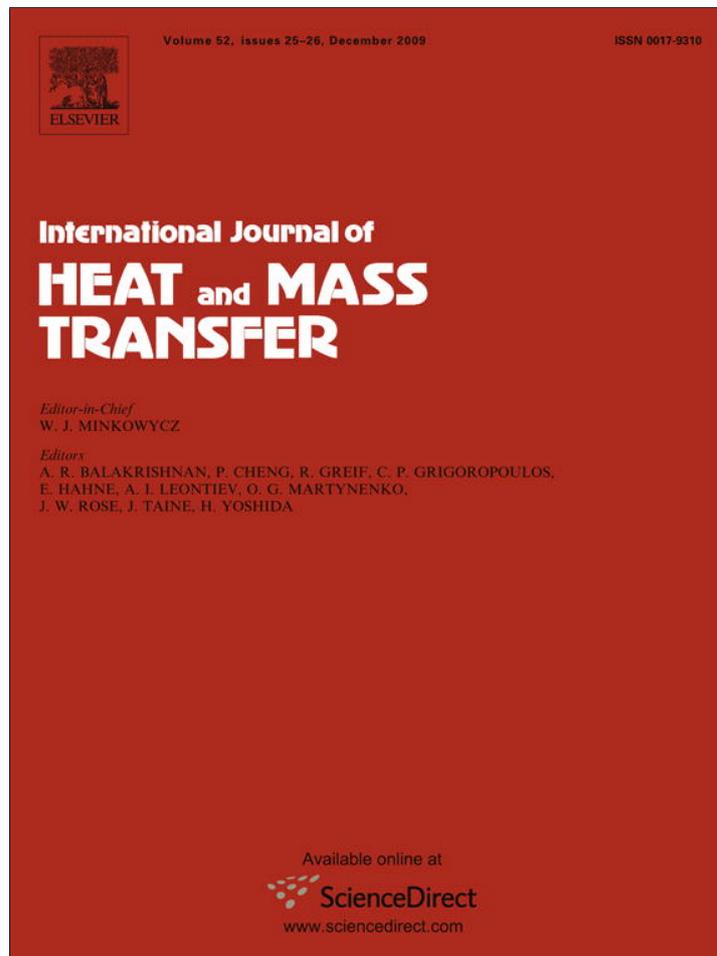


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# Thermal diffusivity estimation using numerical inverse solution for 1D heat conduction

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## ABSTRACT

This work presents an improved apparatus and a numerical approach to obtain the estimate of thermal diffusivity of complex materials. Transient thermal response at the axis of cylindrical sample is measured when boundary temperature is suddenly changed. Instead of assuming an ideal step temperature excitement, a measured temperature of a material boundary was employed. An iterative procedure, based on minimizing a sum of squares function with the Levenberg–Marquardt method, is used to solve the inverse problem. A graphical user interface is built to enable easy use of the inverse thermal diffusivity estimation method. The reference materials used to evaluate the method are Agar water gel, glycerol and Ottawa quartz sand.

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## 1. Introduction

Thermal diffusivity  $a$  ( $\text{m}^2 \text{s}^{-1}$ ) is an important property of material in all problems involving transient heat conduction. It is a measure of rapidity of the heat propagation through a material and combines thermal conductivity ( $\lambda$ ), specific heat ( $c_p$ ) and density ( $\rho$ ) according to:

$$a = \frac{\lambda}{\rho c_p} \quad (1)$$

There are many cases in which thermal diffusivity of the studied material is not available, e.g. cement materials of different compositions during cement setting and hardening [1–8], powdered granular or porous food products with variable heterogeneous structure [9,10], and wide range of composite materials [11]. Furthermore, these materials may be wet and porous and in order to prevent development of humidity gradients under imposed thermal gradients, one finds transient measurement methods as preferable [12,13]. Through such complex heterogeneous, multiphase and polydisperse materials the heat is transferred by a combination of different modes. They include conduction through the solid particles, conduction and convection through the gaseous and liquid phases, evaporation–condensation mechanism [8,13], and radiation at the particle surfaces. However, the overall heat transfer process is usually modelled solely by a heat conduction model considering the conduction parameters as apparent [1–13].

The determination of thermal diffusivity is very challenging since it belongs to a class of inverse problems where an estimated parameter is very sensitive to measured quantities necessary for its calculation. There are several methods to measure the thermal diffusivity of materials. Laser flash method requires relatively complex instrumentation [14,15], thin samples (below  $\sim 1$  cm) and special preparation techniques for testing low-conducting materials [16]. A hot wire method, which is a standard transient method for thermal conductivity determination, is much less sensitive and reliable for thermal diffusivity determination [5,17,18]. A simple experimental technique based on the thermal response of cylindrical samples when boundary temperature is suddenly changed is shown efficient and reliable for estimating thermal diffusivity [1,2,9,10]. The method is based on one-dimensional solution of Fourier's heat balance. Based on the existing experimental transient method the thermal diffusivity can be estimated by fitting the experimental results to a theoretical expression based on a Bessel function [19,20] or its linear (first term) approximation [9,10]. Materials which cannot be molded or require tests under sealed conditions (most of the aforementioned materials) necessitate usage of tube sample holder. However, the accuracy of the estimated value is strongly influenced by a discrepancy between real and conceptual boundary conditions. Therefore, in this work a numerical approach for thermal diffusivity estimation is proposed for better accuracy and precision on materials which require a sample holder. Instead of assuming an ideal step temperature change, a measured temperature for the material boundary condition is employed. A graphical user interface (GUI) built to enable easy use of the presented inverse thermal diffusivity estimation method is freely available by sub-programs that were written for the setting of Matlab.

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**Nomenclature**

|              |  |
|--------------|--|
| 2R           | inner diameter of cylindrical tube (mm)                                |
| $a$          | thermal diffusivity ( $\text{m}^2 \text{s}^{-1}$ )                     |
| $c_p$        | specific heat capacity ( $\text{J kg}^{-1} \text{K}^{-1}$ )            |
| $e$          | user prescribed tolerance ( $^\circ\text{C}$ )                         |
| GUI          | graphical user interface   |
| $l$          | length of temperature vector $\mathbf{T}$                              |
| $\mathbf{J}$ | sensitivity (Jacobian) vector  |
| $k$          | iteration number   |
| $m$          | mass fraction  |
| $q_v$        | volumetric heat generation or sink term ( $\text{W m}^{-3}$ )          |
| $q_v$        | volumetric heat generation ( $\text{W m}^{-3}$ )                       |
| $r$          | radial dimension (m)   |
| $R$          | radius of cylindrical base of specimen (m)                             |
| $S$          | objective (sum of squares) function                                    |
| SD           | standard deviation ( $^\circ\text{C}$ )                                |
| $SD_{fit}$   | standard deviation of fitted temperature response ( $^\circ\text{C}$ ) |
| $T$          | temperature ( $^\circ\text{C}$ )                                       |
| $t$          | time (s)   |
| $\mathbf{T}$ | vector containing the measured temperatures (at a location $r = 0$ )   |

$\mathbf{u}$  vector containing the estimated temperatures (at a location  $r = 0$ )

*Greeks symbols*

|           |  |
|-----------|--|
| $\lambda$ | thermal conductivity ( $\text{W m}^{-1} \text{K}^{-1}$ ) |
| $\mu$     | damping parameter  |
| $\rho$    | density ( $\text{kg m}^{-3}$ )                           |
| $\varphi$ | volume fraction  |
| $\Omega$  | Levenberg–Marquardt parameter                            |

*Subscript*

$i$  time  $t_i$  where  $i = 1, \dots, I$

*Superscripts*

|     |                       |
|-----|-----------------------|
| 0   | initial value         |
| $k$ | iteration number      |
| Tr  | transpose of a vector |

**2. Numerical inverse solution for estimating the thermal diffusivity**

The inverse estimation of thermophysical properties can be classified into two main groups [21]. First is the direct problem usually solved with numerical methods such as finite differences. Second is the inverse problem, which is actually an optimization problem, in this work solved using Levenberg–Marquardt algorithm [22,23]. This iterative method for solving least square optimization is quite stable, powerful and straightforward and is applied in a large variety of inverse heat transfer problems.

The solution of inverse heat transfer problems with the Levenberg–Marquardt method can be suitably arranged in the following basic steps:

- The direct problem
- The inverse problem
- The computational algorithm
- The stopping criteria

The details of each of these steps are presented below. Based on the experimental design (see Section 3.2) we assume that the temperature distribution in the mold can be calculated from the one-dimensional transient heat conduction model. Transient heat conduction in the radial direction in an infinitely long cylinder can be expressed as [24]:

$$\frac{1}{a} \frac{\partial T(r,t)}{\partial t} = \frac{\partial^2 T(r,t)}{\partial r^2} + \frac{1}{r} \frac{\partial T(r,t)}{\partial r} + \frac{q_v}{\lambda} \quad (2)$$

$$\begin{aligned} T(r, t = 0) &= f(r), & \text{initial condition} \\ T(r = R, t) &= g(t), & \text{boundary condition} \\ \frac{\partial T}{\partial r}(r = 0, t) &= 0, & \text{symmetry} \end{aligned} \quad (3)$$

For materials that involve no chemical reactions or phase changes, the volumetric heat generation/sink term,  $q_v$  ( $\text{W m}^{-3}$ ) in Eq. (2) is zero. It should be noted that for a sufficiently constant heat generation rate during a period of testing, this term can be also disregarded. Initially, a material has a defined temperature field  $T(r, t = 0) = f(r)$ . After a required period of stabilization and no heat generation or sink term a uniform initial temperature  $T(r, t = 0) = T^0$  can be assumed. A Dirichlet condition is considered for material boundary

value with imposed measured time-dependant temperature increase.

*2.1. The direct problem*

In the direct problem associated with the physical problem described above, the thermal diffusivity is known. The objective of the direct problem is then to determine the transient temperature field  $T(r, t)$  in the material.

*2.2. The inverse problem*

For the inverse problem considered of the interest here, the thermal diffusivity is estimated, based on obtained transient temperature measurements taken at a location  $r = 0$  and  $r = R$  at times  $t_i$ ,  $i = 1, 2, \dots, I$ , and the minimization of an objective function,  $S$ .

$$S(a) = [\mathbf{T} - \mathbf{u}(a)]^{\text{Tr}} [\mathbf{T} - \mathbf{u}(a)] \quad (4)$$

where  $a$  is unknown thermal diffusivity,  $\mathbf{T}$  and  $\mathbf{u}$  are the vectors containing the measured and estimated temperatures (at a location  $r = 0$ ), respectively, and the superscript Tr indicates the transpose of a vector. The estimated temperatures are obtained from the solution of the direct problem with an estimate for the thermal diffusivity.

Thus, the ordinary least squares norm, given by Eq. (4), can be written as:

$$S(a) = [\mathbf{T} - \mathbf{u}(a)]^{\text{Tr}} [\mathbf{T} - \mathbf{u}(a)] = \sum_{i=1}^I [T_i - u_i(a)]^2 \quad (5)$$

where the subscript  $i$  refers to time  $t_i$ , where  $i = 1, \dots, I$ .

*2.3. The iterative procedure*

To minimize the least squares norm given by Eq. (5) the derivative of  $S(a)$  with respect to the unknown parameter is equated to zero,

$$\frac{\partial S(a)}{\partial a} = 0 \quad (6)$$

Such necessary condition for the minimization of  $S(a)$  can be represented in matrix notation by equating the gradient of  $S(a)$  with respect to parameter  $a$  to zero

$$\nabla S(a) = 2 \left[ -\frac{\partial \mathbf{u}^{\text{Tr}}(a)}{\partial a} \right] [\mathbf{T} - \mathbf{u}(a)] = 0 \quad (7)$$

$$\frac{\partial \mathbf{u}^{\text{Tr}}(a)}{\partial a} = \left[ \frac{\partial}{\partial a} \right] [T_1 T_2 \dots T_i] \quad (8)$$

The sensitivity or Jacobian vector,  $\mathbf{J}(a)$ , is defined as the transpose of Eq. (8), that is,

$$\mathbf{J}(a) = \left[ \frac{\partial \mathbf{u}^{\text{Tr}}(a)}{\partial a} \right]^{\text{Tr}} \quad (9)$$

Therefore, we can write the sensitivity vector in the form

$$\mathbf{J}(a) = \left[ \frac{\partial \mathbf{u}^{\text{Tr}}(a)}{\partial a} \right]^{\text{Tr}} = \begin{bmatrix} \frac{\partial T_1}{\partial a} \\ \frac{\partial T_2}{\partial a} \\ \vdots \\ \frac{\partial T_i}{\partial a} \end{bmatrix} \quad (10)$$

The elements of the sensitivity vector, called the sensitivity coefficients are thus defined as the first derivative of the estimated temperature at time  $t_i$  with respect to the unknown parameter.

$$J_i = \frac{\partial T_i}{\partial a} \quad (11)$$

The estimation of the parameter for a small value of the magnitude of  $J_j$  is difficult, because basically the same value for temperature would be obtained for a wide range of parameter values. The estimation problem is ill-conditioned near the initial guess used for the unknown parameter, creating difficulties in the applications of Gauss method. The Levenberg–Marquardt method [22,23] alleviates such difficulties by utilizing an iterative procedure in the form:

$$a^{k+1} = a^k + (\mathbf{J}^k)^{\text{Tr}} [\mathbf{T} - \mathbf{u}(a^k)] \left[ (\mathbf{J}^k)^{\text{Tr}} \mathbf{J}^k + \mu^k \Omega^k \right]^{-1} \quad (12)$$

where  $\mu^k$  is a positive scalar named damping parameter, and  $\Omega^k$  is a Levenberg–Marquardt parameter.

The purpose of the term  $\mu^k \Omega^k$ , included in Eq (12), is to damp oscillations and instabilities due to the ill-conditioned character of the problem, by making it large as compared to  $\mathbf{J}^{\text{Tr}} \mathbf{J}$  if necessary [21].

#### 2.4. Determining the sensitivity coefficients

There are several different approaches for the computation of the sensitivity coefficients including: the direct analytic solution, the boundary value problem, and the finite-difference approximation [21]. In this work, a boundary value problem for the determination of sensitivity coefficients is obtained by differentiating the original direct problem presented in Eqs. (2) and (3) with respect to the unknown parameter,  $a$ . The sensitivity problem governing the sensitivity coefficients  $J_a = \frac{\partial T}{\partial a}$  is thus:

$$\frac{1}{a} \frac{\partial J_a(r, t)}{\partial t} = \frac{\partial^2 J_a(r, t)}{\partial r^2} + \frac{1}{r} \frac{\partial J_a(r, t)}{\partial r} + \frac{1}{a^2} \frac{\partial T(r, t)}{\partial t} \quad (13)$$

$$\begin{aligned} J_a(r, t = 0) &= 0 \\ J_a(r = R, t) &= 0 \\ \frac{\partial J}{\partial r} &= (r = 0, t) = 0 \end{aligned} \quad (14)$$

#### 2.5. Statistical analysis

The standard deviation of the fitted temperature response,  $SD_{\text{fit}}$  is calculated from the ordinary least squares norm,  $S(a)$  as:

$$SD_{\text{fit}} = \sqrt{\frac{S(a)}{N-1}} \quad (15)$$

The standard deviation for the estimated thermal diffusivity,  $SD_a$  can be obtained from the standard deviation of the measurement error,  $SD_T$  and the sensitivity vector  $\mathbf{J}(a)$  according to:

$$SD_a = SD_T \sqrt{[\mathbf{J}^{\text{Tr}} \mathbf{J}]^{-1}} \quad (16)$$

#### 2.6. Stopping criteria

The iterative procedure of the Levenberg–Marquardt method is stopped after fulfilling the following criterion:

$$SD_{\text{fit}}(a^{k+1}) < e \quad (17)$$

where  $e$  is user prescribed tolerance; used value in this paper is about  $e \sim 0.015$  °C. This criterion results from the fact that the least squares norm is sufficiently small when the method has converged [25].

#### 2.7. Computation algorithm

Different versions of the Levenberg–Marquardt method can be found in the literature, depending on the choice of the value  $\Omega$  and on the form chosen for the variation of the damping parameter  $\mu$  [21]. A procedure with the  $\Omega$  is taken as:

$$\Omega^k = (\mathbf{J}^k)^{\text{Tr}} \mathbf{J}^k \quad (18)$$

An initial guess  $a^0$  and maximal number of iterations,  $k_{\text{max}}$  is set. A value for  $k$  and  $\mu^0$  was set to  $k = 1$ , and  $\mu^0 = 10^{-4}$ . Then

- Step 1. Solve the direct problem given by Eqs. (2) and (3) with the available estimate  $a^k$  to obtain the temperature vector  $\mathbf{u}(a^k) = [u_1, u_2, \dots, u_i]$ .
- Step 2. Compute  $S(a^k)$  from Eq. (5).
- Step 3. Compute the sensitivity vector  $\mathbf{J}^k$  by solving Eqs. (13) and (14) and then the value  $\Omega^k$  with Eq. (18), by using the current values of  $a^k$ .
- Step 4. Solve the following linear system of algebraic equations, obtained from the iterative procedure of the Levenberg–Marquardt method in order to compute vector  $\Delta a^k$ :

$$\Delta a^k = (\mathbf{J}^k)^{\text{Tr}} [\mathbf{T} - \mathbf{u}(a^k)] \left[ (\mathbf{J}^k)^{\text{Tr}} \mathbf{J}^k + \mu^k \Omega^k \right]^{-1} \quad (19)$$

- Step 5. Compute the new estimate as

$$a^{k+1} = a^k + \Delta a^k \quad (20)$$

- Step 6. Solve the direct problem in Eqs. (2) and (3) with the new estimate  $a^{k+1}$  in order to find  $\mathbf{u}(a^{k+1})$ . Then compute  $S(a^{k+1})$ , with Eq. (5).
- Step 7. If  $S(a^{k+1}) \geq S(a^k)$ , replace  $\mu^k$  by  $10 \mu^k$  and return to Step 4, but if  $\mu^k > 10^{20}$  go to Step 8.
- Step 8. If  $S(a^{k+1}) < S(a^k)$ , accept the new estimate  $a^{k+1}$  and replace  $\mu^k$  by  $0.1 \mu^k$ .
- Step 9. Accept the estimated value if the stopping criterion, Eq. (17), is satisfied. Otherwise, replace  $k$  by  $k + 1$  and return to Step 3. If  $k = k_{\text{max}}$  stop the iteration.

### 3. Experimental

#### 3.1. Materials

The reference materials used to evaluate the method and used apparatus are gelatinous water (Agar gel 0.7%), glycerol (p.a.

redistilled) and Ottawa (quartz) sand. For detailed discussion on reference materials used please see further in Section 4.2.

### 3.2. Thermal diffusivity measurement setup

To determine thermal diffusivity of a sample, an improved experimental transient method was used based on existing method [1,2,9,10]. The experimental setup consists of a two thermostated water baths ( $\pm 0.03^\circ\text{C}$ ), the copper tube container, K-type thermocouples wiring, data acquisition unit and a PC. Two sizes of cylindrical copper containers were used, with inner diameter  $2R = 51$  or  $26$  mm, length  $250$  mm. Thickness of the larger ( $2R = 51$  mm) and smaller ( $2R = 26$  mm) container, is  $1$  and  $0.7$  mm, respectively. The copper tube was carefully filled with the material continuously applying vibrations in order to minimize air entrapment. One thermocouple was placed at the axis of the tube. The accurate radial position of the thermocouple measuring end ( $r = 0 \pm 1$  mm) was obtained by using a prestressed plastic thread,  $0.30$  mm thick, supported diagonally at the bases of a cylinder, as shown in Fig. 1. Second thermocouple is placed at the inside surface of the copper tube wall. The copper tube filled with material is carefully sealed with styropore and rubber stoppers and placed vertically in a temperature controlled water bath ( $\pm 0.03^\circ\text{C}$ ). The wires exit through the upper base of the tube. Upper insulating base is above a bath water level, Fig. 1. A cylindrical specimen contained in copper tube, attains a uniform temperature of first bath,  $T_1$ , and then is suddenly immersed in a second bath at temperature  $T_1 + \Delta T$ . The temperature at the tube axis,  $T(r \sim 0, t)$  and the material boundary temperature,  $T(r = R, t)$  are measured as a function of time.

K-type thermocouples  $0.2$  mm thick with grounded twisted-shielded wiring were used to obtain accuracy and eliminate noise. Cable shielding and twisted wire pairs are used to minimize or eliminate capacitive coupled interference and to aid in lowering electro-magnetically induced errors. The thermocouples are calibrated before use (using Pt 100 with overall accuracy  $\pm 0.03^\circ\text{C}$ ), and they are observed to have an accuracy of  $\pm 0.1^\circ\text{C}$  in a temperature range of  $0$ – $90^\circ\text{C}$ . An eight channel data logger (TC-08 pico technology) is used for temperature measurements. The  $20$  bit resolution ensures detection of minute changes in temperature. It

stores the entire set of temperatures once every  $100$  ms. The experimental data is simultaneously transferred to a PC. Thermocouple cold junction is held at room temperature and sensed by a precision thermistor in good thermal contact with the input connectors on thermal block of the measuring instrument. In order to have accurate cold junction compensation a change of its temperature is kept as low as possible.

Prior to loading the mold, the Ottawa sand was dried at  $105^\circ\text{C}$ . The gelatinous water was prepared by mixing  $0.7\%$  of Agar powder (Biolife) by weight with hot ( $85^\circ\text{C}$ ) deionized water in a laboratory glass. The mixture was heated and stirred vigorously using a magnetic stirrer hot plate. Once the gel was melted, it was poured into a mold by tapping it to help the air bubbles rise to the surface. The mold was put in a water bath at  $20^\circ\text{C}$  and waited for at least  $4$  h to attain uniform temperature. For measurements on glycerol care was devoted regarding to its hygroscopic nature.

## 4. Results and discussion

### 4.1. Numerical method for inverse thermal diffusivity estimation

In this work, a numerical approach for thermal diffusivity estimation is adopted to employ a real measured temperature excitement for the material boundary condition,  $T(r = R, t)$ . Fourier's one-dimensional radial heat conduction model in Eqs. (2) and (3) and the sensitivity problem given by Eqs. (13) and (14) was solved using Matlab's built-in solver "pdepe" [26,27]. The Levenberg–Marquardt method [22,23] for optimization was used for the solution of the inverse problem of the parameter estimation [21]. The inverse problem was solved and optimized by using a sub-program that was written for the setting of Matlab 6.5 (The MathWorks, Inc., Natick, MA); script files comprising a specially built graphical user interface (GUI) are freely available upon request to the corresponding author. In order to speed up the iterative computing, the temperature-time input, i.e.  $T(r = R, t)$  and  $T(r = 0, t)$  is previously reduced as follows. First, the accurate onset of excitement was determined from raw temperature data collected each  $100$  ms. Then the temperature records were reduced to about  $1000$  time points for each measurement position.

#### 4.1.1. Graphical user interface (GUI)

By providing an interface between the user and the application's underlying code, GUIs enable the user to operate the application without managing the commands required by a command line interface. Therefore, applications that provide GUIs are much easier to learn and use than those that are run from the command line.

Based on the built GUI shown in Fig. 2 and a guided instructions written by the author even the first time users of Matlab with no programming skills are encourage to use the developed numerical method for inverse thermal diffusivity estimation. After importing and plotting the data the required inputs for the GUI listed in order as shown in Fig. 2 are: radial grid,  $r/m$  (introduced as the axial starting point (always zero), increment and radius of the tube  $R$ ), the initial condition  $T(r, t = 0) = T^0$ , radial position of 'axial' thermocouple, initial guess  $a^0$ , prescribed tolerance for the stopping criteria  $e$  (see Section 2.6), and maximal number of iterations. The output of the numerical model gives a result of thermal diffusivity estimate (example shown in Figs. 3 and 4). Ten iterations for such data run on Intel® Pentium® 4 CPU  $3.00$  GHz,  $1$  GB RAM, take about  $40$  s.

#### 4.2. Evaluation on reference materials

The reference materials used to evaluate the used apparatus and method are gelatinous water (Agar gel  $0.7\%$ ), glycerol (p.a. redistilled) and Ottawa quartz sand.

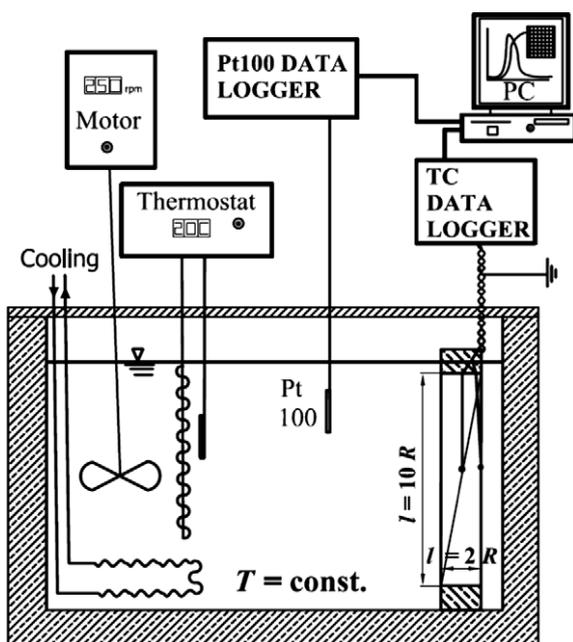


Fig. 1. Thermal diffusivity measuring set-up.

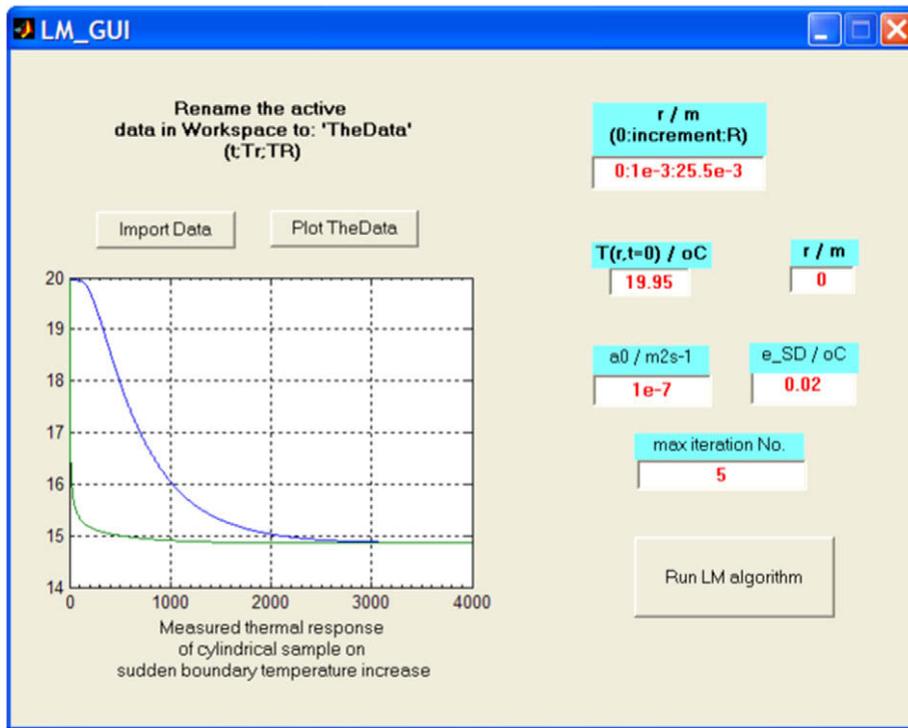


Fig. 2. The developed graphical user interface enables easy usage of the inverse thermal diffusivity estimation method.

Agar is gel-forming polysaccharide, widely used in industry and in scientific applications. Agar form gels at approximately 35 °C and once formed does not melt below 85 °C. Gelation occurs when a chain of macromolecules forms a network capable of entrapping the dispersing medium. Such gel has a composition very close to a pure liquid but resemble a solid. In that way heat transfer through Agar is by conduction solely, excluding natural convection.

Ottawa sand consists of spherical grains (high purity silica) with an accurately graded particle size distribution to pass an 850- $\mu\text{m}$  (US Standard No. 20) sieve and to be retained on a 600- $\mu\text{m}$  (US Standard No. 30) sieve. The solid density of the Ottawa sand was measured by submerging a known mass of sand in water and measuring the volume change of the liquid. The solid density was

found to be 2.69  $\text{g}/\text{cm}^3$ , which is very close to the published value of 2.65  $\text{g}/\text{cm}^3$  [28]. Knowledge of the bulk density is important because the thermal properties can change based on how tightly the sand particles are packed. Measured bulk density of investigated

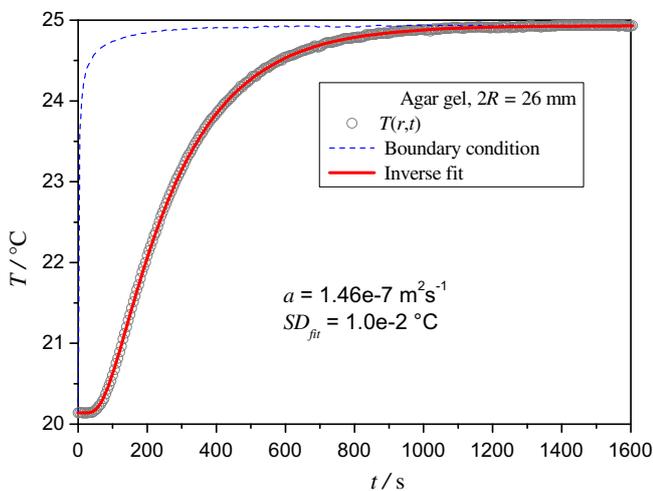


Fig. 3. An example of a result for estimation of thermal diffusivity based on the measured temperature response (Agar gel, 2R = 26 mm) and the radial heat conduction model in Eqs. (2) and (3).

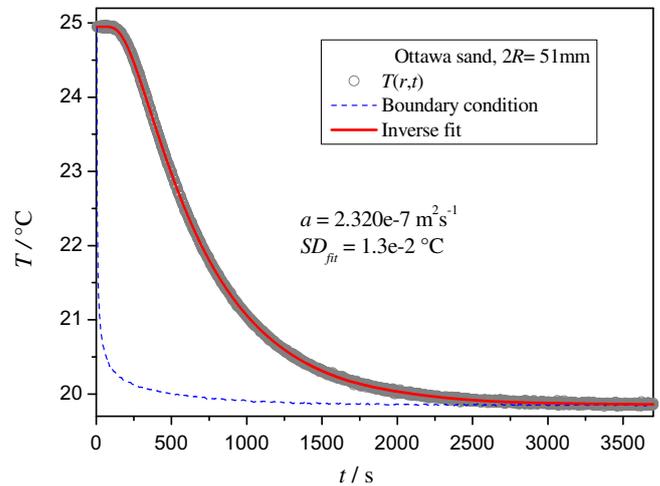


Fig. 4. An example of a result for estimation of thermal diffusivity based on the measured temperature response (Ottawa sand, 2R = 51 mm) and the radial heat conduction model in Eqs. (2) and (3).

Table 1

Literature data on thermal conductivity and thermal diffusivity of reference materials at  $T = 22.5\text{ }^\circ\text{C}$ .

| Material    | $\lambda$ ( $\text{W m}^{-1} \text{K}^{-1}$ ) | $\alpha \times 10^7$ ( $\text{m}^2 \text{s}^{-1}$ ) |
|-------------|---|---|
| Water       | 0.603 [34]                                    | 1.444 [34]  |
| Glycerol    | 0.285 [35]                                    | 0.958 [35]  |
| Ottawa sand | 0.306 [29]                                    | 2.33 <sup>a</sup> [30–33]                           |

<sup>a</sup> Sand in general.

**Table 2**  
Comparison of obtained and published data on thermal diffusivity at  $T = 22.5$  °C.

| Material                | 2R (mm) | $a \times 10^7$ (m <sup>2</sup> s <sup>-1</sup> ) |                            |                           |              |
|-------------------------|---------|---|----------------------------|---------------------------|--------------|
|                         |         | Measured  | Precision <sup>a</sup> (%) | Reported                  | Accuracy (%) |
| Gelatinous water (Agar) | 26      | 1.459   | 0.6                        | 1.444 [34]                | 1.0          |
| Glycerol                | 26      | 0.966   | 0.7                        | 0.958 [35]                | 0.8          |
| Ottawa sand             | 51      | 2.320   | 0.7                        | 2.33 <sup>b</sup> [30–33] | 0.4          |

<sup>a</sup> 95% confidence level.

<sup>b</sup> Sand in general.

Ottawa sand of 1.650 g/cm<sup>3</sup> is close to the published value of 1.615 g/cm<sup>3</sup> [29]. Data on the thermal conductivity of Ottawa sand in air is readily available from several sources [29], although no specific standard exists for this property. However, the thermal diffusivity data on Ottawa sand are not readily found in the literature. Hence, the value obtained is compared to thermal diffusivity of sand in general [30–33]. Literature data [29–35] on thermal conductivity and thermal diffusivity of investigated materials at  $T = 22.5$  °C are given in Table 1.

The low temperature rise of material, obtained by applying low boundary temperature increase, is desirable in terms of minimizing the effects of natural convection, radiation and/or gas evolution. On the other hand, higher excitement reduces the effect of noise and allow for a more precise temperature measurement. In this work, the applied inducement temperature difference was 5 °C, the average temperature being 22.5 °C. Only for measurements on glycerol the excitements were reduced to 3 °C in order to minimize the natural convection effect.

An example of a result for estimation of thermal diffusivity based on the measured temperature response on Agar gel,  $2R = 26$  mm, and the radial heat conduction model in Eqs. (2) and (3) is given in Fig. 3. An excellent fit with a standard deviation of  $1.0 \times 10^{-2}$  °C is obtained.

The higher the value of thermal diffusivity for tested material the higher radius of the cylinder should be used in order to increase the sensitivity of the method, i.e. the sensitivity coefficients  $J_{ia}$ , defined by Eq. (11). In that way higher precision and accuracy can be obtained according to Eq. (16). An example of a result for estimation of thermal diffusivity on Ottawa sand,  $2R = 51$  mm, is given in Fig. 4. A good fit with a standard deviation of  $1.3 \times 10^{-2}$  °C validates the appliance of the heat conduction model used for describing the heat transfer of investigated granular material.

A repeatability analysis was conducted on samples by repeating the measurements 10 times. The mean values of the thermal conductivities and estimated precision at a 95% confidence level are listed in Table 2. Very good agreement was found between the results of the experimental investigation and sources of available data. This finding validates the accuracy of the method and measurement apparatus. It can be concluded that the results of the method evaluation on reference materials indicated an accuracy of 1% and a precision of 0.7% (for 95% confidence).

In order to demonstrate the effect of the thermal inertia of a sample holder, the estimation results on obtained measured responses employing ideal and real boundary temperature increase were compared. An assumption of ideal boundary temperature increase resulted in a systematic error of 7.5% and 9.4% lower value of estimated thermal diffusivity based on measurements done on Ottawa sand ( $2R = 51$  mm) and Agar gel ( $2R = 26$  mm), respectively.

#### 4.2.1. Experimental appliance to various molds and different boundary excitement techniques

The developed method makes possible to estimate the thermal diffusivity based on measured time-dependant boundary and

'axial' temperature increase in one-dimensional heat transfer. This extends the experimental appliance to different variety of molds, e.g. thicker (larger) and even plastic tubes. It should be noted that to obtain accuracy and precision the boundary temperature increase should be as fast as possible, and the cylinder radius as large as possible. The optimal experimental design should be investigated by numerical simulations (developed Matlab '.m' GUI sub-program also freely available upon request). Furthermore, different boundary excitement techniques can be used assuring that the cylinder surface heat transfer is homogenously applied. For example, the thermal diffusivity of Ottawa sand was additionally estimated by experimental method using only one temperature bath. After the temperature stabilization of a material in a temperature bath at  $T = 20$  °C the temperature on thermostat is set to 25 °C and thus provided a bath temperature heating ramp of about 1.1 K/min. The comparison of results for estimated thermal diffusivity obtained by using experimental method with two baths (described in Section 3.2) and one bath with the heating ramp indicated deviation of 1%. This deviation is slightly above the precision of the measurement, Table 2.

## 5. Conclusion

An improved experimental technique and a numerical approach for thermal diffusivity estimation are presented for better accuracy and precision on materials which require a sample holder. The results of the method and used apparatus evaluation on reference materials indicated an accuracy of 1% and a precision of 0.7% (for 95% confidence). The higher the value of thermal diffusivity for tested material the higher radius of the cylinder should be used in order to increase precision and accuracy of the method.

The developed method extends the experimental appliance to different variety of molds (e.g. thicker (larger) and even plastic tubes) and to different boundary excitement techniques (e.g. use of only one temperature bath with a heating ramp). The built GUI enables even the first time users of Matlab with no programming skills to use the presented numerical method for inverse thermal diffusivity estimation.

Results on fitting the temperature response by the numerical method provide a tool for investigating the appliance of the heat conduction model used for describing the complex heat transfer in wet and porous materials.

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