Uncertainty Analysis of Model for Cuboid Shape Samples Applied on Thermopysical Measurement of Stone Porous Material

[1]V. Boháč, [2]P. Dieška, [1]Ľ. Kubičár, [1]V.Vretenár

[1]Institute of Physics, Slovak Academy of Sciences, Bratislava, Slovakia,
[2]Department of Physics, FEI - STU, Ilkovičova 3, Bratislava, Slovakia
Email: bohac@savba.sk

Abstract. *A new model for the sample of square cross section having cuboids geometry including the effect of heat loss from the surface of the sample was tested using the theory of sensitivity coefficients. Theoretical calculation of model uncertainty is presented. Results of the uncertainty analysis set out range of experimental conditions under which the model is valid and the uncertainty of estimated parameters is optimal. The sensitivity coefficients serve to derive the formulas for calculation of analytical error propagation for non-stochastic dynamic measurements. Result shows limitations relating to a restricted range of validity. The analysis improve the accuracy of measurements. Model was used for data evaluation of thermophysical parameters measured on the sandstone from locality Pravčická brána. Evaluation procedure was tested on experimental data measured under different experimental conditions. In conclusion, the discussion of the experiment optimization is presented.*

Keywords:Pulse Transient Method, Sensitivity Coefficient Analysis, Thermophysical Property

1. Introduction

Thermophysical parameters, e.g. thermal diffusivity, specific heat and thermal conductivity reflect thermodynamical state of the material structure. This state changes with the consideration of the atomic structure arrangement, arrangement of crystalline components and consideration on material defects created in time that are responsible for further development of structure change or degradation. Thermodynamical state should be a measure of the quality, stability and durability of material in regarding to industrial use. Process of material deterioration of stones or their state and quality comparing its basic or initial state should be monitored by continuous measurements (monitoring) of thermophysical properties too. Pulse Transient Technique that belongs to a group of dynamic methods for measurement of thermophysical parameters can be used for the quality control in material testing.

The problems connected with deficiency in a large amount of testing material cause some problems in data evaluation as an ideal model assumes already infinitively large specimen or cylinders. The finite geometry of the specimen cause additional effects that harm the accuracy of measurement. The particular contributions to uncertainty comes form additional effects caused by differences in ideal and real sample geometry. The main effects are the heat losses from the sample surface; the heat capacity of the heat source and technically the heat pulse of certain duration that is not ideal Dirac function. In this paper we discuss the given problem for cuboid shape samples.

2. Subject and Methods

Principle of the Pulse transient method

In principle the planar heat source generates the heat pulse. A temperature response to the heat pulse is recorded by thermocouple placed at distance h from the heat source (Fig. 1). In an ideal case of unlimited sample size the temperature response is described by function

$$T(h,t) = \frac{2 \cdot Q}{c\rho\sqrt{\kappa}} \left[\sqrt{t} \cdot i\Phi^* \left(\frac{h}{2\sqrt{\kappa t}} \right) - \sqrt{t - t_0} \cdot i\Phi^* \left(\frac{h}{2\sqrt{\kappa (t - t_0)}} \right) \right] \quad (1)$$

For the maximum of the temperature response there were derived simple formulas for evaluation $\kappa = h^2/2t_m \cdot f_\kappa$, $c = Q/\sqrt{2\pi e}\rho h T_m \cdot f_c$ and $\lambda = \kappa \cdot c \cdot \rho$ [1]. This we call one point evaluation procedure. Description of used variables one can find in [1, 3 and 4].

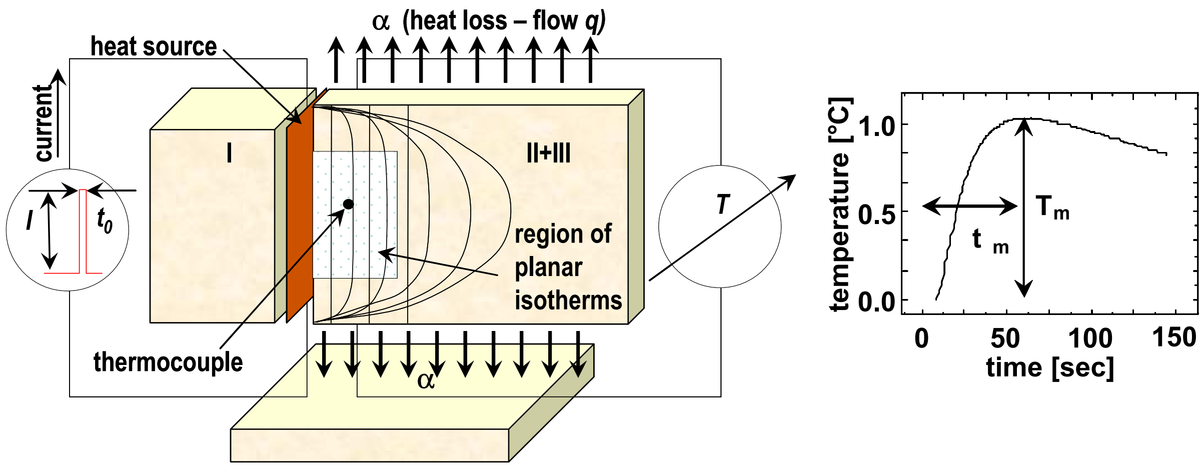

Fig. 1. Wiring diagram and the sample set. In between three parts of a sample set a planar heat source and thermocouple are inserted. In the sample cut it is illustrated a problem with deformed temperature equipotentials. Non influenced temperature equipotentials are planar and are depicted in a white region for shorter times of measurements or lower thicknesses. They are deformed later in time, especially for bigger thicknesses of materials.An example of temperature response in on the right.

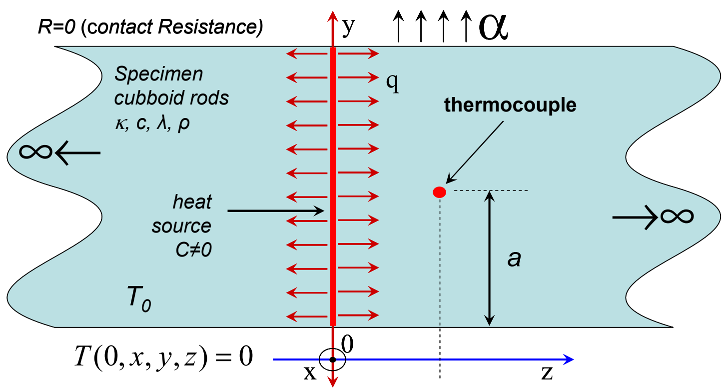

Fig. 2. Initial and boundary conditions for the model. κ, c, λ and ρ are thermaldiffusivity, specific heat, thermal conductivity and density of material. T_0 is stabilizes initial temperature of sample.

Mechanism of deformation of planar temperature equipotentials by heat losses effect is drawn on Fig. 1. This effect is evident for specimens having bigger thicknesses or using longer times of the measurements. For this case it was derived a model for cuboid geometry that accounting a heat transfer coefficient α. The solution of temperature response under initial and boundary conditions drawn in Fig. 2. for basic heat equation is the following:

$$T(t,x,y,z) = T_0 \frac{w}{2} \sum_{n=1}^{\infty} \sum_{m=1}^{\infty} \frac{b_n b_m}{v_{nm}} F(u, v_{nm}) \varphi_n \left(\frac{x}{a}\right) \varphi_m \left(\frac{y}{a}\right) \quad (2)$$

where variables are

$\varphi_n(s) = \sqrt{\frac{2\beta}{\beta + \sin^2 \mu_n}} \cos(\mu_n s)$, $F_n(u,v) = e^{-2uv}\Phi^*(u-v) - e^{2uv}\Phi^*(u+v)$, $T_0 = \frac{qa}{\lambda}$, $\beta = \frac{a\alpha}{\lambda}$,

$u = \frac{z}{\sqrt{\lambda kt}}$, $w = \frac{\sqrt{kt}}{a}$, $v_{nm} = w\sqrt{\mu_n^2 + \mu_m^n}$, $b_n = \varphi_n(0)\frac{\sin(\mu_n)}{\mu_n}$ and T temperature, t time, z axial space coordinate, x,y transversal space coordinates, $2a$ transversal size of the sample, q heat flow density at source, λ thermal conductivity, k thermal diffusivity, α heat transfer coefficient for sample – ambient interface, $\Phi^*(u)$ is the complementary error function, μ_n are the roots of equation $\beta\cos\mu - \mu\sin\mu = 0$.

Theoretical calculation of model uncertainty

This type of uncertainty arising from different sources and includes errors in the data, parameter estimation procedure and model structures. They are all propagated through the model for uncertainty analysis and their relative importance is quantified via sensitivity analysis. This type of uncertainty should be supposed as systematic error. The uncertainty analysis was developed in respect with experimental data set $\{t_n, T_n\}_{n=1}^N$, where N is the number of measurements. Model temperature function $T_{model} = f(t,a,b)$ represents temperature response in time that depend on constants $b = \{b_j\}_{j=1}^{N_b}$ in model are determined by different independent measurements and dependent random variables represented by set of free parameters $a = \{a_i\}_{i=1}^{N_a}$ that are evaluated by fitting procedure. We used least square optimization to search for $\min\left\{\sum_{n=1}^N [T_n - f_n(a,b)]^2\right\}$, where $f_n(a,b) = f(t_n,a,b)$, t_n is deterministic parameter, $b, \{T_n\}_{n=1}^N$ are independent random variables. The least square optimization gives the system of non-linear equations that based on sensitivity coefficients (Fig. 3.).

$$\sum_{n-1}^{N}(T_n - f_n)\frac{\partial f_n}{\partial a_i} = 0, \qquad i = 1,2, \ldots N_a \tag{3}$$

Uncertainty of the model is given by formula $u(a_i)^2 \sim \left\langle (a_i - \langle a_i \rangle)^2 \right\rangle = \left\langle (\Delta a_i)^2 \right\rangle \sim \left\langle (da_i)^2 \right\rangle$.

Differentiating equation (3) we obtain system of equations (correlation matrix)

$$\sum_{i=1}^{N_a} A_{ki} da_i = \sum_{n=1}^{N} dT_n \frac{\partial f_n}{\partial a_k} + \sum_{j=1}^{N_b} B_{kj} db_j, \qquad k = 1,2, \ldots N_a \tag{4}$$

for which we estimated uncertainty contribution of any particular measurement of involved parameters like T or b. Then for relative uncertainties we can write the equation

$$u_r(a_k)^2 = C_{kT}^2 \frac{u(T)^2}{a_k^2} + \sum_{j=1}^{N_b} v_{kj}^2 u_r(b_j)^2 \tag{5}$$

3. Results

Experimental data measured in RTB1.02 chamber with temperature stability of 0.01 K are ploted in Fig. 4. Data under the air and vacuum atmosphere were obtained in the temperature range from -22 up to 70°C. Temperature responses were measured for the pulse width of 3 and 6 seconds. The total time of the recording was up to 370 seconds. Two temperature models were used for data evaluation. First model consider final pulse width and evaluation procedure based on formulas derived for maximum of the temperature response published in several papers [3, 4]. The second one uses fitting procedure and model assuming cuboid samples having squared cross section and heat losses from the sample surface (Eq. 1.). The sandstone specimen set was carved in a form of cuboids having finite length. The dimensions of parts I and III were 50x50x30 part II 50x50x10mm. The volume density was 1738.7 kg m^{-3}. Porosity measured by weighting dry and water saturated specimen was 27.5%.

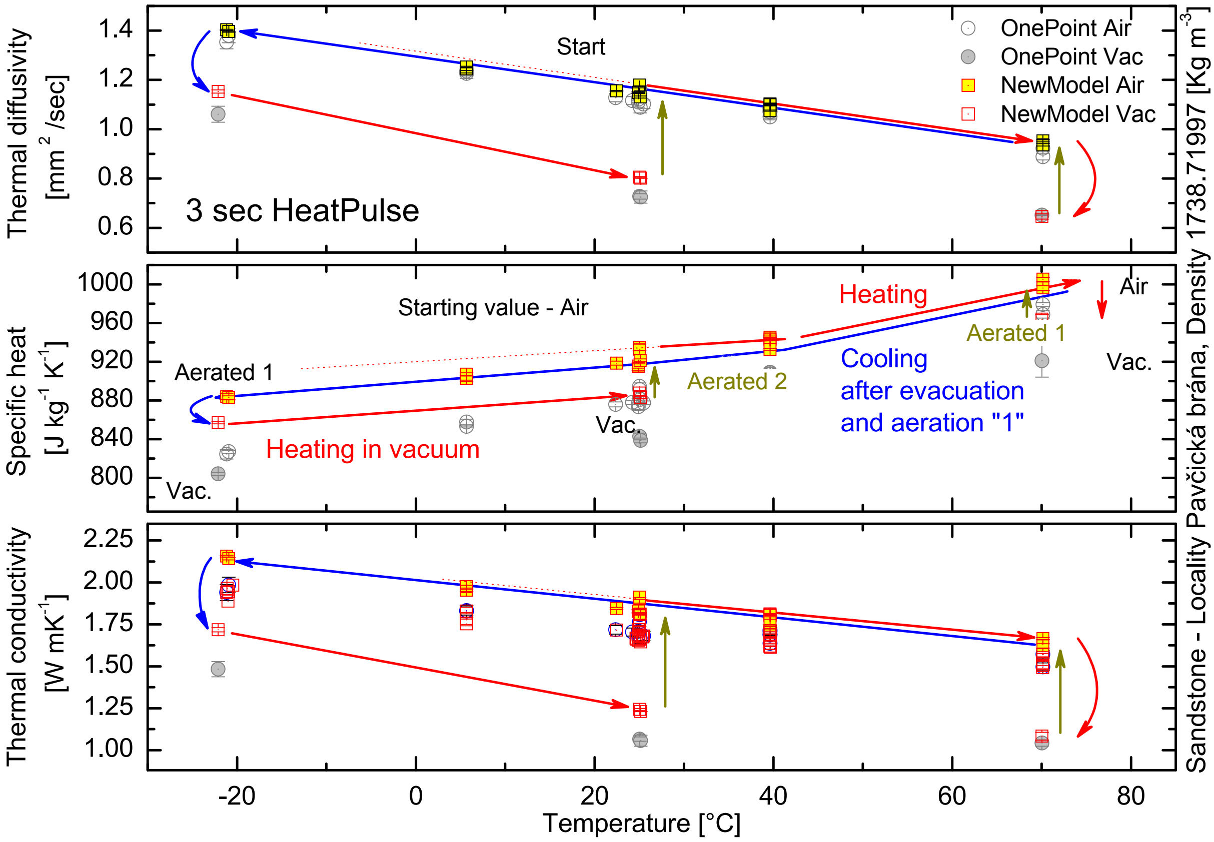

Fig. 3. Thermophysical parameters estimated by model fitting. The temperature history as well as vacuum treatment follows the arrows denoting heating and cooling regime. The shifted temperature dependency of evacuated data follows the measurements performed under the air atmosphere. Annealing and the vacuum treatment causes lowering of all thermophysical parameters due to removing of remanent water from pores and structure of sandstone (compare red and blue arrows of first heatig and cooling).

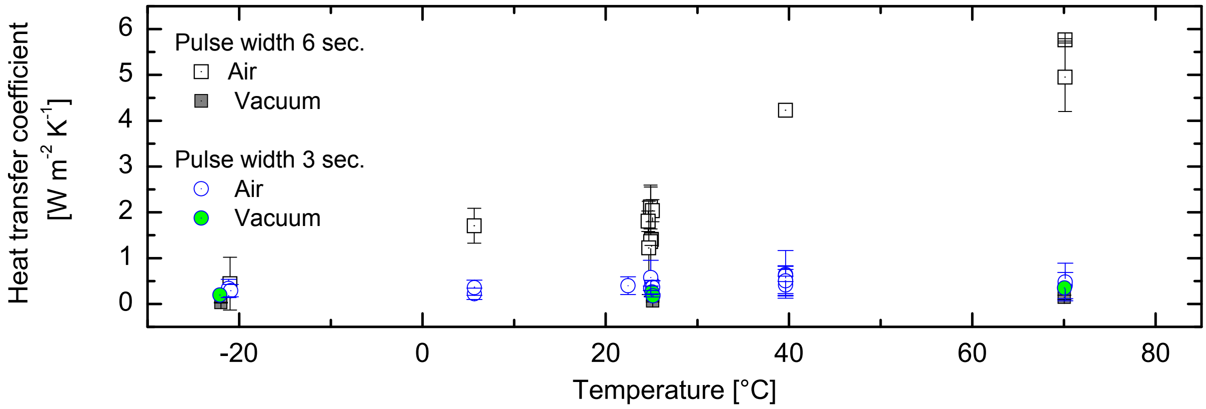

Fig. 4. Heat transfer coefficients calculated by fitting procedure for 3 and 6 second of pulse duration as well as for air and vacuum conditions. The low values are for 3 seconds of pulse duration as well as for data measured under the vacuum. This is the consequence of sensitivity for this parameter only for bigger thicknesses of material or higher times of measurement of temperature response. The positive temperature dependence of this parameter is evident for 6 seconds pulse width. The heat transfer coefficient from the sample surface to the surrounding is temperature dependent.

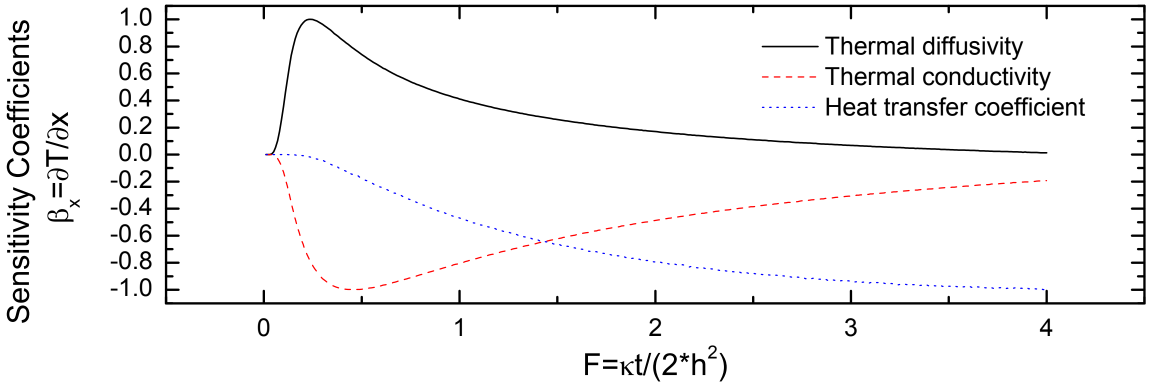

Fig. 5. Normalized sensitivity coefficients derived from temperature function given by Eq. 1. $\beta_p = \partial T/\partial p$ where p denotes free parameter in model, e.g. thermal diffusivity, thermal conductivity and heat transfer coefficient. The normalized sensitivity coefficients in Fig. 5. were calculated for values of thermophysical parameters like those one measured in experimental part ad are give in Fig. 3. Optimized region for data evaluation is up to value of 2*F* in dimensionless time

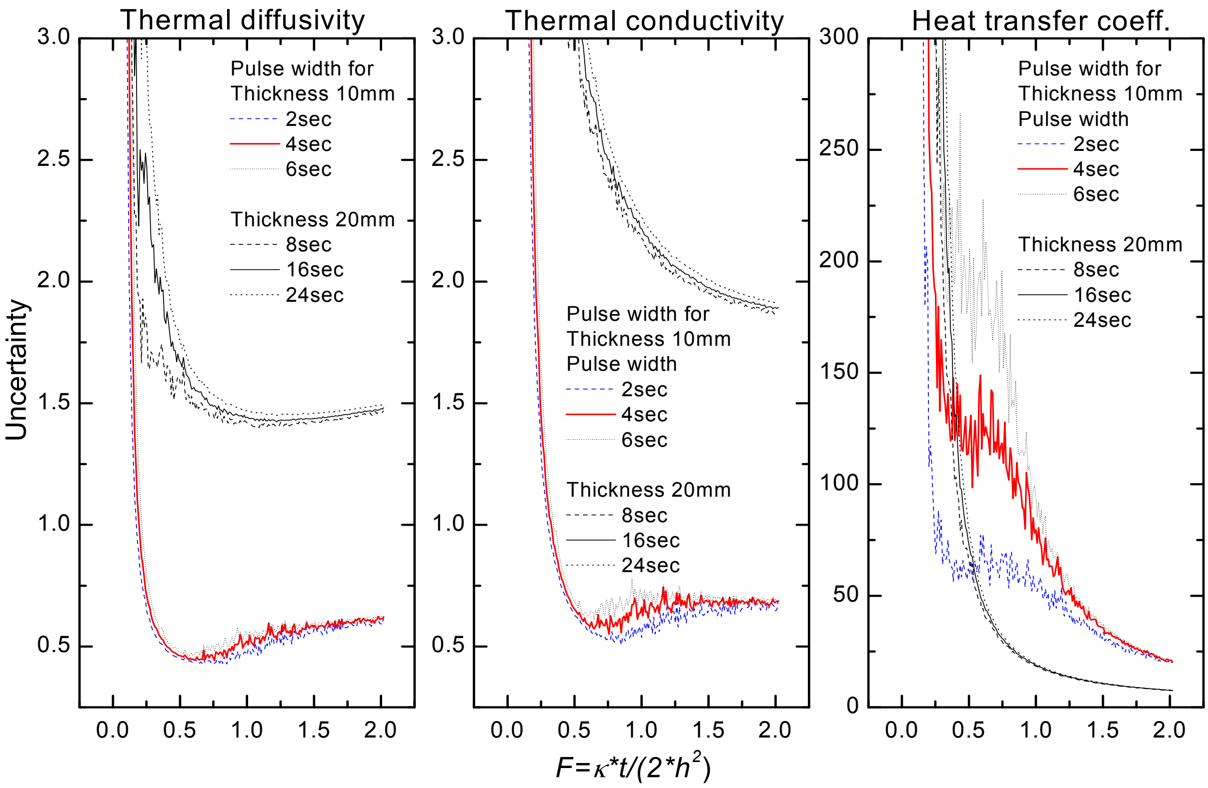

Fig. 6. Uncertainty analysis for sample thickness of 10mm at 2, 4 and 6 seconds of pulse width and thickness 20mm for 8, 16 and 24 seconds of pulse duration. The maximum of temperature response is at 0.5 *F*. This concludes the discussion on the total time of transient record measurement.

Measured thermophysical parameters of porous sandstone depend on quality of material and the moisture content in pores. Temperature dependency of transport parameters (thermal diffusivity and thermal conductivity) are of negative slope, while the specific heat have positive slope in a give temperature range from -22 up to 70°C (Fig. 4). Differences in all parameters after evacuation and aeration were caused by drying process in vacuum as well as at the elevated temperatures. The calculated values of heat transfer coefficients are in Fig. 4.

Analysis of uncertainty (Fig. 6.) was calculated for fixed number of points in evaluation time interval, variable time step and fixed time window that begin at t=0sec. Each data point represents different length of the time interval but the number of points in each interval is the same to preserve statistical weight of the calculation results. Fig.6 illustrates the correlation of uncertainties and the time of the measurement as well as the thickness of the sample. It explains why the heat transfer coefficient is possible to evaluate only from data measured at longer times (6 sec. pulse width data at Fig. 5.). The reason is that the sensitivity for this parameter is higher for longer times of measurements. Heat loss effect starts to influence the measurement at higher times and at higher thicknesses of material. Uncertainty of estimation of the heat transfer coefficient is decreasing with increasing thickness of the sample as well as with increasing time of the measurement. The similar situation we found at uncertainty calculations for thermal diffusivity and thermal conductivity. Their values are decreasing with time and after getting maximum of the temperature response at $0.5F$, e.g. about 42 seconds in real time axes it starts to slightly increase. This means that there is no reason for the increase of the measurement time higher than $1.5 \div 2F$ (280÷370sec. for this sandstone material).

4. Conclusions

The thermophysical properties of sandstone were investigated by pulse transient method for dry state. The data were evaluated by procedures of one point evaluation and by fitting by new model for cuboid samples that accounts heat losses from the sample surface.

The uncertainty analysis of the new model was analyzed in the Fig. 5 and Fig. 6. Data illustrates the sensitivity on the heat transfer coefficient in regarding the time of the measurement as well as geometry of the specimen. This parameter affects the measurement with increasing time of the measurement or at higher thicknesses of the sample. The heat transfer coefficient is not possible to estimate unambiguously for lover times of measurement. The values measured in vacuum are practically the same over all temperature range and also for the data measured for shorter pulse width (3sec.).

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References

[1] Kubičár Ľ, 1990, *Pulse Method of Measuring Basic Thermophysical Parameters*. in Comprehensive Analytical Chemistry Vol. XII Thermal Analysis Part E Ed Svehla G, Amsterdam, Oxford, New York, Tokyo: Elsevier, pp 350.

[2] Beck, J.V.;Arnold, Kenneth J., Parameter *Estimation in Engineering and Science*, ISBN: 9780471061182, Better World Books (Mishawaka, IN, U.S.A.) John Wiley & Sons, 1977

[3] Boháč V., Kubičár Ľ., Vretenár V., *Investigation of heat transport in porous materials by pulse transient method*, HIGH TEMP-HIGH PRESS. 2003/2004, Vol. 35/36, pp 67-74

[4] Boháč V, Dieška P, Kubičár Ľ. *The Heat Loss Effect at the Measurements by Transient Pulse Method*, MEASUREMENT SCIENCE REVIEW, Volume 7, Section 3, No. 2, 2007 pp 24-27