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DEVELOPMENT AND QUALIFICATION OF AN EXPERIMENTAL METHOD FOR PCMs LIQUID PHASE THERMAL EXPANSION COEFFICIENT MEASUREMENT

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ABSTRACT

The development and industrialization of latent heat thermal energy storage technologies require a thorough mastery of the thermophysical properties of phase change materials (PCMs) used for the storage. These properties are often dependent on the material's temperature, and the literature highlights the influence of their variation on the performance of latent storage systems. In this study, the influence of the variation of the thermal expansion coefficient on the performance of a latent storage unit was analyzed. Following this numerical analysis, an experimental setup was proposed to accurately measure the temperature dependence of the liquid phase density and the thermal expansion coefficient of PCMs. A precision of more than 99 % was reached for the liquid phase density when compared to data obtained using the D5-METTLER TOLEDO densimeter and to water data from the literature. The experimental procedure was then applied to the characterization of six PCMs for building applications.

1. INTRODUCTION

Latent Heat Thermal Energy Storage (LHTES) technology is of great interest for energy transition through the valorization of solar energy as a substitute for fossil fuels. It allows for the storage of energy in both sensible and latent forms within Phase Change Materials (PCMs). This offers a higher storage density, reducing the amount of material needed for sensible storage and compacting the systems. Unfortunately, despite its promise, latent heat storage technology suffers from low charging and discharging rates due to the low thermal conductivity of PCMs. As a result, several techniques have been proposed in the literature to enhance heat transfers in LHTES systems (Kaboré *et al.*, 2023-1), (Dekhîl *et al.*, 2022). To successfully mature and industrialize LHTES systems, it is imperative to understand the various physical, thermal, and hydraulic phenomena during the PCM phase change through the use of Computational Fluid Dynamics (CFD) tools. Numerical modeling and simulation of physical phenomena in LHTES systems require knowledge of the thermophysical properties of the PCMs used, and the accuracy of the results requires mastery of these data. While some PCM suppliers provide these properties through product datasheets, they are often incomplete and those available are provided for a sample product from industrial production; it is preferable to characterize the PCM oneself in order to access the complete thermophysical properties. Several researchers in the literature have characterized the thermophysical properties of various PCMs, and some have determined the dependence of certain properties on temperature. Soares *et al.* (2023) characterized ten low-temperature commercial PCMs and provided data on specific and latent heat, as well as the temperature dependence of thermal conductivity and diffusivity. No data were given according the density. Longo *et al.* (2022) characterized five PCMs for refrigeration application, focusing mainly on density, conductivity, and viscosity of the liquid phase. A densimeter was used for measuring the temperature dependence of the density. Kraiem *et al.* (2023) and Duquesne *et al.* (2021) conducted experimental studies that established the temperature dependence of certain thermophysical properties including liquid density, conductivity, as well as transition temperatures and latent

and specific heats. The authors used a pycnometer in the former case, and a method similar to the present study but with PCM volumes in the order of a few milliliters, leading to possible high uncertainty values compared to the present study. The literature analysis shows a significant lack of PCMs data required for accurate phase change modeling, especially concerning the thermal expansion coefficient, and PCMs for which data are available are often incomplete regarding the thermophysical properties.

The present study proposes a simple, validated, and cost-effective experimental procedure for accurately measuring the temperature dependence of the liquid phase density of low-temperature PCMs ($<100^{\circ}\text{C}$), in order to deduce the thermal expansion coefficient necessary for modeling the natural convection in the liquid phase PCM during the phase change process. Six new low-temperature PCMs not characterized in the literature were subsequently characterized, and the thermal expansion coefficients were determined. Prior to these experimental tests, a numerical study highlighted the influence of varying this parameter on the melting kinetics of a PCM as well as the melting performance of a LHTES unit.

2. ANALYSIS OF THE INFLUENCE OF THE THERMAL EXPANSION COEFFICIENT ON THE MELTING KINETICS OF A LHTES UNIT

The objective of this numerical study is to highlight the influence of the thermal expansion coefficient of a PCM on its phase change kinetics, the performance of an LHTES unit, and the necessity of precise knowledge of this parameter for the development of LHTES systems.

2.1. Geometry description and boundary conditions

2.1.1. Numerical geometry

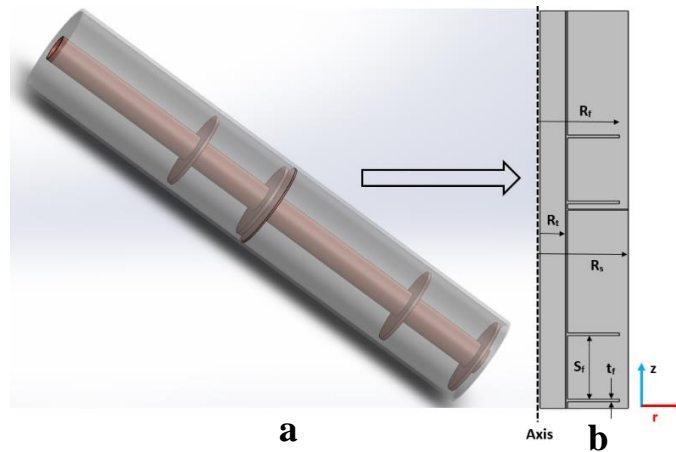


Figure 1. Numerical geometry: (a) 3D view, (b) vertical 2D axisymmetric model.

The geometry under study is a shell-and-tube heat exchanger consisting of two cylindrical tubes with inner radius $R_t = 15 \text{ mm}$ and $R_s = 51 \text{ mm}$. The heat transfer fluid flows through the inner tube which has a thickness $t = 1 \text{ mm}$, while the PCM is stored in the annular space between the two tubes, which is divided into two equal compartments. The PCM considered is the commercial bio-based product PT23. The aim of this study is to highlight the influence of the value of the thermal expansion coefficient on the natural convection of the liquid PCM and therefore on the kinetics of phase transition. It is therefore necessary for the geometry to allow for highly developed natural convection. To achieve this, four identical fins with radius $R_f = 46 \text{ mm}$ and $t_f = 2 \text{ mm}$ thickness were added to the shell to enhance heat transfer and natural convection. In each compartment, the fins are spaced by $S_f = 48.5 \text{ mm}$ and the lower fin is located at 5 mm from the bottom corner of the shell. This geometry was found to enhance the natural convection during the melting process in a previous study (Kaboré 2023-2). The heat transfer fluid flowing through the central tube is considered to have a very high flow rate, allowing for the axial thermal gradient to be neglected, and thus assuming a constant tube wall temperature. The LHTES unit is studied in a vertical orientation, allowing for simplification of the 3D geometry into a 2D axisymmetric geometry. The studied geometry is presented in Figure 1.

2.1.2. Physical formulation and numerical procedure

The phase change phenomenon was modeled using the enthalpy-porosity model. The conservation equations for mass, momentum, and energy were solved in the computational domain using the commercial CFD code STAR-CCM+. A second-order spatial and temporal discretization was adopted. In order to ensure a good balance between result accuracy and computational cost, a preliminary sensitivity study on mesh and time step was conducted, and a mesh and time step pair that achieved this balance was determined. The conservation equations, simplifying assumptions, and numerical procedure were detailed in a previous study by Kaboré *et al.* (2023-1).

Buoyancy forces are modeled by implementing a User Field Function using the Boussinesq approximation. These forces are given by eq. (1) and allows the modeling of the natural convection phenomena induced by temperature and therefore density gradients in the liquid PCM.

$$\vec{F}_b = \rho_{ref} \vec{g} \beta (T_{ref} - T) \quad (1)$$

F_b (N) is the buoyancy force, ρ_{ref} (kg/m^3) is a reference density of the PCM considered constant, \vec{g} (m/s^2) is the acceleration due to gravity, β ($1/K$) is the thermal expansion coefficient of the liquid PCM, T_{ref} ($^{\circ}C$) and T ($^{\circ}C$) respectively represent the liquidus temperature and the instantaneous temperature of the PCM. The aim of this study is to determine the influence of β on the melting performance of the PCM in an LHTES unit. Therefore, four values of β ranging from $2 \times 10^{-4} /K$ to $1.6 \times 10^{-3} /K$ were considered, based on the assumption that this property is the only property missing in the datasheet of the PCM supplier, which is in general at least the case for most PCM suppliers.

2.1.3. Initial and boundary conditions

In this study, the influence of the variation of the thermal expansion coefficient of the PCM on the natural convection was analyzed; therefore, the melting process involving more developed natural convection was considered. The PCM is initially assumed to be solid at a temperature $T_i = 22^{\circ}C$, corresponding to the solidus temperature of the PCM; $T(r, z, t = 0) = 22^{\circ}C$. Making an assumption of a very high flow rate of the heat transfer fluid, allowing the axial temperature gradient along the tube to be neglected, a constant temperature was imposed on the inner wall of the heat transfer tube: $T(r = 15 \text{ mm}, z, t) = 40^{\circ}C$.

2.2. Results and discussions

The Boussinesq approximation assumes a linear dependence of the density on temperature, thus approximating buoyancy forces by eq. (1), which also shows a linear dependence on the thermal expansion coefficient β . Figure 2a qualitatively presents the local distribution of natural convection velocity for the different values of β analyzed. The various contour maps show an increase in the velocity of the liquid PCM as the value of β increases. This is due to the proportionality of the buoyancy forces to temperature gradients through this coefficient; thus, as β increases, the liquid PCM is more capable of moving rapidly. Indeed, the β coefficient reflects the material's ability to expand with changes in temperature. The higher the thermal expansion coefficient, the more sensitive the material is to temperature variations. Therefore, as β increases, even small temperature variations within the PCM can induce significant changes in density, leading to more intense and vigorous convective movements. Therefore, configuration D represented by $\beta = 1.6 \times 10^{-3} /K$ exhibits higher natural convection velocities than configurations A ($\beta = 2 \times 10^{-4} /K$), B ($\beta = 4 \times 10^{-4} /K$), and C ($\beta = 8 \times 10^{-4} /K$).

The immediate consequence of the intensity or absence of natural convection is the enhancement or delay of the phase change kinetics. Figures 2b and 3a respectively present the distribution of the liquid fraction in the shell at time $t = 2400 \text{ s}$, and its evolution over time during the melting process. These figures show that the variation of the thermal expansion coefficient leads to a variation in the global performance of the LHTES unit, notably the melting kinetics and the full melting duration. Indeed, one of the driving physical phenomena leading the PCM melting process is natural convection. Thus, the enhancement or reduction of natural convection induced by the variation of the thermal expansion coefficient leads to a more or less significant variation in the PCM full melting time, and thus the charging performance of the LHTES unit. Figure 3b shows that this dependence of the full melting duration on the variation of β is not linear. Strong variations in the full

melting duration with a significant slope are observed for low values of β in the order of 10^{-4} while when the values of β become large, on the order of 10^{-3} the variations are less pronounced. The full melting durations are respectively 6650 s, 5100 s, 3930 s, and 3070 s for values of β of $2 \times 10^{-4}/K$, $4 \times 10^{-4}/K$, $8 \times 10^{-4}/K$ and $1.6 \times 10^{-3}/K$, representing a variation of up to 117 %.

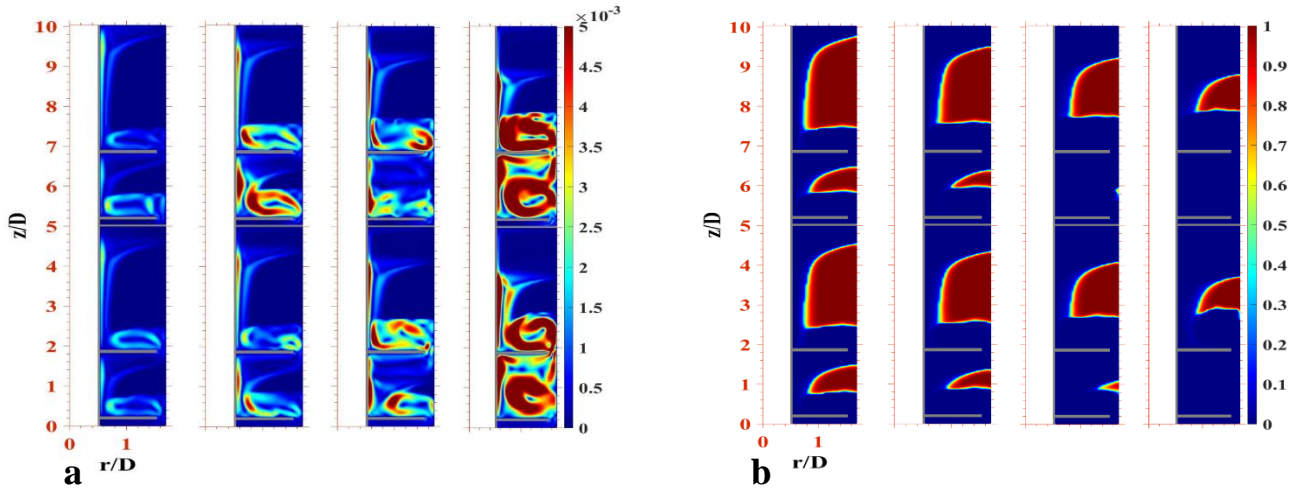


Figure 2. Natural convection velocity (a) and liquid fraction (b) distributions at $t = 2400$ s.

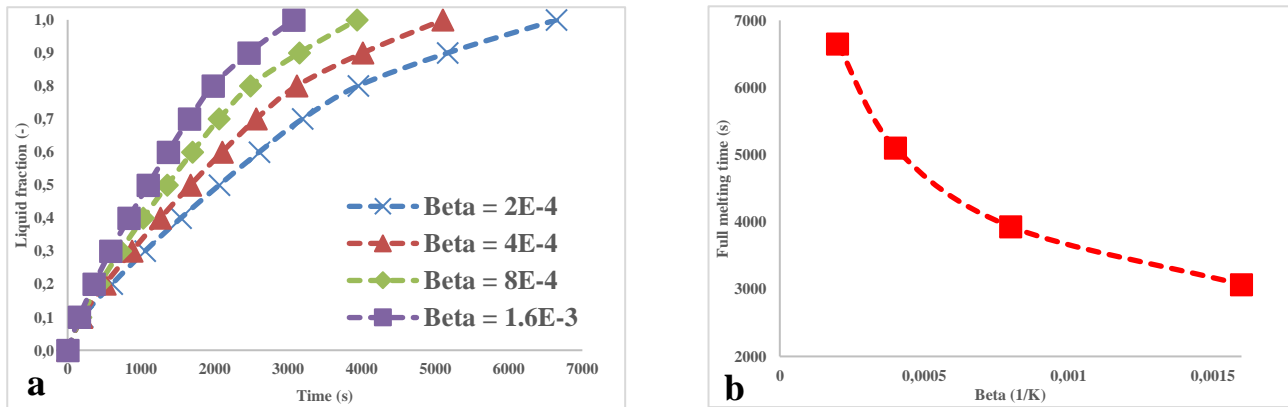


Figure 3. Influence of the thermal expansion coefficient on the full melting time.

In the literature, many PCMs have a thermal expansion coefficient lower than $10^{-3}/K$ (Kraiem *et al.*, 2023) placing them in the highly sensitive zone of performance for LHTES unit in relation to the value of β . This underscores the necessity to minimize errors associated with estimating the value of β for PCMs, to ensure a better precision in numerical modeling of LHTES systems intended for large-scale deployment. For this purpose, an experimental method is proposed hereafter to precisely determine the thermal expansion coefficient of PCMs.

3. DEVELOPMENT OF AN EXPERIMENTAL METHOD FOR THERMAL EXPANSION COEFFICIENT MEASUREMENT

3.1. Material and methods

3.1.1. Experimental procedure

The determination of the temperature dependence of the liquid phase density of the PCMs was performed using an in-house method developed in the laboratory. The equipment used, as shown in Figure 4, mainly

consist of a flat-bottomed glass flask, a silicone stopper, graduated pipettes, a balance and a water thermal bath of hundredth regulation precision.

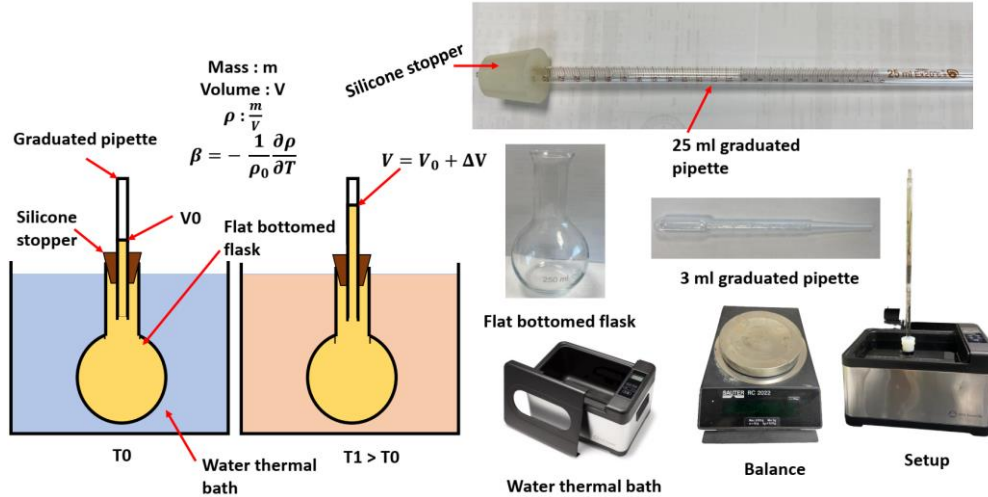


Figure 4. Experimental setup.

The masses of the main components, namely the flask, pipette, and stopper, were determined separately and after assembly to ensure accuracy, using a precise balance SAUTER RC 2022, with a precision of 0.1 g . In the flat-bottomed glass flask of 250 ml , a known volume of the liquid PCM, measured using a graduated beaker, was introduced up to the neck of the flask. The flask was sealed with a silicone stopper, perforated with a hole in the middle through which the 25 ml graduated pipette was inserted. A second 3 ml graduated pipette was used to top up the volume of PCM already in the flask until it overflows into the first 25 ml pipette. This allows for the most precise determination of the initial volume of PCM, denoted as $V_0\text{ (m}^3\text{)}$. Its associated mass, denoted as $m\text{ (kg)}$, was determined through the difference between the total mass including the PCM and the mass of the equipment already known. The assembly was then immersed in a circulating and temperature-regulated bath, sweeping the temperature range above the melting temperature up to $60\text{ }^\circ\text{C}$ in $5\text{ }^\circ\text{C}$ increments. At each stabilized temperature, the variation in the volume of the PCM $\Delta V\text{ (m}^3\text{)}$ was recorded by a reading of the PCM level in the 25 ml pipette. The density $\rho\text{ (kg/m}^3\text{)}$ was then determined for each temperature using the eq. (2).

$$\rho = \frac{m}{V_0 + \Delta V} \quad (2)$$

The thermal expansion coefficient, which is important for numerical modelling of natural convection, was determined through eq. (3).

$$\beta = -\frac{1}{\rho_0} \frac{\partial \rho}{\partial T} \quad (3)$$

$\beta\text{ (1/K)}$ is the thermal expansion coefficient, $\rho_0\text{ (kg/m}^3\text{)}$ is the liquid density at a reference temperature, $T\text{ (}^\circ\text{C)}$ is the temperature of the PCM. Seven PCMs including distilled water, paraffinic products from Rubitherm GmbH Ltd (Rubitherm GmbH) RT18HC, RT21HC, RT24HC, and bio-based PCMs from PureTemp LLC PT20, PT23, PT25 (Puretemp LLC) were characterized.

3.1.2. Uncertainties quantification

In order to verify the relevance of the results and the accuracy of the experimental procedure, an uncertainty analysis was conducted in accordance with Recommendation 1 (CI-1981) of the International Committee for Weights and Measures (CIPM) and Recommendation INC-1 (1980) of the Working Group on the Expression of Uncertainties (Iso 1995). Density measurement tests were carried out multiple times for the case of water to ensure result repeatability, followed by a single test for each other PCM. The uncertainties to be considered in these tests are therefore of type B. The uncertainty in density is a composed uncertainty integrating those of mass and volume. The total uncertainty $\delta\rho$ is thus given by eq. (4).

$$\delta\rho = \sqrt{\left(\frac{\partial\rho}{\partial m} \delta m\right)^2 + \left(\frac{\partial\rho}{\partial V} \delta V\right)^2} = \sqrt{\left(\frac{1}{V} \delta m\right)^2 + \left(\frac{m}{V^2} \delta V\right)^2} \quad (4)$$

The uncertainty resulting from the determination of the mass of PCM, δm is notably linked to the precision of the balance. The balance used has an error of $e = 0.1 \text{ g}$. Considering that the measured value of the mass necessarily falls within the interval $[(m - e); (m + e)]$ with a probability of 1, and assuming that m is equally likely to occur at any point within the interval, the uncertainty associated with the mass, according to the ISO norm (Iso 1995) is determined by eq. (5).

$$\delta m = \frac{e}{\sqrt{3}} \quad (5)$$

The uncertainty associated to the volume determination is decomposed into five main uncertainties:

- The uncertainty related to the graduation of the flat-bottomed flask provided by the supplier and assessed by eq. (5) at $\pm 0.17 \text{ ml}$.
- The uncertainty associated with the reading in the flat-bottomed flask, which is considered to be 1 ml .
- The uncertainty related to the graduation of the pipette estimated at $\pm 0.05 \text{ ml}$.
- The uncertainty related to the reading, estimated at $\pm 0.05 \text{ ml}$, which is half a graduation.
- The uncertainty related to the expansion of the flat-bottomed flask and the pipette, calculated with a maximum of $\pm 0.027 \text{ ml}$ considering an expansion coefficient of $3.3 \times 10^{-6} / \text{K}$ for glass.

The uncertainty associated to the mass of PCM is estimated at $\delta m = \pm 0.058 \text{ g}$, and the uncertainty associated with the volume of PCM is evaluated at $\pm 1.3 \text{ ml}$. Based on these analyses, the maximum uncertainty assessed on the density was $\pm 4 \text{ kg/m}^3$ for all the PCMs analyzed; the maximum value of the uncertainty was considered.

3.2. Results and discussions

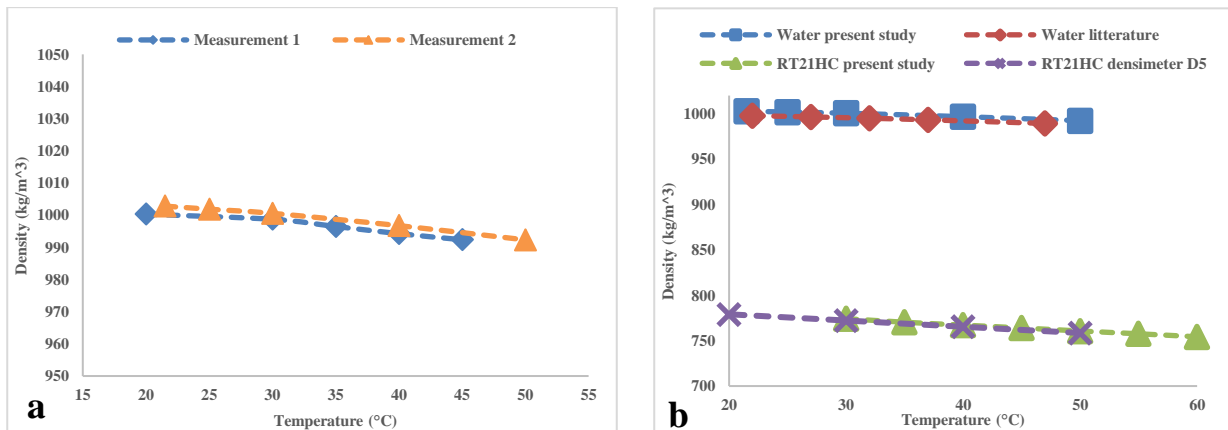


Figure 5. Validation of the experimental procedure: (a) repeatability, (b) comparison with reference data.

To validate the developed experimental method, validation test cases were conducted using distilled water as PCM, for which the temperature dependence of the liquid phase density is known, as well as the thermal expansion coefficient. The test on distilled water was performed twice, considering different temperatures. Figure 5a shows the evolution of the density of distilled water for the two separate tests. A near superposition of the curves is observed, indicating the repeatability of the tests and results. To quantify the relevance of the obtained results, a comparison was made between the measured data of distilled water and literature data (Lienhard, 1981). Furthermore, a characterization of the density of RT21HC was carried out using the D5 densimeter from METTLER TOLEDO. Figure 5b presents the measured data of the liquid density of distilled water and RT21HC by the present method, overlaid with literature data for water (Lienhard, 1981), and experimental data for RT21HC. Both for RT21HC and distilled water, the data from this present study almost perfectly overlap with the data obtained by the D5 densimeter, with a relative deviation at each temperature of less than 0.5% . The maximum uncertainty associated to the density measurement was estimated at $\pm 4 \text{ kg/m}^3$ both for water and RT21HC. These comparisons validate the experimental procedure, which demonstrates very high precision in measuring the liquid density of PCMs, as well as their temperature dependence.

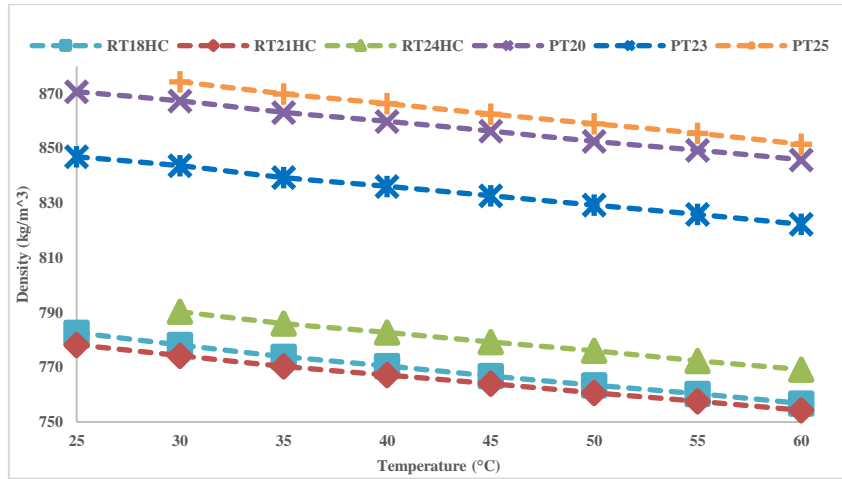


Figure 6. Temperature-dependence of the liquid density of the six PCMs studied.

Once the experimental procedure was validated using literature and experimental data, it was then applied to characterize the density of the 5 other PCMs, namely RT18HC, RT24HC, PT20, PT23, PT25. Figure 6 depicts the evolution of the liquid phase density of the PCMs as a function of temperature. A nearly linear behavior is observed for all PCMs. It is noteworthy that PCMs from Puretemp exhibit higher densities ranging from 820 kg/m^3 to 880 kg/m^3 compared to those from Rubitherm, which range from 750 kg/m^3 to 800 kg/m^3 within the same temperature ranges. Referring to eq. (1), buoyancy forces in these materials are more significant during the phase change process. The evolution of liquid density with temperature can be approximated by a linear correlation given by eq. (6).

$$\rho(T) = aT + b \quad (6)$$

The coefficients $a \text{ (kg/m}^3/\text{°C)}$ and $b \text{ (kg/m}^3)$ of the function as well as the regression coefficient R^2 are presented in Table 1. The thermal expansion coefficient β is evaluated by eq. (3), taking ρ_0 as the density at temperature $T = 30 \text{ °C}$ for all PCMs, with $\frac{\partial \rho}{\partial T} = a$ which represents the slope of the curve obtained by linear regression. The calculated values of β shown in Table 1, with RT18HC exhibiting the highest thermal expansion coefficient, and PT20 the lowest. To compare the natural convection performances of different PCMs, the product $\rho_m \times \beta$ was evaluated and presented in Table 1, with $\rho_m \text{ (kg/m}^3)$ being the mean density. This intrinsic term of the material indicates its convective performance when subjected to a temperature gradient. Thus, under the same operating conditions, the PT25 PCM exhibits the most significant natural convection potential.

Table 1. Thermal expansion coefficient of some PCMs and estimated values of a and b in eq. (6).

Materials	$\beta \text{ (1/K)}$	$\rho_m \times \beta \text{ (kg/m}^3/\text{K)}$	A $\text{(kg/m}^3/\text{°C)}$	B $\text{(kg/m}^3)$	R^2	$\sigma_{min} \text{(kg/m}^3)$	$\sigma_{max} \text{(kg/m}^3)$
RT18HC	0.00093	0.7182	-0.7267	799.95	0.9968	3.07	3.29
RT21HC	0.00087	0.6632	-0.6705	794.27	0.9985	3.05	3.25
RT24HC	0.00088	0.6849	-0.6945	810.65	0.9986	3.14	3.32
PT20	0.00081	0.6933	-0.7007	887.82	0.9991	3.45	3.66
PT23	0.00083	0.6954	-0.703	864.37	0.9993	3.35	3.56
PT25	0.00085	0.7388	-0.7472	896.36	0.9993	3.45	3.66

4. CONCLUSIONS

The objective of this study was to propose an experimental method for characterizing the liquid phase density and thermal expansion coefficient of PCMs. A numerical analysis first highlighted the influence of the thermal expansion coefficient on the performance of PCMs during the melting process and the full charging time of the LHTES unit. Subsequently, a simple, cost-effective experimental procedure, validated with high

precision, was proposed for determining this coefficient. The main findings of this study can be summarized as follows:

- The natural convection of the PCM and the performance of the LHTES unit are proportional to the thermal expansion coefficient. These performances are even more sensitive to the variation of β when its value is less than 10^{-3} , corresponding to a large portion of PCMs. The full melting time varies by nearly 30 % when a value of $\beta = 0.0004 /K$ is considered instead of $\beta = 0.0008 /K$.
- The proposed experimental procedure allows to determine the temperature dependence of the liquid phase density of PCMs with a relative maximum uncertainty of 0.5 %, providing a precision compared to literature data for distilled water and data obtained by the D5 densimeter.
- The product of the average density by the thermal expansion coefficient $\rho_m \times \beta$ representing the average density-temperature gradient into the PCM allows for the assessment of natural convection potential and comparison between PCMs. Based on this, among the characterized PCMs, PT25 exhibits the highest natural convection potential and RT21HC the lowest potential, with respective values of $0.7388 \text{ kg/m}^3\text{K}$ and $0.6632 \text{ kg/m}^3\text{K}$.

Following this study, the other thermophysical properties of the various materials studied will be determined through DSC, thermal conductivity, and viscosity measurements. Mastery of the thermophysical properties of materials allows for a judicious selection of the material according to the intended application for the development of an LHTES system.

5. ACKNOWLEDGMENTS

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