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A Hybrid Numerical-Experimental Method for Determining Thermal Conductivities

Determining the thermal conductivity of a material from temperature measurements in a cooling or heating process belongs to the class of inverse problems. In this article, we present a method for a simple experimental setup, consisting of a glass tube containing the material under investigation, two thermistors for temperature monitoring (one at the central axis and the other attached on the outer surface of the tube), and a water heat bath maintained at a desired temperature. We solve the direct problem, i.e., the transient heat conduction equation, treating the thermal conductivity as a parameter whose value is determined by minimizing the difference between the calculated and the experimentally measured temperatures. The method is based on the numerical solution of the one-dimensional transient heat conduction equation in cylindrical coordinates that accurately describes the temperature evolution of a material in a narrow, long glass tube. The technique has been validated by applying it to the lauric and capric acids, whose thermal conductivities are accurately known and therefore it could be a valuable tool for the determination of the thermal properties of phase change materials suitable for thermal storage applications. [DOI: 10.1115/1.4048468]

1 Introduction

Thermal properties of phase changing materials (PCM) are usually determined by means of the so-called T-History method [1], according to which sample and reference material (typically water) are preheated above the PCM melting temperature, and subsequently, during cooling, the temperature evolution is monitored. The thermal properties, i.e., melting point, specific and latent heats are evaluated by simple application of energy balance equations. However, the method, although very simple and easy to implement, suffers from several drawbacks: the accurate determination of the critical times of the T-History curve upon cooling, i.e., the time entering in the solidification and the time at which the solid starts to cool down is difficult to assess, especially in mixed PCMs. Hong et al. [2] tried to overcome this issue using time derivatives of the temperature evolution curve, which proved to work satisfactorily in the case of pure substances with clearly defined phase change zones, but it is troublesome in mixtures, which are the most common case of industrial PCMs in which the limits of solid and liquid phases do not appear clearly in the T-History curve. An alternative approach based on the numerical determination of the enthalpy versus temperature by successive integrations of the T-History curve was proposed [3]. Although this method tackles rather well the problem of critical time determination, it has problems in the phase change zone at which the temperature remains almost constant and therefore the numerical evaluation of the enthalpy becomes challenging. To overcome this issue, Sandnes et al. used a quadratic polynomial adjusted to the T-History curve to evaluate the heat loss upon cooling, thus providing a more stable procedure [4]. However, in all these approaches the thermal conductivity, which is of major importance, e.g., in applications of thermal storage cannot be determined.

Aiming in providing an answer to these issues, we devised an easy and flexible experimental procedure in conjunction with a robust and accurate computational tool that is based on the numerical solution of the well-known transient heat conduction equation. In what follows, we describe the experimental setup and the computational approach, to continue with representative results and end up with some concluding remarks.

2 Experimental Setup and Computational Details

Contrary to the T-History method, we use only one cylindrical tube having length of 25 cm and external diameter and thickness of 1.2 cm and 3 mm, respectively. The tube was of Borosilicate glass DURAN having thermal conductivity of 1.2 W/(mK) at 90 °C and a density of 2.23 g/cm^3, while the specific heat (not provided from the producer) was evaluated as a weighted average of the specific heat of its constituents, i.e., 81% SiO_2, 13% B_2O_3, 4% Na_2O+K_2O, and 2% Al_2O_3. For the validation of the method, we used two different prototypes PCMs to fill the tube (typical using about 10 g), namely the lauric and capric acid purchased from Quartzy. Two negative temperature coefficient 5-mm thermistors of 14 KOhms purchased from Mouser Corporation UK were used for the temperature measurements (accuracy ±0.01 °C), one at the center of the tube containing a PCM and the second one attached

Contributed by the Heat Transfer Division of ASME for publication in the JOURNAL OF HEAT TRANSFER. Manuscript received July 31, 2019; final manuscript received August 31, 2020; published online October 30, 2020. Assoc. Editor: Thomas Beechem.

at the tube's external surface. The central thermistor was supported by a Teflon thin stick (diameter 0.6 mm) and attached at the top at upper end of the tube by means of solid support, thus ensuring correct positioning within 0.2 mm, Fig. 1(*a*). We evaluated that such a misalignment results in meaningless differences in the evaluated thermal quantities. The temperature acquisition was obtained by means of the measurement computing and counter USB data acquisition card. The tube with the PCM was initially equilibrated in a water bath at a certain temperature, below the solidification or above the melting temperature, depending on whether we wished to evaluate the thermal conductivity of the solid or the liquid phase, to subsequently immerse the tube into a cold water bath with a constant temperature at 10 °C. Taking into account the geometry of the tube, i.e., that its length is approximately 20 times longer than its diameter, we may assume that we are dealing with 1D transient heat conduction problem in cylindrical coordinates. We will proceed by first solving the heat conduction equation for a thin and long cylindrically shaped material with its peripheral surface kept at a constant temperature. The purpose of this is to illustrate the numerical technique and leave out the complexities due to the cylindrical glass tube and the associated interface conditions. Subsequently, the actual problem will be tackled, i.e., that of a material filling a cylindrical glass tube of finite width, with its outer surface maintained at a temperature that is known as a function of time. We note here that the proposed approach is substantially different from those used previously: we solve numerically the differential equation of heat transfer and at the same time we perform minimization for the determination of the thermal conductivity. The T-history method and its variations employ energy balance, they do not compare to a solution of the heat transfer equation, and therefore, the conductivity is not determined by fitting the experimental temperature values.

2.1 Governing Equation. The heat conduction is described by the following partial differential equation:

$$\frac{\partial T(r,t)}{\partial t} = \alpha \mathcal{L} T(r,t), \quad \alpha = \frac{k}{\rho c_p}, \quad \mathcal{L} = \nabla^2, \quad r \in [0,b] \tag{1}$$

With the conditions

$$T(r,0) = \Theta_0 \text{ (initial condition)} \tag{2}$$

$$\left.\frac{\partial T(r,t)}{\partial r}\right|_{r=0} = 0, \; t > 0 \quad \text{(boundary condition 1)} \tag{3}$$

$$T(b,t) = \Theta_1, \; t > 0 \quad \text{(boundary condition 2)} \tag{4}$$

In cylindrical coordinates with no z-dependence and azimuthal symmetry

$$\mathcal{L} \equiv \frac{\partial^2}{\partial r^2} + \frac{1}{r}\frac{\partial}{\partial r} \tag{5}$$

The Crank–Nicolson scheme reads

$$T(r,t+dt) = T(r,t) + \frac{adt}{2}\mathcal{L}\left[T(r,t) + T(r,t+dt)\right] \tag{6}$$

Or if rewritten

$$\left[1 - \frac{adt}{2}\mathcal{L}\right] T(r,t+dt) = \left[1 + \frac{adt}{2}\mathcal{L}\right] T(r,t) \tag{7}$$

This equation provides the scheme that permits the time propagation while the spatial evolution is inherently included.

The space coordinate r ranging in [0,b] may be discretized as

$$r_i = ih, \; \forall\, i = 0, 1, \; \ldots, \; n+1, \text{ with } h = \frac{b}{n+1} \tag{8}$$

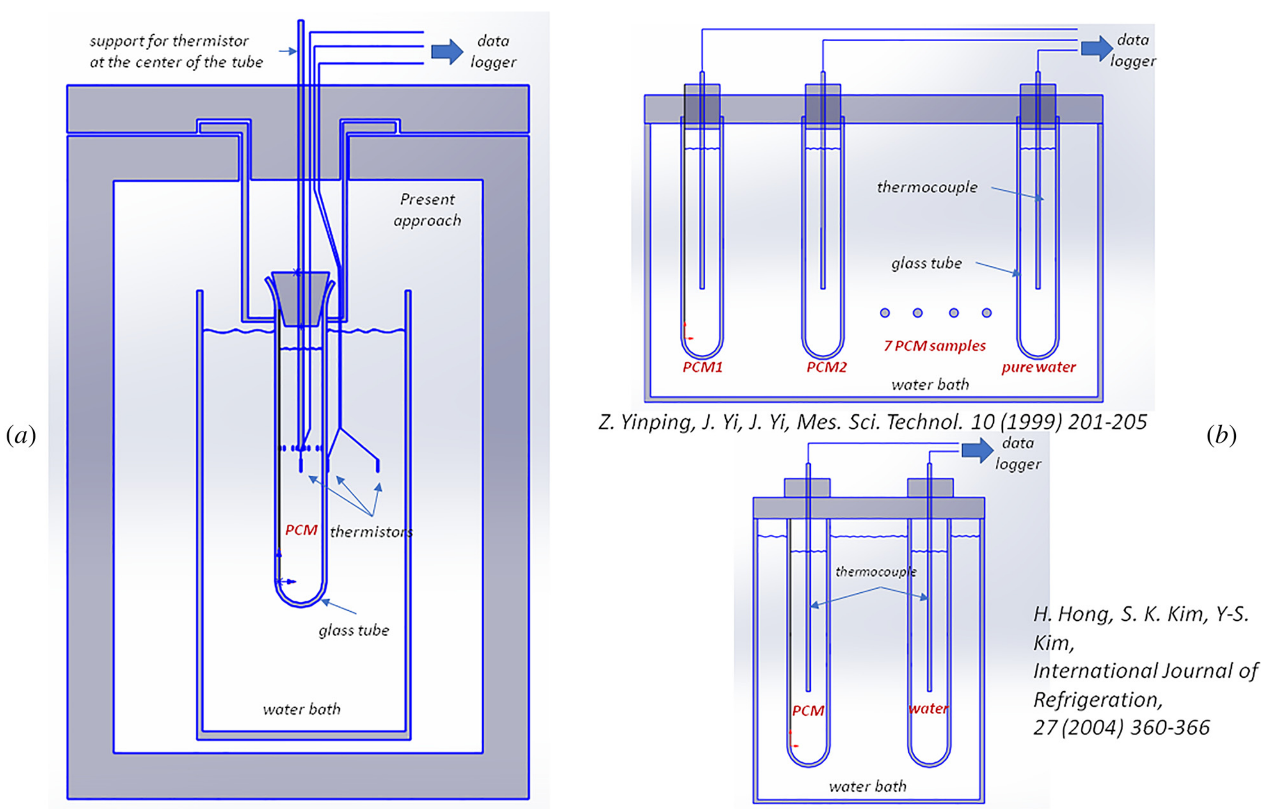

Fig. 1 (*a*) Experimental setup of the present approach and (*b*) experimental setup of T-history methods

We make the following conventions for convenience:

$$T_i \equiv T(r_i,t), \quad T_i' \equiv \frac{\partial T(r_i,t)}{\partial r}, \quad T_i'' \equiv \frac{\partial^2 T(r_i,t)}{\partial r^2} \text{ and likewise} \tag{9}$$

$$T_i^+ \equiv T(r_i,t+dt), \quad T_i^{+\prime} \equiv \frac{\partial T(r_i,t+dt)}{\partial r}, \quad T_i^{+\prime\prime} \equiv \frac{\partial^2 T(r_i,t+dt)}{\partial r^2} \tag{10}$$

At $r = r_{n+1} = b$ we know that $T_{n+1} = \Theta_1$, $\forall t > 0$. Hence T will be represented by a vector $T \in R^{n+1}$ with components T_i, $i = 0, 1, \ldots, n$ (and similarly for T^+).

The action of the $\mathcal{L}$ operator on may be represented by

$$\mathcal{L}T = AT + d, \; A \in R^{(n+1)\times(n+1)}, \; d \in R^{n+1} \tag{11}$$

The first and second derivatives at r_i with $i \neq 0$ and $i \neq n$ may be approximated by

$$T_i' \equiv \frac{T_{i+1} - T_{i-1}}{2h} \tag{12}$$

$$T_i'' \equiv \frac{T_{i+1} - 2T_i + T_{i-1}}{h^2} \tag{13}$$

and

$$\mathcal{L}\mathcal{T}_i = \frac{1}{h^2}\left(\left(1 - \frac{1}{2i}\right)T_{i-1} - 2\mathcal{T}_i + \left(1 + \frac{1}{2i}\right)\mathcal{T}_{i+1}\right) \tag{14}$$

$$\forall i = 1, \cdots, n-1$$

2.2 Treating the Boundary Conditions. At $r = 0$, there is an apparent singularity in calculating $\frac{1}{r}\partial T(r,t)/\partial r$. Hence we will make use of the boundary conditions at $r = 0$, namely, Eq. (3)

$$\frac{\partial^2 T(0,t)}{\partial r^2} \equiv \lim_{r\to 0} \frac{\dfrac{\partial T(r,t)}{\partial r} - \dfrac{\partial T(0,t)}{\partial r}}{r - 0} = \lim_{r\to 0} \frac{1}{r}\frac{\partial T(r,t)}{\partial r} \tag{15}$$

Hence $\frac{1}{r_0}T_0' = T_0''$ and so $\mathcal{L}T_0 = 2T_0''$. From Taylor expansion, we obtain

$$T_1 = T_0 + hT_0' + 0.5h^2T_0'' + O(h^3) \tag{16}$$

$$T_2 = T_0 + 2hT_0' + 2h^2T_0'' + O(8h^3) \tag{17}$$

Using the boundary condition at $r = 0, T_0' = 0$ and eliminating the third-order terms from Eqs. (16) and (17), we obtain to second order the following:

$$T_0'' = \frac{1}{2h^2}(8T_1 - T_2 - 7T_0) \tag{18}$$

Again from the same equations by eliminating second-order terms, we obtain

$$T_0' = \frac{4T_1 - T_2 - 3T_0}{2h} \tag{19}$$

Since $T_0' = 0, \; 4T_1 - T_2 - 3T_0 = 0$ and hence

$$T_0'' = \frac{1}{2h^2}(T_1 - T_0) \text{ and} \tag{20}$$

$$\mathcal{L}T_0 = \frac{1}{h^2}(-4T_0 + 4T_1) \tag{21}$$

For the $\mathcal{L}T_n$ we proceed in the following manner: From Eq. (14) for $i = n$ and replacing $T_{n+1} = \Theta_1$ using Eq. (4), we obtain

$$\mathcal{L}T_n = \frac{1}{h^2}\left(\left(1 - \frac{1}{2n}\right)T_{n+1} - 2T_n\right) + \frac{1}{h^2}\left(1 + \frac{1}{2n}\right)\Theta_1 \tag{22}$$

From Eqs. (21), (14), (22), and (11), we may deduce the expression for the matrix $A_{i,j}$ and the vector d_i by direct comparison, i.e.,

$$A_{0,0} = -4/h^2, \quad A_{0,1} = 4/h^2, \quad A_{0,j} = 0, \quad \forall j \geq 2 \tag{23}$$

$$A_{ij} = \frac{1}{h^2}\left(\left(1 - \frac{1}{2i}\right)\delta_{i-1,j} - 2\delta_{i,j} + \left(1 + \frac{1}{2i}\right)\delta_{i+1,j}\right) \quad \forall i \neq 0, j \in \{0, 1, \ldots, n\} \tag{24}$$

$$d_i = \frac{1}{h^2}\left(1 + \frac{1}{2n}\right)\Theta_1\delta_{i,n}, t > 0 \text{ and } d_i = \frac{1}{h^2}\left(1 + \frac{1}{2n}\right)\Theta_0\delta_{i,n}, t = 0 \tag{25}$$

2.3 Implementation The matrix

$$\text{The matrix } B \equiv I - \frac{adt}{2}A \text{ with elements} \tag{26}$$

$$B_{i,j} = \delta_{i,j} - \frac{adt}{2}A_{i,j}, \quad \forall i,j \in \{0, 1, \ldots, n\} \tag{27}$$

is a tridiagonal matrix that will be repeatedly used in solving linear systems of the kind: By $= z$. Therefore, it is important, in order to efficiently solve the linear systems, to maintain it in lower-upper decomposition (LU) factors. Explicitly the B-matrix elements may be written as

$$\forall j = 0, 1, \ldots, n$$

$$B_{0,j} = (1 + 4\beta)\delta_{0,j} - 4\beta\delta_{1,j}, \text{ and } \beta \equiv \frac{adt}{2h^2} \tag{28a}$$

$$B_{i,j} = (1 + 2\beta)\delta_{i,j} - \beta\left(1 - \frac{1}{2i}\right)\delta_{i-1,j} - \beta\left(1 + \frac{1}{2i}\right)\delta_{i+1,j}, \quad \forall i = 1, \ldots, n \tag{28b}$$

The main diagonal $B_{i,i}$ of B and likewise its lower $B_{j,j-1}$ and upper $B_{k,k+1}$ off diagonals may be written as

$$a_i = B_{i,i}, \quad \forall i = 0, \ldots, n \tag{29a}$$

$$b_i = B_{j,j-1}, \quad \forall j = 1, \ldots, n \tag{29b}$$

$$c_k = B_{k,k+1}, \quad \forall k = 0, \ldots, n-1 \tag{29c}$$

$$B = \begin{bmatrix} a_0 & c_0 & 0 & \cdots & \cdots & 0 \\ b_1 & a_1 & c_1 & 0 & \cdots & 0 \\ 0 & \ddots & \ddots & \ddots & \ddots & \vdots \\ \vdots & \ddots & \ddots & \ddots & \ddots & \vdots \\ \vdots & 0 & \ddots & b_{n-1} & a_{n-1} & c_{n-1} \\ 0 & \cdots & \cdots & 0 & b_n & a_n \end{bmatrix} \tag{30}$$

Hence

$$a_0 = 1 + 4\beta, \quad a_i = 1 + 2\beta, \quad \forall i = 1, \ldots, n \tag{31a}$$

$$c_0 = -4\beta,\ c_k = -\beta\left(1+\frac{1}{2k}\right), \quad \forall k = 0,\ \ldots,\ n-1 \tag{31b}$$

$$b_j = -\beta\left(1-\frac{1}{2j}\right), \quad \forall j = 1,\ \ldots,\ n \tag{31c}$$

The LU decomposition of B is easily performed by a matrix L

$$L_{i,j} = \delta_{i,j} + l_i\delta_{i,j+1} \tag{32}$$

$$\text{with } l_{i,} \forall i = 1,\ \ldots,\ n \tag{33}$$

to be determined, and a matrix U

$$U_{i,j} = u_i\delta_{i,j} + c_i\delta_{i,j-1} \tag{34}$$

i.e., with identical upper off diagonal as that of B, and diagonal elements $u_i = U_{i,i}$ to be determined

$$u_0 = a_0 \tag{35a}$$

$\forall i = 1,\ \ldots,\ n$ (Next two lines indented)

$$l_i = b_i/u_{i-1} \tag{35b}$$

$$u_i = a_i - l_i c_{i-1} \tag{35c}$$

Combining Eqs. (7), (11), and (27), the time development scheme becomes

$$BT^+ = \left(I + \frac{adt}{2}A\right)T + \frac{adt}{2}(d^+ + d) \tag{36}$$

Note that $(^+)$ superscripts denote quantities calculated at time $t + dt$

$(I + \frac{adt}{2}A)$ is tridiagonal and it can be obtained from B by the following substitutions:

$$a_i \to a'_i = 2 - a_i \tag{37a}$$

$$b_i \to b'_i = -b_i \tag{37b}$$

$$c_i \to c'_i = -c_i \tag{37c}$$

2.4 Linear Solver. Since we have decomposed

$$B = LU \tag{38}$$

the system

$$BT^+ = g \tag{39}$$

$$\text{with } g = \left(I + \frac{adt}{2}A\right)T + \frac{adt}{2}(d^+ + d) \tag{40}$$

may be implemented by introducing a vector z as

$$z = UT^+ \tag{41}$$

$$\text{Then } Lz = g \text{ is solved by} \tag{42}$$

$$z_0 = g_0 \tag{43a}$$

$$z_i = g_i - l_i z_{i-1}, \quad \forall i = 1, \ldots, n \tag{43b}$$

Subsequently, T^+ is obtained by solving $UT^+ = z$ as

$$T_n^+ = z_n/u_n \tag{44a}$$

$$T_i^+ = (z_i - c_i T_{i+1}^+)/u_i, \quad \forall i = n-1,\ \ldots,\ 1,\ 0 \tag{44b}$$

To calculate g, a matrix–vector multiplication is needed, which may be simplified considering Eq. (37), namely

$$\left[\left(I + \frac{adt}{2}A\right)T\right]_i = \begin{cases} (2-a_0)T_0 - c_0T_1, & i = 0 \\ (2-a_n)T_n - b_nT_{n-1}, & i = n \\ (2-a_i)T_i - c_iT_{i+1} - b_iT_{i-1}, & i = 1,\ \ldots,\ n-1 \end{cases} \tag{45}$$

2.6 Interface of Two Materials. Once we have the framework given above, we now turn to a cylindrical glass tube with internal and external radii R_1 and R_2. The tube is filled with a material and the whole system (glass–material) is initially brought at temperature Θ_1. The tube's external surface in maintained at a time-dependent temperature

$$\Theta_2 = \Theta_2(t) \tag{46}$$

At the glass–material interface, we assume that there is no boundary resistance and therefore the temperature is continuous, i.e., approaches the same value from both sides. Also since heat cannot be stored on the interface, heat flux is balanced out. The heat flux is given by $-k\partial T(r,t)/\partial r$ and therefore at the interface $k_1\partial T(R_1-\epsilon,t)/\partial r = k_2\partial T(R_1+\epsilon,t)/\partial r$, i.e., there is a discontinuity in the temperature gradient across the interface

The governing equation is again Eq. (1) with the remark that a, k, ρ, c_p are different for $r \in [0, R_1)$ and $r \in (R_1, R_2]$

$$a = \begin{cases} a^{(1)}\ \forall r < R_1 \\ a^{(2)}\ \forall r > R_1 \end{cases}, \quad k = \begin{cases} k^{(1)}\ \forall r < R_1 \\ k^{(2)}\ \forall r > R_1 \end{cases},$$
$$\rho = \begin{cases} \rho^{(1)}\ \forall r < R_1 \\ \rho^{(2)}\ \forall r > R_1 \end{cases}, \quad c_p = \begin{cases} c_p^{(1)}\ \forall r < R_1 \\ c_p^{(2)}\ \forall r > R_1 \end{cases} \tag{47}$$

The grid in r uses two steps, one for $0 \le r \le R_1$ and another for $R_1 \le r \le R_2$

$$r_i = ih, \text{ with } h = \frac{R_1}{n}, \quad \forall i = 0, 1,\ \ldots,\ n \tag{48a}$$

$$r_i = R_1 + (i-n)\delta, \text{ with } \delta = \frac{R_2 - R_1}{m+1-n}, \quad \forall i = n,\ \ldots,\ m+1 \tag{48b}$$

Again the tridiagonal matrix is comprised of

$$a_0 = 1 + 4\frac{a^{(1)}dt}{2h^2},\ a_i = 1 + 2\frac{a^{(1)}dt}{2h^2},\ \forall i = 1,\ \ldots,\ n-1 \tag{49a}$$

$$a_i = 1 + 2\frac{a^{(2)}dt}{2\delta^2}, \quad \forall i = n+1,\ \ldots,\ m,$$
$$a_n = 1 - \frac{dt}{s_2 - s_1}\left(\frac{a^{(2)}s_1}{\delta^2} - \frac{a^{(1)}s_2}{h^2}\right) \tag{49b}$$

with

$$s_1 = \frac{a^{(1)}}{c_p^{(1)}}\left(\frac{1}{R_1} + \frac{2}{h}\right), \quad s_2 = \frac{a^{(2)}}{c_p^{(2)}}\left(\frac{1}{R_1} - \frac{2}{\delta}\right) \tag{50}$$

$$b_i = -\frac{a^{(1)}dt}{2h^2}\left(1 - \frac{1}{2i}\right), \quad \forall i = 1,\ \ldots,\ n-1, b_n = -\frac{a^{(1)}s_2dt}{(s_2 - s_1)h^2} \tag{51a}$$

$$b_i = -\frac{a^{(2)}dt}{2\delta^2}\left(1 - \frac{1}{2\left(i - n + \frac{R_1}{\delta}\right)}\right), \quad \forall i = n+1, \ldots, m \tag{51b}$$

$$c_0 = -4\frac{a^{(1)}dt}{2h^2}, \; c_i = -\frac{a^{(1)}dt}{2h^2}\left(1 + \frac{1}{2i}\right), \; \forall i = 1, \ldots, n-1 \tag{52a}$$

$$c_n = \frac{a^{(2)}s_1 dt}{(s_2 - s_1)\delta^2}, \quad c_i = -\frac{a^{(2)}dt}{2\delta^2}\left(1 + \frac{1}{2\left(i - n + \frac{R_1}{\delta}\right)}\right),$$
$$\forall i = n+1, \ldots, m-1 \tag{52b}$$

And for the d-vector

$$d_i = \delta_{m,i}\frac{\Theta_2(t)}{\delta^2}\left(1 + \frac{1}{2\left(m - n + \frac{R_1}{\delta}\right)}\right) \tag{53}$$

The above coefficients satisfy at the interface $r = R_1$ the following conditions:

$$T(R_1 - \varepsilon, t) = T(R_1 + \varepsilon, t) \text{ and } k^{(1)}\frac{\partial T(R_1 - \varepsilon, t)}{\partial r} = k^{(2)}\frac{\partial T(R_1 + \varepsilon, t)}{\partial r}, \; \varepsilon \to 0 \tag{54}$$

We have again to solve repeatedly Eq. (36) with B reconstructed using the above values for a_i, b_i, c_i .

3 Results and Discussion

The numerical procedure described above was implemented in a homemade code written in Fortran and first tested against an analytical solution that is available for the case of transient heat conduction in 1D cylindrical tube shaped materials, i.e., no interface [5]. Figure 1 depicts the analytical and the numerical results referring to the time evolution of the temperature in this case. It turns out that the model reproduces very well the analytical solution.

We then passed in the more realistic cases of lauric and capric acids. In Figs. 2(*a*) and (*b*), we provide the experimental data along with the numerical predictions for these two cases.

As it can be seen, the model reproduces very well the experimental data, yielding thermal conductivities of 0.23±0.02 W/mK and 0.19±0.03 W/mK for the lauric and capric acids, in line with available reference values, e.g., 0.215±0.01 and 0.15 W/mK, [6] and [7], respectively. We note that the error bars represent the standard deviations evaluated over 30 independent measurements and calculations, and the provided values are obtained from the mean values. In addition, we verified that the specific heat capacity we used for the glass tube (by varying its value by as much ±50%) does not affect the obtained thermal conductivity values, a result that is somehow expected taking into account its small thickness and the fact that its conductivity value is 5–6 times larger than those of the PCMs.

4 Concluding Remarks

We presented a method based on the numerical solution of the one-dimensional transient heat conduction equation in cylindrical coordinates that describes accurately the temperature evolution of a material in a narrow and long glass tube. The experimental setup permitting the application of the method is very simple and easy to implement.

The thermal conductivity is determined by fitting the numerically simulated temporal profile to the one measured experimentally. The procedure was applied successfully in the representative

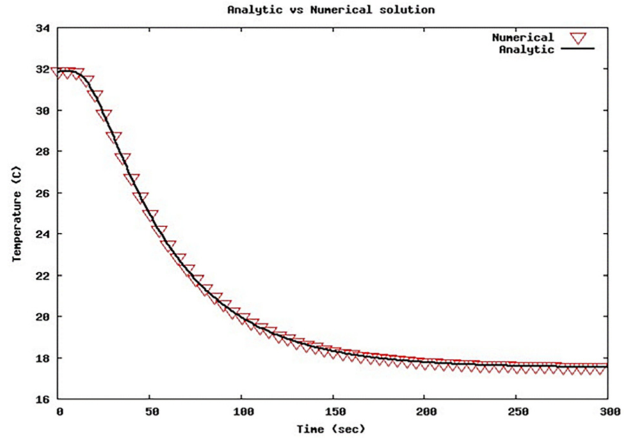

Fig. 2 Comparison between analytical and numerical solutions for the particular case of a material with long cylindrical shape

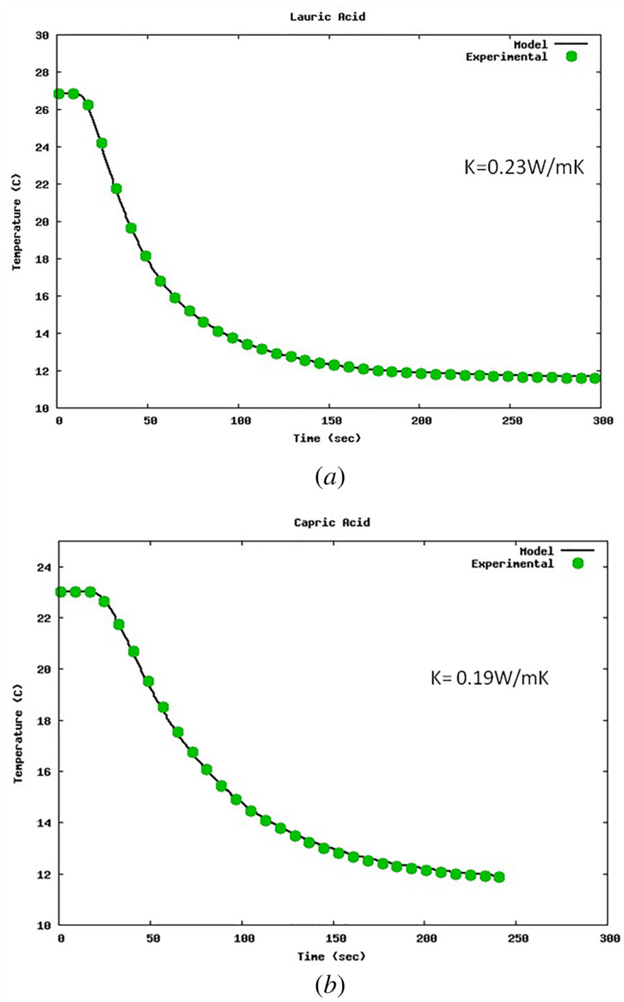

Fig. 3 (*a*) Numerical evaluation of the time evolution of the temperature according to the numerical solution of the transient heat conduction method adjusted to the experimental data in the case of lauric acid and (*b*) the same as Fig. 2(*a*) but for capric acid

cases of lauric and capric acids, suggesting that it could be of valuable use in choosing phase change materials to be used in thermal storage applications.

Acknowledgment

This work was financially supported by the TESSe2b project that has received funding from the European Union's Horizon 2020 research and innovation program under Grant Agreement No 680555. This article reflects only the authors' view and the European Commission is not responsible for any use that may be made of the information it contains.

Funding Data

- European Union's Horizon 2020 (Funder ID: 10.13039/100010661).

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