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Effect of Moisture on Thermal Conductivity of Lime-Based Composites

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Abstract The effect of moisture content on the thermal conductivity of lime-based composites is investigated both experimentally and theoretically. Experimental measurements of thermal conductivity as a function of moisture content from a dry state to a fully water saturated state are performed using an impulse technique. The obtained experimental data are analyzed at first with fundamental theoretical models, and it is found that they conform to both Wiener's and Hashin–Shtrikman's bounds. The thermal conductivity of wet lime-based composites is then analyzed using several homogenization techniques, among them, Lichtenecker's, Polder and van Santen's, and Dobson's formulas. The validity of applied effective-media treatments is assessed comparing the measured and calculated data. Lichtenecker's equation is found to achieve the best agreement with experimental measurements.

Keywords Homogenization procedure · Impulse technique · Lime-based composites · Moisture content · Thermal conductivity

1 Introduction

The envelope of any building continuously responds to changes in indoor and outdoor temperatures, air pressure, and humidity conditions. This results in an exchange of energy and mass (air as well as moisture) between the indoor and outdoor environments through the envelope. Building physicists refer to these phenomena as "heat, air, and moisture transport" through building materials and structures [1]. Designers and builders are always interested, especially for economical reasons and durability and

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service-life problems, in knowing the long-term performance of a building envelope, as subjected to transport processes.

Thermal properties of building materials appear to be of particular importance for their practical applications, whereas the majority of them contains significant amount of pores that can be, in specific cases, filled by water. Every catalog list of any material producer of building materials contains thermal conductivity, sometimes also specific heat capacity, but they give only single characteristic values that mainly represent properties of dry materials. However, absolutely dry materials occur at the conditions of building sites very rarely. Also, the materials already inbuilt in the structures and exposed to climatic loading exhibit the dependence of their properties on moisture changes. If the material is wet, heat transferred by moisture in the capillaries adds to the density of the heat flow rate. The thermal conductivity of water is $0.60\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ [2], which is more than 20 times higher than that of air. Therefore, if water is present in the pore space, its effect competes with the effect of air, and the thermal conductivity of a composite material can be considered as a result of this competition, together with the effect of the solid matrix [3]. So, the motivation for determination of the thermal conductivity of porous materials as a function of moisture content appears very significant. Since the experimental measurement of thermal properties as a function of moisture is very time consuming, new approaches are explored and tested in materials research.

In this article, we report on the application of homogenization theory for the assessment of the moisture-dependent thermal conductivity of lime-based composite materials. The measured values of thermal conductivity are analyzed using several types of homogenization formulas originally derived for application in electromagnetic field theory, namely, Lichtenecker's equation [4], mixing formulas proposed by Polder and van Santen [5], Looyenga's model [6] and Dobson's four-phase model [7]. The limiting Wiener's [8] and Hashin-Shtrikman's bounds [9] are taken into account as well.

2 Experimental Methods

The moisture content as well as thermal properties of porous building materials depends mainly on the pore structure of the tested material. Therefore, basic material properties of all tested lime-based plasters were studied. As fundamental physical material characteristics, bulk density $\rho_b (\mathrm{kg \cdot m^{-3}})$, matrix density $\rho_{\mathrm{mat}} (\mathrm{kg \cdot m^{-3}})$, and total open porosity $\psi (\%)$ were determined. The bulk density was measured using a gravimetric method. The matrix density was obtained using a Pycnomatic ATC, automatic helium pycnometer with fully integrated temperature control with a precision of $\pm\,0.01\,^{\circ}\mathrm{C}$ and a real multi-volume density analyzer. The samples for determination of the basic material parameters had dimensions of $40\ \mathrm{mm} \times 40\ \mathrm{mm} \times 20\ \mathrm{mm}$.

Thermal conductivity belongs together with thermal diffusivity and specific heat capacity as the most important material parameters for the description of the thermal transport properties of a material or component. The thermal conductivity can be experimentally determined either by steady-state methods or by transient methods. A measurement method has to be selected depending on the following criteria [10]: possible sample size and shape, temperature range (limited for individual techniques), and thermal-conductivity range (low-conductivity materials like insulating materials or foams need different methods than high-conductivity materials such as metals).

For our measurements, the commercially produced device ISOMET 2104 (Applied Precision, Ltd.) was used as a typical representative of transient impulse methods. ISOMET 2104 is a multifunctional instrument for measuring thermal conductivity, thermal diffusivity, and volumetric heat capacity. It is equipped with various types of optional probes; needle probes are for porous, fibrous, or soft materials; and surface probes are suitable for hard materials. In our case we have chosen the surface probe.

The measuring device applies a dynamic measurement method which enables reduction of the period of thermal-conductivity measurements to approximately (10 to 16) min. The measurement is based on analysis of the temperature response of the analyzed material to heat flow impulses. The heat flow is induced by electrical heating using a resistor heater having direct thermal contact with the surface of the sample. A built-in menu system with four-line alphanumeric display enables effective interactive communication with the device and recalibration of measurement probes by means of reference materials. Calibration data in the internal memory ensure interchangeability of probes without affecting the measurement accuracy [11]. The basic measuring uncertainty of ISOMET 2104 for thermal conductivity measurements is 3 % of reading $+0.001\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$.

The measurements in this article were done in laboratory conditions at an average temperature of (23 ± 1) °C. The material samples were first dried, and after that, exposed to liquid water for specific time intervals. In this way, the different moisture contents of the studied samples were reached. The sample size for thermal conductivity measurement was 70 mm × 70 mm × 70 mm.

3 Studied Materials and Samples

Five different types of lime-based composites that should find use as plasters in the restoration and reconstruction of historical buildings were investigated. As the chemical analyses of many plasters from historical buildings show, the past centuries' external plasters that are preserved until today contained products formed by lime reaction with pozzolanic or hydraulic admixtures. Pozzolanic admixtures appeared to have a positive effect on properties of lime binder in the past [12]. On this account, metakaolin as a pozzolanic admixture was used as a partial binder replacement in mixtures denoted S1–S4. Mixture S5 was produced as a reference lime-based composite without metakaolin admixture. The compositions of mixtures for sample preparation are presented in Table 1.

Four different types of lime hydrate CL 90 were used. The lime hydrates were produced by Czech limekilns in Mokrá, Štramberk, Vitošov, and Čertovy Schody. Metakaolin MEFISTO K05 is a product of "České lupkové závody Inc., Nové Strašecí". The reason for using MEFISTO in lime plasters is the supposed increase of compressive and flexural strength and frost resistance, decrease of water absorption, and reduction of the occurrence of efflorescence. The average particle size of metakaolin is in the interval of 3 μm to 5 μm. As aggregates, a mixture of silica and basalt sands was used. The mass contents of particular sand fractions are given in Table 2.

Table 1 Compositions of the studied lime-based plaster mixtures

Type of mixture	Lime hydrate type	Mass (kg)			Water dosing per 10 kg of mixture (kg)
		Lime hydrate	Metakaolin	Aggregates	
S1	Mokrá	2.08	0.48	7.5	2.5
S2	Štramberk	2.08	0.48	7.5	2.5
S3	Vitošov	2.08	0.48	7.5	2.5
S4	Čertovy Schody	2.08	0.48	7.5	2.5
S5	Mokrá	2.50	0	7.5	2.5

Table 2 Contents of particular aggregate fractions in percentage of mass

Material	Fraction	(%)
Silica sand	PR 33C	25
	PR 33H	30
	PR 1-2	25
	PR 1.6-4	10
Basalt sand	PDK 2/4	10

Plaster mixtures were prepared using a laboratory mixing machine with forced rotation for 3 min and then compacted using a vibrating machine. Each mixture was cast into a standard prism form; after 2 days all prisms were taken out of the forms and then cured for 28 days in a high relative humidity environment.

4 Homogenization Procedure

In terms of a homogenization procedure, a porous material can be considered basically as a mixture of three phases, namely, solid, liquid, and gas [13] (in four-phase systems, the effect of bound water can be included) that form the solid matrix and porous space of the material. The solid phase is formed by the materials of the solid matrix. The liquid phase is represented by water and the gaseous phase by air. In the case of a dry material, only the solid and gaseous phases are considered. The volumetric fraction of air in a porous body is given by the measured total open porosity. In case of penetration of water, a part of the porous space is filled by water. For evaluation of the thermal conductivity of the whole material (i.e., the effective thermal conductivity), the thermal conductivities of the particular constituents forming the porous body have to be known.

In the lime-based plasters studied in this article, the solid phase is formed by the products of joint hydration of lime hydrate CL 90 and metakaolin and by the aggregates. The homogenization was performed in two steps. At first, it was necessary to determine the thermal conductivity of the solid matrix of particular studied materials. In this study, the thermal conductivity of the solid matrix was calculated using the Rayleigh [14] mixing rule,

$$\frac{\lambda_M - 1}{\lambda_M + 2} = f_s\left(\frac{\lambda_s - 1}{\lambda_s + 2}\right) + f_c\left(\frac{\lambda_c - 1}{\lambda_c + 2}\right) + f_b\left(\frac{\lambda_b - 1}{\lambda_b + 2}\right), \tag{1}$$

where λ_M is the thermal conductivity of solid lime-based matrix, λ_s is the thermal conductivity of silica aggregates ($3.5\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$), λ_c is the thermal conductivity of hydrated lime—calcium carbonate ($5.5\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$), λ_b is the thermal conductivity of basalt aggregates ($3.0\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$), f_s is the volumetric fraction of silica aggregates, f_c is the volumetric fraction of calcium carbonate, and f_b is the volumetric fraction of basalt aggregates. The values of thermal conductivities of particular components of lime-based composites were taken from the CRC Handbook of Chemistry and Physics [2].

The second step within the homogenization procedure represents evaluation of the thermal conductivity of the whole material, where the mixing is performed for the solid matrix, air, and water.

The effective thermal conductivity λ_{eff} of a multi-phase composite cannot exceed the bounds given by the thermal conductivities and volumetric fractions of its constituents. The upper bound is reached in a system consisting of plane-parallel layers disposed along the heat flux vector. The lower bound is reached in a similar system but with the layers perpendicular to the heat flux. These bounds are usually called Wiener's bounds, according to Wiener's original study [8] and can be expressed by the following relations:

$$\lambda_{\text{eff}} = \frac{1}{\frac{f_1}{\lambda_1} + \frac{f_2}{\lambda_2} + \frac{f_3}{\lambda_3}}, \tag{2}$$

$$\lambda_{\text{eff}} = f_1\lambda_1 + f_2\lambda_2 + f_3\lambda_3, \tag{3}$$

where Eq. 2 represents the lower limit and Eq. 3 the upper limit of the effective thermal conductivity (f_j is the volumetric fraction of the particular phase, λ_j its thermal conductivity).

For verification and certain limitation of the effective thermal conductivity values, the Hashin–Shtrikman's bounds [9] can also be applied, which were originally derived for theoretical determination of the effective magnetic permeability of macroscopically homogeneous and isotropic multiphase materials. In [9], the authors conclude that for reasons of mathematical analogy, the derived bounds also hold for the dielectric constant, electrical conductivity, thermal conductivity, and mass diffusivity of such materials.

Hashin–Shtrikman's bounds were originally proposed for two-phase systems only. However, their extension to three- or four-phase systems is quite simple. The lower limit of the effective thermal conductivity can be expressed as:

$$\lambda_{\text{lower}} = \lambda_1 + \frac{3\lambda_1}{\frac{1}{\sum_{i=2}^{n} f_i \frac{\lambda_i - \lambda_1}{2\lambda_1 + \lambda_i}} - 1}, \tag{4}$$

and the upper limit as

$$\lambda_{\mathrm{upper}} = \lambda_n + \frac{3\lambda_n}{\frac{1}{\sum_{i=1}^{n-1} f_i \frac{\lambda_i - \lambda_n}{2\lambda_n + \lambda_i}} - 1}. \tag{5}$$

In Eqs. 4 and 5, $f_1 – f_n$ are the volumetric fractions of the particular phase ($f_1 + f_2 + \cdots + f_n = 1$), and $\lambda_1 - \lambda_n$ are their thermal conductivities, whereas $\lambda_1 < \lambda_2 \cdots < \lambda_n$.

The mixing of phases resulting in effective thermal conductivity functions falling between Wiener's and Hashin–Shtrinkman's bounds can be done using many different techniques. In this study, the mixing was done by Lichtenecker's equation [4], Polder and van Santen's models [5] derived from the original Bruggeman's formula [15], and Dobson's four-phase model [7] taking into account the effect of bound water. All these models and formulas were successfully applied by various scientists especially for dielectric mixing in the past, and we introduce them modified for thermal-conductivity calculations. Only self-consistent formulas were used as they allowed modeling the material behavior in a sufficiently wide moisture range.

Lichtenecker's equation [4],

$$\lambda_{\mathrm{eff}}^k = \sum f_j \lambda_j^k \tag{6}$$

is a straightforward generalization of Wiener's formulas. The parameter k in Eq. 6 varies within the $[-1, 1]$ range. Thus, the extreme values of k correspond to the Wiener's boundary values. The parameter k may be considered as describing a transition from the anisotropy at $k = -1.0$ to another anisotropy at $k = 1.0$. However, Lichtenecker's equation may be also applied for isotropic composites. Note also that Eq. 6 is consistent with some other approximations (e.g., Looyenga [6] with $k = 1/3$).

Polder and van Santen [5] extended the Bruggeman formula [15] to elliptical inclusions and formulated its three useful simplifications (given in somewhat different algebraic form). First, the original one, is valid for spherical inclusions; Second for needle-shape inclusions; and the third for their board shape. The resulting mixing formulas can be written as:

$$\lambda_{\mathrm{eff}} = \lambda_{\mathrm{M}} + \sum f_j (\lambda_j - \lambda_{\mathrm{M}}) \frac{3\lambda_{\mathrm{eff}}}{2\lambda_{\mathrm{eff}} + \lambda_j}, \tag{7}$$

$$\lambda_{\mathrm{eff}} = \lambda_{\mathrm{M}} + \sum f_j (\lambda_j - \lambda_{\mathrm{M}}) \cdot \frac{5\lambda_{\mathrm{eff}} + \lambda_j}{3\lambda_{\mathrm{eff}} + 3\lambda_j}, \tag{8}$$

$$\lambda_{\mathrm{eff}} = \lambda_{\mathrm{M}} + \sum f_j (\lambda_j - \lambda_{\mathrm{M}}) \cdot \frac{2\lambda_j + \lambda_{\mathrm{eff}}}{3\lambda_j}. \tag{9}$$

Because of the large difference between the thermal conductivity of free and bound water in porous medium, Dobson et al. [7] extended Lichtenecker's [4] power-law formula and arrived at the relation,

$$\theta = \frac{\lambda_{\mathrm{eff}}^{\beta} - \theta_{\mathrm{bw}} (\lambda_{\mathrm{bw}}^{\beta} - \lambda_{\mathrm{fw}}^{\beta}) - (1 - \psi)\lambda_{\mathrm{s}}^{\beta} - \psi \lambda_{\mathrm{a}}^{\beta}}{\lambda_{\mathrm{fw}}^{\beta} - \lambda_{\mathrm{a}}^{\beta}}, \tag{10}$$

Table 3 Basic material properties of studied materials

Material	ρ (kg · m^{-3})	ρ_{mat} (kg · m^{-3})	ψ (%)
S1	1637	2478	33.9
S2	1716	2669	35.7
S3	1756	2582	35.4
S4	1767	2638	33.2
S5	1745	2663	34.5

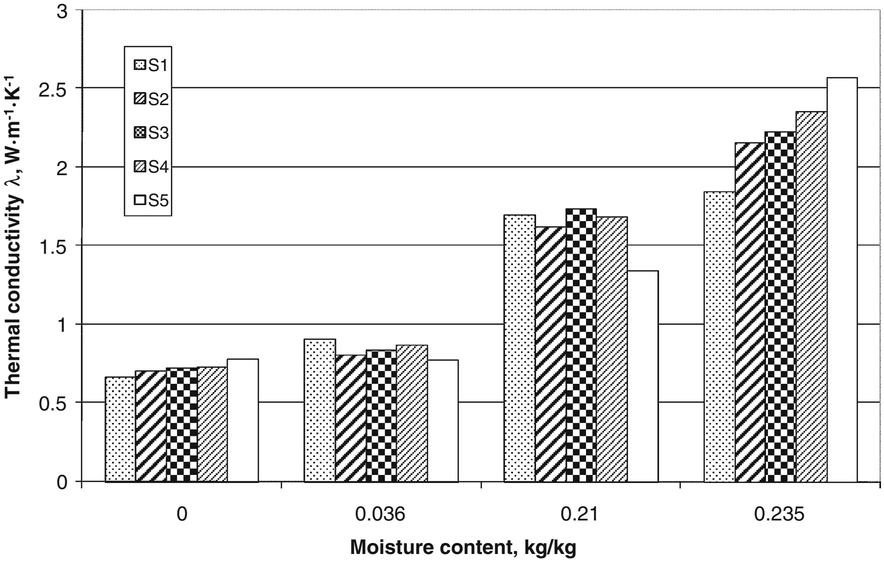

Fig. 1 Experimentally determined thermal conductivity of the studied materials as a function of moisture content

where θ_{bw} is the amount of water bonded on pore walls (m^3/m^3), λ_{bw} is the thermal conductivity of bound water (according to [16], the bound water can be assumed to have the same thermal conductivity as ice, so near −20 °C it is equal to $2.4\,\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$), λ_{fw} is the thermal conductivity of free water ($0.6\,\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$), λ_{a} is the thermal conductivity of air ($0.026\,\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$), ψ is the total open porosity, and β is an empirical parameter.

5 Results and Discussion

Table 3 shows the basic material properties of the studied lime-based composites. We can see that only material S1 exhibits a certain difference in comparison with the other studied materials. The total open porosity of the tested materials is quite high and gives a good prerequisite for application in restoration of the historical building facades.

Experimental results of the moisture-dependent thermal conductivity of studied materials are presented in Fig. 1. The measured data give information on a very substantial effect of the moisture content on the thermal conductivity of the studied material

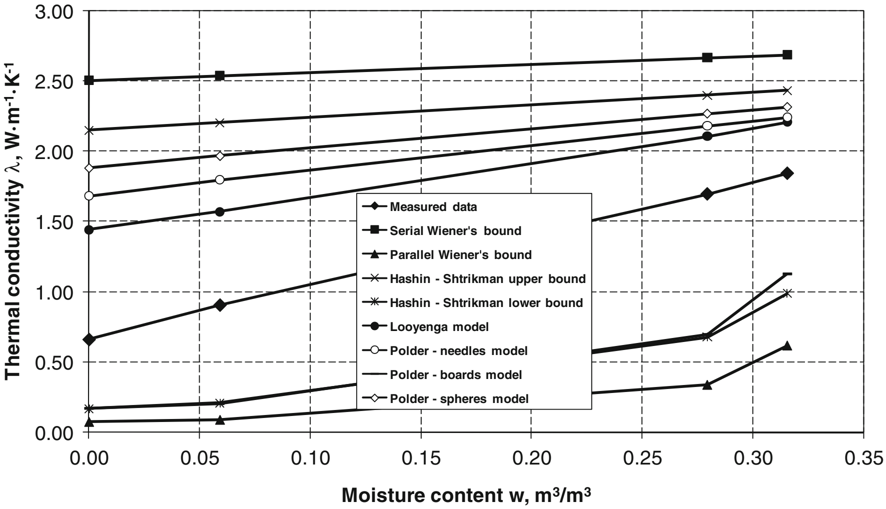

Fig. 2 Measured and calculated thermal conductivity of the lime-pozzolana plaster S1

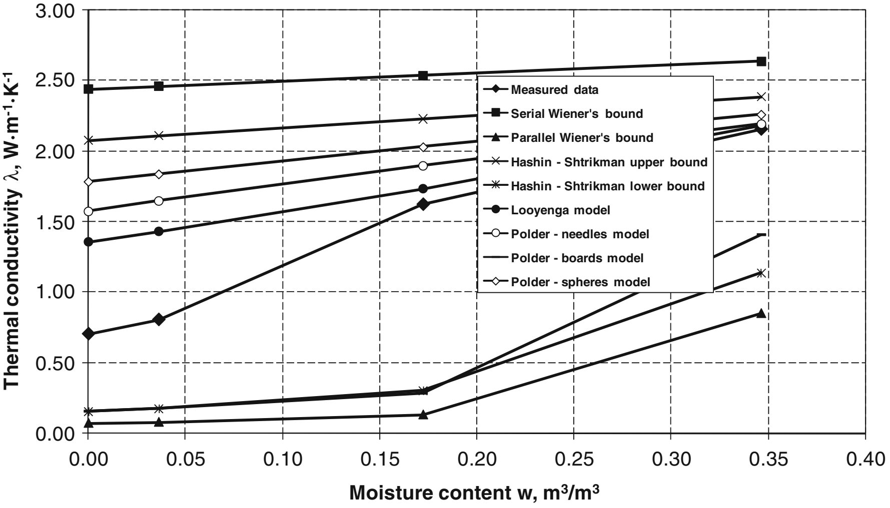

Fig. 3 Measured and calculated thermal conductivity of the lime-pozzolana plaster S2

which is in basic agreement with its relatively high total open porosity. The thermal-conductivity values for water-saturated samples are approximately three times higher than those for dry materials.

In Figs. 2, 3, 4, 5, and 6, thermal conductivity versus moisture content functions for particular studied materials are presented which are calculated using different types of the above-described homogenization models and mixing formulas. The limiting Wiener's and Hashin–Shtrikman's bounds are presented as well. From the point of view of Wiener's and Hashin–Shtrikman's bounds, we can see that the calculated

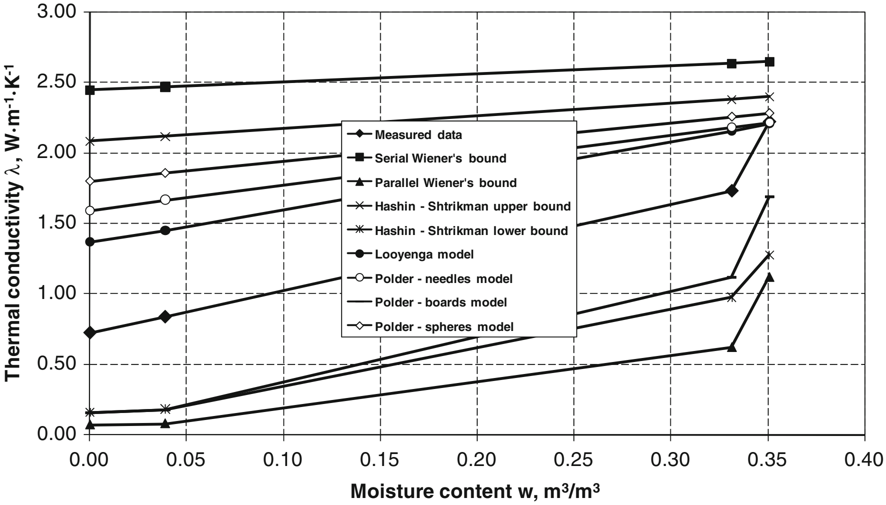

Fig. 4 Measured and calculated thermal conductivity of the lime-pozzolana plaster S3

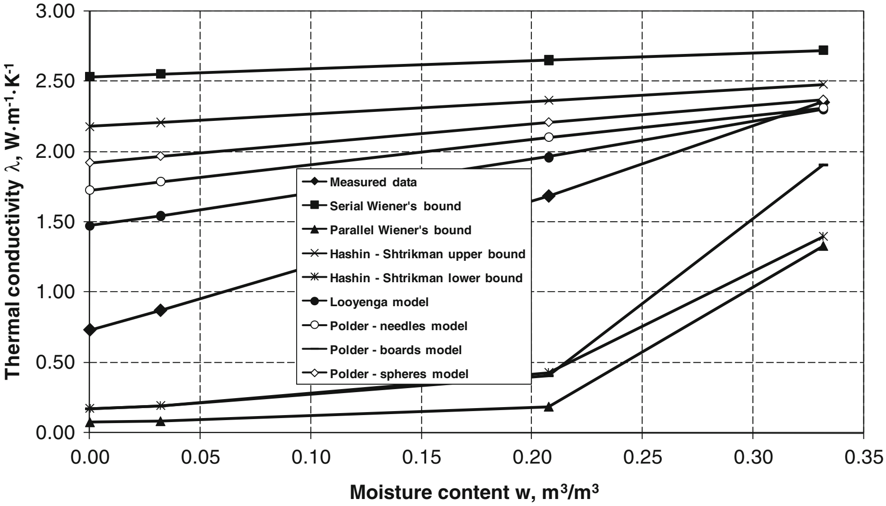

Fig. 5 Measured and calculated thermal conductivity of the lime-pozzolana plaster S4

results as well as the experimentally measured data lie between the serial and parallel models, which basically justify the reasonable accuracy of both experiment and calculations. Looking at the data from the point of view of accuracy of the applied homogenization techniques, we can see large differences between the particular types of formulas, shapes of inclusions in the case of Polder and van Santen's model, and measured results. The highest differences can be found for lower moisture contents, whereas for higher moisture contents, the measured and calculated values are in slightly better agreement. The differences in the range of lower moisture contents are probably caused by the inaccuracies in the thermal conductivity values of the particular

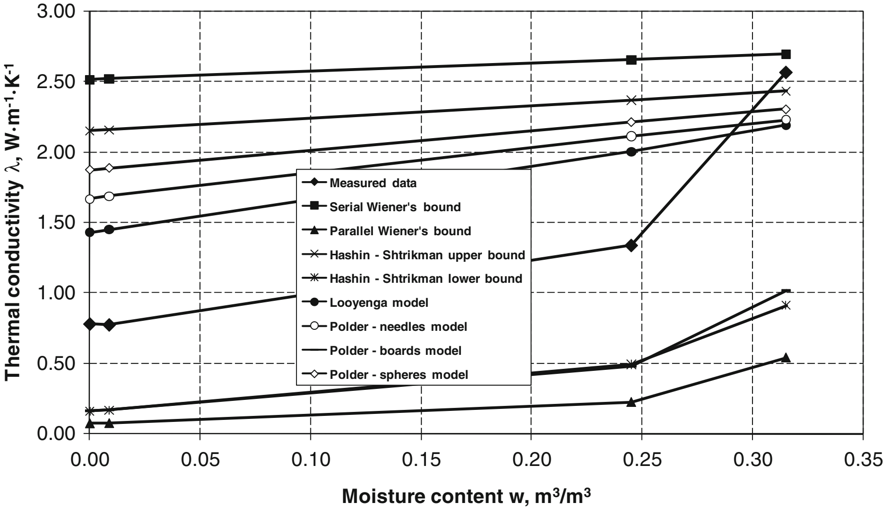

Fig. 6 Measured and calculated thermal conductivity of the reference pure lime plaster S5

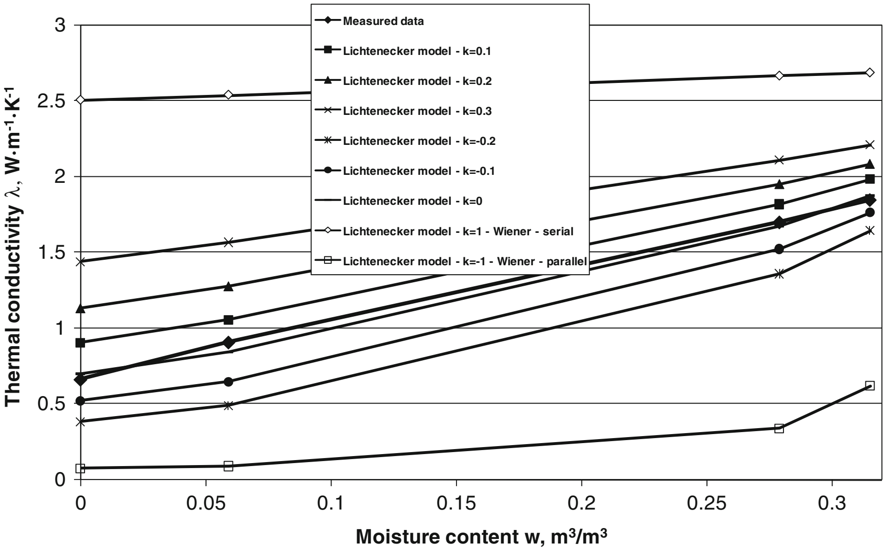

Fig. 7 Thermal conductivity of the lime-pozzolana plaster S1 calculated by Lichtenecker's equation

components forming the matrix of the material taken from the CRC Handbook of Chemistry and Physics [2]. The better agreement in the range of higher moistures can be attributed to the higher effect of water thermal conductivity (measured with sufficient accuracy in many references) on the thermal conductivity of the studied lime-based composites.

In Polder and van Santen's models the effect of shape of applied inclusions was very significant. Systematically, the highest values of thermal conductivity were obtained

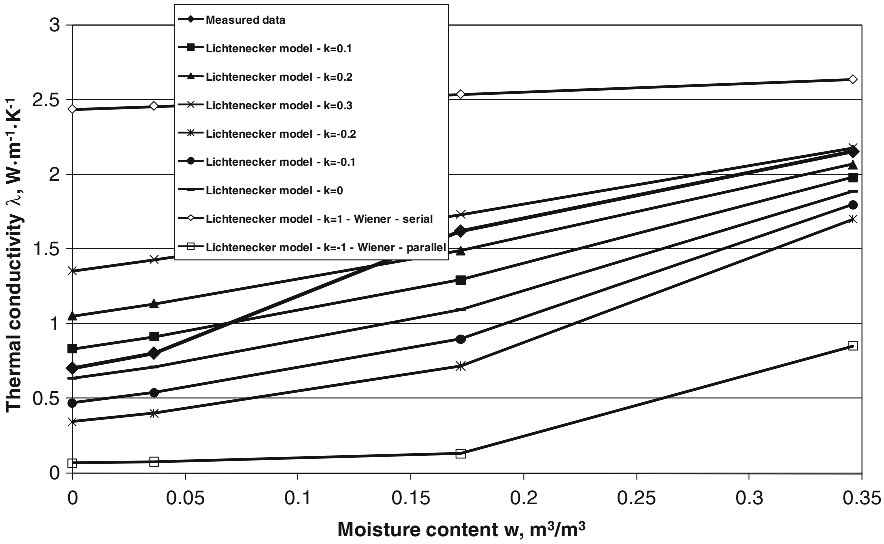

Fig. 8 Thermal conductivity of the lime-pozzolana plaster S2 calculated by Lichtenecker's equation

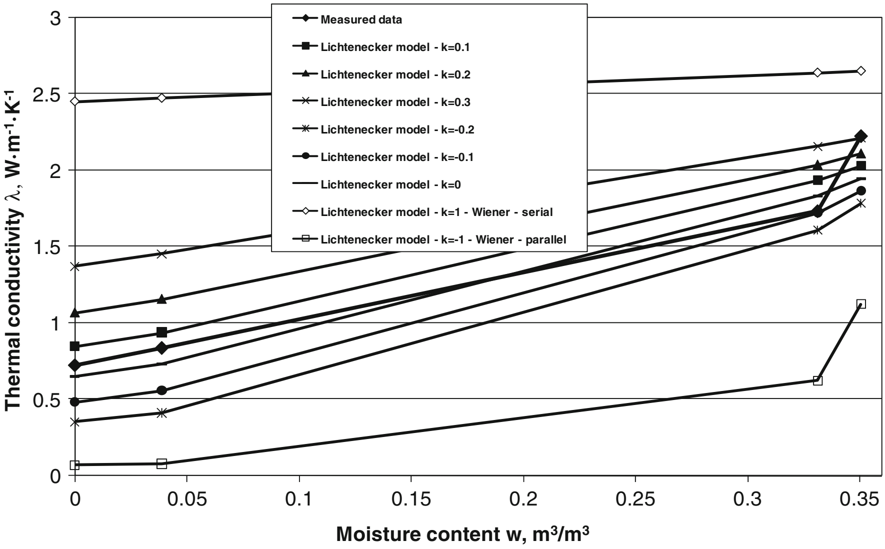

Fig. 9 Thermal conductivity of the lime-pozzolana plaster S3 calculated by Lichtenecker's equation

for the spherical and needle models, and the lowest values were reached using the board model. This feature we have already observed in our previous studies, e.g., in [13] and [17]. This is a logical result because the board-shaped inclusions should lead to results closer to the parallel model [17] than to the serial model (taken from the point of view of mixing thermal conductivities in Eqs. 2 and 3, for the thermal resistances the serial-parallel orientation is naturally quite opposite, i.e., the serial model

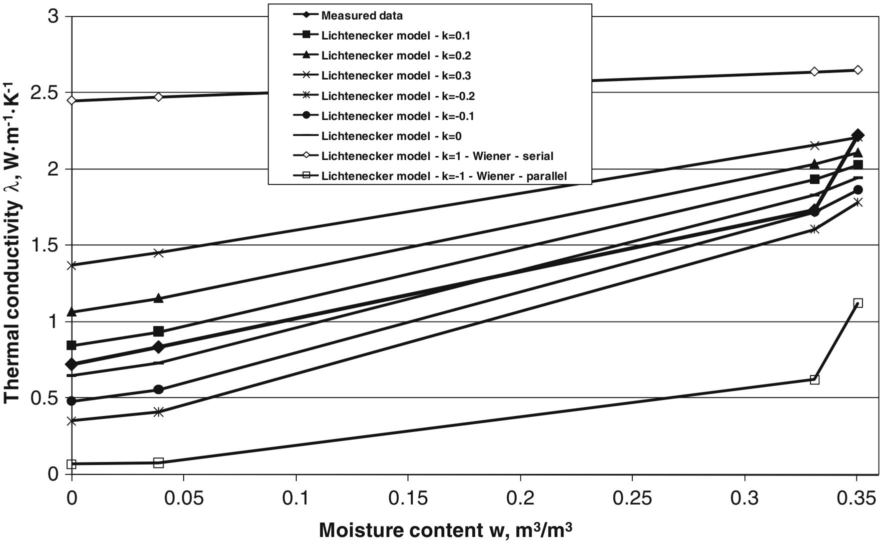

Fig. 10 Thermal conductivity of the lime-pozzolana plaster S4 calculated by Lichtenecker's equation

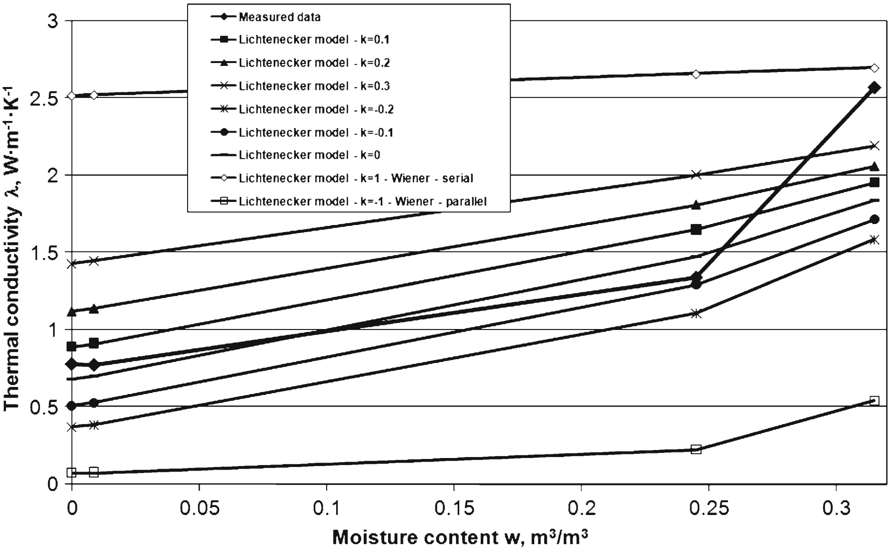

Fig. 11 Thermal conductivity of the lime-pozzolana plaster S5 calculated by Lichtenecker's equation

should be closer to the board-type inclusions). The measured data were found in all cases between the spherical and board-shaped inclusion variants of the Polder and van Santen's models. The very interesting finding is that Haskin–Shtrikman's bounds give in lower moisture contents almost the same values of thermal conductivity as the board-shape inclusion variant of Polder and van Santen's model.

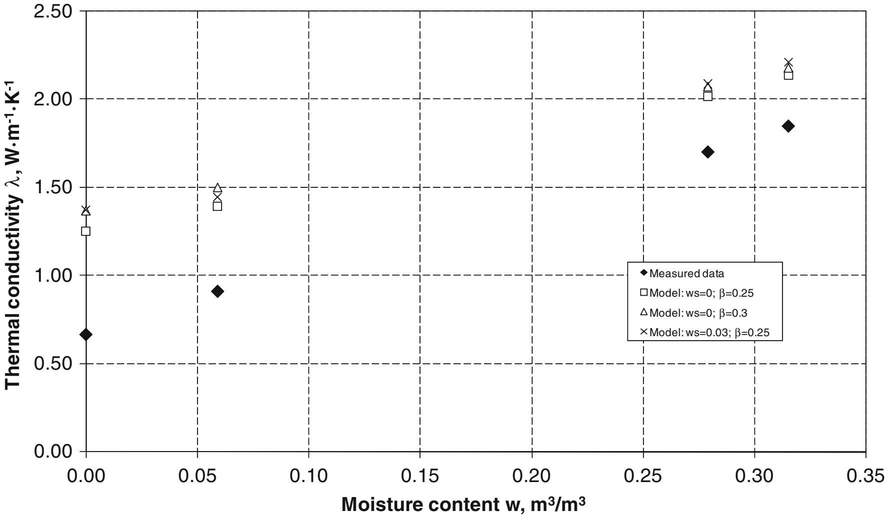

Fig. 12 Thermal conductivity of the lime-pozzolana plaster S1 calculated by Dobson's model

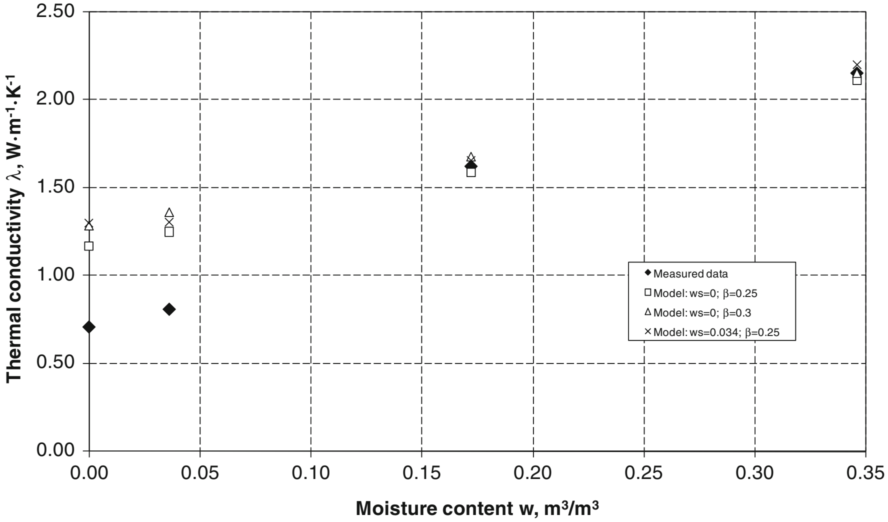

Fig. 13 Thermal conductivity of the lime-pozzolana plaster S2 calculated by Dobson's model

The results calculated by Lichtenecker's equation are presented together with measured data in Figs. 7, 8, 9, 10, and 11. The effect of fitting parameter k on the accuracy of obtained data is clearly demonstrated. Using the proper parameter k, we obtained the sufficient agreement of calculated and experimental results. In most cases the choice of $k \rightarrow 0$ led to the best results (application of the limiting procedure to Lichtenecker's formula (4) for $k \rightarrow 0$ results in): $\ln \lambda_{\mathrm{eff}} = \sum_{i=1}^{n} f_i \ln \lambda_i$.

Figures 12, 13, 14, 15, and 16 describe the results of the application of Dobson's model, where w_{s} is the amount of bound water in $(\mathrm{m}^3/\mathrm{m}^3)$ and β is an empirical

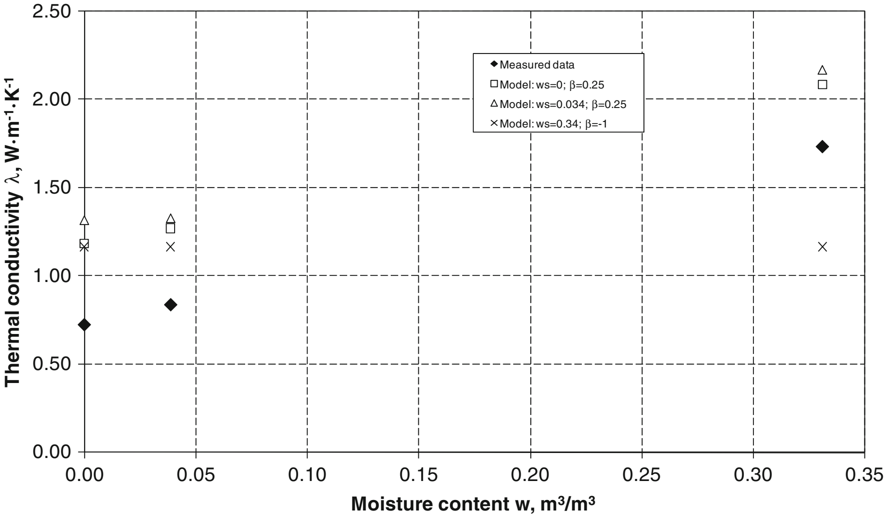

Fig. 14 Thermal conductivity of the lime-pozzolana plaster S3 calculated by Dobson's model

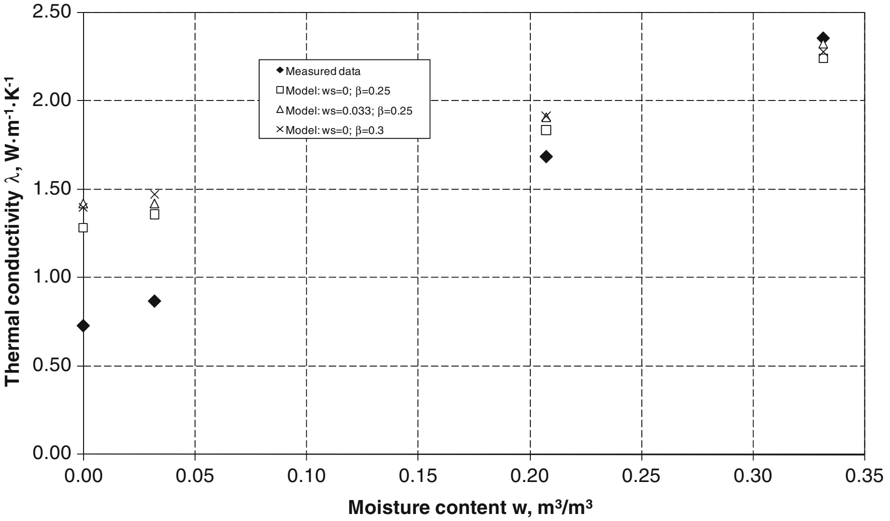

Fig. 15 Thermal conductivity of the lime-pozzolana plaster S4 calculated by Dobson's model

parameter. We can see that the thermal conductivity and amount of bound water had significant influence on the results. However, distinctively better results than for the three-phase models were not achieved. Systematically the better agreement with experimental data was observed again for higher moisture contents.

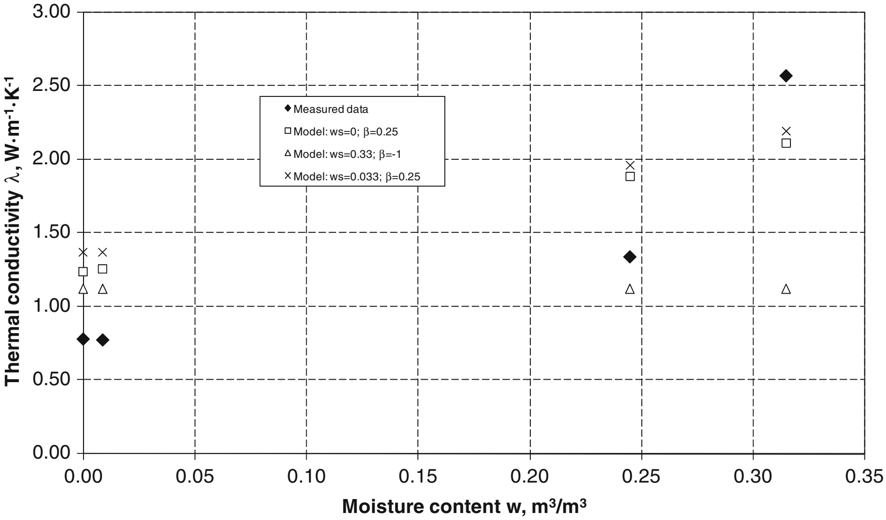

Fig. 16 Thermal conductivity of the lime-pozzolana plaster S5 calculated by Dobson's model

6 Conclusions

The results of theoretical and experimental investigations presented in this article have shown that effective media theory can be successfully used for estimation of the dependence of thermal conductivity of lime-based composites on moisture content. Lichtenecker's power-law formula in its limiting form was shown to work with reasonable accuracy for all five studied composites. The three-phase Lichtenecker model provided better results than its four-phase Dobson modification. The application of Polder–van Santen's formulas, which appeared useful in some previous applications for composite materials, was not a successful solution in this case.

Despite the relative success which was achieved in the application of homogenization procedures for thermal-conductivity estimates, it should be noted, however, that the theoretical treatment is not yet developed to such extent that it would be possible to make substantial generalizations immediately. An extension to other types of materials is still not a straightforward task, and in the current state of knowledge, it seems that some preliminary study will always be necessary if the gained experience is to be utilized for other porous materials.

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