HEAT OF HYDRATION BY INVERSE METHOD

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INTRODUCTION

Numerous laboratory techniques have been developed for determination of the heat of hydration of cement based materials, ranging from sophisticated (micro)calorimeters to (semi-)adiabatic temperature measurements at the centre of a large insulated block of representative sample. Determining the heat of hydration of cement material will enable, in practice, the realistic simulation of temperature distribution in material and easier planning of curing in the early stage of hydration with the purpose of achieving better durability and functionality of placed material.

METHODOLOGY

By solving a system of partial differential equations by a method of finite difference, beside material temperature distribution, a sensitivity of the developed method regarding the influential parameters was estimated and a measurement uncertainty analyses performed. For estimating the heat generation two approaches were adopted: *Conjugate Gradient Method with Adjoint Problem for function estimation* and *Levenberg-Marquardt Method for parameter estimation* of assumed function. A computer code for solving the inverse problem is written in MATLAB®. This paper examines the hydration of commercial aluminate cement (AC) ISTRA 40 (producer: Istra Cement, Pula; Croatia)

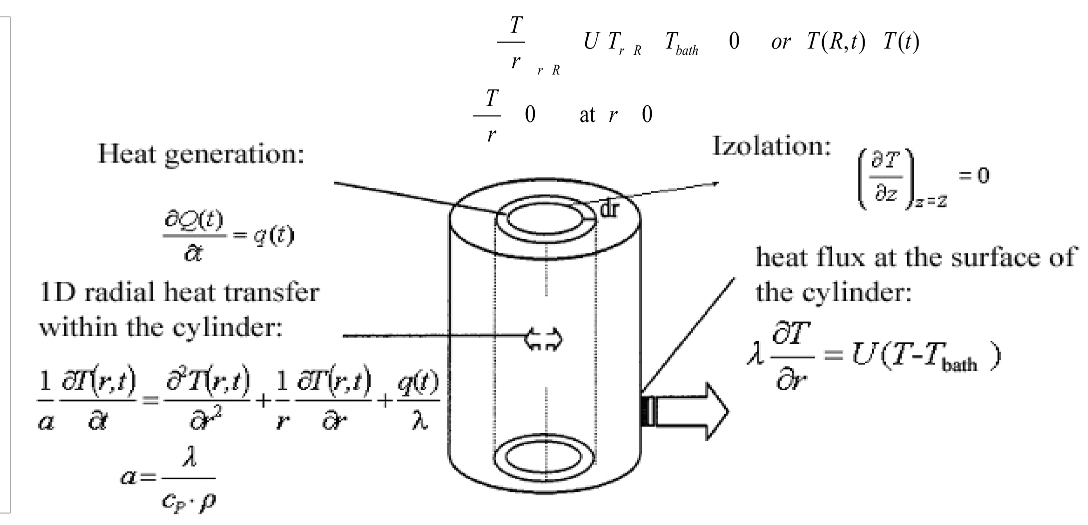


Fig. 1. Mathematical formulation of the transient heat conduction with internal volumetric heat source in infinite cylinder.

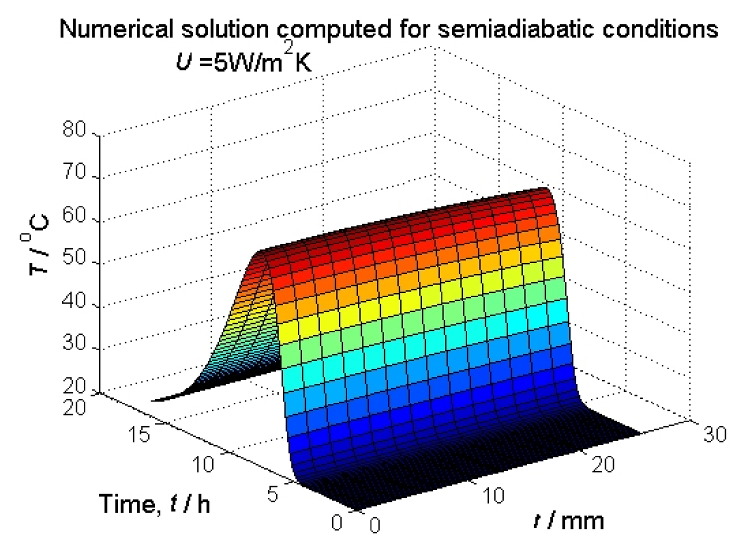


Fig 2. FD Numerical solution for semiadiabatic calorimeter.

Tab. 1. Impact of thermocouple measuring uncertainty and baseline accuracy to the estimated heats. *The reported uncertainty is calculated using a coverage factor of 2 (level of confidence 95%).

Inverse isothermal calorimeter			
Uncertainty*			Maximal relative error (Q = 250 J/g), U_Q/Q
TC, U_T, °C	Heat power, U_q, mW/cm³	Cumulative heat, (t=12h, Δt=0.012h), U_Q, J/g	
± 0.2	± 1.17	± 0.979	0.39 %
± 0.5	± 2.93	± 2.45	0.98 %
Inverse semi-adiabatic calorimeter			
Uncertainty*			Maximal relative error (Q = 300 J/g), U_Q/Q
TC, U_T, °C	Heat power, U_q, mW/cm³	Cumulative heat, (t=12h, Δt=0.012h), U_Q, J/g	
± 2.0	± 0.42	± 0.35	0.12 %
± 0.5	± 0.10	± 0.09	0.03 %

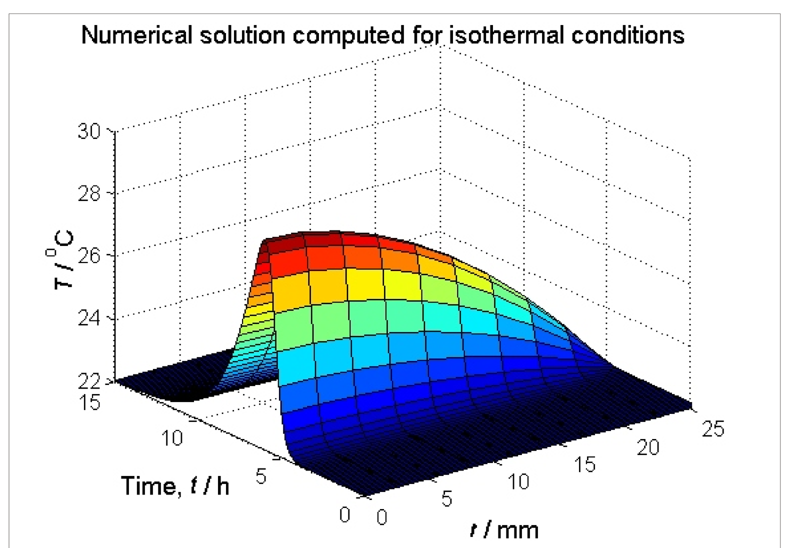


Fig 3. FD Numerical solution for isothermal calorimeter.

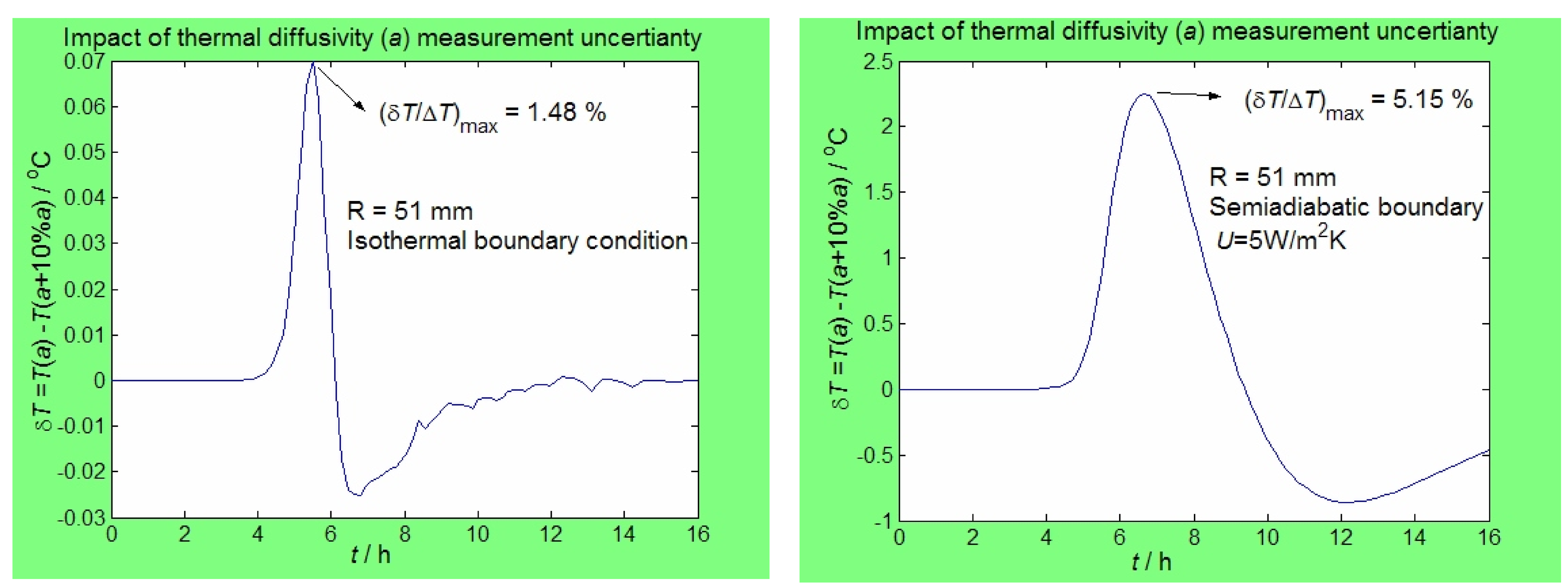
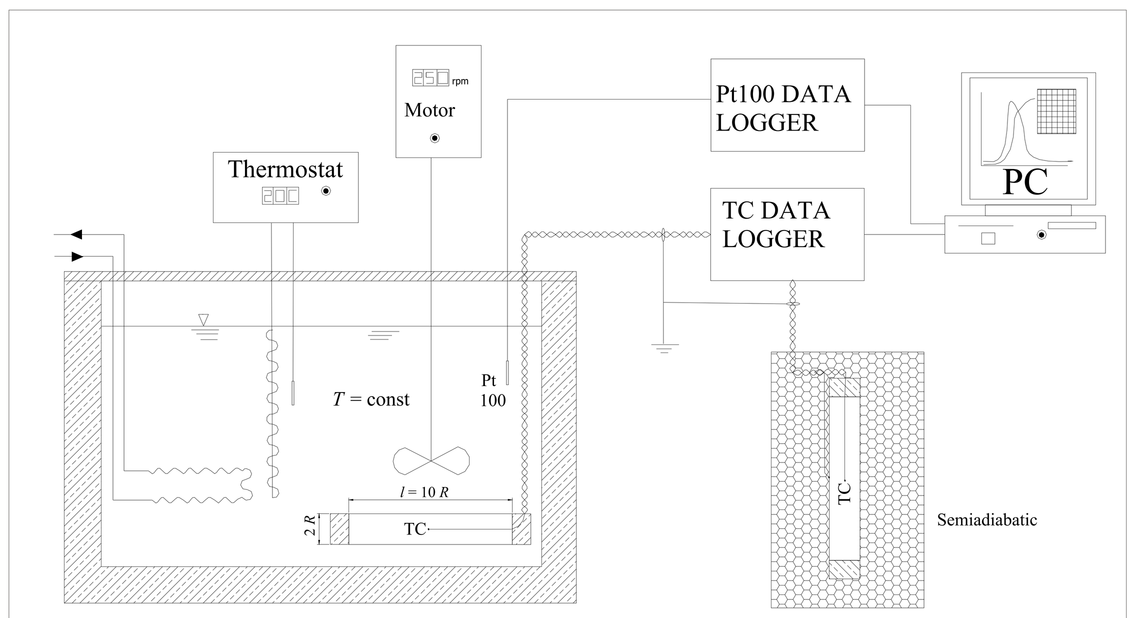


Fig. 6. Simulation of the changes of thermal diffusivity onto the temperatures during isothermal and semiadiabatic measurement.



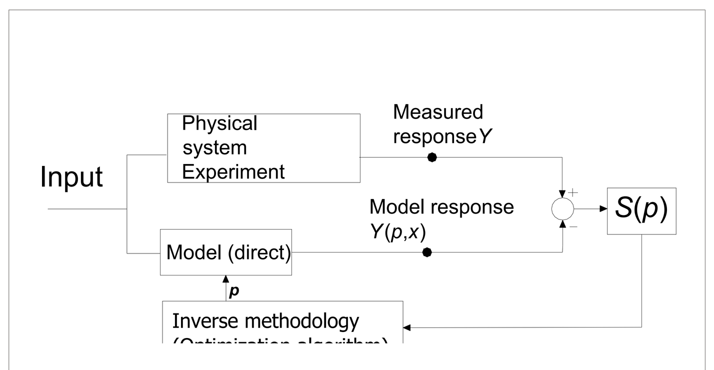


Fig. 4. Schematic of parameter estimation inverse methodology.

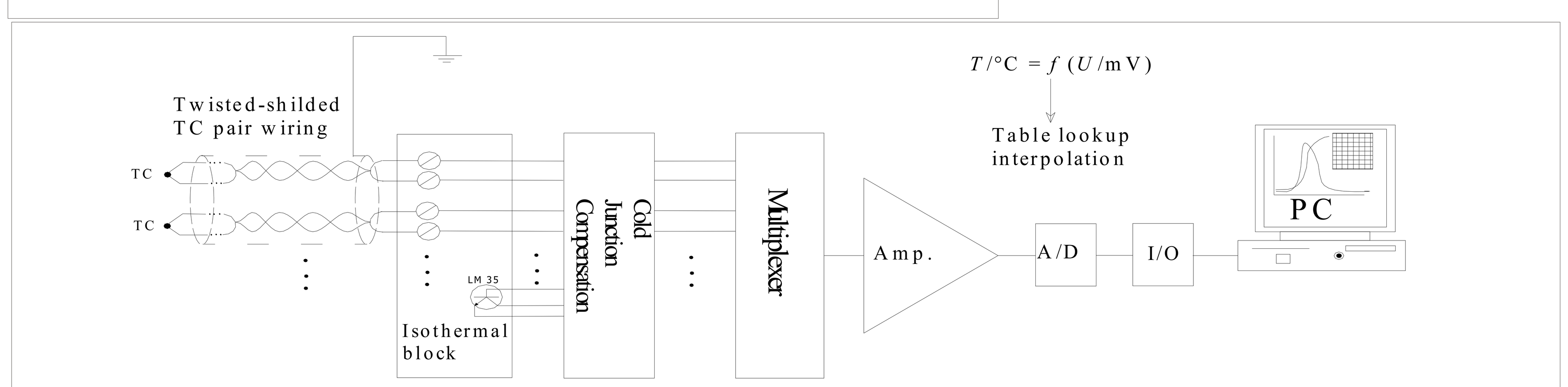


Fig 5. Apparatus and detailed view of thermocouple data logger scheme.

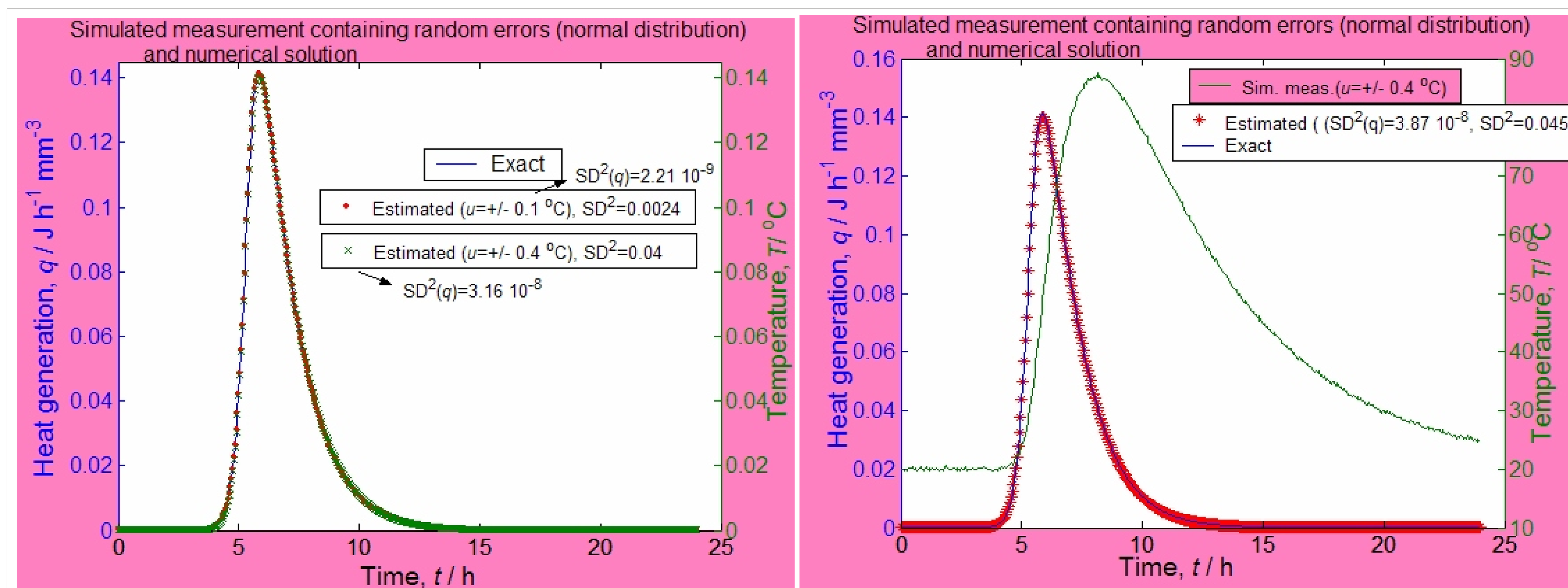


Fig 7. Exactness and accuracy examination of the inverse algorithm on simulated measurements containing random errors.

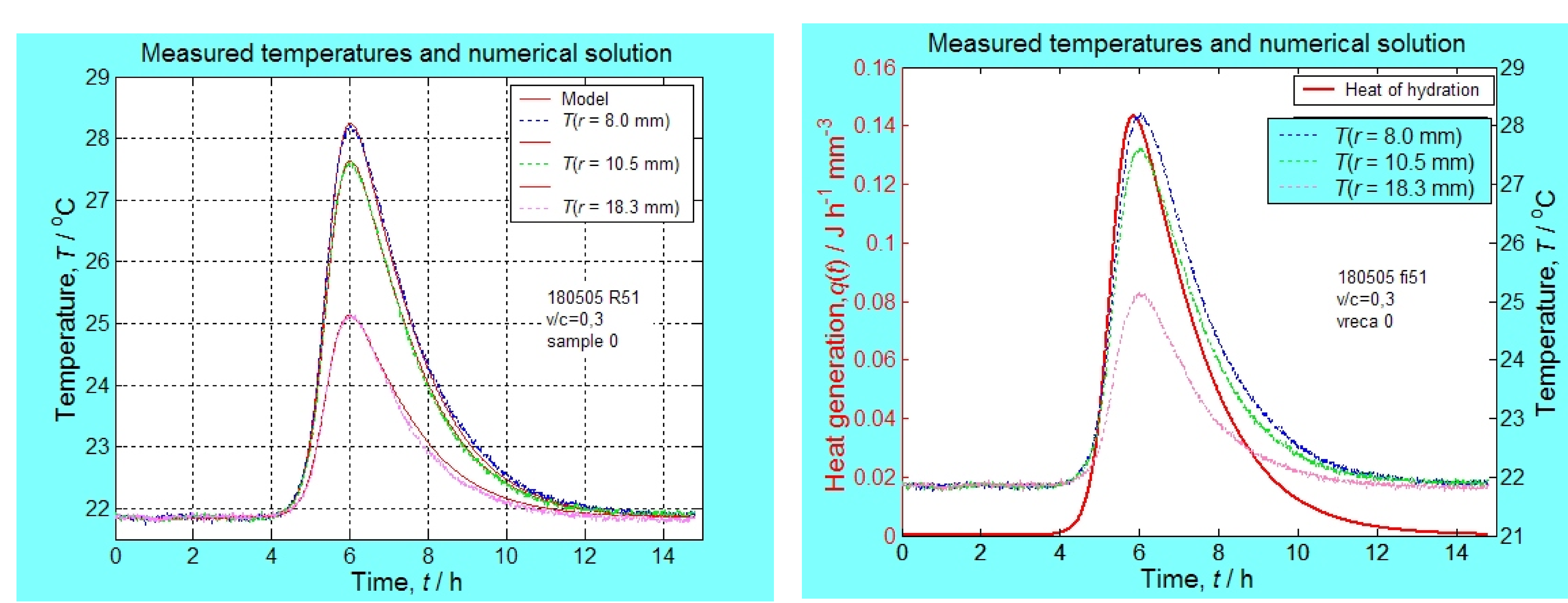


Fig. 8. Estimated heat of hydration by isothermal calorimetry.

CONCLUSION

The novel calorimetric method is based on the inverse method implementation for determination of heat generation from known (measured) material temperature distribution by solving transient heat conduction problem. The advantages of developed calorimetric method are: direct estimation of term q/λ = heat generation/thermal conductivity [(Wm^{-3})/(Wm^{-1}K^{-1})], robustness to thermal diffusivity measurement uncertainty, the knowledge of boundary condition and determination of heat loss from actual measurement. Moreover, the simple experimental technique which measures representative samples in isothermal and semi-adiabatic conditions enables the investigation of the temperature dependence of physico-chemical hydration processes. The evolution of thermal conductivity and diffusivity is possible to determine by dynamic methods and can be used for determination of heat generation and temperature distribution in cement based material. The heats of hydration determined by the developed method agree with literature data and differential micro-calorimeter method.

Reference

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