

THERMAL CHARACTERISATION OF PRODUCTS UNDERGOING PHASE CHANGE : SOLIDIFICATION AND FUSION

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Abstract. The aim of this work is the thermal characterization of phase change material (PCM). The thermophysical properties are identified simultaneously by using Levenberg-Marquardt's method. Simulated transient measurements of the temperature at the extremities of the PCM are used to solve the parameter estimation problem. The preliminary results present a relative error lower than 1% with respect to the exact values of the required parameters.

Keywords. Inverse problem, parameter estimation, thermophysical properties, phase change

1. Introduction

The advance of computer technology has contributed to the development of powerful numerical programs to simulate the processes of solidification and fusion for a wide range of *phase change materials* (PCMs). However, the optimum performance of these programs depends on the accurate knowledge of the products' thermal properties.

In this paper, we present one of the steps concerning the development of an experimental set-up that allows the simultaneous thermal characterization of an industrial product undergoing phase change, i.e., solidification and fusion. The thermophysical properties considered are thermal conductivities and heat capacities for liquid and solid states, the phase change temperature and the latent heat. These properties are determined by using the Levenberg-Marquardt's method (Beck and Arnold, 1977, Orlande and Ozisik, 2000). This technique, which is an improvement of the Gauss's method, is widely employed in parameter estimation problems.

Here, the experimental set-up is numerically simulated, in order to obtain the transient measurements of temperature and heat flux at the limits of the sample. These values are introduced as boundary conditions in the *direct model*. By applying this model the phase change phenomenon is simulated. The Levenberg-Marquardt's method use the *direct model* to identify the thermophysical properties of the PCM.

2. Experimental set-up modeling

The experimental set-up has been designed and realized. It's principle is schematized in Fig. (1). It has a cylindrical geometry that ensure heat transfer only in the axial direction y . Initially, the PCM is in the liquid phase, and is placed between two metallic blocks (1 and 2), forming a cavity of thickness L , and it is laterally locked up by a thermal insulated cell. The temperatures at the limits of the two blocks are controlled by heat exchangers (third *kind* boundary conditions). The assembly is vertical and the T_{oo1} temperature is always smaller than T_{oo2} , in order to avoid natural convection in the sample. A system of vertical guides allows the displacement of *block-2* according to the density variation of the sample. *Block-1* remains fixed at the base of the device.

We assume that the PCM is homogeneous and opaque. Heat transfer in the two metallic blocks and in the sample is described by the transient heat conduction equations. Between the metallic blocks and the sample, the thermal contact is assumed to be perfect. The phase change phenomenon in the sample is modelled using an enthalpy formulation proposed by Voller and Cross (1981). The moving boundary problem is given as follows:

$$\rho_1 \frac{\partial}{\partial t}(H) = \lambda_1 \frac{\partial^2 T}{\partial y^2} \quad \text{in } 0 < y < y_1, \text{ for } t > 0 \quad (1)$$

$$\rho(T) \frac{\partial}{\partial t}(H) = \frac{\partial}{\partial y}(\lambda(T) \frac{\partial T}{\partial y}) \quad \text{in } y_1 < y < y_2(t), \text{ for } t > 0 \quad (2)$$

$$\rho_2 \frac{\partial}{\partial t}(H) = \lambda_2 \frac{\partial^2 T}{\partial y^2} \quad \text{in } y_2(t) < y < y_3(t), \text{ for } t > 0 \quad (3)$$

$$-\lambda_1 \frac{\partial T}{\partial y} \Big|_{y=0^-} = -h(T(y=0^+) - T_{\infty 1}(t)) \quad \text{at } y=0, \text{ for } t > 0 \quad (4)$$

$$-\lambda_1 \frac{\partial T}{\partial y} \Big|_{y=y_1^-} = -\lambda(T) \frac{\partial T}{\partial y} \Big|_{y=y_1^+} \quad \text{at } y=y_1, \text{ for } t > 0 \quad (5)$$

$$-\lambda(T) \frac{\partial T}{\partial y} \Big|_{y=y_2(t)^-} = -\lambda_2 \frac{\partial T}{\partial y} \Big|_{y=y_2(t)^+} \quad \text{at } y=y_2(t), \text{ for } t > 0 \quad (6)$$

$$-\lambda_2 \frac{\partial T}{\partial y} \Big|_{y=y_3(t)^-} = h(T(y=y_3(t)^+) - T_{\infty 2}(t)) \quad \text{at } y=y_3(t), \text{ for } t > 0 \quad (7)$$

$$T(y,0) = T(y) \quad \text{in } 0 < y < y_3(t) \quad (8)$$

where ρ_1 , $\rho(T)$, and ρ_2 are the densities of the *block-1*, the sample and the *block-2*. The thermal conductivities of the *block-1*, the PCM and the *block-2* are given by λ_1 , $\lambda(T)$ et λ_2 respectively.

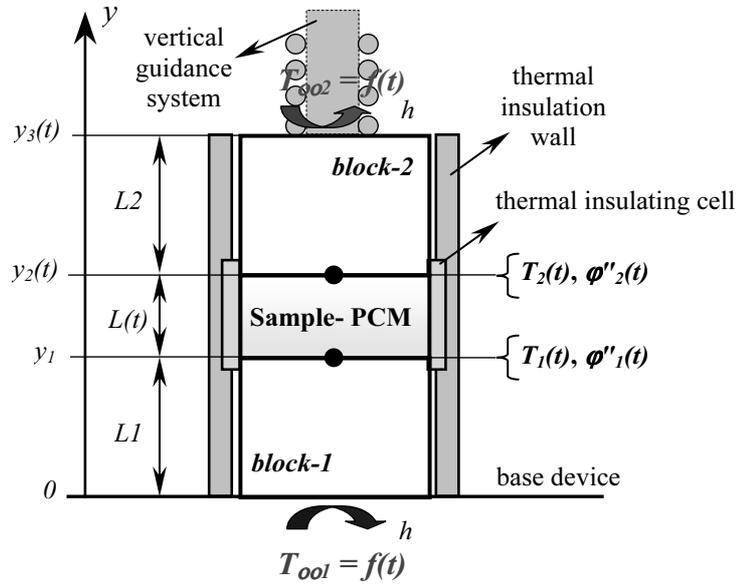


Figure 1. Experimental set-up

For the two metallic blocks, the thermophysical properties are supposed perfectly known and constant with respect to the temperature variation. The enthalpy, H , relation to temperature, T , for the blocks is given as:

$$H(T) = Cp_1[T - T_{refH}] + H_{ref} \quad \text{in } 0 < y < y_1, \text{ for } t \geq 0 \quad (9)$$

$$H(T) = Cp_2[T - T_{refH}] + H_{ref} \quad \text{in } y_2(t) < y < y_3(t), \text{ for } t \geq 0 \quad (10)$$

where Cp_1 and Cp_2 are the specific heats of the *block-1* and *block-2*. H_{ref} is a reference enthalpy which corresponds to an reference temperature, T_{refH} (here $H_{ref} = 0$ when $T_{refH} = 223.15$ K). For the PCM, Chiu and Caldwell (1996) proposed a general enthalpy formulation (a mixed enthalpy) given by:

$$H(T) = \int g Cp_s dT + \int (1-g) Cp_1 dT + (1-g) \Delta H \quad \text{in } y_1 < y < y_2(t), \text{ for } t \geq 0 \quad (11)$$

where Cp_s and Cp_l are the specific heats in the solid and liquid phases respectively. Here, they are assumed to be constants with respect to the temperature variation. The phase change latent heat is indicated by ΔH . The solid fraction, g , is a variable which depends on a number of factors, including the temperature and the material composition. Here, for simplicity, g is a linear function that depends only the temperature variation, such as:

$$g(T) = \frac{T_f - T}{\Delta T} \quad \text{with } 0 \leq g \leq 1 \tag{12}$$

where T_f is the phase change temperature and ΔT is the temperature variation during the phase change.

For a PCM, in some cases, the density and thermal conductivity are strongly dependent of the temperature. Here, the variation of the density is considered known. From experimental and bibliographical observations (Bonacina and Comini, 1974, Woinet, 1997), we adopted linear functions to represent the thermal conductivity variation. This choice has the objective to reduce the number of parameters to be estimated. They are given as follows :

i. for the solid phase, $g = 1$ (indice s) :

$$\lambda_s(T) = \alpha_s [T - T_{ref\lambda}] + \lambda_{ref} \tag{13}$$

ii. for the mushy region, $0 < g < 1$ (indice m) :

$$\lambda_m(T) = \lambda_s(T_f - \Delta T) + [1 - g] \Delta \lambda \tag{14}$$

iii. for the liquid phase, $g = 0$ (indice l) :

$$\lambda_l(T) = \lambda_m(T_f) + \alpha_l [T - T_f] \tag{15}$$

where α_l and α_s are the slopes of the thermal conductivity for liquid and solid phases. $\Delta \lambda$ is the jump of the thermal conductivity when the phase change occur. The variable λ_{ref} is a reference thermal conductivity which corresponds to an reference temperature $T_{ref\lambda}$. Figure (2) resumes the equations above.

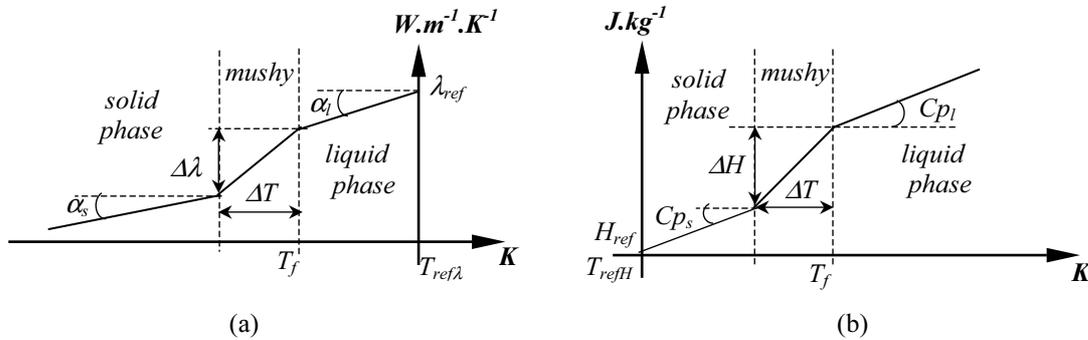


Figure 2. (a) PCM thermal conductivity - temperature relationship. (b) PCM Enthalpy- temperature relationship.

The experimental set-up described in this paragraph is modeled to provide the simulated transient measurements of the temperatures and heat fluxes at the limits of the sample: $T_1(t)$, $\phi''_1(t)$, $T_2(t)$ and $\phi''_2(t)$, Fig. (1). These simulated data will be introduced as boundary conditions in the *direct model*, paragraph (3).

2.1. Validation of the experimental set-up model

The numerical solution of the model is calculated using MATLAB software. A finite difference scheme (Voller and Cross, 1981, Ozisik, 1993) is used to discretize the differential equations of heat conduction of the PCM and the two metallic blocks, Eq. 1-8.

The total volume variation of the PCM is taken into account by modifying the grid size according to the variation of the density. An implicit formulation of the Runge-Kutta's method (Hosea and Shampine, 1996), available in MATLAB, is used to solve the matrix system. The numerical results in solidification are validated by comparing the temperature fields obtained by the approximate Neumann's analytical solution (Ozisik, 1993) and the numerical solution under the

same conditions as the analytical solution. The thermal properties used for the simulations are taken from n-Hexadecane. Figure (3) shows the temperature-time variations for two points inside of the PCM (these points are taken from the bottom side of the sample). The maximum relative errors found between the numerical and analytical results are smaller than 0.1%.

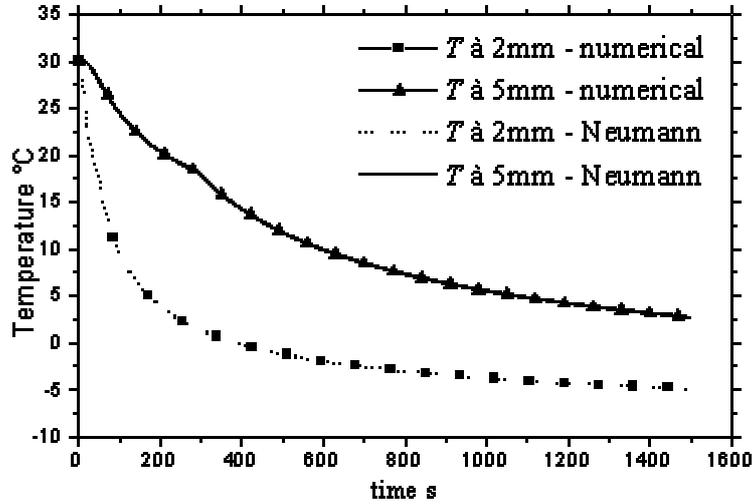


Figure 3. Numerical validation

3. Direct model

For the direct model only the heat transfers in the product is considered. Here, the assumptions adopted for the PCM are the same as those used in paragraph (2). Figure (4) presents this model.

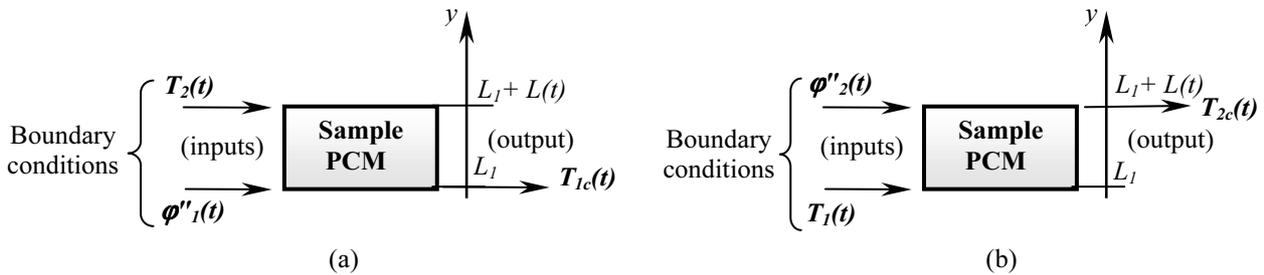


Figure 4. Direct model

In Fig. (4), we can notice that the outputs of the direct model, $T_{1c}(t)$ and $T_{2c}(t)$, are determined in two steps. Initially, $T_{1c}(t)$ is evaluated from the boundary conditions $\varphi''_1(t)$ et $T_2(t)$, Fig. (4.a). Afterwards, $T_{2c}(t)$ is calculated from the inputs $T_1(t)$ and $\varphi''_2(t)$, Fig. (4.b). These boundary conditions are generated by the numerical simulations of the experimental set-up, paragraph (2). The system of equations below describes the direct model.

$$\rho(T) \frac{\partial}{\partial t} (H) = \frac{\partial}{\partial y} (\lambda(T) \frac{\partial T}{\partial y}) \quad \text{in } L_1 < y < L_1 + L(t), \text{ for } t > 0 \tag{16}$$

$$-\lambda(T) \frac{\partial T}{\partial y} \Big|_{y=L_1} = \varphi''_1(t) \quad \text{at } y=L_1, \text{ for } t > 0, \text{ Fig. (4.a)} \tag{17.a}$$

$$T(y=L_1+L(t), t) = T_2(t) \quad \text{at } y=L_1+L(t), \text{ for } t > 0, \text{ Fig. (4.a)} \tag{17.b}$$

$$T(y=L_1, t) = T_1(t) \quad \text{at } y=L_1, \text{ for } t > 0, \text{ Fig. (4.b)} \tag{18.a}$$

$$-\lambda(T) \frac{\partial T}{\partial y} \Big|_{y=L_1+L(t)} = \varphi''_2(t) \text{ at } y=L_1+L(t), \text{ for } t > 0, \text{ Fig. (4.b)} \quad (18.b)$$

$$T(y,0) = T(y) \text{ in } L_1 < y < L_1+L(t) \quad (19)$$

For the system of equations above, Eq. 16-19, the same thermophysical properties-temperature relationship used in paragraph (2) are applied here. Its numerical solution is similarly as those described in paragraph (2.1).

4. Parameter estimation

The parameter estimation problem is solved by minimizing the least-squares norm with respect to the unknown parameters. The objective function, $S(\mathbf{P}, t)$, is written in matrix form as:

$$S(\mathbf{P}, t) = [\mathbf{Y}(t) - \mathbf{T}(\mathbf{P}, t)]^T \cdot [\mathbf{Y}(t) - \mathbf{T}(\mathbf{P}, t)] \quad (20)$$

where \mathbf{P} is the vector that contains the parameters to be estimated (the thermophysical properties of the PCM); \mathbf{Y} is the matrix of the measured data, Eq. (22), at time t ; and \mathbf{T} is the matrix of the calculated data, Eq. (23), which is a function of \mathbf{P} and t . The superscript T denotes the transposed.

From Fig. (2) we can show the values of \mathbf{P} , so :

$$\mathbf{P} = [\lambda_{ref}, \alpha_i, \alpha_s, \Delta\lambda, Cp_s, Cp_l, \Delta H, \Delta T, T_f] \quad (21)$$

The measured data, \mathbf{Y} , are obtained by numerical simulations of the experimental set-up, paragraph (2). These simulations provide the transient temperatures measurements on the interfaces of the metallic blocks/sample, as shown in Fig. (1). So:

$$\mathbf{Y} = [T_1(t), T_2(t)] \quad (22)$$

By the direct model, Fig. (4), we can obtain the values of \mathbf{T} :

$$\mathbf{T} = [T_{1c}(t), T_{2c}(t)] \quad (23)$$

The Levenberg-Marquardt's method (Beck and Arnold, 1977, Orlande and Ozisik, 2000) is used to optimize the values of the vector \mathbf{P} so as to minimize $S(\mathbf{P}, t)$. The interactive algorithm is given by:

$$\mathbf{P}^{k+1} = \mathbf{P}^k + [(\mathbf{J}(\mathbf{P}^k))^T \cdot \mathbf{J}(\mathbf{P}^k) + \mu^k \cdot \mathbf{\Omega}^k]^{-1} \cdot (\mathbf{J}(\mathbf{P}^k))^T \cdot [\mathbf{Y} - \mathbf{T}(\mathbf{P}^k)] \quad (24)$$

where the superscript k denotes the number of iterations, μ is a damping parameter and $\mathbf{\Omega}$ is a diagonal matrix:

$$\mathbf{\Omega}^k = \text{diag}[(\mathbf{J}(\mathbf{P}^k))^T \cdot \mathbf{J}(\mathbf{P}^k)] \quad (25)$$

where $\mathbf{J}(\mathbf{P})$ the sensitivity matrix or *Jacobian matrix*:

$$\mathbf{J}(\mathbf{P}) = [\nabla_{\mathbf{P}} [\mathbf{T}(\mathbf{P}, t)]]^T \quad (26)$$

and its reduced form is:

$$\mathbf{J}^*(\mathbf{P}) = \mathbf{P} \cdot \mathbf{J}(\mathbf{P}) \quad (27)$$

The analysis of $\mathbf{J}^*(\mathbf{P})$ is important (Raynaud, 1999, Orlande and Ozisik, 2000) because its columns reveal the sensitivity of the outputs variables of the direct model, $\mathbf{T} = [T_{1c}(t), T_{2c}(t)]$, with respect to each required property (λ_{ref} , α_i , α_s , $\Delta\lambda$, Cp_s , Cp_l , ΔH , ΔT , T_f).

5. Sensitivity coefficient analysis

We chose the paraffin PPW-20 as material-test whose the thermophysical properties are modeled according to the functions showed in paragraph (2) with the followings parameters: $\lambda_{ref} = 0.1412 \text{ W.m}^{-1}.\text{K}^{-1}$ (for $T_{ref\lambda} = 413.15 \text{ K}$), $\alpha_i = -0.000193 \text{ W.m}^{-1}.\text{K}^{-2}$, $\alpha_s = -0.000287 \text{ W.m}^{-1}.\text{K}^{-2}$, $\Delta\lambda = -0.0801 \text{ W.m}^{-1}.\text{K}^{-1}$, $Cp_s = 2500 \text{ J.kg}^{-1}.\text{K}^{-1}$, $Cp_l = 2670 \text{ J.kg}^{-1}.\text{K}^{-1}$, $\Delta H = 160000 \text{ J.kg}^{-1}$, $\Delta T = 9 \text{ K}$ et $T_f = 329.15 \text{ K}$.

The experimental methodology is as follows: at the beginning, the temperature in the sample and the two metallic blocks, Fig. (1), is uniform and higher than the phase change temperature, T_f . The sample is then solidified by decreasing simultaneously the temperatures $T_{oo1}(t)$ and $T_{oo2}(t)$. After its total solidification, these temperatures are increased until the sample returns to the liquid phase. All this process is simulated by the model described in paragraph (2). During the simulation, transient temperatures, $T_1(t)$ and $T_2(t)$, and densities of the heat fluxes, $\varphi''_1(t)$ et $\varphi''_2(t)$, are calculated at the extremities of the PCM. These values are introduced as boundary conditions in the direct model, paragraph (3).

The solution of the direct model, paragraph (3), allowed the numerical determination of the sensitivity matrix, Eq. (27). Each column of this matrix is plotted as shown in Fig. (5), (6) and (7). All of the curves present five important regions. Region I: the sample is in the liquid phase (liquid zone 1). Region II: the solid and liquid phases coexist (mushy zone 1). Region III: the sample is in the solid phase (solid zone). Region IV: the liquid and solid phases coexist (mushy zone 2). Region V: the sample returns to the liquid phase (liquid zone 2).

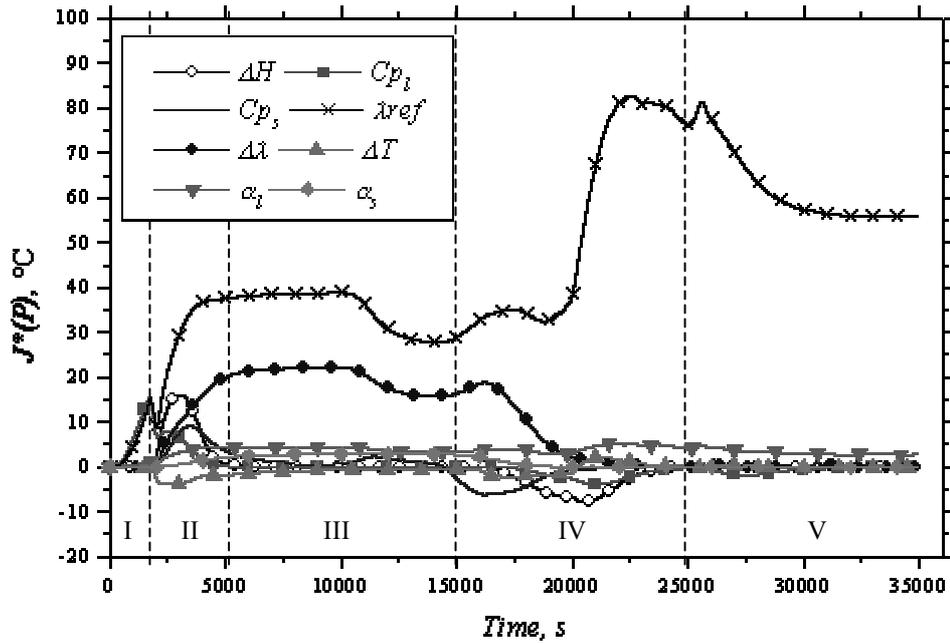


Figure 5. Sensitivity coefficients at the bottom of the sample: $y = L_1$, Fig. (4)

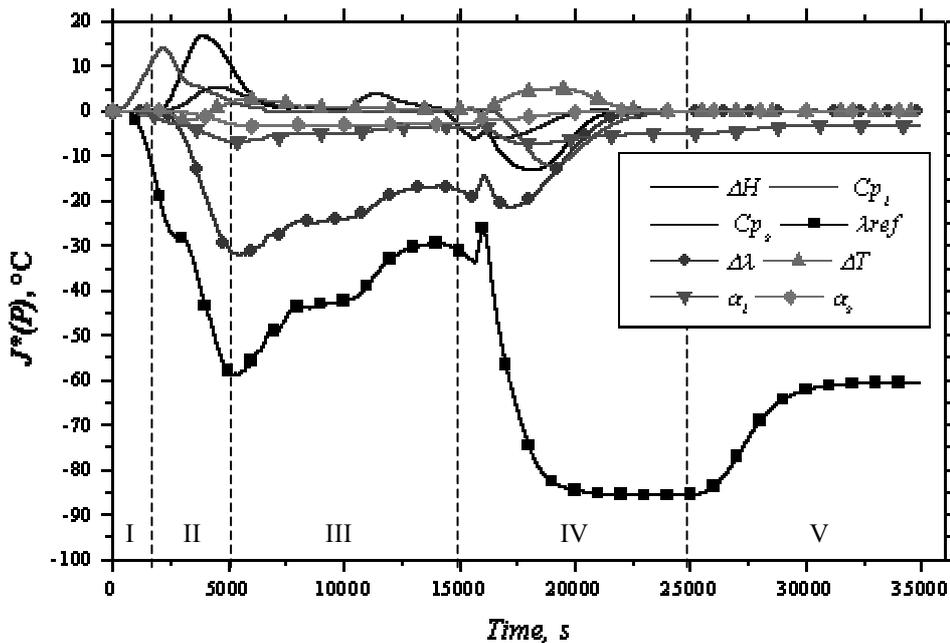


Figure 6. Sensitivity coefficients at the top of the sample: $y = L_1 + L(t)$, Fig. (4)

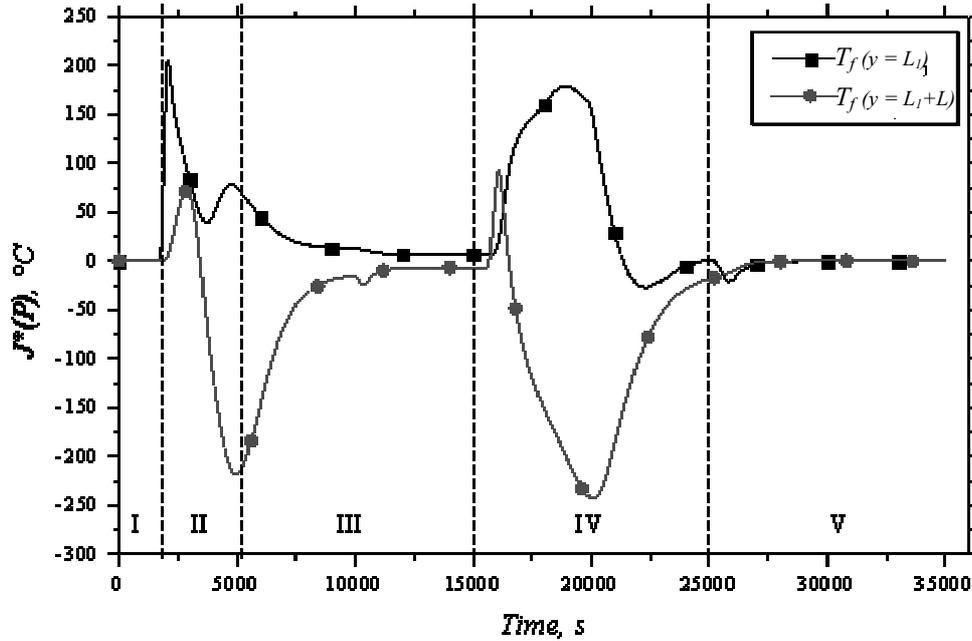


Figure 7. Sensitivity coefficients of $T_f(y=L_1)$ (at the bottom of the PCM) and $T_f(y=L_1+L(t))$ (at the top of the PCM)

In Fig. (5) and (6), we can notice that the parameters $\Delta\lambda$ and λ_{ref} cannot be simultaneously estimated in region III because they are linearly dependent. However, $\Delta\lambda$ can be estimated from the temperature measurements in region III, and λ_{ref} from the times corresponding to the regions IV and V.

The values of the sensitivity coefficients of the parameters $\Delta\lambda$, ΔH , T_f , and ΔT vanish in the liquid zones 1 and 2. In fact, these properties characterize only the phase change of the product.

There is no linear dependence among the parameters ΔH , Cp_l , Cp_s . Moreover, the direct model has a good sensitivity to these parameters. We can easily estimate them in the regions where their sensitivity coefficients are more important, i.e., in the regions I, II and IV.

The values of the sensitivity coefficients of the parameters α_s and α_l are not important. This is because the thermal conductivity of this PCM varies weakly in the solid and liquid phases. In fact, the values of these parameters represent only 2% of variation of the thermal conductivities (in a 50 K temperature range) in the two phases.

The Fig. (7) shows that the direct model has a strong sensitivity to the phase change temperature. In fact, T_f is the parameter which determines the moment when the phase change latent heat is released or absorbed. In spite of its important sensitivity, its estimation is delicate because an important error in its initial value (more than 7%) can generate a divergence in the program. Then, it is important to have a priori information of this parameter.

6. Results and discussion

Thanks to the sensitivity coefficients analysis, paragraph (5), the Levenberg-Marquardt's algorithm (Beck and Arnold, 1977, Orlande and Ozisik, 2000) was optimized. The identification iterative methodology that has been used is resumed in the following basic steps:

Step 0. calculate $S(\mathbf{P}^i, t)$, Eq. (20), by using the initials values adopted to the vector \mathbf{P}^i , Eq. (21).

Step 1. estimates λ_{ref}^{i+1} by using Levenberg-Marquardt's method with the temperature values, Eq. (22-23), corresponding the region V, see Fig. (5) and (6).

Step 2. estimates Cp_l^{i+1} by using Levenberg-Marquardt's method with temperature values, Eq. (22-23), corresponding the region I, see Fig. (5) and (6).

Step 3. estimate α_l^{i+1} , α_s^{i+1} , $\Delta\lambda^{i+1}$, Cp_s^{i+1} , ΔH^{i+1} , ΔT^{i+1} and T_f^{i+1} by using Levenberg-Marquardt's method with temperature values, Eq. (22-23), corresponding the regions II, III and IV, see Fig. (5), (6) and (7).

Step 4. repeat steps 1, 2 and 3 until satisfy the stop criteria below:

$$N^{-1}[S(\mathbf{P}^{i+1}, t)]^{-1/2} < \varepsilon \tag{28}$$

where N is the number of measured data and i denotes the number of iterations of the identification methodology. ε is tolerance value (here, $\varepsilon \approx 2e-2$).

For the test-case presented in Tab. (1), we suppose that the parameters α_l and α_s are known. The other parameters are optimized by minimizing the objective function, $S(\mathbf{P}, t)$, using the Levenberg-Marquardt's interactive procedure, Eq.

(24), and the iterative methodology described above. In spite of the initial values being distant 60% approximately of the real data, the maximal relative error is smaller than 0.5%.

Table 1. Estimated parameters – slopes (α_s and α_i) are assumed to be known.

	λ_{ref} $W.m^{-1}.K^{-1}$	C_{p_l} $J.kg^{-1}.K^{-1}$	ΔH $J.kg^{-1}$	C_{p_s} $J.kg^{-1}.K^{-1}$	$\Delta\lambda$ $W.m^{-1}.K^{-1}$	ΔT K	T_f K
Real data	0.1412	2670	160000	2500	-0.0801	9.00	329.15
Initial values	0.0847	1602	95000	3000	-0.0481	7.00	323.15
Iteration, $i=1$	0.1410	2719	174659	2657	-0.0777	19.19	338.22
Iteration, $i=3$	0.1412	2669	162303	2502	-0.0802	9.86	329.63
Iteration, $i=5$ (estimated)	0.1412	2669	160155	2499	-0.801	9.04	329.17
Relative error (%)	0.00	0.04	≈ 0.00	≈ 0.00	0.00	0.44	≈ 0.01

The second test-case, Tab. (2), takes into account all of the parameters suggested for the thermal characterization of the paraffin PPW-20. We can notice that the initial values for the required parameters are far from the real ones in 20% approximately. The Levenberg-Marquardt's method presents a good convergence, from the third iteration of the iterative methodology, the average of the relative errors of all of the parameters is 0.1% approximately.

Table 2. Estimated parameters – slopes (α_s and α_i) are identified.

	λ_{ref} $W.m^{-1}.K^{-1}$	C_{p_l} $J.kg^{-1}.K^{-1}$	ΔH $J.kg^{-1}$	C_{p_s} $J.kg^{-1}.K^{-1}$	$\Delta\lambda$ $W.m^{-1}.K^{-1}$	α_i $W.m^{-1}.K^{-2}$	α_s $W.m^{-1}.K^{-2}$	ΔT K	T_f K
Real data	0.1412	2670	160000	2500	-0.0801	-0.000193	-0.000287	9.00	329.15
Initial values	0.1694	2136	128000	3000	-0.0641	-0.000150	-0.000137	7.00	323.15
Iteration, $i=1$	0.1429	2720	158675	2522	-0.0788	-0.000195	-0.000197	10.75	330.05
Iteration, $i=3$ (estimated)	0.1412	2669	160340	2499	-0.0801	-0.000193	-0.000287	9.09	329.20
Relative error (%)	0.00	0.04	0.21	0.04	0.00	0.00	0.00	1.00	0.02

7. Conclusions

The sensitivity coefficients analysis provided the essential informations for the optimization of the estimation algorithm. (i) We saw the regions where their variations were more important. (ii) We noticed the linear dependence between $\Delta\lambda$ and λ_{ref} . (iii) We observed that a possible estimation of T_f can be done if we have a priori informations of this parameter.

Initially, the estimations results (obtained with simulated transient measurements of the temperatures) were very satisfactory. Indeed, the Levenberg-Marquardt's method seems to provide accurate estimates for the thermophysical properties. However, the results presented in Tab. (1) and (2) consider that the simulated temperatures have not measurement errors. A complementary study, which take into account the measurement errors, is in progress.

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