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# Measuring thermal conductivity and specific heat capacity values of inhomogeneous materials with a heat flow meter apparatus



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# ABSTRACT

The aim of this study was to observe the suitability of the heat flow meter apparatus for thermal conductivity and specific heat capacity tests of concrete, as well as to determine specific heat capacity values for 14 different plasters. In total, two concrete types, five floor screed plasters, two fixing plasters, six wall screed plasters, and one specialized plaster that had small EPS spheres added into it were tested.

The main novelty value of this research is studying how well heat flow meter apparatus can determine specific heat capacities of inhomogeneous materials. Also, how precisely thermal conductivity could be measured from small concrete specimens of interest. The results measured with the most suitable methods were in line with published values, which indicate that the apparatus was suitable for both tests.

#### 1. Introduction

Thermal conductivity and specific heat capacity are among the most essential material properties of a building material. Thermal conductivity describes the ability of a material to conduct heat, and the specific heat capacity tells how much heat energy is absorbed or released depending on the temperature difference and mass [1]. These values are needed, among other uses, in thermal performance calculations. However, the published values for material properties are usually not sufficient for more accurate calculations. That is due to the fact that individual products in a product group, such as plasters, may have widely varying material properties. As many material properties are listed generically for whole material groups, values determined from the actual products are required to achieve a high level of accuracy in the calculations for a specific case.

There are different kinds of methods to determine thermal conductivity. In the study [2] there is one method presented to determine thermal conductivity of insulation materials. The aim of this study was to observe the suitability of the heat flow meter apparatus for thermal conductivity and specific heat capacity tests of concrete, so it is known whether or not test results of inhomogeneous materials acquired with this apparatus are reliable. Also, specific heat capacity values for 14 different plasters were measured to get more accurate material properties.

Specific heat capacity is usually tested with a calorimeter. One kind of calorimeter for defining specific heat capacity is used in the study [3]. However, the use of a heat flow meter apparatus has recently

become possible as well. This is due to the approval of an American standard ASTM C1784-13 [4]. The standard describes how to use a heat flow meter apparatus for measuring thermal storage properties, i.e. specific heat capacity. Research on the subject is conducted by the developer of the apparatus, as well as other instances [5-7] and instructions on how to perform the tests have been given [4,8,9]. Excluding thermal contact resistance is one of the key factors when measuring thermal conductivity since it greatly impacts the accuracy and reliability of results [10–12].

# 2. Material and methods

## 2.1. Heat flow meter apparatus FOX50

In this study a heat flow meter apparatus FOX50 that runs on WinTherm50 software was used. The apparatus is developed and manufactured by an American company LaserComp, Inc. The apparatus consists of upper and lower plates, two heat flow meters and protective casing which is preventing the heat losses. Sample to be measured is placed between the upper and lower plates. The upper plate is stationary and the lower plate moves vertically to provide good contact with the sample and minimize interface resistance [13].

The specimen size in this apparatus is rather small, from 50 to 61 mm in diameter and 0-25 mm thick, and the thermal conductivity range is from 0.1 to 10 W/(m K) with an absolute thermal conductivity test accuracy of  $\pm 3\%$  [13]. The apparatus has been calibrated to four different ranges of thermal conductivity values and the most suitable

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Nomenclature	T	temperature (K)
	$T_{cal}$	known temperature for calibration (K)
<i>c</i> specific heat capacity at a constant pressure (J/(kg K))	$T_m$	average test temperature (K)
d thickness of specimen (m)	$\Delta T_{ext}$	temperature difference between external thermocouples
$d_{total}$ thickness of both the rubber sheets and the specimen		(K)
together (m)	x	thickness of specimen (m)
<i>H</i> amount of heat energy per square meter $(J/m^2)$	δx	depth of the groove (mm)
$H_{hfm}(T)$ correction factor to remove the effect of the plates (J/	$\Delta x_1, \Delta x_2$	thicknesses of two separate specimens (m)
(m <sup>2</sup> K))	λ	thermal conductivity (W/(m K))
m mass (kg)	$\lambda_{cal}$	known thermal conductivity for calibration (W/(m K))
q heat flux flowing through the specimen (W/m <sup>2</sup> )	$\lambda_{total}$	thermal conductivity of both the rubber sheets and the
Q heat energy (J)		specimen together (W/(m K))
$Q_1, Q_2$ signal values of two separate tests ( $\mu$ V)	ho	material's density (kg/m <sup>3</sup> )
$Q_{Lequil}$ Heat Flow Meter signal at the final steady-state, lower	τ	time interval (s)
plate (µV)		
$Q_{Li}$ Heat Flow Meter signal value of lower plate ( $\mu$ V)	Subscripts	S
$Q_{Uequil}$ Heat Flow Meter signal at the final steady-state, upper		
plate (µV)	r	rubber sheets
$Q_{Ui}$ Heat Flow Meter signal value of upper plate ( $\mu$ V)	cal	known thermal properties for calibration
$S_{cal}$ temperature dependent calibration factor (W/(m <sup>2</sup> $\mu$ V))	U	upper plate
$S_{Lcal}$ calibration factor of lower plate (W/(m <sup>2</sup> $\mu$ V))	L	lower plate
$S_{Ucal}$ calibration factor of upper plate (W/(m <sup>2</sup> $\mu$ V))	equil	value at the final steady-state

file based on material's supposed thermal conductivity ought to be used [13]; here the calibration file Pyrex 7740 was used.

The procedure to determine thermal conductivity is described in the European standards SFS-EN 12664 and SFS-EN 12667 [14,15], and test equipment requirements for the heat flow meter apparatus are stated in standards SFS EN 1946-1, SFS EN 1946-3 and ISO 8301:1991 [16–18]. The thermal conductivity tests were conducted at the mean temperature of 10 °C. The temperature difference between the upper and lower plate should have been 20 K; however, the difference was greater (26 K) when using external thermocouples, due to a recommendation of the developer. The thermal conductivities of concrete specimens were tested to decipher how reliable thermal conductivity values can be gotten from a small specimen size with relatively large (16 mm) maximum particle size.

When measuring specific heat capacity, the apparatus is suitable for building materials such as concrete, wood and insulating materials with absolute accuracy of  $\pm 5\%$  [13] and is in accordance with standard ASTM C1784-13 [4]. Specific heat tests were conducted with plain specimens and PID coefficient alterations. Test temperatures were 10 °C, 20 °C, 30 °C and 40 °C as instructed by the apparatus developer [13]. The average result is therefore the value at 25 °C.

#### 2.2. Specimens

There were two types of concrete specimens: C20/25 and C32/40 specimens. Both had maximum particle size of 16 mm and both were in consistence tolerance class S3. The concrete masses were tested in laboratory: Concrete C20/25 was a mixture of 249.10 kg of cement (CEM I 42.5 R/2), 69.30 kg of ash, 503 kg of rock 0–8, 524 kg of rock 0–8/6, 186 kg of rock 3–8, 394 kg of rock 6–16, 200 kg of filling and 128.30 kg of cold water. Water-cement ratio (w/c) was 0.57. Volume fraction of rock material in concrete C20/25 was 67.91% and mass fraction 77.75%.

Concrete C32/40 was a mixture of 721.60 kg of cement (CEM I 42.5 R), 204.10 kg of ash, 805 kg of rock 0-8, 734 kg of rock 0-8/6, 238 kg of rock 3-8, 1,114 kg of rock 6-16, 437 kg of filling and 326,3 kg of cold water. Water-cement ratio (w/c) was 0.44. Volume fraction of rock material in concrete C32/40 was 61.34% and mass fraction 70.80%.

In addition to concrete tests five floor screed plasters, two fixing plasters, six wall screed plasters, and one specialized plasters that had

1-3 mm EPS spheres added into it were also tested in specific heat capacity tests with heat flow meter apparatus according to standard ASTM C1784-13 [4]. Floor screed plaster 1 was a mixture of resincement-quartz sand-based powder and water and its maximum particle size was 3 mm. Floor screed plaster 2 was a mixture of 0.1 kg of water and 0.57 kg of fiber-polyester-special cement-quartz-based powder with 1 mm maximum particle size. Water-powder ratio for Floor screed plaster 2 was 5.70. Floor screed plaster 3 was made of 1.11 kg of fibersynthetic resin-aluminate/Portland cement-quartz-based powder and 0.2 kg of water. Powder had 1 mm maximum particle size and waterpowder ratio was 5.55. Floor screed plaster 4 was a mixture of 0.80 kg of resin-cement-quartz-based powder and 0.2 kg of water. Plaster's maximum particle size was 0.3 mm and water-powder ratio 4.00. Floor screed plaster 5 was a mixture of 0.44 kg of resin-special cement-quartz sand-based powder and 0.1 kg of water. The maximum particle size of Floor plaster 5 was 0.5 mm and water-powder ratio 4.40.

Fixing plaster 1 was made of polymer-white cement-lightweight filler-quartz sand-based powder and water and its maximum particle size was 0.5 mm. Fixing plaster 2 was a mixture of 0.2 kg of water and 0.941 kg of polymer-cement-quartz-based powder which maximum particle size was 0.5 mm. Plaster O1 was made of water and EPS-granulate-cement-based powder, which EPS granulate size was 1–3 mm but maximum rock particle size was unknown.

Wall screed plaster 1 was a mixture of 0.842 kg of polymer-cementsand-based powder and 0.2 kg of water and its maximum particle size was 3 mm. Wall screed plaster 2 was a mixture of water and polymercement-sand-based powder with maximum particle size 2 mm. Wall screed plaster 3 was a light weight smoothing plaster with maximum particle size 2 mm. Wall screed plaster 4 was made of 0.15 kg of water and 0.667 kg of polymer-cement-sand-based powder with maximum particle size 1.5 mm. Wall screed plaster 5 was a mixture of 0.941 kg of polymer-cement-limestone-based powder and 0.2 kg of water. Plaster's maximum particle size was 0.5 mm. Wall screed plaster 6 was a mixture of 0.645 kg of polymer-cement-limestone-based powder and 0.2 kg of water. Plaster's maximum particle size was 0.2 mm.

More specific product formulations are a trade secret of the plaster manufacturer. The manufacturing properties of plasters are also presented in Table 1. Plasters with no exact information about the batch are made by way of manufacturer's instructions. Ingredients are weighted with the scale with accuracy of  $\pm 0.1$  g and mixed with a laboratory mixer.

#### Table 1

The manufacturing properties of plasters.

Material	Manufacturer's recommended ratio	Batch (plaster + water)	Ratio
Plaster FL1	8.00	-	_
Plaster FL2	5.714	0.57 kg + 0.1 kg	5.70
Plaster FL3	5.556	1.11 kg + 0.2 kg	5.55
Plaster FL4	4.00	0.80 kg + 0.2 kg	4.00
Plaster FL5	4.444	0.44 kg + 0.1 kg	4.40
Plaster FIX1	2.31	_	-
Plaster FIX2	4.44	0.941 kg + 0.2 kg	4.71
Plaster W1	4.211	0.842 kg + 0.2 kg	4.21
Plaster W2	4.17	_	-
Plaster W3	4.00	_	-
Plaster W4	4.444	0.667 kg + 0.15 kg	4.45
Plaster W5	4.706	0.941 kg + 0.2 kg	4.71
Plaster W6	3.226	0.645 kg + 0.2 kg	3.23
Plaster O1	-	-	-

Every concrete and plaster type had thick and thin specimens tested; the thick specimens were about 20 mm thick and the thin specimens were about 10 mm thick. All specimens were stored at 23 °C and relative humidity of 50% RH before tests to reach a steady-state [14,15]. The steady-state was reached when the change in the mass of the test specimen over a 24 h period was random and less than the equivalent of 0.1 kg/m<sup>3</sup>. However, the eventual water contents were not measured.

#### 2.3. Equations to determine thermal conductivity

The temperature field in a specimen is considered to be uniform throughout the whole volume of the specimen when the specimen is placed between two flat isothermal plates that are at different temperatures, and a uniform one-dimensional temperature field had been stabilized [19].

The general principle of the FOX50 apparatus is based on onedimensional Fourier law Eq. (1) that shows the relation between the heat flux flowing through the specimen and thermal conductivity [19].

$$q = q_x = -\lambda \frac{\partial T}{\partial x} \approx \lambda \frac{\Delta T}{\Delta x} = \lambda \frac{T_1 - T_2}{d}$$
(1)

where

q= heat flux flowing through the specimen (W/m²). $\lambda$ = thermal conductivity (W/(m K)).T= temperature (K).d and x= thickness of specimen (m).

T/x = temperature gradient (K/m).

However, before testing a specimen with an unknown thermal conductivity, the FOX50 apparatus must be calibrated with a material that has a known thermal conductivity value  $\lambda_{cal}$  at the temperature  $T_{cal}$ . The apparatus has been calibrated temperature dependently with Perspex for 0.1860–0.1933 W/(m K), Vespel DuPont for 0.374–0.394 W/(m K), Pyrex 7740 for 1.058–1.20 W/(m K) and Pyroceram 9606 for 3.710–4.149 W/(m K) [13]. The calibration factors are characteristic for the apparatus. Calibration factors are determined from Eq. (2). They are used in actual tests as presented in Eq. (3) [6,9,20].

$$S_{cal} = \frac{\lambda_{cal} \cdot \Delta T \cdot (Q_1 - Q_2)}{(\Delta x_2 - \Delta x_1) \cdot (Q_1 \cdot Q_2)}$$
(2)

where

$$S_{cal}$$
 = temperature dependent calibration factor (W/(m<sup>2</sup> µV)).  
 $Q_I, Q_2$  = signal values of two separate tests (µV).  
 $\Delta x_I, \Delta x_2$  = thicknesses of two separate specimens (m)

$$\lambda_{test} = S_{cal}(T_{test}) \cdot Q \cdot \frac{d_{test}}{\Delta T_{test}}$$
(3)

Because there are two plates, the apparatus gets two different thermal conductivity values for the specimen. Final thermal conductivity is the average value of these two values and is declared at the average test temperature  $T_m$  [9,20].

The thermal conductivity tests in this study were conducted in the following ways: with plain specimens, specimens with rubber sheets, specimens with external thermocouples and specimens with both rubber sheets and external thermocouples. The procedure to test plain specimens follows the above-mentioned equations, whereas the methods with rubber sheets and external thermocouples use other equations as well (see Eqs. (4) and (5)).

The main reason to use rubber sheets or thermocouples is to eliminate errors caused by air gaps and poor contact between the specimen's surfaces and the apparatus' plates. Elastic rubber sheets fill out the air gaps and smoothens the surface which improves the contact [21].

In Eq. (4) subscript "total" indicates the result of such a test where the specimen was tested with the rubber sheets, i.e. thickness and thermal conductivity of both the rubber sheets and the specimen together. Subscript "r" indicates the rubber sheets, i.e.  $d_r$  is the thickness of two rubber sheets and  $\lambda_r$  is the thermal conductivity of two rubber sheets. These values can be obtained by doing additional thermal conductivity tests solely with the rubber sheets.

$$\lambda = \frac{d_{total} - d_r}{\left(\frac{d_{total}}{\lambda_{total}} - \frac{d_r}{\lambda_r}\right)}$$
(4)

Another way to improve the accuracy of test results is to use external thermocouples. Grooves on the specimen surfaces, in which the ends of thermocouples are placed, should be made along the diameter line of specimen. The WinTherm50 software uses the following Eq. (5) to determine the thermal conductivity of a specimen when external thermocouples are used [22].

$$A = S_{cal} \cdot Q \cdot \frac{(x - \delta x)}{\Delta T_{ext}}$$
(5)

where

 $\delta x$  = depth of the groove (mm).

 $\Delta T_{ext}$  = temperature difference between external thermocouples (K).

# 2.4. Equations to determine specific heat capacity

The specific heat capacity of a substance is the amount of energy needed to change the temperature of 1 kg of the substance by 1 °C. Specific heat capacity at constant pressure is determined by Eq. (6).

$$c_p = \frac{1}{m} \frac{dQ}{dT} = \frac{Q}{m \cdot \Delta T} \tag{6}$$

where

Q

 $c_p$  = specific heat capacity at a constant pressure (J/(kg K)).

$$m = mass (kg)$$

T = temperature (K).

The energy fed into the specimen will not cause only an increase in temperature, but also thermal expansion. However, the expansion of solid objects and fluids is so meaninglessly small that the expansion can be disregarded [19,23].

In this study the heat flow meter apparatus was used in specific heat tests as well. The method follows the American standard ASTM C1784-13 (2013) [4] and is based on that the amount of heat absorbed by the specimen can be calculated from the heat flow meter's readings as shown in Eq. (7). Time interval  $\tau$  depends on the used gain level. In the tests the constant default gain level value 7 was used which corresponds to  $\pm$  20,000 microvolts of heat flow meters signals limits [6]. Also the time interval was constant in these thermal conductivity and specific heat capacity tests.

$$H = \sum_{i=1}^{n} \tau \cdot [S_{Ucal} \cdot (Q_{Ui} - Q_{Uequil}) + S_{Lcal} \cdot (Q_{Li} - Q_{Lequil})]$$
(7)

where

 $\begin{array}{ll} H & = \mbox{ amount of heat energy per square meter } (J/m^2). \\ \tau & = \mbox{ time interval } (s). \\ S_{Ucal} & = \mbox{ calibration factor of upper plate } (W/(m^2 \, \mu V)). \\ S_{Lcal} & = \mbox{ calibration factor of lower plate } (W/(m^2 \, \mu V)). \\ Q_{Ui} & = \mbox{ Heat Flow Meter signal value of upper plate } (\mu V). \\ Q_{Li} & = \mbox{ HFM signal value of lower plate } (\mu V). \\ Q_{Uequil} & = \mbox{ HFM signal at the final steady-state, upper plate } (\mu V). \\ \end{array}$ 

 $Q_{Lequil}$  = HFM signal at the final steady-state, lower plate ( $\mu$ V). The heat absorbed by the two plates of the heat flow meter themselves is included in the calculated sum and needs to be

eliminated from the final results. The energy conservation equation Eq. (8) shows the heat capacity of the heat flow meters. (These equations are more extensively described in references [5,6].)

$$c_p \rho \cdot x + c_p \cdot \rho' 2\delta x' = \frac{H}{\Delta T}$$
(8)

$$\rightarrow \quad c_p \rho = \left(\frac{H}{\Delta T} - c_p \rho' 2\delta x'\right) \cdot \frac{1}{x} \tag{9}$$

where

 $c_p \rho$  = specific heat of the specimen (J/(m<sup>3</sup> K)) ( $\rho$  is density (kg/m<sup>3</sup>)).

 $c_p \rho$  = specific heat of two the heat flow meters (J/(m<sup>3</sup> K)).

x =thickness of the specimen (m).

 $2\delta x$  = thickness of the two heat flow meters (m).

 $\Delta T$  = temperature change (K).

The apparatus calculates the material's specific heat capacity from Eq. (10). Finally, the specific heat capacity at a constant pressure is calculated from the result by dividing it by the material's density  $\rho$  [8].

$$c_p \rho = \frac{H_{last} - H_{hfm}(T) \cdot \Delta T}{x \cdot \Delta T}$$
(10)

where

 $H_{hfm}(T)$  = correction factor to remove the effect of the plates (J/(m<sup>2</sup> K)).

#### 3. Results

# 3.1. Thermal conductivity of rubber sheets

The average combined thickness of two rubber sheets by three separate measurements was 1.55 mm (no deviation, i.e.  $\pm 0.00 \text{ mm}$ ) and the average thermal conductivity was 0.2185 W/(m K) ( $\pm 0.0005 \text{ mm}$ ). The average values were used in determining thermal conductivities of specimens as described in Eq. (4).

#### 3.2. The thermal conductivity of small concrete specimens

The specimens were conditioned before tests and material humidity of the tested specimens was 50 ( $\pm$ 10) % RH. Density of both C20/25 and C32/40 concrete specimens that were used in these tests were between 2,240 and 2,250 kg/m<sup>3</sup>, which means that their thermal conductivity should settle between 1.70–1.73 W/(m K) [24]. For 2,200 kg/m<sup>3</sup> concrete standard gives the value 1.65 W/(m K) for thermal conductivity and for 2,400 kg/m<sup>3</sup> concrete 2.00 W/(m K) [25]. The interpolated standard values for thermal conductivity for concrete with densities between 2,240 and 2,250 kg/m<sup>3</sup> are 1,72–1,74 W/(m K).

When measuring thermal conductivity, the heat flow throw the specimen is measured by heat flow meters and the temperatures are measured by sensors attached to upper and lower plates. Individual test results are presented in Tables 2, 3 and Figs. 1 and 2. One presented value means the average value of upper and lower plate. In total ten specimens from concrete C20/25 and 12 from concrete C32/

40 were tested; both 20 mm thick ("thick#") specimens and 10 mm thick specimens were tested ("thin#"). The manufacturer of the apparatus had suggested using ten specimens when measuring the thermal conductivity of inhomogeneous materials to decrease error caused by diverse specimens.

Although test results of plain specimens, specimens with rubber sheets, and specimens with thermocouples are somewhat in line with each other, the thermal conductivity values are lower than declared for concrete. On the other hand, values of specimens with both rubber sheets and thermocouples were of a correct magnitude. The average thermal conductivity value for the C20/25 concrete measurement with rubber sheets and thermocouples is 2,09 W/(m K). However, the measurement for the specimen "thick1" gives significantly bigger value than all the other measurements. If that measurement is left out of the calculations the average thermal conductivity value measured also with rubber sheets and thermocouples is 1,82 W/(m K). These two values are near to the published values which vary from 1,70 to 1,74 W/(m K).

The main reason why rubber sheets and thermocouples together gives the best result is that the method eliminates errors caused by air gaps and poor contact between the specimen's surfaces and the apparatus' plates. Also when using thermocouples, the real temperature of the specimen surface is measured. Without thermocouples the temperature is measured from the apparatus' plates which is not exactly the same as the temperature of the specimen.

Please note that the variation in results is caused by inhomogeneous material and specimens. The specimens were different even to the naked eye and it was clear that the amount of rock and cement varied in specimens: some had more large particles on their surface and some had a few or none. Since the maximum size of aggregate was 16 mm and specimens were either 10 mm or 20 mm thick, one could assume that there were not many large particles in the specimen if there were none to be seen on the surface. And since the thermal conductivity values of rock and cement are different (granite 2.8 W/m K and cement 0.7-1.0 W/m K), this content variation in specimens causes that the measured thermal conductivity values vary, too.

## 3.3. Specific heat capacity of concrete specimens

The results of the specific heat capacity tests of concrete specimens are presented in Fig. 3. There was different amount of specimens when defining specific heat capacity because the method is also different compared to thermal conductivity method and the major aim of this test was to find out if the heat flow apparatus is also suitable for measuring specific heat capacity of relatively small specimens made of inhomogeneous materials. That is why there were four specimens

#### Table 2

Thermal conductivity values of C20/25 concrete specimens. "Thick#" specimens were about 20 mm thick and "thin#" 10 mm thick.

Specimen	Thermal conductivity [W/(m K)]			
	Plain specimen	With rubber sheets	With thermocouples	With rubber sheets and thermocouples
Thick1	1.14	1.25	0.94	5.18
Thick2	0.34	0.66	0.54	1.86
Thick3	0.48	0.69	0.61	1.20
Thick4	0.64	0.86	0.92	1.53
Thick5	0.72	1.12	0.92	1.89
Thick6	0.80	1.16	1.01	1.75
Thick7	0.66	0.84	0.80	1.38
Thin1	0.74	0.88	0.83	1.70
Thin2	0.81	1.07	0.94	2.33
Thin3	0.77	1.03	1.06	2.11
Average	0.71	0.96	0.86	2.09

#### Table 3

Thermal conductivity values of C32/40 concrete specimens. "Thick#" specimens were about 20 mm thick and "thin#" 10 mm thick.

Specimen	Thermal conductivity [W/(m K)]			
	Plain specimen	With rubber sheets	With thermocouples	With rubber sheets and thermocouples
Thick1	0.72	0.92	0.74	2.34
Thick2	0.59	0.68	0.89	2.08
Thick3	0.50	0.73	0.70	1.68
Thick4	0.41	0.72	0.57	1.86
Thick5	0.75	1.00	0.87	1.84
Thick6	0.60	0.82	0.84	1.36
Thick7	0.65	1.06	0.90	1.79
Thick8	0.60	0.75	0.85	1.52
Thin1	0.67	0.95	0.82	1.91
Thin2	0.54	0.69	0.99	1.16
Thin3	0.99	1.24	0.94	2.41
Thin4	0.84	1.10	1.01	1.91
Average	0.65	0.89	0.84	1.82







Fig. 2. Thermal conductivity values of C32/40 concrete specimens. "Thick#" specimens were about 20 mm thick and "thin#" 10 mm thick.

chosen randomly out of all the same kind of specimens in order to represent actual measurement where the specimens used in tests cannot be chosen. In specific heat capacity measurements, the computer is calculating the time used to raise the temperatures to steady state and recording the cumulative amount of energy entering the specimen while temperatures are trying to reach the steady state. With this information the program calculates specific heat capacity value for the specimen.



Fig. 3. Specific heat capacity values of concrete specimens in different temperatures. Presented specific heat capacity values have been divided by density.

The specimens were conditioned before tests and material humidity of the tested specimens was 50 ( $\pm 10$ ) % RH. Table 4 shows the rounded specific heat capacity values for concrete at 25 °C. The last column in the Table 4 contains the volumetric specific heat capacity values that have been divided by density (J/(kg K)), whereas the preceding column has the measured value in  $J/(m^3 K)$ . Accurate specific heat capacity results of concrete specimens at 25 °C (average value) range between 715-841 J/(m<sup>3</sup> K). This is close to published values which vary from 840 to 1170 J/(m<sup>3</sup> K) [24]. Specific heat capacity value 1,000 J/(m<sup>3</sup> K) is given for all concrete densities in standard [25]. Even if the densities of different concrete specimens are quite the same specific heat capacity values can vary because of inhomogeneous materials. The relatively small specimens contain a big share of rock which can have a significantly smaller specific heat capacity value than cement [1,19]. That is one reason why measured specific heat capacity values are a bit lower than published values. Also, the values increase quite steadily as the temperature rises.

# 3.4. Specific heat capacity of plaster specimens

The average results of the specific heat capacity tests of plaster specimens as the test temperature rises are presented in Fig. 4.

The numerical results of the specific heat capacity tests of plasters are presented in Table 2. It is noticeable that plasters have similar values with their kind, i.e. the values of different floor screed plasters are close to one another and same with fixing plasters and wall screed plasters. The only clear exception in its group is Plaster W6 but compared to other wall screed plasters its composition was very fine, powdery.

Accurate results of plaster specimens range between 820 and 1,370 J/(kg K). In theory, materials with a larger particle size should get lower specific heat capacity values [1,19]. Here plasters whose maximum size of aggregate was 1 mm or larger had accurate values of 819–1009 J/(kg K), and those which had under 1 mm had 846–1,237 J/(kg K) (note: only one below 1,000 J/(kg K)). Published values are 1,000 J/(kg K) for lighter plasters and 900 J/(kg K) for denser sandy plasters [24].

Fig. 5 presents the relation between specific heat capacity and the maximum particle size of plasters.

## 3.5. Uncertainty of the methods

Uncertainty of the methods can be calculated when the quantities which are used in calculations of thermal conductivity and specific heat capacity are known. The manufacturer of the apparatus provided uncertainty values for measured quantities. Surface temperatures of samples can be measured within 0.01 °C and heat flux within 0.6  $\mu$ V. The thickness of the specimen is determined within ±0.025 mm resolution and the apparatus can maintain temperature difference

#### Table 4

Specific heat capacity values of concrete at 25 °C. Material humidity was (50 ± 10) % RH. The presented values have been rounded but the accurate values have been used in calculations.

Concrete sample	Density $\rho$ [kg/m <sup>3</sup> ]	Specific heat capacity $c_{\mathbf{p}}$ at 25 °C [J/(m <sup>3</sup> K)]	Specific heat capacity $c_{\mathbf{p}}at \ 25 \ ^{\circ}C \ [J/(kg \ K)]$
C20/25_sample 1	2260	$1.86 \cdot 10^{6}$	800
C20/25_sample 2	2250	$1.60 \cdot 10^{6}$	700
C20/25_sample 3	2200	$1.73 \cdot 10^{6}$	800
C20/25_sample 4	2260	$1.81 \cdot 10^{6}$	800
C32/40_sample 1	2250	$1,89 \cdot 10^{6}$	850
C32/40_sample 2	2240	$1.81 \cdot 10^{6}$	800
C32/40_sample 3	2240	$1.72 \cdot 10^{6}$	750
C32/40_sample 4	2250	$1.81 \cdot 10^{6}$	800



Fig. 4. Specific heat capacity values of plaster specimens in different temperatures Presented specific heat capacity values have been divided by density.



Fig. 5. The relation between specific heat capacity and maximum particle size of plasters. The maximum particle size of plaster O1 was unknown.

between the plates with the accuracy of  $\pm 0.02$  °C. The error estimation for the calibration factor is calculated by manufacturer of the apparatus and it is 3.27%. With these values manufacturer has calculated thermal conductivity error estimation which is 3.5%. Overall the given absolute accuracy for the apparatus is  $\pm 5\%$  and reproducibility  $\pm 2\%$  [13].

# 4. Conclusions and discussion

In this research, both thermal conductivity and specific heat capacity tests on concrete were made as well as specific heat capacity tests on different plasters.

In order to obtain reliable results, it is important to use the correct and the most suitable measurement method in the thermal conductivity tests, as can be noted by comparing different methods in Figs. 1 and 2. In these figures three out of four methods give results that are close to each other but the fourth one gives results of a correct magnitude. Please note that the variation in results in Figs. 1 and 2 is caused by inhomogeneous material and specimens. The apparatus is designed for testing of relatively small specimens which in case of inhomogeneous materials might cause problems if the specimens have any imperfections since these imperfections may become more obvious. Significance of inhomogeneous of the specimens is accentuated by that the maximum size of aggregate of concrete here was 16 mm and specimen thickness bare 10 mm or 20 mm which means that specimens that happened to have larger particles differ significantly from those that happened to have none. However, one of the goals of this study was to examine how well the apparatus could test inhomogeneous materials and therefore this variation in specimens was not considered to be a too crucial deficiency but rather an important observation point.

When doing actual thermal conductivity tests with a heat flow meter apparatus the method should be chosen accurately. This study with the recommendation of the apparatus manufacturer shows that the best way to measure the thermal conductivity of tested specimens is to use both the rubber sheets and thermocouples because it is the best way to eliminate errors caused by air caps and poor contact between the specimen's surfaces and the apparatus's plates especially with small specimens with large maximum particle size. It is recommended to test at least ten specimens when testing thermal conductivity of inhomogeneous materials and to consider the test method every time when measuring new material.

Overall, thermal conductivity results were in accordance with published values and theory when using both the rubber sheets and thermocouples. The average thermal conductivity value for the C20/25 concrete measurement without one value which is significantly bigger than the others is 1,75 W/(m K) and for the C32/40 concrete 1,82 W/(m K). Published values vary from 1,70 to 1,74 W/(m K) [24,25]. That indicates that the apparatus is suitable for testing inhomogeneous materials such as concrete. At the same time, the results vary significantly between specimens (results of C20/25: 1.20-2.32 (5.18) W/(m K) and C32/40: 1.16-2.41 W/(m K)) as seen in Figs. 1 and 2 due to that some

Table 5

Specific heat capacity values of plasters at 25 °C. Material humidity was (50  $\pm$  10) % RH. The presented values have been rounded but the accurate values have been used in calculations.

Material	Density ρ [kg/m <sup>3</sup> ]	<b>Specific heat</b> capacity c <sub>p</sub> at 25 °C [J/(m <sup>3</sup> K)]	<b>Specific heat</b> <b>capacity c<sub>p</sub>at 25 °C</b> [J/(kg K)]
Plaster FL1	2 130	$1.95 \cdot 10^{6}$	900
Plaster FL2	2 070	$1.97 \cdot 10^{6}$	950
Plaster FL3	2 000	$2.02 \cdot 10^{6}$	1 000
Plaster FL4	1 770	$1.91 \cdot 10^{6}$	1 100
Plaster FL5	1 930	$2.06 \cdot 10^{6}$	1 050
Plaster FIX1	1 240	$1.37 \cdot 10^{6}$	1 100
Plaster FIX2	1 570	$1.68 \cdot 10^{6}$	1 050
Plaster W1	1 710	$1.48 \cdot 10^{6}$	850
Plaster W2	1 490	$1.25 \cdot 10^{6}$	850
Plaster W3	1 180	$0.97 \cdot 10^{6}$	800
Plaster W4	1 320	$1.13 \cdot 10^{6}$	850
Plaster W5	1 230	$1.04 \cdot 10^{6}$	850
Plaster W6	1 520	$1.87 \cdot 10^{6}$	1 250
Plaster O1	470	$0.51 \cdot 10^{6}$	1 050

specimens have more and some fewer pieces of rock on the surface and inside them. Thermal conductivity of rock and cement differ from each other (granite 2.8 W/m K and cement 0.7-1.0 W/m K). The same inhomogeneity of the specimens explains also why 10 mm and 20 mm thick specimens' results have differences between them. Because of this the reliability of results must be evaluated whenever tests are done.

The main novelty value of this research is that heat flow meter apparatus can determine specific heat capacities of inhomogeneous materials well as shown in Table 5 and Fig. 4 if the test method has been chosen correctly. The measured specific heat capacities comply with the theory that materials that have a larger particle size should have lower specific heat capacity values [1,19], as illustrated in Fig. 5. The results also follow the theory that specific heat capacity should increase steadily as the temperature rises [1,19], as shown in Fig. 4. However, the values of some plasters do not rise perfectly steadily. One must bear in mind that the accuracy of the apparatus is  $\pm 5\%$ , so there might be some imprecisions in the measurements that might explain why the rise is not perfectly steady.

Overall, specific heat capacity results of concrete and plasters were in accordance with published values and theory. That indicates that the apparatus is suitable for testing inhomogeneous materials such as those tested here.

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