sample for PCM content of 2 %, 5 %, 10 % and 20 %, respectively. In fact, at this level, specific heat capacity increases from $576\text{ J.kg}^{-1}.\text{K}^{-1}$ for the sample without PCM, to around $2762\text{ J.kg}^{-1}.\text{K}^{-1}$ for the mixture containing 20 % PCM. It should be noticed that in the phase change range, the specific heat capacities represent a combination of two contributions: latent heat of phase change and sensible heat.

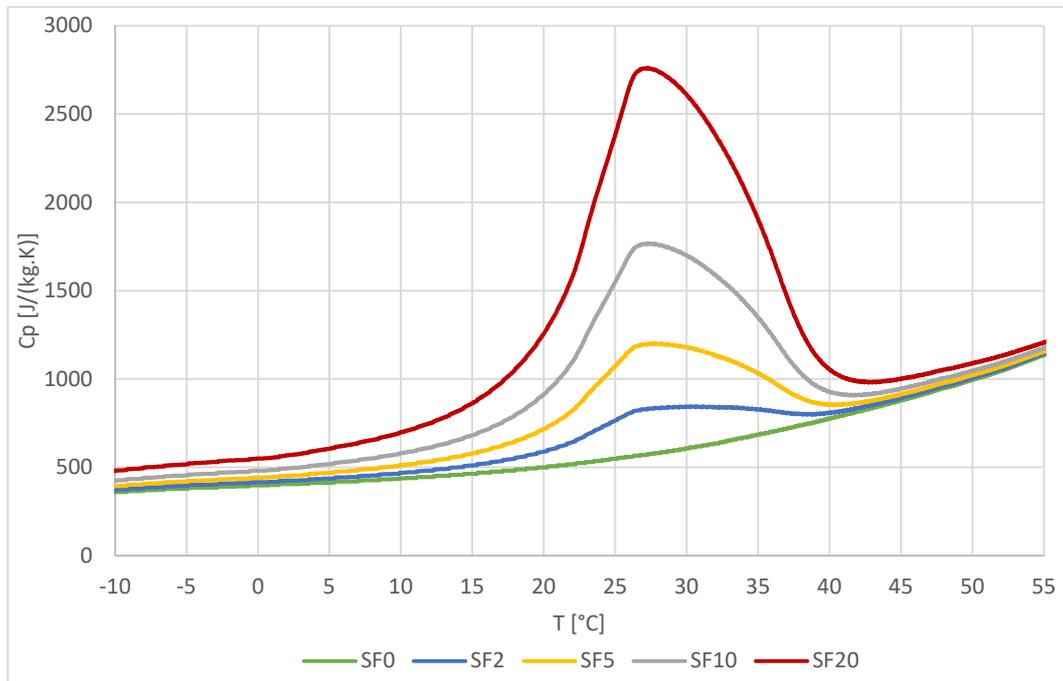


Figure 7: Evolution of the lightened earth specific heat capacity as function of temperature and PCM content in heating.

4.5 Mechanical behavior

To understand the impact of PCM introduction on the mechanical resistance of soil-fiber mixtures, the stress-strain relationships are reported on Figure 8. From this figure, it can be noticed that stress-strain curves in compression tend to have a degree 2 polynomial form. Since studied materials are ductile, no particle breakage was observed during the mechanical test. Consequently, the maximum stress is not a relevant indicator to study the samples compressive strength [10,65–67]. At the best of our knowledge, no standard sets a strain value at which compressive strength should be studied. Consequently, Unconfined Compressive Strength (UCS) is determined at a strain of 2 %. UCS at chosen strain of the studied mixtures presents similarity to those already observed on other bio-based mixtures [44].

Also, the addition of PCM has almost no effect on mechanical strength of the soil-fiber mixtures. As it can be observed on Figure 8, stress-strain graphs corresponding to the different mixtures are very close with an evident shape similarity. This may be explained by the high porosity in such type of mixtures. Thus, adding PCM of a few tens of micrometers size in high porous materials do not influence their mechanical behavior, see Table 9. This is not the case on the compressive strength that can be observed in cementitious matrices [39,68]. In these latter, PCM incorporation in mortar or concrete will increase porosity and consequently degrade the mechanical resistance.

Considering that the primary function these mixtures *i.e.* soil-fiber-PCM in building, is purely thermal. Then, given that these materials are self-supporting. It can be assumed that the obtained weak compressive strength does not constitute a limitation for the use of these mixtures in building (or housing) envelopes.

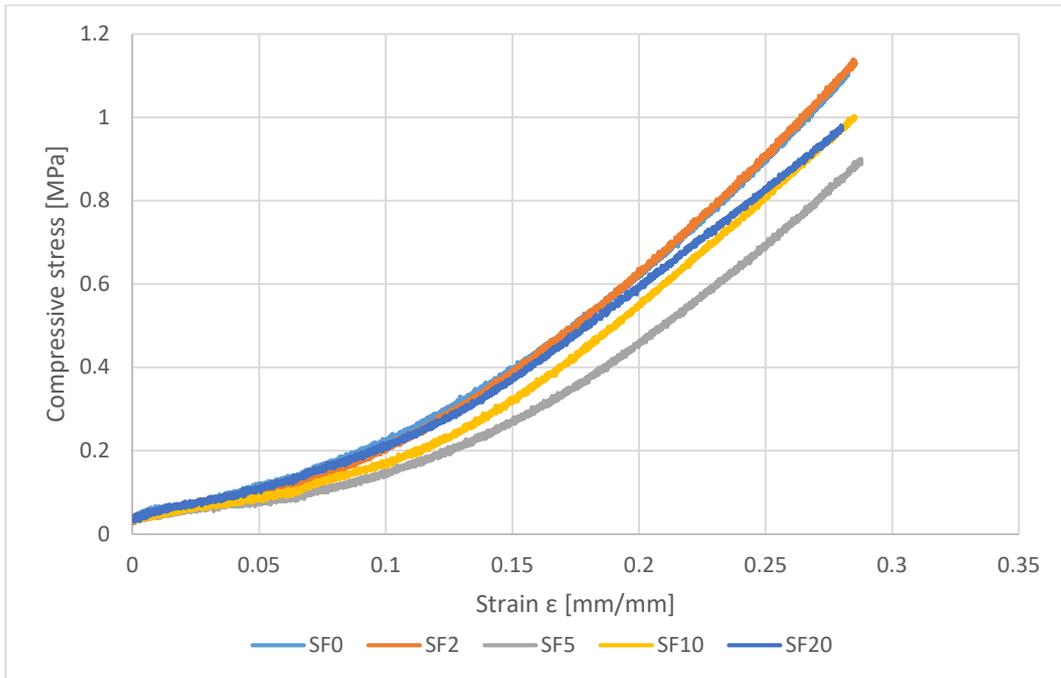


Figure 8: Stress-strain relationship for soil-fiber mixtures as function of PCM content.

Table 9: Unconfined compressive strength parameters for soil-fiber mixtures as function of PCM content.

Soil-fiber mixture	SF0	SF2	SF5	SF10	SF20
Strain [mm/mm]	0.02	0.02	0.02	0.02	0.02
UCS [MPa]	0.07	0.07	0.06	0.06	0.06
Young's modulus [GPa]	1.347	1.153	0.841	1.010	1.368

4.6 Literature comparison

To be able to evaluate performances of the developed mixtures, their hygrothermal behavior is compared to literature. In Table 10, hygrothermal properties for different earth-based materials are reported: lightened earth with different densities, cob, earth bricks, etc. It should be noted that reported properties have been obtained in experimental conditions (temperature and relative humidity) which are sometimes different. This fact was not considered in present comparisons.

From the lightened earth section in Table 10, it can be seen that mixtures investigated in present study are hygroscopically and thermally (thermal conductivity) less efficient than those reported in other works [9,24,25]. This may mainly be due to the differences in the used materials and preparation/conditioning methods. In addition, differences in density can be observed. In fact, materials in present study are denser when compared to those reported in refs [9,24,25]. This difference in density can explain, in part, the disparities between here-obtained results and those reported in literature. Indeed, this parameter is of a crucial importance. Niang *et al.* [24] reported that thermal conductivity increases from 0.065 to 0.112 W/(m.K) when lightened earth density increases from 323 to 586 kg/m³. This increase in density also affects the lightened earth hygroscopic properties. In fact, maximum moisture sorption decreases, and water vapor resistance factor increases. Moreover, Colinart *et al.* [9] reported that thermal conductivity clearly increases with density. However, no effect of density on water vapor resistance factor was clearly observed.

Even though PCM introduction degrades lightened earth hygroscopic properties, as detailed in section 4.1 and 4.2, these properties remain comparable or better than those of the other earth-based materials considered as suitable to moderate buildings indoor humidity. In the case of water vapor permeability, here investigated materials behave as Cob or earth bricks, see Table 10. Regarding maximum sorption value, lightened earth-PCM mixtures are more efficient when compared to these latter. Otherwise, due to their high fiber content, soil-fiber-PCM mixtures present a lower thermal conductivity than Cob or earth bricks.

Finally, as expected, the main enhancement brought by PCMs is noticeable on the specific heat capacity of the lightened earth, near phase change temperature, see Table 10.

Table 10: Measured hygrothermal properties for different earth-based materials.

Earth-based materials	Mix	Density [kg/m ³]	Sorption max [%]	μ (dry cup)	Cp [J/(kg.K)]	λ [W/(m.K)]	Ref
Lightened earth	Earth + 25 % Reed + 0 – 20 % PCM	650 - 700	6 - 7,7 at RH=90 %	7,10 - 8,67	537 - 2130 at 24 °C	0,134 – 0,156 at 24 °C	Present study
	Earth + Straw	241 - 531	12 at RH=93 %	4,8	-	0,071 – 0,120 at 25 °C	[25]
	Earth + Typha Australis (20 %, 33 %)	323 - 586	12,9 at RH=97 %	3,748 - 7,057	-	0,065 – 0,112 at 23 °C	[24]
	Earth + hemp shiv 34 % - 67 %	200-350	5,3 - 7,56 at RH=80 %	2.24 - 4.14	965 - 1105	0.06 – 0.12 at 23 °C	[9]
Other earth-based materials	Unfired earth bricks	1761 - 1797	5,3 at RH=95 %	-	817,6 at 40°C	0,77 – 0,95	[18]
	Extruded earth brick	1940 - 2070	4 - 6 at RH=97 %	7 - 9	900 - 960	0,47 – 0,59 at 25 °C	[17]
	Cob	1462 - 2011	2,8 – 4,1 at RH=90 %	7 – 9,98	-	0,616 – 1,933	[12]

5. Conclusion

In this work, we were interested in the study of PCM effect on thermal, hygroscopic and mechanical properties of a lightened earth. Samples with different PCM content were elaborated. Thermal conductivity, specific heat capacity, sorption behavior, water vapor permeability and compressive strength were studied.

Regarding thermal properties, the study showed that mixtures thermal conductivity decreases when PCM content increases. This is supposed due to the weak thermal conductivity of the PCM and to the disorder increase when phase change takes place inside microcapsules. At 24 °C, the mixture with 20 % PCM was approximately 14 % less conductive than the one without PCM. Also, PCM incorporation showed an interesting effect on the lightened earth specific heat capacity. In fact, we saw that C_p increases linearly with PCM content. Near the phase-change temperature, mixture with 20 % PCM presented a C_p value 4 times larger than the one of the sample without PCM.

Regarding the hygroscopic properties, results indicate that PCM incorporation degrades the lightened earth hygroscopic properties. Indeed, soil-fiber mixtures sorption behavior and water vapor permeability are inversely proportional to the PCM content. The drop is supposed due to the PCM hydrophobic nature and porosity decrease which results in degradation of both properties. These two properties drop approximately in the same proportions, for a PCM content of 20 %, the decrease is about 20 %.

Looking changes in lightened earth hygrothermal behavior, a numerical study might be relevant to evaluate the PCMs real impact on indoor air and thermal comfort.

Furthermore, lightened earth presented a weak mechanical resistance but sufficient to be self-supporting. The addition of PCM has almost no effect on mechanical strength of the soil-fiber mixtures.

Finally, it can be stated that incorporation of phase change materials in lightened earth improves their insulation level, their heat storage capacity and keep their compressive strength almost unchanged. From another side, PCM incorporation degrades the mixtures hygroscopic properties. However, whether it is sorption behavior or water vapor permeability, the soil-fiber-PCM mixtures remain comparable to other earth-based materials and much more hygroscopic than conventional building materials.

Nextek 24D is composed by a paraffinic core and a polymeric shell. It is highly probable that its introduction into lightened earth will increase the environmental impact of the material. Consequently, the following question is worth asking: Does the reduction of the environmental impact due to energy savings generated by PCMs remain interesting in view of the impact generated by the production, transport, and disposal of this material?

In continuity of this work, simulations of energy saving and environmental impact due to PCM incorporation into lightened earth will be performed. The case of biobased PCMs will be carefully considered.

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