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Thermophysical Parameters of Carbonate Rock estimated by Slab Model Developed for Pulse Transient Technique

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The slab model has been used for parameter estimation from the measurement performed by the Pulse Transient Technique. The estimation of thermophysical parameters was done on carbonate rock sample. In addition to basic thermal parameter for example thermal diffusivity, thermal conductivity and specific heat capacity, the slab model takes into account the heat capacity of the heat source, as well as the heat transfer coefficient between the heat source and the sample. The thermophysical parameters were estimated for the case when thermal conductive paste as a heat contact agent was not used for the measurements. The paste contains silicone oil that penetrates into the porous stone material and thus causes irreversible changes of properties during the measurement so we decided not to use it. The experiment was done with dry contacts at the interfaces that causes the disturbances in the measurement that have been introduced and resolved using the slab model. Uncertainty analysis of the estimation of the parameters by the slab model was done for real measurements conducted on the carbonate rock. In this paper we analyzed the quality of the temperature response fit in dependency on the originally free fitted parameters of the heat transfer coefficient and the heat capacity of the heat source that was replaced by constant values in two steps. The heat capacity of the heat source was calculated from the material properties, e.g., the nickel and Kapton. The fit results obtained by the slab model were compared with the data obtained by the ideal and cuboid model. The analysis of the sensitivity coefficients and calculated uncertainties of estimated parameters with the slab model help to improve the accuracy of parameter estimation.

Keywords: Pulse Transient Technique, sensitivity coefficient, uncertainty analysis, thermal property, slab and cuboid model

1. INTRODUCTION

Today's science is more focused on developing simple and efficient models for reading the thermal properties of materials, especially in the field of non-polluting energy sources. The motivation of research will provide more use of the energy-efficient building in performance to improve the thermal comfort of its occupants while least affecting the environment and surroundings.

Therefore in the field of geoscience, the thermal properties of building construction materials or rocks depending upon the composition of minerals, rock type, geometry, porosity, location, granularity, etc. [1], [2], [3] plays a major role in defining its thermal behavior with the surroundings. Thermal properties such as thermal conductivity, thermal diffusivity, specific heat capacity, heat transfer coefficient in between heat source and specimen, and heat capacity of heat source are major parameters to focus on in this article.

Scientific laboratories and industries have already developed numerous methods for the study of the thermal parameters of materials to probe heat transport via material structures, predominantly they can be separated into two groups namely steady-state and transient methods. Transient methods [4], [5], [6], [7], [8], [9], [10] compared to the steady-state techniques, require a significantly shorter time for the measurement, so the data obtained corresponds to the thermodynamic state in the real world. The experimental problems attached to the specimen geometry are induced sometimes by the limiting size of the tested material, atomic structure arrangement, decoration of crystalline components quality, stability, durability, polishing of specimen surface, and arrangements of devices during experiment setup, noise from the thermocouple.

In paper, we have used the Pulse Transient Technique for the measurement of thermophysical parameters of the carbonate rock sample estimated by the slab model that helped to provide the most reliable and correct data with less than 0.4 % of accuracy when the data was compared with previously published data in literature.

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2. METHODOLOGY AND MODELS

2.1. *Pulse Transient Technique*

The Pulse Transient Technique is based on the principle of generation of the heat pulse by the heat source and recording the thermal response to this heat pulse by the thermocouple that is placed at the distance ,*h* apart from the heat source (Fig.1.). The thickness *h* represents the active part of the specimen represented by the slab. It is inserted in between semi-infinitely large specimen surroundings marked as part I and III in Fig.1. Also in the experiment technically the heat pulse used is not an ideal Dirac function but a heat pulse of limited duration.

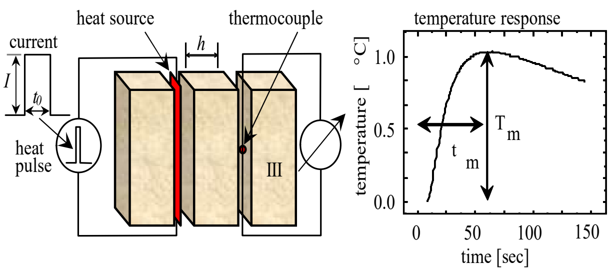

Fig.1. Principle of Pulse Transient Technique and the example of the maximum of thermal response (T_m) is recorded in time (t_m) to the heat pulse on the right [15], [16], [17], [18], [19].

2.2. *Slab model*

The slab model was derived for the sample consisting of the two semi-infinitive parts of the specimen with the infinitely large and thin plane heat source inserted in between them.

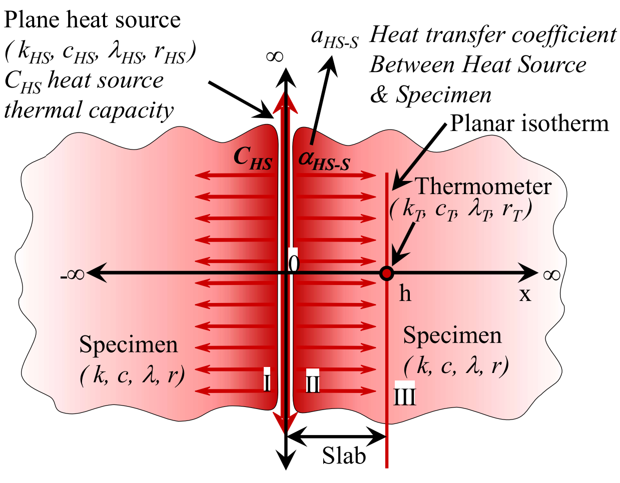

Fig.2. The slab model for infinitely large specimens accounting the heat source heat capacity as well as the heat transfer coefficient in between the heat source and specimen body is drowned with the experimental parameters and geometry of arrangement [15], [16], [17], [18], [19].

The model accounts for the heat contact problems that are represented by the heat contact resistance realized in a form of the heat transfer coefficient and by the heat capacity of the heat source. The solution of heat transfer equation for the given initial and boundary conditions is the temperature function that describes the thermal response to the step- wise heat pulse H(t).q(t) for the times t>0 in equations (1), (2), (3), (4).

$$T(0,x) = T_S = 0 \qquad (1)$$

$$-\lambda \left.\frac{\partial T}{\partial x}\right|_{x=o} = \alpha\left(T_S - T|_{x=0}\right) \qquad (2)$$

$$qH(t) = C\frac{\partial T_S}{\partial t} + \alpha\left(T_S - T|x=0\right) \qquad (3)$$

$$T(t,\infty) = 0 \qquad (4)$$

The temperature function is given by the following equations (5), (6), (7).

$$T(t,x) = T_1(t,x) + \Delta T(t,x) \qquad (5)$$

$$T_1(t,x) = T_0 e^{-u^2}\left[\frac{1}{\sqrt{\pi}u} - w(iu)\right]H(t) \qquad (6)$$

Where

$$\Delta T(t,x) = -\frac{T_0}{2a\xi}e^{-u^2}\left\{w(iu) + \frac{z_1^2 w\left[i(u - z_2\frac{\xi}{u})\right] - z_2^2 w\left[i(u - z_1\frac{\xi}{u})\right]}{z_1 - z_2}\right\}H(t) \qquad (7)$$

The meaning of the variables is the following:

$$u = \frac{x}{2\sqrt{kt}}\ ;\ \xi = \frac{x\alpha}{2\lambda}\ ;\ z_{1,2} = \frac{-1 \pm \sqrt{1-4a}}{2}\ ;$$

$$a = \frac{\lambda^2}{kC\alpha}\ ;\ T_0 = \frac{qx}{\lambda}$$

and $w(z) = e^{-z^2}erfc(-iz)$ is the Fadeeva function. erfc() is the complementary error function of a complex argument, T_1 is the ideal temperature function, ΔT is the modified part of the temperature function accounting parameters α and C, where $2C$ is the heat capacity per unit area of the heat source ($J.K^{-1} m^{-2}$) (only one half of it is accounted by the slab model in positive x-direction because of the model geometry for semi-infinitely large specimen halves), α is the heat transfer coefficient for heat source - sample interface ($W.m^{-2}.K^{-1}$), T_S is the temperature of the heat source, $H(t)$ is the heaviside step function, or the unit step function also denoted by 1 or θ. (H=0 for t<0 and H=1 for t≥0, so for t=0 the value $H(0)$=1), $2q$ is the power per unit area of the source (the generated heat is split into two halves in the model due to symmetrical geometry regarding from the heat source), t is time (s), x cartesian coordinate (equal to h in Fig.2.) (m), λ is the thermal conductivity ($W.m^{-1}.K^{-1}$), k is the thermal diffusivity ($mm^2.s^{-1}$).

Equation (5) describes the step-wise function, so the pulse regime of the measurement is evaluated from two subsequent stepwise functions shifted on the duration of the heating pulse t_0.

$$T_{pulse}(t,x) = T(t,x) - T(t - t_0, x) \quad (8)$$

2.3. Data analysis of uncertainty and sensitivity coefficients

Usually, the shape of the specimen is cylindrical or cuboid form. The finite geometry of the specimen caused additional effect and errors of different origin specified in GUM and JCGM [11], [12], which harms the accuracy of the measurement. The main effects are the heat transfer coefficient between the heat source and specimen and the heat capacity of the heat source.

The slab model used in this paper is accounting for these two effects by using the calculation and analysis of the sensitivity coefficients that are serving for the calculation of the uncertainties of free parameters [13], [14], [15] see Fig.5. and Fig.6. Thus it is compulsory to evaluate the assessment of the results by analysis of the sensitivity coefficient $\beta(a_n)$ and then calculate parameter uncertainties also. Sensitivity coefficient analysis is based on the first derivatives of temperature function *T (t, x)* according to the free parameters ($a_n = \lambda$, κ, α and *C*) in the model [13], [14] (Fig.5., Fig.6.).

$$\beta(a_n) = a_n \frac{dT(t,x)}{da_n} \quad (9)$$

Then for relative uncertainties (U_r) for the parameters can be written in form equation (10) for which the definition of all parameters as well as the mathematical background was described in the paper [14].

$$U_r(a_n)^2 = C_{nT}^2 \frac{u(T)^2}{a_n^2} + \sum_{j=1}^{N_b} v_{nj}^2 u_r(b_j)^2 \quad (10)$$

2.4. Data estimation procedure

Simple formulas for evaluation based on the maximum of the temperature response have been derived from the ideal model. It is so called one point evaluation procedure, where the correction factors f_κ and f_c are described in [15], [16].

$$\kappa = h^2 / 2t_m \cdot f_\kappa \quad (11)$$

$$c = Q / \sqrt{2\pi e} \rho h T_m \cdot f_c \quad (12)$$

$$\lambda = \kappa \cdot c \cdot \rho \quad (13)$$

The meanings of the next variables are the same as for the slab model. Data values calculated from the one-point procedure (Refer to equations (11), (12) and (13)) were used as the initial guess values for the fitting procedures based on the slab model.

The cuboid model that has been already published, it was developed for accounting heat losses from the sample free surface in a radial direction. The value of the heat transfer coefficient from the sample surface to the surrounding evaluated by the cuboid model is low and thus we expect no or little effect on measurement using the slab model [15], [16], [17], [18], [19]. Using the slab model it is not possible to estimate heat transfer coefficient and heat source heat capacity unambiguously because of their low sensitivity coefficients and thus high relative uncertainty (Fig.3. and Fig.4.)Therefore, in the slab model due to high uncertainty values they are substituted by constant values.

3. EXPERIMENT

Experimental data were measured in the RTB1.02 chamber. The used sample belongs to the class of Carbonate rock. It was carved in a form of cuboids having a finite length. with finite cuboid geometry having the base dimension 50 mm x 50 mm for all parts and the thickness of part I and III were 30 mm, while part II was 14.62 mm. The volume density was 2812.20 kg.m^{-3}. The plane heat source was etched of Ni foil and insulated by Kapton foil. Data from the samples were tested under the room temperature ranges from 18 to 25°C.

4. RESULTS

For the data evaluation by fitting procedure, two discussed models with temperature response were used, the slab model (Fig.3. up) and the cuboid model (Fig.3. bottom). In Fig.3. both the theoretical models are compared with the experimental curve (black ink)

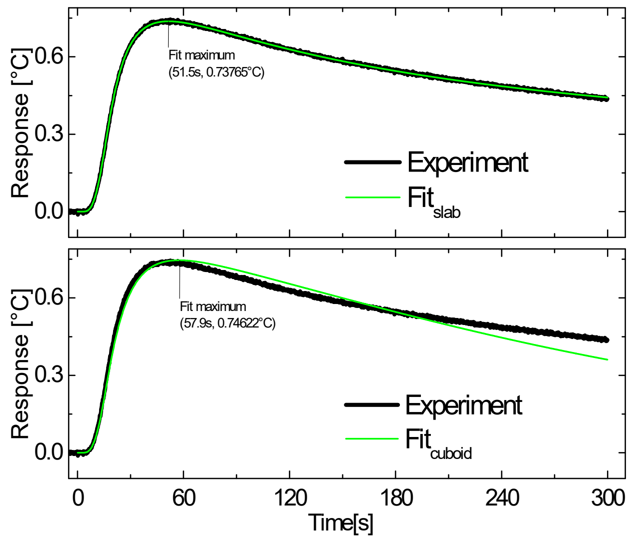

Fig.3. Fitted curve of Temperature response for time window 0-300 seconds using the slab model (top) compared with the cuboid model (bottom) for 10 s pulse width.

The maximum of the temperature response was found at 56.6 seconds as well as for the fit by slab model. The cuboid model shifted maximum to a higher value at 57.9 seconds that is wrong As the sensitivity coefficient has the extreme values at the times below the maximum point, one has to use the time interval from the calculations of the uncertainties that are getting acceptable values after the maximum of the temperature response is reached (Fig.5., Fig.6.).

The differences in the theoretical curves obtained by fitting of the experimentally measured thermal responses on the Carbonate sample set for the 10 seconds heat pulse duration using the slab model (top) and cuboid model (bottom) are clear from Fig.3. The total time for the recording of the temperature response was 300 s.

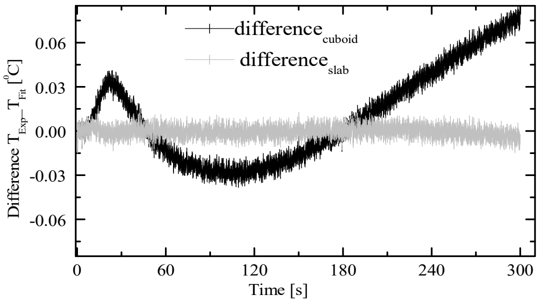

Fig.4. Comparison of residual plots of fitted curves from Fig.4. obtained using the slab and cuboid models. The slab model fits the data within the background noise on a scale of ±0.005°C.

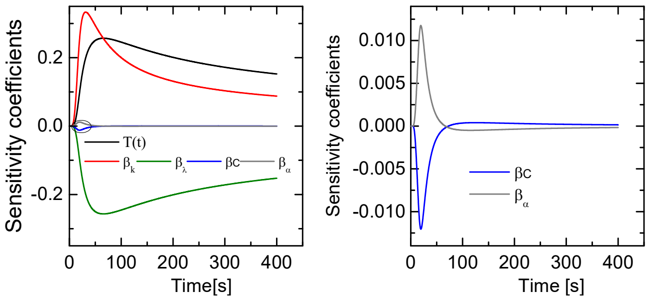

Fig.5. The temperature response T(t) and sensitivity coefficients $\beta(a_n)$ (where a_n=*k*, *λ*, *C* and *α*) calculated for 14.62 mm sample thickness and pulse width 10 s on the left side. The sensitivity coefficients for the heat capacity of source heat (β_C) and heat transfer coefficient (β_α) in between the heat source and specimen are marked by ellipse on the left and re-plotted in enlarged scale on the right as they are lower by three orders of magnitude comparing the β_λ, β_κ.

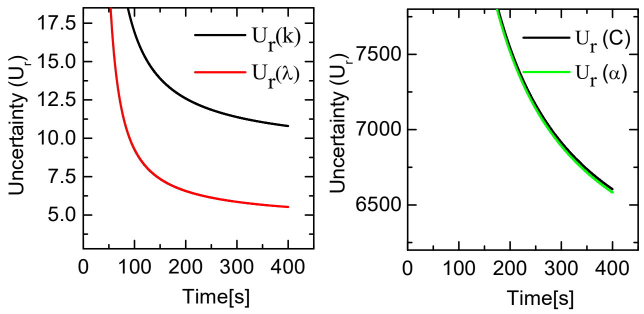

Fig.6. Time dependency of parameters uncertainty for the thermal diffusivity $U_r(\kappa)$, thermal conductivity $U_r(\lambda)$ (left), and the heat capacity of the heat source $U_r(C)$ and heat transfer coefficient $U_r(\alpha)$ (right) for the heat pulse duration of 10 seconds.

The fit quality of the slab model is represented by the difference graph in Fig.4. The residual plot was calculated by subtraction of the theoretically calculated thermal response by the slab or cuboid model from the experimentally measured thermal response.

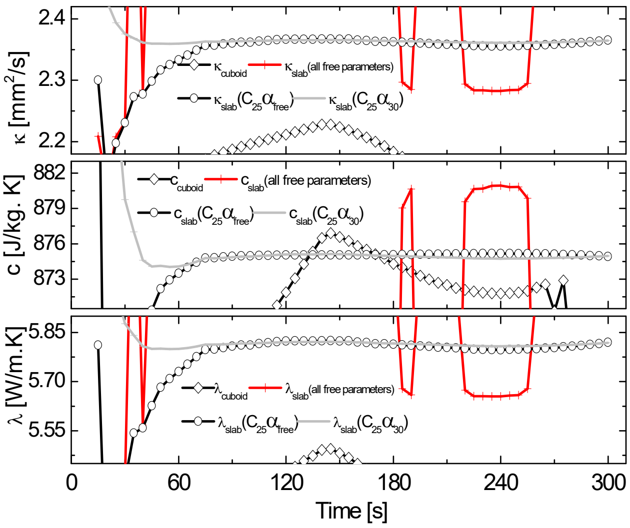

Fig.7. The window interval analysis of slab and cuboid model (rhombus) for thermal diffusivity (k), thermal conductivity (λ), and specific heat capacity (c). Analysis for slab model is given for fitting leaving all four parameters as free (+, red), then with the *C* parameter as constant (hollow circle), the third step of evaluation (grey line).

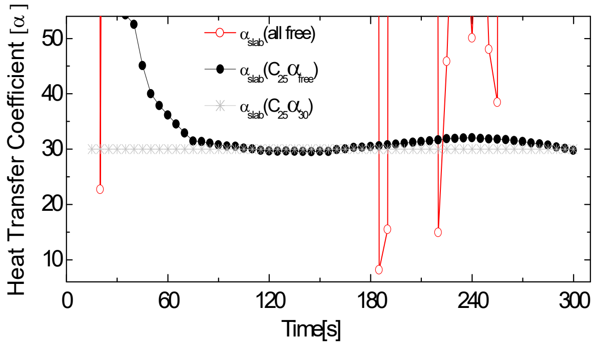

Fig.8. Window interval analysis of slab model for estimation of heat transfer coefficient in between the heat source and specimen calculated by three variations of fitting procedure depending on the variability and substitution of the free parameters. Heat transfer coefficient (α) values for the fitting with all free parameters are out of a given scale because of high uncertainty (open circles). The plateau with minimum scattering (black circles) represents an average 30 W.K^{-1}.m^{-2} (asterisk) for window times from 0-105 up to 0-140 s.

For the assessment of model reliability and the values of thermophysical parameters for the carbonate rock sample evaluated by the slab model, we used the window interval analysis applying three variations of the fitting procedure. The window interval analysis is based on fitting data in different time window intervals ranging - from 0-15, 0-20, 0-25 s up to 0-300 s with the time increment step 5 (Fig7.,

Fig.8., Fig.9.). In the first level of analysis, we set all 4 parameters in the slab model as free for the fitting (Fig.7., red ink data). In the second level, we replace the parameter C (free heat capacity of heat source) with the constant value that was calculated from the real parameters of construction materials that are used for the heat source construction (Fig.7., black hollow circle). The value of the calculated heat capacity (thermal capacity) per unit area is $C = 25\ J.K^{-1}.m^{-2}$.

From the results of this window analysis for the heat transfer coefficient from the second step, we calculated the average value $30\ W.K^{-1}.m^{-2}$ for the desired time window interval and used it as a constant in the third and final level of analysis. In the final level of parameter estimation both the parameters are kept as constant (C, α) (Fig.7., gray ink)

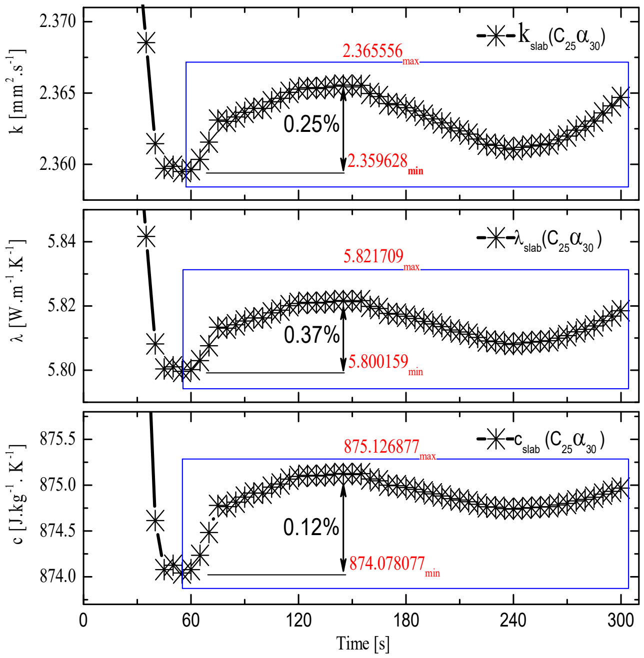

Fig.9. Sinusoidal form of window interval analysis re-plotted only for fitting using constant values of C and α with marked differences from maximum to minimum value for thermal diffusivity (κ), thermal conductivity (λ), and Specific heat Capacity (c). The difference value in percentage is given at the line drawn with arrows.

Fig.8. clearly shows all the three steps of evaluation done by the slab model for the heat transfer coefficient with time. The average value of α as a constant number comes from the window time interval 0-105 s up to 0-140 s.From Fig.8. and Fig.9., based on the final analysis (third step) we can see that the end time of the fitting interval has to exceed the maximum of the temperature response. Therefore, data reliability interval represents the fitting intervals where the end of interval time was in the range from 0-105 s up to 0-140 s which shows the least scattering of data with the highest stability of values. The values of thermophysical parameters for carbonate rock sample evaluated by the slab, cuboid and one-point evaluation procedure are marked in Table 1.

Table 1. Thermophysical data calculated by three different models.

Models	**Thermal diffusivity [mm^2.s^{-1}]**	**Thermal conductivity [W.m^{-1}.K^{-1}]**	**specific heat [J.kg^{-1}.K^{-1}]**
One-point	2.05	5.04	876.43
Cuboid	2.14	5.20	865.54
Slab	2.36	5.82	875.07

5. DISCUSSION & CONCLUSIONS

The uncertainty analysis has been done for Carbonate rock sample by using Pulse transient method at room temperatures. Two models were used to estimate the parameters by fitting procedure. The thermophysical parameters obtained using the slab and cuboid model fits were compared. The parameters estimated by slab model are higher in comparison with the cuboid model. The results show that contact parameters C and α are affecting the measurements by shifting the onset of the temperature response to higher times (Fig.3., Fig.4. and Fig.5.).

The residual plot obtained from the experimental thermal response subtracted by the slab model response represents thermal noise scattering around the zero. The residual plot from the cuboid model curve is of sinusoidal shape (Fig.4.).

The scattering of the thermophysical parameters obtained by window interval analysis is used for the statistical analysis to study the measurement uncertainty. The assessment of model reliability for the time window interval analysis was done in three steps using a fitting procedure of parameter estimation.

The analysis facilitates to estimate the accuracy of the parameters by using the sensitivity coefficients theory and uncertainty analysis. The slab model was successful in suppressing the high uncertainties of data by replacing the values of heat capacity of the heat source and the heat transfer coefficient with the constants which are otherwise supposed as the disturbing parameters described in previous papers [15], [16].

The thermophysical data reliability proves from the Fig.9. when the difference in between the maximum and minimum values within the marked range decreased to acceptable values that were observed below 0.4 %. The validity of the used methodology for the estimation of the parameters was confirmed by measurement on PMMA laboratory standard material [17], [18], [19].

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