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1 Investigation and Properties of a Novel Composite Bio-PCM to Reduce Summer Energy

2 Consumptions in Buildings of Hot and Dry Climates.

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8 ABSTRACT

9 The use of bio-based Phase Change Materials (bio-PCM) is an effective mean to enhance the building's 10 Thermal Energy Storage (TES) capacity with low-cost and eco-friendly materials. The present study encompasses 11 the results of thermal, physico-chemical and mechanical characterizations of a novel composite bio-PCM 12 developed in the laboratory to improve the thermal comfort in the building's indoor environments. We tested a 13 non-cocoa Vegetable Fat (VF) recovered from confectionery manufacturing industry. The VF is incorporated into 14 a mineral board prepared from Natural Clay (NC) and Cellulose Fibers (CF). Phase change temperatures and TES 15 capacity of the composite at melting and freezing cycles are measured with Differential Scanning Calorimetry 16 (DSC) to be at 34.83 °C, 22.34°C and 60.41J/g, 62.39 J/g respectively. The Scanning Electron Microcopy (SEM) 17 showed that the VF was evenly distributed into the microstructure of the mineral board. Results of thermal 18 stability tests (more than 2000 cycles) confirmed that the composite bio-PCM is thermally and chemically stable. 19 The compressive strength of the supporting material was slightly reduced after the incorporation of the bio-PCM, 20 but it remains within the accepted limits for non-structural building applications. This research on thermal and 21 mechanical properties has revealed that this homemade PCM panel could be a potential candidate for an 22 application in buildings of hot and dry climates.

23 Key words

24 Solar energy storage; Bio-based PCM; Bio-composite material; Thermal comfort; Building envelope.

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25 **1. Introduction**

26 Globally, the success of a building depends on its energy consumption and the thermal comfort provided to 27 its occupants [1]. Thermal Energy Storage (TES) systems take an important place in cooling/ heating buildings 28 and reducing or time shifting their peak energy demand [2]; one solution that has been identified as an effective 29 way to improve the TES capability of building envelopes is the use of Phase Change Material (PCM) [3]. These 30 PCMs lead to a passive lowering of the indoor temperature fluctuations and peaks thanks to the Latent Heat 31 Thermal Energy Storage capacity (LHTES) [4]. During their melting process, PCMs absorb the excessive heat 32 from the ambient air. In contrast, it restores the heat during the solidification process when the temperature drops 33 and reaches the solidification point of the substance.

The conventional substances commonly used for LHTES are paraffin waxes, salt hydrates, eutectics (organicorganic, inorganic-inorganic and organic-inorganic) and fatty acids [5], [6]. Several studies investigated the use of these chemical substances in building applications and explored experimentally and numerically their main advantages and disadvantages [7, 11]. Today, the challenge is to find the ideal bio-based PCM or bio-composite-PCM working in a similar manner and making its application in buildings easy, cost-effective and practicable with low-environmental impact [12].

40 In recent years, several research studies demonstrated that the use of bio-based PCMs for LHTES in 41 buildings is greatly appreciated because of the fact that these substances are not expensive, renewable, non-toxic, 42 available and accessible [13]. Supes et al. [14] stated that fat and vegetable oils in their natural form have a useful 43 phase change behavior for LHTES applications in buildings and could constitute a biomaterial alternative to 44 paraffin and salt hydrates. Kosny et al. [15] studied the dynamic thermal performance of an insulation material 45 enhanced with a bio-based PCM; Jeong et al. [16] investigated the thermal performance of a composite Boron 46 Nitride/ bio-based PCM with a melting point of 29.38°C. Hu et al. [17] studied the thermal and mechanical 47 properties of a composite bio-based PCM when Kang et al. [18] experimented a composite Silica fume/ Bio-based 48 PCM with a melting point of 29.24 °C for building applications. Boussaba et al. [19], [20] explored the thermal 49 and physico-chemical properties of 100 % composites bio-based PCMs with 26.53 °C and 22.63 °C melting 50 points respectively. Coconut fat and Palm Kernel vegetable fat were used in their natural forms as bio-based 51 PCMs.

According to their melting and solidification points, bio-based PCMs could be manufactured for various passive building applications, their thermal potential is highly related to the local weather of the building [21]. The incorporation of these substances in the building skin materials is a key parameter to control the interior temperature with a minimum usage of electricity or any other source of energy.

Although a large number of studies have focused on the investigation of bio-based PCMs for building applications under various climatic conditions, there remains a broad range of unexplored substances and their TES behavior especially after several repetitive heat storage/discharge cycles.

The present study points to investigate experimentally and numerically the potential of a 100 % bio-based composite-PCM prepared from non-cocoa-vegetable fat recovered from manufacturing industry of confectionery. The use of this substance in the food industry is more into disuse because its amount in the confectionery manufacturing becomes increasingly limited [22].

The DSC results show the phase change properties of the non-cocoa vegetable fat, before and after its incorporation into the microstructure of an eco-friendly supporting material prepared in the laboratory from natural clay and cellulose fibers. The preparation process of the supporting material and its compounds was detailed in our previous publications [19], [20].

The bio-composite PCM melts at 34.83 °C and solidifies at 22.34 °C. This phase change property makes its application suitable in buildings of hot and dry climates where temperatures are usually very high (above 40 °C) during the day and drop brutally during the night by around 15°C-20°C especially in the summer [23], [24]. These weather conditions lead to activate the potential of the bio-PCM to switch repeatedly between the two phases in order to limit the indoor thermal fluctuations.

Prior experimental and numerical modeling studies were carried out respectively on a small-scale wall mounted on a test bench and on COMSOL Multiphysics[®]. The aim is to get tangible indications about the potential of the novel composite-PCM in enhancing the thermal performance of a building envelope model subjected to a thermal condition scenario of a hot climate.

Furthermore, the novel composite bio-PCM was subject to more than 2000-repeated thermal cycling test in order to verify the reliability of the building material after several melting and freezing cycles. Both DSC and Fourier Transformed Infrared Spectroscopy (FT-IR) were applied to the novel composite-PCM before and after
the thermal cycling test in order to assess respectively the thermal and the chemical stability of the material.

Scanning Electron Microscopy (SEM) images showed that the bio-based PCM was successfully incorporated into the microstructure of its supporting material. The compressive strength characterization aimed to assess the mechanical compressive resistance of the novel material and the effect of incorporating the bio-based PCM into its microstructure on the mechanical stability of the bio-composite PCM.

84 **2. Materials and method**

85 2.1. Materials

The non-cocoa vegetable fat (Fig.1) was used in its natural form to assess its potential for LHTES applications. The substance was recovered from Fine Inddustrie Food (FIF), Algiers (Algeria). It is a blend of several vegetable fats (Illipe butter, Shea butter, Kokum butter and Palm oil) traditionally used in confectionery manufacturing, but each day more in disuse because of its impact on health. The primary fatty acids present in these fats are: Palmitic, Stearic Oleic and Linoleic acid [25].

The phase change temperatures of this vegetable fat were found to be 34.94 °C and 22.69 °C for melting and solidification cycles respectively, with respective latent heat thermal energy storage capacities of 108.83 J/g and 109.73 J/g. The experimented PCM substance solidifies at a temperature below the melting temperature by 12.27 °C, such phenomenon is known as super-cooling of the PCM [48], [49], and [50]. In general, super-cooling is disadvantageous in LHTES using PCMs but it depends on the thermal condition of the intended application. The investigation of thermal properties of the explored PCM showed that the material could be a potential candidate bio-based PCM for passive LHTES applications in buildings.

The bio-based PCM was incorporated with a rate of 56 wt. % into the microstructure of a mineral board prepared in the laboratory essentially from natural clay; cellulose fibers obtained by recycling cardboards and recycled graphite to improve the thermal conductivity of the final material. The addition of recycled graphite enhanced its thermal conductivity by 415 % (from 0.2 W.m⁻¹.K⁻¹ to 0.83 W.m⁻¹.K⁻¹) to approach the thermal conductivity values of the conventional building materials. 103 The composite bio-based PCM panel was covered with an aluminum foil to prevent leakages or seepages of 104 the PCM in its melted state without affecting its thermal conductivity (Fig.2). The final composite-PCM 105 underwent 2000 accelerated thermal cycles that demonstrated its good stability after repeated and accelerated 106 melting/solidification cycles.

107 K-type thermocouples connected to a computer by means of a data logger were placed on an experimental
 108 test bench to register the evolution of temperatures in a model of a building envelope integrating the novel
 109 composite PCM panel subjected to an artificial solar radiation.

110 2.2. Procedure

111 The validation process of the potential of the novel candidate composite bio-based PCM for LHTES 112 applications in buildings of hot and dry climates has gone through several stages:

- The non-cocoa vegetable fat was left under laboratory conditions at room temperature in its natural form without any treatment for more than two years before the beginning of the tests.
- The substance did not exhibit any sign of chemical degradation or oxidation.
- A DSC test done on this substance revealed its potential as a bio-based PCM for passive LHTES in
 buildings.

The phase change behavior of the VF gives an indication about the weather conditions that could activate the potential of the PCM for an optimal passive LHTES.

- The bio-based PCM was incorporated into the microstructure of an eco-friendly supporting material
 to prevent leakages or seepages during the melting process.
- Thermal, physico-chemical and mechanical characterization means revealed the properties of the
 novel composite bio-based PCM and its thermo-chemical stability after thermal cycling test.
- The novel material was tested experimentally in a vertical wall model subjected to an artificial heat flux mounted on a test bench (Fig.3) which reproduced experimentally a summer temperature condition scenario of a hot and dry climate. The temperature profiles on both the exposed temperature (P1) and that on the internal surface areas of the walls (P2) were registered by means of K-type thermocouples. In addition, the two walls were modeled in COMSOL Multiphysics ®

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software to compare the two results obtained numerically and experimentally. Some results of this final part are presented, but not in full details in this paper due to the lack of space.

131 2.3. Characterization

132 Differential Scanning Calorimetry (METTLER TOLEDO DSC 1 STAR^e SYSTEM calorimeter) analyzed the 133 phase change properties such as melting and freezing points and latent heat capacity of the bio-based PCM and 134 the bio-composite PCM. This device measures the difference between the heat flows from two sides of a very 135 sensitive sensor (FRS 5) placed in a silver-furnace as a function of the temperatures imposed. The Reference side 136 "R" of the sensor receives the crucible that contains the reference material. In contrast the characterized material 137 is placed in another crucible placed in the Sample side "S" of the same sensor. Each sample (8-10 mg) was tested 138 from 5 °C to 50 °C for heating cycle and from 50 °C to 5 °C for cooling cycle with 1°C/min scan rate in order to 139 identify the phase change properties of the novel material in the working temperature range of the intended 140 application. Each sample test was repeated three times to ensure the validity of the results. The latent heat 141 capacity of melting and freezing of each sample was determined by numerical integration of the area under the 142 peaks of phase change transitions using the DSC1 STAR^e software.

The thermal conductivity of the bio-composite PCM was measured using a Hot-Disk thermal constant analyzer (Hot-Disk TPS 500, Perkin Elmer Company) at room temperature (18 °C). The thermal conductivity of the composite-PCM is an important parameter responsible for activating the latent heat capacity potential of the PCM.

147 The thermal performance of the characterized composite PCM shaped as a panel was tested experimentally 148 on a test-bench described previously and then modeled numerically on COMSOL Multiphysics® by reporting the 149 same boundary conditions as the test-bench. Both numerical and experimental works were done on a reference 150 model that does not contain the composite-PCM panel and on a model with a composite PCM Panel. The 151 chemical stability analysis of the novel bio-composite PCM was performed by Fourier Transformed Infrared 152 Spectroscopy (FT-IR) using IR Affinity-1 Fourier Transform Infrared Spectrophotometer model of Shimadzu under the wave range 4000 cm⁻¹ - 400 cm⁻¹ with 4 cm⁻¹ resolution. The samples were shaped as circular thin 153 154 pellets using clear Potassium Bromide (KBr) as a background and then scanned 43 times in transmission mode.

The morphology and the microstructure of the bio-based composite PCM and that of the mineral panel prepared to contain the bio-based PCM into its microstructure without seepages or leakages were observed using the scanning Electron Microscopy (FEI, Quanta 650 SEM) with 30 mV of accelerating voltage at low vacuum mode and 19 °C of room temperature.

159 2.3.1. Thermal cycling test

160 Thermal cycling test is important to assess the long-term stability of the PCM's latent storage. This test 161 allows studying the thermal reliability of the bio-composite PCM after several repetitive phase change cycles; 162 since the potential of a PCM depends on its melting and freezing cycle's behavior [26].

Many devices have already been used for thermal cycling tests in the literature such as: thermal bath [27], thermal cycling chamber [28] and tubes with water circulation setup [29]. The newly developed thermal cycling test device shown in Fig.4 and its software was used in this study to operate more than 2000 thermal cycles on the prepared bio-composite PCM. The thermal cycling suitcase is controlled by a computer via Wi-Fi connection. A software adapted to the use of this device was developed to launch the thermal cycles, to configure the heating and cooling cycle's conditions and to retrieve the results of the test as a CSV file format.

The thermal cycling suitcase is equipped with the Peltier effect (Fig.4) using a thermo-electric module that generates a difference of temperature according to the applied polarity (positive to negative or vice versa); accordingly the temperature is transiently varied on each side of the module.

The thermal cycling test was operated on the novel bio-composite PCM for 2000 successive cycles from 5 °C to 50 °C for heating cycles and from 50°C to 5 °C for cooling cycles. A thin layer of thermal paste was applied as an interface between the suitcase's sample holder and the sample in order to eliminate air gaps that act as thermal insulation at the interface sample/container. A K-type thermocouple was placed in the testing sample to record the temperature evolution in the novel material during the test. The upper side of the sample holder was completely insulated (Fig.4) to prevent any influence of the laboratory temperature conditions on the experience.

178 2.3.2. Compressive strength test

The compressive strength of the bio-composite PCM was performed on cube specimens of sizes (50 x 50 x 50 mm³) (Fig. 5a). The bio-composite PCM cubes were prepared in the laboratory using a specific stainless steel mold manufactured for this specific purpose. The compressive strength result is the average of five repeated measurements done on the bio-composite cubes before and after the incorporation of the bio-based PCM. The test was performed by means of electromechanical testing machine Ibertest Model Elib-200 W (Fig. 5b). The load was applied gradually on each cube with a rate of 0.5 MPa/s at room temperature (20°C) until the destruction of the sample (Fig. 5c).

186 **3. Results and discussion**

187 3.1. Thermal properties

Fig.6 shows the DSC thermograms of the bio-based PCM (VF) and the bio-composite PCM obtained after the incorporation of the VF in the microstructure of its matrix (the mineral panel). The DSC analysis was run on the full samples exactly at the same test conditions. The thermograms were superimposed in order to compare the phase change properties of the bio-based PCM before and after its incorporation into the bio-composite mineral panel.

The phase change temperatures of the vegetable fat were found to be at 34.94 °C for melting cycle and 22.69 °C for solidification cycle, when those of the bio-composite PCM were found approximately at the same phase change temperatures. The bio-composite PCM melts at 34.83 °C and solidifies at 22.34 °C; the difference between the melting and solidification temperatures is 12.37 °C \pm 0.12 °C. This leads to classify the VF and the bio-composite PCM among super-cooled materials [30]. Materials that experience super-cooling can be employed in a broad range of building applications according to their phase change temperatures [31], [32].

From Fig.6, we can observe the latent heat reduction on the DSC curves after the incorporation of the biobased PCM in its matrix. The latent heat decreased from 108.39 J/g to 60.41 J/g for endothermic cycles and from 109.73 J/g to 62.39 J/g for exothermic cycles. This could be explained by the amount of the bio-based PCM retained by the matrix without leakages (around 56 wt. %) and with various theories such as interfacial liquid layering, Brownian motion and particle clumping [32], [33]. In contrast, the discrepancies recorded between the latent heat of fusion and solidification of both the bio-based PCM and the composite PCM could be related to the 205 thermal hysteresis phenomenon during phase transitions [53], [54]. This is induced by super-cooling in the case of 206 this study.

The thermal conductivity is a key parameter for composites-PCM, this physical property is essential to conduct the heat flux, so as to start the latent heat storage process [34]. In this study, the addition of recycled graphite led to increase the thermal conductivity of the novel material from 0.2 W.m⁻¹.K⁻¹ to 0.83 W.m⁻¹.K⁻¹. Graphite could also act an important role in enhancing the roughness of the supporting material as well as the flammability property, so it helps to prevent the problems of leakages of the PCM from its matrix in its liquid state and to improve the fire resistance of the composite-PCM [51], [52].

The DSC results show that the melting and solidification temperatures of the novel bio-composite PCM make its application suitable for passive solar TES in buildings of hot and dry climates, especially during the summer where temperatures are usually above 40 °C during the day and drop down during the night to meet temperatures around 20 °C. Consequently, the bio-composite PCM could shift some peak-cooling load to off peak periods. Table 1 compares the phase change properties of the bio-composite PCM prepared in this study with those of some other bio-composites-PCMs in the literature.

Fig.7 and Fig.8 gives prior indications about the impact of the characterized composite-PCM on a model of a building envelope submitted to an artificial solar radiation that led to heat the exposed surface area (P1) of the wall from 26 °C to 51 °C over a period of 6 hours. Both numerical and experimental studies were carried out on the reference model that does not integrate the composite PCM panel and then on the model with the composite PCM panel under the same conditions.

Fig. 7 shows the superposition of the temperature profiles registered on (P1) and on (P2) before and after the integration of the composite PCM panel respectively, over a period of 6 h. The temperature of the internal surface area (P2) in the reference model increased more quickly than on the model integrating the composite-PCM panel. It reaches 35 °C approximately at 6h; accordingly, the temperature of this internal surface (P2) increased by around 9 °C. In contrast, the temperature of the surface P2 in the model integrating the composite PCM panel evolves less quickly from 26 °C to around 29 °C on 6h. Its temperature increased by around 3 °C. Hence, we recorded a difference of 5 °C between the two models at the end of the temperature registration. These prior results give us a first indication about the impact of the novel composite PCM panel in improving the thermal performance of building envelopes.

233 Fig.8 shows the resulted thermographs from the numerical modeling on COMSOL Multiphysics ® of the 234 reference wall model (Fig. 8 B) and that of the wall integrating the composite PCM panel (Fig. 8 A) after 6 h 235 (22800 s). All boundary conditions are modeled to be the same as in the test bench. The numerical results show 236 that the minimum temperature recorded on P2 (A) was found to be at 28.08 °C and that registered on P2 (B) was 237 at 34.64 °C. The difference between the temperatures of the internal face area (P2) in the two models (with and 238 without the composite-PCM panel) is of 6.56 °C. A short discrepancy of about 1.5 °C was found compared to the 239 experimental model. Despite this, the results confirm that the composite PCM panel could be a potential candidate 240 to reduce peak load temperatures in buildings under hot thermal conditions.

241 3.2. Chemical characterization

The chemical analysis was operated by FT-IR on the bio-based PCM (VF) and on the bio-composite PCM in order to investigate their respective chemical structures and possible combinations (chemical compatibility) after the incorporation of the VF in its matrix. Fig. 9 shows the FT-IR spectra of the bio-based PCM and the biocomposite PCM before the thermal cycling test.

The peaks that were observed in the IR spectrum of the VF at 2919 cm⁻¹ and 2859 cm⁻¹ are related to the stretching vibrations of $-CH_3$ groups. The peak recorded at 1744 cm⁻¹ was caused by C=O bands. The stretching vibrations of $-CH_2$ groups led to observe specific peaks at: 1551 cm⁻¹, 1460 cm⁻¹, 967 cm⁻¹ and 723 cm⁻¹.

The IR spectrum of the bio-composite shows that each distinguishing peak recorded on the bio-based PCM spectrum reappeared clearly without wave-number shifts or changes. These outcomes confirm that the relation between the PCM and its matrix is only physical, there were no chemical reactions. Accordingly, the bio-based PCM presents a good compatibility with its matrix, so the novel bio-composite PCM is chemically stable.

253 3.3. Morphology and microstructure of the novel bio-composite PCM

The morphology and microstructure of the bio-PCM's supporting material and that of the final biocomposite-PCM were studied by means of Electron Scanning Microscopy (SEM) in order to verify if the biobased PCM was incorporated successfully in its matrix. Fig. 10 displays the SEM images of the bio-composite with different magnifications (x 100, x700 and x 9519) before and after the incorporation of the bio-PCM in its
 matrix for every magnification.

Fig. 10 a shows that the microstructure of the supporting material consists essentially of intertwined fibers. As seen from Fig.10 b, the fibrous appearance diminished significantly after the incorporation of the bio-based PCM. Moreover, Fig. 10 (b, c) and Fig. 10 (e, f) confirm the first observations and show more clearly with magnification x700 and x 9519 the presence of holes among the intertwined fibers and an important roughness on the surface of the fibers of the matrix (Fig. 10 c and e).

As seen from Fig. 10 (d, f), the micro-holes among the intertwined fibers were fully filled with the bio-based PCM and the roughness was smoothed after the incorporation of the PCM in its matrix. Through SEM observations, we conclude that the bio-based PCM was successfully incorporated into the microstructure of its matrix and uniformly distributed both on micro-gaps and on the fiber's surfaces. This was effective thanks to the capillary and surface tension forces between the bio-composite PCM's components and the accidental (rough) microstructure of the matrix.

270 3.4. Accelerated melting/cooling cycles results

Fig.11 shows the temperature profile curves of the novel bio-composite PCM during the first 10/2000 melting/cooling cycles (Fig.11 A, a) compared with that of the latest 10/2000 thermal cycles (Fig.11 B, b). The temperature profiles were recorded by means of a K-type thermocouple placed in the bio-composite PCM during the thermal cycling test. The sample was cooled down to 5 °C then heated to 50 °C periodically. The applied setpoint temperatures were respectively below the solidification temperature and above the melting temperature in order to allow the material to accomplish complete melting and cooling cycles.

The trend of recorded temperature profiles is directly related to the phase change temperatures and the heat capacity of the bio-composite PCM [35], [36]. This is why we compared the recorded curves during the first 10/2000 heating/cooling cycles with that of the latest 10/2000 of the same thermal cycling test.

As seen from Fig. 11 A, the ascending side of each peak representing the heating step shows that temperatures were nearly maintained stable between 30 °C to 34 °C, which corresponds to the melting temperature range of the bio-composite PCM. The descending side of each peak representing the cooling step illustrates a shoulder shape on the curve at around 25 °C to 15 °C that corresponds to the solidification range temperatures. Moreover, we observed a time delay in the temperature decrease during the cooling cycle. This is due to the fact that the bio-composite-PCM is not sensitive to the temperature variations during its phase change processes.

Fig. 11 B shows that the temperature profile curve of the bio-composite recorded during the above latest heating/cooling cycles is in accordance with that of the first cycles. This result means that the phase change properties of the bio-composite PCM have not changed after more than 2000 cycles. This result confirms the good stability of the material during thermal cycling. An analysis by DSC was operated on this sample after thermal cycling test in order to verify this result.

292 3.5. Thermal and chemical stability test

The long-term stability of the composite-PCM used for LHTES in buildings is a key parameter since the material should have essentially good thermal and chemical properties stability after several repeated melting/solidification cycles. Tests of DSC and FT-IR were carried out on the novel bio-composite-PCM sample, which underwent the 2000 accelerated thermal cycles in order to investigate its thermal and chemical reliability after these successive repeated cycles.

Fig. 12 shows the DSC curves of the VF and the bio-composite PCM before thermal cycling superimposed with that of the bio-composite PCM after the accelerated thermal cycling test. The melting and solidification temperatures changed insignificantly after accelerated thermal cycling from 34.83 °C to 34.47 °C and from 22.34 °C to 22.10 °C respectively. The latent heat capacity changed from 60.41 J/g to 60.75 J/g (+0.34 J/g) for melting cycle and from 62.39 J/g to 64.38 J/g (+1.99 J/g) for cooling cycle. This slight increase in the latent heat capacity after thermal cycling could be related to the elimination of some impurities contained in the VF during the accelerated and repeated heating/cooling cycle's runs.

This result means that the minor shifts in the phase change properties after thermal cycling are negligible for LHTES applications, so the novel bio-composite PCM has good thermal properties stability after thermal cycling.

Fig. 13 shows the FT-IR spectra of the novel bio-composite PCM before and after the thermal cycling test.
 The IR spectrum of the cycled bio-composite PCM is in perfect accordance with that of un-cycled one. We did

309 not record any shift in the shapes of the peaks and in their frequency, so accelerated thermal cycling test did not 310 affect the chemical structure of the novel bio-composite PCM. This result means that the novel material is 311 chemically stable.

312 3.6. Compressive strength properties

The compressive strength data of the novel bio-composite before and after the incorporation of the bio-based PCM into its microstructure are presented in Fig. 14. As seen from the two curves, the compressive strength decreased from 4.6 MPa to 2.8 MPa after the incorporation of the VF into its matrix. Moreover, the curve of the supporting material (matrix) presented ductility under compressive stress before breaking. This property decreased significantly after the incorporation of the bio-based PCM.

The bio-composite PCM has less ability to undergo deformations before breaking, it became more brittle. This could be explained by the redistribution of the micro-holes in the composite caused by the incorporation of the bio-based PCM. In contrast, the reduction of the compressive strength of the bio-composite PCM can be explained by the fact that the VF has a very low compressive strength, and then its incorporation into the microstructure of its matrix affected the structure and crystal size of the novel material on the interface (bio-based PCM/ matrix).

Despite the decrease in the compressive strength of the bio-composite PCM, it remains among acceptable values for non-load-bearing applications in buildings. Table 2 compared the compressive strength value of the bio-composite PCM experimented in this study with that of some other composites-PCM dedicated to building applications in the literature.

328 4. Conclusions

In this study, we investigated experimentally and numerically the thermal potential of a novel bio-composite PCM prepared from eco-friendly and low-cost waste bio-based PCM (The non-cocoa vegetable fat) recovered from the food industry. Developing cost effective composites-PCM for solar thermal energy became an important challenge for future solar thermal technology applications [55]. The bio-composites PCM prepared from recovered components were recognized as economically as well as environmentally friendly materials with sufficient thermal performance for building applications [26], [56], [57]. We focused on the use of a bio-based PCM in its natural form without any prior treatment before its incorporation in a mineral panel prepared
essentially from natural clay and cellulose fibers obtained by recycling waste cardboards.

The DSC results showed that the phase change properties of the novel material and the difference of ± 12 °C between the melting and freezing cycles make its application suitable for passive LHTES in buildings of hot and dry climates where the difference between night and day temperatures are in the range of 15 °C to 20 °C. The thermal behavior of the novel material confirms that its building application in such thermal conditions could be effective to enhance the thermal comfort essentially by delaying the temperature heat wave between the indoor and outdoor environments especially in the summer.

343 FT-IR results demonstrated that the relation between the bio-based PCM and its supporting material is only 344 physics; the material could be used for solar TES in buildings without any risk of chemical degradation. This 345 result was confirmed by SEM observations; the bio-based PCM was successfully retained by its matrix thanks to 346 the capillary and surface tension forces. The thermal cycling test confirmed that the novel bio-composite PCM has 347 good thermal and chemical stability after 2000 accelerated and repeated melting/ freezing cycles. Furthermore, the 348 mechanical characterization showed that the incorporation of the PCM in its supporting material led to decrease 349 its compressive strength. Despite this, it remains within accepted limits for non-structural building applications. 350 Further characterization could be important to look on the flammability and the toxicity of the composite as well 351 as the impact of the addition of fire retardants on its thermal properties.

Ongoing research on the prepared bio-composite PCM aims to study the thermal performance enhancement after the application of the novel material in a real-scale building located in southern Algeria. An experimental test bench dedicated to investigating the thermal performance of a real-scale wall with the novel bio-composite PCM board under summer temperature scenarios of hot and dry climate is currently understudy.

14

- 356 Table 1: Comparison between the thermal properties results of the present study with that of some other results in 357 the literature.
- 358 Table 2: Comparison between the compressive strength values of the prepared bio-composite PCM with that of
- 359 some other composites-PCM proposed for building applications in the literature.
- 360 Figure 1: The image of the non-cocoa vegetable fat used as a bio-based PCM.
- 361 Figure 2: The images of the final shape of the elaborated bio-composite-PCM board.
- 362 **Figure 3:** a) The image of the experimental test bench; b) Schematic longitudinal section of the test bench; A)
- 363 Schematic representation of the test wall with the composite PCM; B) Schematic representation of the test wall
- 364 without the composite PCM (The reference model).
- 365 **Figure 4:** Thermal cycling test representation.
- 366 Figure 5: Compressive strength test: a) The samples, b) The compressive strength test device, c) Some
- 367 compression destructive test steps.
- **Figure 6:** DSC thermograms of the bio-based PCM (VF) and the bio-composite PCM before thermal cycling.
- 369 Figure 7: Temperature profiles recorded on the respective inside and outside surface areas (P1), (P2) of the test
- 370 wall before and after inserting the composite PCM panel.
- 371 Figure 8: The numerical temperature profiles thermographs: A) The model integrating the composite-PCM panel;
- B) The reference model (without the composite-PCM panel).
- 373 **Figure 9:** FT-IR spectra of the bio-based PCM and the bio-composite PCM before thermal cycling.
- Figure 10: SEM microstructure images of the PCM's supporting material (a, c, e) and that of the bio-composite
- 375 PCM (b, d, f) under different magnifications (x 100 and x700 and x9519) respectively.
- **Figure 11:** Temperature (time) profiles of the bio-composite heating/cooling cycles run. A, a) the first ten cycles;
- B, b) the latest ten cycles.
- Figure 12: Comparison between the DSC thermograms of the bio-composite PCM before and after thermalcycling test.
- **Figure 13:** Comparison between the IR- spectra of the bio-composite PCM before and after thermal cycling.

Figure 14: Comparison between the compressive strength of the bio-composite before and after the incorporationof the bio-based PCM.

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387 References

- [1] R. Stropnik, R. Koželj, E. Zavrl, U. Stritih, Improved thermal energy storage for nearly zero energy buildings
 with PCM integration, Sol. Energy. 190 (2019) 420–426.
- 390 [2] Y.P. Zhang, K.P. Lin, R. Yang, H.F. Di, Y. Jiang, Preparation, thermal performance and application of shape 391 stabilized PCM in energy efficient buildings, Energy Build. 38 (2006) 1262–1269.
- 392 [3] G. Zhou, Y. Zhang, X. Wang, K. Lin, W. Xiao, An assessment of mixed type PCM-gypsum and shape-
- 393 stabilized PCM plates in a building for passive solar heating, Sol. Energy. 81 (2007) 1351–1360.
- 394 [4] R. Yao, V. Costanzo, X. Li, Q. Zhang, B. Li, The effect of passive measures on thermal comfort and energy
- conservation. A case study of the hot summer and cold winter climate in the Yangtze River Region, J. Build.
 Eng. 15 (2018) 298-310.
- K. Peippo, P. Kauranen, P.D. Lund, A multicomponent PCM wall optimized for passive solar heating, Energy
 Build. 17 (1991) 259–270.
- 399 [6] K.O. Lee, M.A. Medina, X. Sun, X. Jin, Thermal performance of phase change materials (PCM)-enhanced
- 400 cellulose insulation in passive solar residential building walls, Sol. Energy. 163 (2018) 113–121.
- 401 [7] R. Arivazhagan, N.B. Geetha, P. Sivasamy, P. Kumaran, M. Kumara Gnanamithra, S. Sankar, G.
- 402 BabuLoganathan, A. Arivarasan, Review on performance assessment of phase change materials in buildings
- 403 for thermal management through passive approach, Mater. Today Proc. 22 (2020) 419–431.

- 404 [8] D. Li, Y. Zheng, C. Liu, G. Wu, Numerical analysis on thermal performance of roof contained PCM of a
 405 single residential building, Energy Convers. Manag. 100 (2015) 147–156.
- 406 [9] E.M. Alawadhi, H.J. Alqallaf, Building roof with conical holes containing PCM to reduce the cooling load:
 407 Numerical study, Energy Convers. Manag. 52 (2011) 2958–2964.
- 408 [10] E.I. Mohamed Moussa, M. Karkri, A numerical investigation of the effects of metal foam characteristics
- 409 and heating/cooling conditions on the phase change kinetic of phase change materials embedded in metal
- 410 foam, J. Energy Storage. 26 (2019) 100985.
- 411 [11] L. Royon, L. Karim, A. Bontemps, Thermal energy storage and release of a new component with PCM
- 412 for integration in floors for thermal management of buildings, Energy Build. 63 (2013) 29-35.
- 413 [12] R. Saxena, D. Rakshit, S.C. Kaushik, Phase change material (PCM) incorporated bricks for energy
- 414 conservation in composite climate: A sustainable building solution, Sol. Energy. 183 (2019) 276–284.
- 415 [13] F. Frota de Albuquerque Landi, C. Fabiani, A.L. Pisello, Palm oil for seasonal thermal energy storage
- 416 applications in buildings: The potential of multiple melting ranges in blends of bio-based fatty acids, J.
- 417 Energy Storage. 29 (2020) 101431.
- 418 [14] G.J. Suppes, M.J. Goff, S. Lopes, Latent heat characteristics of fatty acid derivatives pursuant phase
 419 change material applications, Chem. Eng. Sci. 58 (2003) 1751–1763.
- 420 [15] J. Kosny, E. Kossecka, A. Brzezinski, A. Tleoubaev, D. Yarbrough, Dynamic thermal performance
- 421 analysis of fiber insulations containing bio-based phase change materials (PCMs), Energy Build. 52 (2012)
 422 122–131.
- 423 [16] S.-G. Jeong, J.-H. Lee, J. Seo, S. Kim, Thermal performance evaluation of Bio-based shape stabilized
 424 PCM with boron nitride for energy saving, Int. J. Heat Mass Transf. 71 (2014) 245–250.
- 425 [17] W. Hu, X. Yu, Thermal and mechanical properties of bio-based PCMs encapsulated with nanofibrous
 426 structure, Renew. Energy. 62 (2014) 454–458.
- 427 [18] Y. Kang, S.-G. Jeong, S. Wi, S. Kim, Energy efficient Bio-based PCM with silica fume composites to
- 428 apply in concrete for energy saving in buildings, Sol. Energy Mater. Sol. Cells. 143 (2015) 430–434.
- 429 [19] L. Boussaba, A. Foufa, S. Makhlouf, G. Lefebvre, L. Royon, Elaboration and properties of a composite
- 430 bio-based PCM for an application in building envelopes, Constr. Build. Mater. 185 (2018) 156–165.

- 431 [20] L. Boussaba, S. Makhlouf, A. Foufa, G. Lefebvre, L. Royon, The Palm Kernel Vegetable Fat: a Low-cost
- Bio-based Phase Change Material for Thermal Energy Storage in Buildings, J. Build. Eng. 21 (2019). 222–
 229.
- 434 [21] C. Castellón, A. Castell, M. Medrano, I. Martorell, L.F. Cabeza, Experimental Study of PCM Inclusion in
 435 Different Building Envelopes, J. Sol. Energy Eng. 131 (2009).
- 436 [22] M. Lipp, E. Anklam, Review of cocoa butter and alternative fats for use in chocolate—Part A.
 437 Compositional data, Food Chem. 62 (1998) 73–97.
- 438 [23] A. Waqas, S. Kumar, Thermal performance of latent heat storage for free cooling of buildings in a dry
 439 and hot climate: An experimental study, Energy Build. 43 (2011) 2621–2630
- 440 [24] M. Sovetova, S.A. Memon, J. Kim, Thermal performance and energy efficiency of building integrated
 441 with PCMs in hot desert climate region, Sol. Energy. 189 (2019) 357–371.
- 442 [25] V.K.S. Shukla, Confectionery fats, in: R.J. Hamilton (Ed.), Dev. Oils Fats, Springer US, Boston, MA,
- 443 1995: pp. 66–94. https://doi.org/10.1007/978-1-4615-2183-9_3.
- 444 [26] A. Sarı, G. Hekimoğlu, V.V. Tyagi, Low cost and eco-friendly wood fiber-based composite phase change
- 445 material: Development, characterization and lab-scale thermoregulation performance for thermal energy
 446 storage, Energy. 195 (2020) 116983.
- 447 [27] A. Sari, R. Eroglu, A. Biçer, A. Karaipekli, Synthesis and Thermal Energy Storage Properties of
- 448 Erythritol Tetrastearate and Erythritol Tetrapalmitate, Chem. Eng. Technol. 34 (2011) 87–92.
- 449 [28] C. Arkar, S. Medved, Free cooling of a building using PCM heat storage integrated into the ventilation
- 450 system, Sol. Energy. 81 (2007) 1078–1087.
- 451 [29] H. Kimura, J. Kai, Phase change stability of CaCl2·6H2O, Sol. Energy. 33 (1984) 49–55.
- 452 [30] A. Safari, R. Saidur, F.A. Sulaiman, Y. Xu, J. Dong, A review on supercooling of Phase Change
- 453 Materials in thermal energy storage systems, Renew. Sustain. Energy Rev. 70 (2017) 905–919.
- 454 [31] X. Jin, H. Hu, X. Shi, X. Zhang, Energy asymmetry in melting and solidifying processes of PCM, Energy
- 455 Convers. Manag. 106 (2015) 608–614.
- 456 [32] B. Zalba, J.M. Marín, L.F. Cabeza, H. Mehling, Review on thermal energy storage with phase change:
- 457 materials, heat transfer analysis and applications, Appl. Therm. Eng. 23 (2003) 251–283.

- [33] N. Putra, M. Amin, E.A. Kosasih, R.A. Luanto, N.A. Abdullah, Characterization of the thermal stability
 of RT 22 HC/graphene using a thermal cycle method based on thermoelectric methods, Appl. Therm. Eng.
 124 (2017) 62–70.
- 461 [34] A. Zabalegui, D. Lokapur, H. Lee, Nanofluid PCMs for thermal energy storage: Latent heat reduction
 462 mechanisms and a numerical study of effective thermal storage performance, Int. J. Heat Mass Transf. 78

463 (2014) 1145–1154.

- 464 [35] E. Elnajjar, Using PCM embedded in building material for thermal management: Performance assessment
 465 study, Energy Build. 151 (2017) 28–34.
- 466 [36] S. Lashgari, H. Arabi, A.R. Mahdavian, V. Ambrogi, Thermal and morphological studies on novel PCM
 467 microcapsules containing n-hexadecane as the core in a flexible shell, Appl. Energy. 190 (2017) 612–622.
- 468 [37] M. Fashandi, S.N. Leung, Preparation and characterization of 100% bio-based polylactic acid/palmitic
 469 acid microcapsules for thermal energy storage, Mater. Renew. Sustain. Energy. 6 (2017) 14.
- 470 [38] W. Hu, X. Yu, Thermal and mechanical properties of bio-based PCMs encapsulated with nanofibrous
 471 structure, Renew. Energy.62 (2014) 454–458.
- 472 [39] M. Amin, N. Putra, E.A. Kosasih, E. Prawiro, R.A. Luanto, T.M.I. Mahlia, Thermal properties of
- beeswax/graphene phase change material as energy storage for building applications, Appl. Therm. Eng. 112
 (2017) 273–280.
- 475 [40] A. Karaipekli, A. Sarı, Development and thermal performance of pumice/organic PCM/gypsum
- 476 composite plasters for thermal energy storage in buildings, Sol. Energy Mater. Sol. Cells. 149 (2016) 19–28.
- 477 [41] L. Li, H. Yu, R. Liu, Research on composite-phase change materials (PCMs)-bricks in the west wall of
 478 room-scale cubicle: Mid-season and summer day cases, Build. Environ. 123 (2017) 494–503.
- 479 [42] B. Srinivasaraonaik, L.P. Singh, S. Sinha, I. Tyagi, A. Rawat, Studies on the mechanical properties and
 480 thermal behavior of microencapsulated eutectic mixture in gypsum composite board for thermal regulation in
- 481 the buildings, J. Build. Eng. 31 (2020) 101400.
- 482 [43] Y. He, X. Zhang, Y. Zhang, Q. Song, X. Liao, Utilization of lauric acid-myristic acid/expanded graphite
- 483 phase change materials to improve thermal properties of cement mortar, Energy Build. 133 (2016) 547–558.

- 484 [44] X. Kong, Y. Zhong, X. Rong, C. Min, C. Qi, Building Energy Storage Panel Based on Paraffin/Expanded
- 485 Perlite: Preparation and Thermal Performance Study, Materials. 9 (2016)70.
- 486 https://doi.org/10.3390/ma9020070
- 487 [45] L.O. Afolabi, Z.M. Ariff, P.S.M. Megat-Yusoff, H.H. Al-Kayiem, A.I. Arogundade, O.T. Afolabi-
- 488 Owolabi, Red-mud geopolymer composite encapsulated phase change material for thermal comfort in built489 sector, Sol. Energy. 181 (2019) 464–474.
- 490 [46] P. Jana, V. Fierro, A. Celzard, Sucrose-based carbon foams with enhanced thermal conductivity, Ind.
 491 Crops Prod. 89 (2016) 498–506.
- 492 [47] L. Zhang, J. Zhu, W. Zhou, J. Wang, Y. Wang, Thermal and electrical conductivity enhancement of
- 493 graphite nanoplatelets on form-stable polyethylene glycol/polymethyl methacrylate composite phase change
 494 materials, Energy. 39 (2012) 294–302.
- 495 [48] H. Hu, X. Jin, X. Zhang, Effect of Supercooling on the Solidification Process of the Phase Change
 496 Material, Energy Procedia. 105 (2017) 4321–4327.
- 497 [49] A. Bland, M. Khzouz, T. Statheros, E.I. Gkanas, PCMs for Residential Building Applications: A Short
- 498Review Focused on Disadvantages and Proposals for Future Development, Buildings. 7 (2017) 78.
- 499 [50] A. Safari, R. Saidur, F.A. Sulaiman, Y. Xu, J. Dong, A review on supercooling of Phase Change
- 500 Materials in thermal energy storage systems, Renew. Sustain. Energy Rev. 70 (2017) 905–919
- 501 [51] Z.A. Qureshi, H.M. Ali, S. Khushnood, Recent advances on thermal conductivity enhancement of phase
- 502 change materials for energy storage system: A review, Int. J. Heat Mass Transf. 127 (2018) 838–856.
- 503 [52] P. Zhang, Y. Hu, L. Song, J. Ni, W. Xing, J. Wang, Effect of expanded graphite on properties of high-
- density polyethylene/paraffin composite with intumescent flame retardant as a shape-stabilized phase change
 material, Sol. Energy Mater. Sol. Cells. 94 (2010) 360–365.
- 506 [53] A. Buonomano, F. Guarino, The impact of thermophysical properties and hysteresis effects on the energy
- 507 performance simulation of PCM wallboards: Experimental studies, modelling, and validation, Renew.
- 508 Sustain. Energy Rev. 126 (2020) 109807.
- 509 [54] T. Barz, A. Sommer, Modeling hysteresis in the phase transition of industrial-grade solid/liquid PCM for
- 510 thermal energy storages, Int. J. Heat Mass Transf. 127 (2018) 701–713.

- 511 [55] Y. Jiang, M. Liu, Y. Sun, Review on the development of high temperature phase change material
 512 composites for solar thermal energy storage, Sol. Energy Mater. Sol. Cells. 203 (2019) 110164.
- 513 [56] A. Sarı, T.A. Saleh, G. Hekimoğlu, M. Tuzen, V.V. Tyagi, Evaluation of carbonized waste tire for
- 514 development of novel shape stabilized composite phase change material for thermal energy storage, Waste
- 515 Manag. 103 (2020) 352–360.
- 516 [57] J. Yoo, S.J. Chang, S. Wi, S. Kim, Spent coffee grounds as supporting materials to produce bio-composite
- 517 PCM with natural waxes, Chemosphere. 235 (2019) 626–635.
- 518



Figure 1:







Figure 3:



Figure 4:



Figure 5:



Figure 6:



Figure 7:



Figure 8:



Figure9:



Figure10:



Figure11:



Figure12:



Figure13:



Figure14:

Ta	ble	1:

The composite PCM	Melting points (°C)	Freezing points (°C)	Latent Heat (J/g)	References
PLA-PA Micro PCM 24%	61.90	-	40.70	[37]
Soy wax/ Nano- fibrous material	50.38	-	36.47	[38]
Beeswax/ Graphene	60.2	59.4	91.1	[39]
Palm kernel fat composite	27.33	26.88	40.27	[20]
Coconut fat composite	23.79	21.99	60.72	[19]
FS-CPCMs	23.27	20.98	55.40	[40]
Composite PCM/ Brick	15.80	22.28	61.44	[41]
Bio-composite Non cocoa vegetable fat	34.83	22.34	62.39	This study

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Table 2:

Samples	Compressive strength (MPa)	References
Gypsum/M-PCM	5- 6.5	[42]
Graphite/ lauric-acid Myristic-acid	0.2- 5.39	[43]
Paraffin/ expanded perlite	4.61	[44]
Red-mud geopolymer PCM	(6.6-8.5).10-9	[45]
Carbon foam/PCM	0.89	[46]
Graphite nano- patelets/polyethylene glycol	3-6	[47]
Bio-composite natural clay- cellulose fibers/ VF (bio-PCM)	2.83-4.6	This study