TCi SYSTEM FOR NON-DESTRUCTIVE DETERMINATION OF THERMAL PROPERTIES OF MATERIALS

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INTRODUCTION

The use of thermal insulation is regarded as one of the most effective means of energy conservation in buildings. The thermal resistance, R, offered by an insulation layer increases with increasing layer thickness and decreasing thermal conductivity. The R-values used to design building walls and roof structures depend strongly on the thermal conductivity of insulation materials. Besides, thermal analysis procedures of building components or the building as a whole, which provide alternatives to heat transmission measurements in laboratory and prototype situations, need thermal property values as input to their calculations. Many types of insulation materials are available, but they differ with regard to thermal properties and many other material properties as well as cost.

Tabulated values of thermal properties of insulation materials are available in the open literature. Accuracy of these property values is sometimes questionable since complete and important information are often lacking. It can be concluded that such data values are very useful but must be used with extreme care because often, thermal conductivity values are quoted for insulation types without giving the density and temperature of the materials tested. Material with aging effects also depends upon manufacturing and storing conditions. In general, the ''effective'' conductivities depend on density, temperature, moisture content as well as the constituents and voids present in their structures.

There are five main types of commercially available instruments to measure thermal conductivity: guarded hot plate (GHP), hot wire, modified plane source, laser flash diffusivity and modulated differential scanning calorimetry (DSC). They can all be broadly classified as steady states methods and transient state methods [1].

All this methods differ in technique, sample size, testing time, capability, and methodologies of measurement. The sample size and test times of many techniques have positioned thermal conductivity analysis within the research and development operation, or to a limited extent, Quality assurance laboratories. Traditional technologies used for thermal properties analysis have been expensive and/or destructive, as well as very time consuming in both sample preparation and testing.

Among the steady state methods, the guarded hot plate (GHP) may be regarded as the most commonly used technique for measuring the thermal conductivity of insulation materials. GHP can be regarded as absolute measurement method, because by measuring the temperature, power of electric energy and thickness the sample thermal conductivity can be calculated. In principle, its operation is based on establishing a steady temperature gradient over a known thickness of a sample and to control the heat flow from one side to the other. Drawbacks of GHP and other steady state techniques are that they require a long time to establish a steady state temperature gradient across the sample, at least 8 hours, and that this temperature gradient is required to be large. The sample size is also required to be large, the thickness of the sample has to be from 20 to 200 mm, and the width and length of the sample are to be between 500 mm and 1 000 mm. The contact resistance between the thermocouple and the sample surface is considered a major source of error, the surface of the sample should be as smooth as possible. [2]

The transient techniques, on the other hand, measure a response as a heat signal is sent into the sample, and the sample response is monitored. Therefore, these techniques are distinguished mainly by the short time required to obtain the desired results.

The study presented in this paper uses a new and accurate experimental technique for measuring a wide range of thermal conductivity of some commonly used insulation and building materials produced by Mathis instruments

PRINCIPLE AND EXPERIMENTAL DEVICE

The TCi system measures thermal conductivity and effusivity of materials directly, based on the modified transient plane source method, and can provide user-inputted capabilities in the calculation of thermal diffusivity and heat capacity [3].
The system is comprised of a sensor, control electronics and computer software, (Figure 1).

Figure 1: Mathis TCi Thermal Property Analyzer   Figure 2: The sensor

The sensor (Figure 2) employs a one-sided, interfacial heat reflectance device that applies a constant current heat source to the sample. The interfacial sensor heats the sample by approximately 1-3°C during the testing. The sample absorbs some of the heat, and the rest causes a temperature rise at the sensor interface. The voltage drop on the spiral heater is measured before and during the transient. The voltage data is then translated into the effusivity value of the tested material. The conductivity is calculated from the voltage data by iterative method.
Sensor does not physically alter or affect the sample being tested, reducing possible contamination as much as possible. Less than 5°C of heat is introduced to the sample by the interfacial sensor, which must only come in contact with one side of the material. As a result, samples remain intact, undisturbed, and reusable. Complete testing requires only 0.8 to 5 seconds what is only a fraction of the duration needed by some traditional methods. In addition, by varying the test time, materials can be evaluated at their surface or to a range of depths. There are practically no restrictions on sample size, the external sensor can obtain accurate readings from samples as small as 17 mm diameter, with a minimum thickness of 0.5 mm, in this case only a good contact needs to be achieved. The maximum sample size and thickness is unlimited [4]. The sensors need to come in contact with only one side of the sample, and have the added capability of "seeing through" layers into the actual sample. For example, liquid samples can be tested in plastic bags, without the thermal value of the bag skewing the results.

Theory

The heat equation in one dimension with a constant supply of heat per time per volume, G', is given with equation (1).

$$\rho c_p \frac{\partial T}{\partial t} = \lambda \frac{\partial^2 T}{\partial x^2} + G' \tag{1}$$

Where ρ is density of a material, c_p is specific heat capacity of a material, T is temperature, t is time, λ is thermal conductivity of a material.

If one-dimensional heat flow and no thermal contact resistance at the interface between the sensor and sample under test are assumed, the temperature change on the sensor surface *(x=0)* is given by equation (2).

$$\Delta T\left(x=0,t\right)=\frac{1.1284\cdot G\sqrt{t}}{e_1+e_2} \tag{2}$$

Where ΔT is a change in sensor surface temperature (°C), G is power flux supplied to sensor (W/m^2), t is time measured from start of process (sec), e_1 is equivalent effusivity of sensor (W√s/m^{2}K), e_2 is effusivity of measured material (W√s/m^{2}K).

To convert the temperature change into equivalent voltage signal change, the temperature coefficient of resistivity (TCR) as a property of the sensor is being used. Assuming perfect linearity of the sensor TCR, platinum wire, the relationship between the sensor resistance and its temperature is given in equation (3).

$$R(T)=R_0(1+\alpha T)=R_0+AT \tag{3}$$

Where $R(T)$ is the resistance of the sensor at temperature T, R_0 is the sensor resistance at $0\ ^oC$, T is the sensor temperature (°C), α is the TCR and A is the slope of resistance versus temperature, Table 1. If Ohm´s law I=V/R is applied to connect equations (2) and (3), the following is derived:

$$\Delta V(t)=\frac{1.1284\cdot I\cdot A\cdot G\cdot \sqrt{t}}{e_1+e_2} \tag{4}$$

Equations (4) can also be written as:

$$\Delta V(t)=m\sqrt{t} \tag{5}$$

where m is the slope of the sensor voltage change versus square root of time, with the value according to equation (6).

$$m=\frac{1,1284\cdot I\cdot A\cdot G}{e_1+e_2} \tag{6}$$

Thermal effusivity calibration

In the short period of time all parameters on the right side in equation (6) have a constant value, and effusivity of the sample material can be calculated as follows in equation (7).

$$\frac{1}{m}=M_2\cdot e_2^2+M_1\cdot e_2+C \tag{7}$$

Where:
- M1, M$_2$ are sensor constants, they depend of material tested, and are provided by the manufacturer for a wide range of materials, Table 1.
- C is sensor property, and it depends of the temperature T during the test

$$C=C_0+C_1\cdot T+C_2\cdot T^2 \tag{8}$$

- C0, C1, C2, -constants of the sensor (Table 1)

Calibration of the sensor signal against materials with known effusivity and conductivity is done by the manufacturer for a specific sensor. All parameters that were used in this study are presented in table 1.

Table 1: Sensor calibration parameters

Calculation Method	Sensor parameters					Effusivity parameters			Conductivity parameters		
	A	R0	C0	C1	C2	M0	M1	M2	a	m*	z
Polymers (Effusivity)	0,09342802	25,7482938	43,72	0,158439	-0,000171	0,057758	-	-	-	-	-
Polymers (Conductivity)	0,09342802	25,7482938	43,72	0,158439	-0,000171	-	-	-	108,6257	0,002689	76,9508
Ceramics (Conductivity)	0,09342802	25,7482938	43,72	0,158439	-0,000171	-	-	-	43,52218	0,000821	96,1518
Ceramics (Effusivity)	0,09342802	25,7482938	43,72	0,158439	-0,000171	-	0,053096	1,90E-06	-	-	-
Foams (Conductivity)	0,09342802	25,7482938	43,72	0,158439	-0,000171	-	-	-	704,0875	0,011648	85,5685
Foams (Effusivity)	0,09342802	25,7482938	43,72	0,158439	-0,000171	0,037944	-	-	-	-	-

Thermal conductivity calibration

Calibration and measurements of thermal conductivity λ are based on same data acquired for effusivity. The algorithm to calibrate and calculate λ is the $m*$, outlined in [4]. It uses an iterative process to calibrate the sensor against measured materials with known thermal conductivity. The calibration process generates a value called $m*$, such that:

$$\frac{1}{m - m^{*}} = a \cdot \lambda + z \qquad (9)$$

Where **a** is a slope and **z** interception value of the calibration line, m* is an iteration algorithm, according to [4], the values for polymers, ceramics and foams are presented in table 1.

Material groups and power levels

The wide range of effusivity and thermal conductivity requires grouping of materials with similar behavior and effusivity/conductivity range, and operation of the TCi sensor at different power levels. Each group of materials has its own calibration curve. Table 2 summarizes the material groups and their attributes.

Table 2: Material groups and their attributes

Material groups	Effusivity range ($W\sqrt{s}/m^2K$)	Conductivity range (W/mK)
Liquids & Powders	0-1660	0-0,6
Foams	50-200	0,04-0,09
Polymers	600-1400	0,25-1,1
Ceramics	1400-8000	1,1-29
Metals	4500-19000	6-110

Contact agent

The TCi sensor has a solid surface optimally engineered for the testing of fluids. When measuring solids a contact agent is required as there is some contact resistance that may significantly affect the results if not addressed within the measurement protocol. The quality of contact and therefore the heat transfer depends on many parameters such as type of material, surface quality and the degree of wetting.

According to the manufacturer, the best contact agent available is water, since it has a relatively high thermal conductivity (~0.6 W/mK), low viscosity, and is easy to apply and clean. Water can be used in a limited temperature range though, from ~5°C to ~70°C. At temperatures lower than 5°C and higher than 70°C alternative contact agents like glycol and glycerin are needed. Calibrations of solids (except for foams) are all done with water.

Limitations for conductivity, λ, measurements

As mentioned above, the method for determining the thermal conductivity of the material is based on the effusivity measurement [4]. Therefore, different calibration curves are required for materials with dissimilar thermo physical properties. For instance, it is necessary to calibrate solids and liquids separately, and use the applicable calibration when measuring a certain material. Measuring a liquid with a calibration made for solids, or vice versa, will generate an invalid result.

Since effusivity includes conductivity (λ), density (ρ) and specific heat capacity (c), equation (10),

$$e = \sqrt{\lambda \cdot c \cdot \rho}$$
(10)

it is possible that two different materials will have the same effusivity, but different λ if their (ρc) product is different. In such cases, the calculated λ may be incorrect and to avoid such them, a sample should always be measured with the test method that uses a calibration group which is closest to the sample's type of material.

MEASUREMENT PROCEDURE

The first part of the experiments involved measuring the thermal conductivity of insulation material samples, table 3, at three different temperatures, 15, 22 and 30°C, and the humidity of 30 and 71,2 %. This was done in order to test the apparatus sensitivity in relatively close temperature and humidity conditions.

Table 3: Insulation materials tested in different temperature and humidity conditions

Material name	Material label	Density [kg/m³]
LGF 488/08	I 1	17,83
LGF 463/08	I 2	103,43
LGF 047/09	I 3	90,21
PTP	K 1	171,23
DDP X2	K 2	153,65
DDP X1	K 3	159,59
DP-7	K 4	70,63
DP3	K 5	41,06
TP	K 6	107,45
FP-GVs	K 7	85,29

Measurements were carried out for three specimens of each test sample, this was repeated three times for each specimen, mean values of thermal conductivity and effusivity were obtained by averaging. The density of a specimen was determined by dividing the measured mass of the specimen by its volume. The mass of the specimen was measured by a precision balance having a resolution of 0.1 g. The length, width and thickness of the specimen were measured by using a steel caliper having a resolution of 0.1 mm.

In figures 3 and 4, thermal conductivity of materials measured at temperatures of 15, 22 and 30 °C and humidity of 30 and 71,2 % is presented.
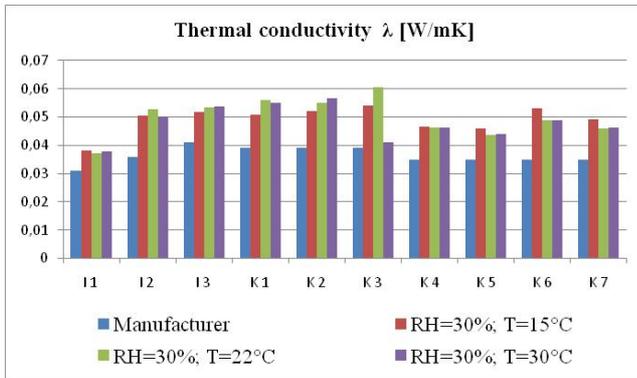
Figure 3: Thermal conductivity measured at the humidity of 30 %, compared to manufacturer data
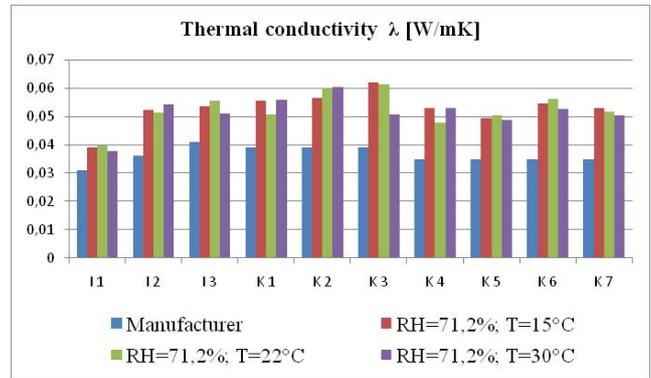
Figure 4: Thermal conductivity measured at the humidity of 71,2 %, compared to manufacturer data

In figures 5 and 6, effusivity of materials measured at temperatures of 15, 22 and 30 °C and humidity of 30 and 71,2 % is summarized.
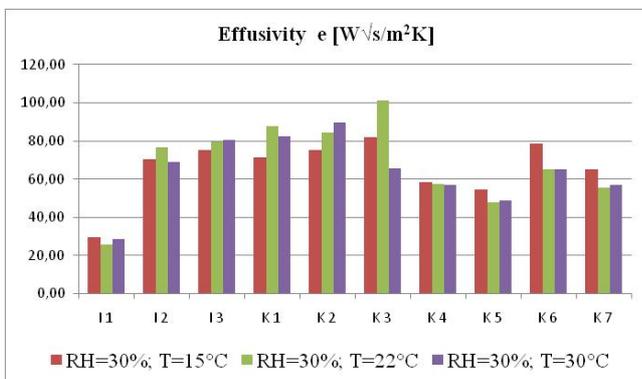
Figure 5: Effusivity measured at the humidity of 30 % compared to manufacturer data
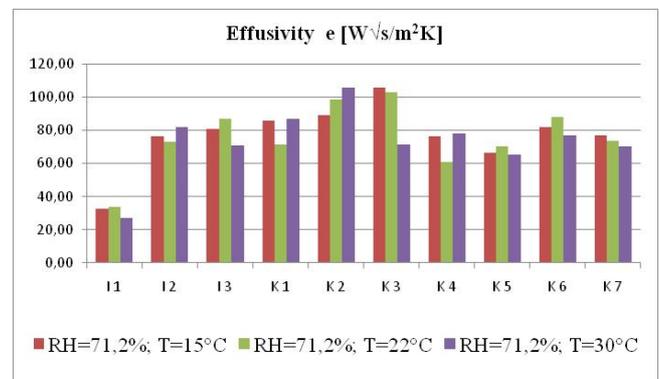
Figure 6: Effusivity measured at the humidity of 71,2%, compared to manufacturer data

From figures 3 and 4 it can be seen that thermal conductivity of insulation materials measured with TCi is higher than that claimed by the manufacturers. The difference varies from 15 to 35 % for different materials, respectively. It was noticed that measured difference is smaller for homogeneous materials than the one for non-homogeneous materials. This can be explained by the fact that calibration curve for foams, provided by the manufacturer of the TCi sensor, was used to determine thermal conductivity of materials. According to the sensor calibration data, "Foams" calibration curve is intended to be used to test materials with effusivity range from 50 to 200 $W\sqrt{s}/m^2K$ and thermal conductivity range from 0,04 to 0,09 W/mK, and by examining the

calibration material, "Foam" it can be seen that it is very homogenous. Also, it can be noted that influence of temperature and humidity changes cannot be described as expected, hence that thermal conductivity rises with rising temperature of the sample [5,6,7]. The results of measurements are changing from sample to sample of the same material, and this is explained with great sensitivity of the sensor and its dependence of the contact with the material and alignment of the sensor.

Other building materials were also tested within presented study, with the intention to observe the influence of contact agent on test results. Materials and their characteristics chosen for this purpose are summarized in table 4.

Table 4: Materials

Material name	Density ρ [kg/m³]	Label
Brick	1831,8	O1
Concrete	1728,2	B1
Marble	2314, 33	M1
Granite	2869,47	G1

The measurements were performed first with dry samples at the temperature of 24,3 °C and humidity of 50,2 %, with glycol as a contact agent, as recommended [3], then, samples were saturated in order to reduce the influence of absorption of contact agent that was noticed during the testing. Saturation is justified because sensor manufacturer provided calibration curves that already take into consideration existence contact agent, therefore it should not be a problem. Saturation was done by submerging the samples into water for 24 h, the water temperature was 24°C. After the saturation of the samples, their surface was wiped and they were tested again with water and glycol as contact agents.

In figures 7 and 8, test results for thermal conductivity and effusivity are summarized and compared with literature values [6] for materials with approximately same density as materials that were tested. It can be seen that dry samples with glycol as a contact agent have smaller values of thermal conductivity than literature values, respectively. This could be explained by the use of inadequate calibration curve and by the fact that contact agent was absorbed into the sample during time and with absorption, measured value of thermal conductivity and effusivity were dropping because air was entering between sensor and material surface. Results presented in figures 7 and 8 are mean values, hence the result differ from literature values. The difference is greater for materials that have smaller density and are more porous than for those with greater density. With saturated samples the results were uniform in time, because there was no absorption of contact agent, and the results are more realistic, the difference is again explained by the use of inadequate calibration curve and by the fact that the literature values are for similar but not the same materials.
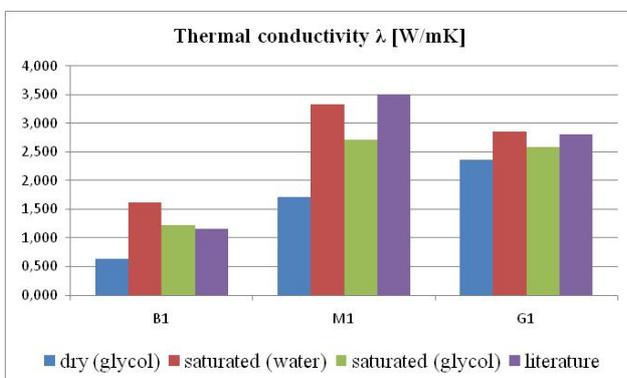

Figure 7: Thermal conductivity compared to literature data
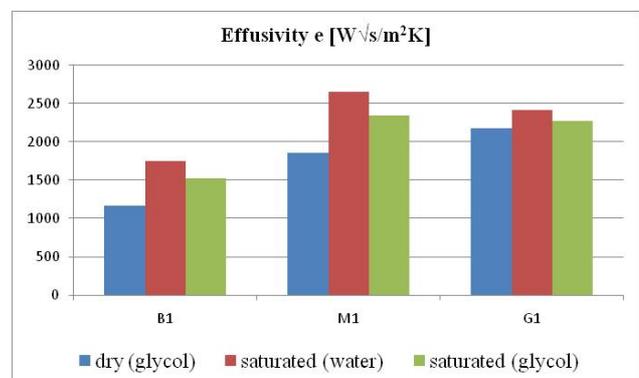
Figure 8: Effusivity data

CALIBRATION CURVE ADJUSTMENT

The brick sample is taken out of above consideration because test results were extremely unusual, in explanation, thermal conductivity value gained for brick was λ= -0,57 W/mK and effusivity was 366 W√s/m²K, while expected values were 0,76 W/mK and 1100 W√s/m²K, respectively. The obvious solution for gaining this kid of results was the use of inadequate calibration curves. All available calibration curves were then applied to test brick samples, table 5.

Table 5: Mean values of thermal properties testing of brick by using different calibration curves

Calibration curve	Effusivity [W√s/m²K]	Thermal conductivity [W/mK]	1/m	Mean value 1/m	Standard deviation 1/m
Foam	497	0,30	66,11		
Polymer	322	0,03	65,88	66,45	0,79
Ceramics	366	-0,57	67,35		

From table 5, it can be seen that, no matter which calibration curve is applied for testing of the samples, parameter **1/m** is always approximately the same. In this case **1/m** is equal 66,45 with the standard deviation of 0,79. All of this is understandable if one knows the principle of TCi system, explained above. It was decided that calibration curve should be adjusted in order to gain correct results. To do the calibration adjustment, some literature values had to be entered as input data, for brick density of ρ = **1800 kg/m³** thermal conductivity is λ = **0,76 W/mK** and heat capacity is **c = 920 J/kgK**. By using the equation (11), effusivity was calculated, **e = 1121,86 W√s/m²K.**
According to [3], by using equations (7 and 8) and sensor calibration parameters (table 1), equation of calibration curve for ceramics can be written as (11), figure 9:

$$\frac{1}{m} = 0,0000019 \cdot e_2^2 + 0,05309609 \cdot e_2 + 47,37896 \tag{11}$$

Because of the fact that M_1 and M_2 from equation (7) are sensor parameters (table 1), and the shape of the curve was intended to be the same, only parameter C from equation (7) was changed to C'. Value **C' = 4,49234** is derived when value of effusivity **e = 1121,86 W√s/m²K** and **1/m = 66,45** is entered into equation (7) with M_1 and M_2 taken from table 1. Adjusted equation for calibration curve is then (12), figure 9.

$$\frac{1}{m} = 0,0000019 \cdot e_2^2 + 0,05309609 \cdot e_2 + 4,49234 \tag{12}$$
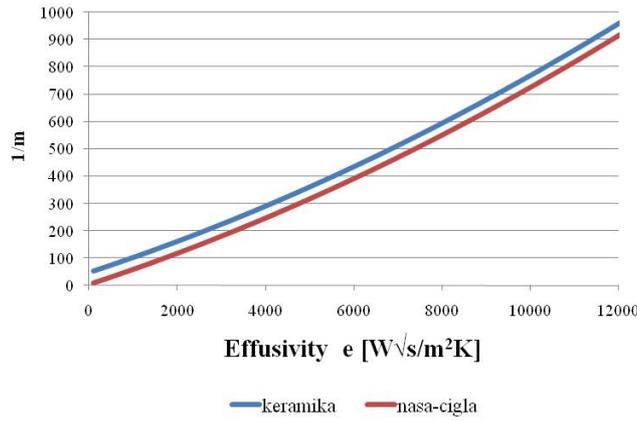
Figure 9: Calibration curve comparison, ceramics – Tci and adjusted curve - brick

Thermal conductivity calibration curve was adjusted in similar way. Calibration curve equation is based on equation (9) and sensor parameters **z**, **a**, **m***, table 1, and is equal to (13):

$$\lambda = \frac{\dfrac{1}{m - 0,000821} - 96,15176}{43,52218} \tag{13}$$

Just to translate vertically the calibration curve, the parameter **z** needed to be changed to **z'**, and was equal to **z' = 37,20754** if **λ = 0,76 W/mK,** and **1/m = 66,45**. Equation of adjusted calibration curve was then (14):

$$\lambda = \frac{\dfrac{1}{m - 0,000821} - 37,20754}{43,52218} \tag{14}$$

Figure 10 is showing dependence of the parameter 1/m and the thermal conductivity for expected interval (0,4 – 2,0 W/mK), hence, it is showing adjusted calibration curve for brick samples tested under described conditions. Figure 11 is showing the dependence of effusivity and thermal conductivity, and the difference between theoretical values and values gained from adjusted calibration curve in the thermal conductivity interval from 0,4 – 1,5 W/mK.
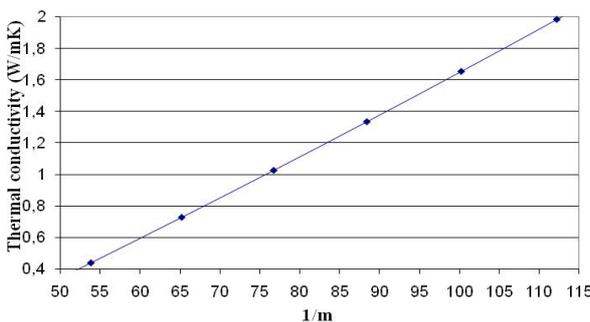

Figure 10: Dependence thermal conductivity to parameter 1/m
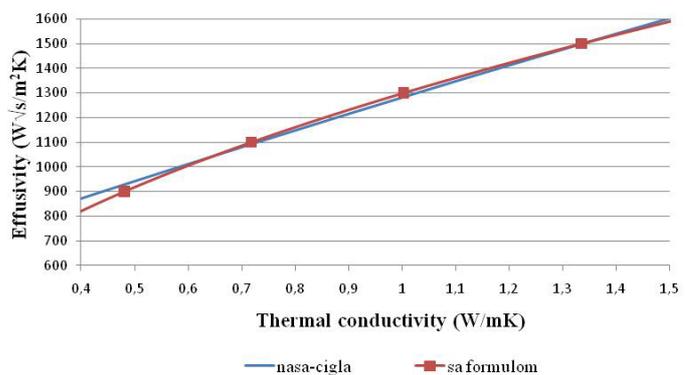
Figure 11: Dependence effusivity to thermal conductivity

From figure 11 it can be seen that, in the expected thermal conductivity interval for a brick from 0,4 – 1,5 W/mK, there aren't any significant deviation of adjusted calibration curve from theoretical curve.

CONCLUSIONS

Thermal properties of different building materials were tested by using TCi system. Experimentally gained results were compared to those claimed by the manufacturers and literature values. Deviation can be explained by the use of inadequate calibration curves for specific materials, respectively, absorption of the contact agent, great sensitivity of the sensor and the influence from the researcher. Main advantages of the TCi system are simple and cheap measuring equipment, test time is much shorter than in traditional stationary methods, testing can be performed in situ. On the other hand, main shortcomings of the TCi system are subjectivity in determination of measuring interval, researcher, contact between the sensor and material, sensor placing, and the fact that calibration curves for every material to be tested have to be known, also non-homogenous materials cannot be tested. From all of the above, some applications of the TCi system can be drawn, system can be used in quality control procedure in factories where thermal properties of homogeneous materials can be controlled if the expected values are known. Quantitative values of thermal properties are difficult to gain while qualitative change of properties in different conditions can be registered.

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