WARSAW UNIVERSITY OF TECHNOLOGY Index 351733
FACULTY OF CIVIL ENGINEERING
COMMITTEE FOR CIVIL AND WATER ENGINEERING
POLISH ACADEMY OF SCIENCES ISSN 1230-2945

DOI: 10.24425/ace.2020.135214
ARCHIVES OF CIVIL ENGINEERING
Vol. LXVI ISSUE 4 2020

ANALYSIS OF THE THERMAL PROPERTIES AND STRUCTURE OF GYPSUM MODIFIED WITH CELLULOSE BASED POLYMER AND AEROGELS

K. PRAŁAT[1], R. JASKULSKI[2], J. CIEMNICKA[3], G. MAKOMASKI[4]

The work presents results of research on the influence of micro materials on the thermal conductivity λ of gypsum. In the research, cellulose-based polymer and aerogel were used as the modifying micro materials. For the purpose of measuring the thermal conductivity, a non-stationary method was used based on the "hot wire method". A very precise set of devices for measuring and recording the temperature of the heating wire was used. In the presented solution, a single measurement took only one minute. Measurements were recorded with the help of a computer measuring system, with a sampling time of 0.01s. During the 60-second-long test, 6000 measurements of the heating wire temperature were collected. A decrease of the thermal conductivity and density of hardened gypsum with added micro materials was observed due to modifications of the structure of the final product. Experimental values of the thermal conductivity of the gypsum specimens with the addition of polymer and aerogel were respectively over 23% and 6% lower than the non-modified gypsum specimen.

Keywords: thermal conductivity, micro additives, structure, hot wire method

[1] DSc., PhD., Eng., Warsaw University of Technology, Faculty of Civil Engineering, Mechanics and Petrochemistry, Ul. Łukasiewicza 17, 09-400 Płock, Poland, e-mail: Karol.Pralat@pw.edu.pl, Orcid: 0000-0001-5116-0379

[2] PhD., Eng., Warsaw University of Technology, Faculty of Civil Engineering, Mechanics and Petrochemistry, Ul. Łukasiewicza 17, 09-400 Płock, Poland, e-mail: Roman.Jaskulski@pw.edu.pl, Orcid: 0000-0002-2318-9323

[3] PhD., Eng., Warsaw University of Technology, Faculty of Civil Engineering, Mechanics and Petrochemistry, Ul. Łukasiewicza 17, 09-400 Płock, Poland, e-mail: Justyna.Ciemnicka@gmail.com, Orcid: 0000-0002-9022-7545

[4] PhD., Eng., Warsaw University of Technology, Faculty of Civil Engineering, Mechanics and Petrochemistry, Ul. Łukasiewicza 17, 09-400 Płock, Poland, e-mail: Grzegorz.Makomaski@pw.edu.pl, Orcid: 0000-0002-5172-424X

1. INTRODUCTION

In accordance with the principles of sustainable development, more and more bi- or multifunctional building mortars are appearing on the building materials market. Such mortars combine the performance of at least two materials with different characteristics or functions. A good example of these types of mortars are thermal insulating gypsum mortars. They are characterized by good thermal insulation parameters, while also maintaining the very beneficial functional properties of standard mortars. Currently, a very wide field in this area is the search for insulation and building composites with low values of thermal conductivity coefficient λ. An innovative way to reduce this parameter is to create composite building materials using additives in form of micro particles. These additives, which are built into the structure of the substance, allow composites with new interesting thermal properties to be obtained [1,2].

1.1. LITERATURE REVIEW

In this chapter, selected literature items published in recent years related to the use of micro additives (up to 1 wt.%) in building materials have been reviewed. Although there are numerous applications of micro additives in concrete technology, there is a large lack of knowledge in the case of gypsum materials. Research work has led to the development of new thermal insulation materials with insulating properties that are much better than that of foamed polystyrene. Such materials are aerogels. The different available possibilities of applying aerogels in the building industry are presented in the paper [3]. Due to its thermal insulation properties, aerogel is currently the subject of some studies on the use of its potential in material engineering [3–6], but due to the relatively high price of this material, scientific research on aerogels as micro additives for building materials is quite limited. However, there are some works. The authors of papers [3,7–10] tested cement composites modified with aerogel and confirmed that this additive significantly improves the thermal properties of the modified materials. The authors of paper [11] observed changes in the thermal conductivity and mechanical properties of modified materials in terms of the percentage of application of hydrophobic aerogel particles in the mixture. Fickler [12] carried out research on high-strength aerogel-doped concrete.

In the paper of Strzałkowski and Garbalińska [13], the results of work on the application of aerogel as an additive to concrete are presented. The results of the conducted tests confirm that the addition of aerogel particles improves, in comparison with the control sample without aerogel, the thermal conductivity coefficient by placing it in the range of 0.36-0.53 W/(m·K).The thermal conductivity of the control sample was λ = 1.198 W/(m·K).

Different types of polymers are used in manufacturing of building materials. Some studies on the influence of polymeric additives to cement [14,15] and concrete [16] were carried out. It was noticed that the addition of inorganic polymers causes an increase in the size of pores in the material, which results in a change in thermal conductivity. An attempt to determine the influence of polymer addition on the thermal conductivity of gypsum was made by Heim et al. [17]. In order to determine the effect of the additive on the thermal properties of gypsum, gypsum-based composites with a 0.1% and 1% mass of polymer (hydroxylethylmethylcellulose - HEMC) were prepared. Thermal conductivity was measured using the "hot wire" method. Results showed that the 1% of polymer in gypsum specimens causes a decrease of thermal conductivity by more than 20% when compared with the specimens without its presence. The thermal conductivity values of the modified samples were within the range of 0.29÷0.36 W/(m·K). The authors of the paper suggested a continuation of the studies with different amounts of polymer in the samples in relation to the applied gypsum (p/g), and also a different weight ratio of water to gypsum (w/g).

The authors of paper [18] investigated the influence of cellulose ethers (methyl cellulose) on the properties of gypsum building materials. They showed that despite its small addition of up to 0.4% in relation to the base material, the effect on the physical properties and microstructure of mortars is very high. However, in the paper no attempt was made to assess the influence of the applied additive on the thermal properties of modified gypsum.

In paper [19], the authors presented the results of laboratory experiments concerning the influence of the viscosity of hydroxypropylmethylcellulose (HPMC) on selected physical properties of gypsum plaster mortars and the microstructure of hardened gypsum mortars. Microscopic investigations of the hardened gypsum mortar indicated the influence of the methylcellulose on the microstructure. Gypsum mortars without the addition of HPMC had large, well-developed crystalline forms of gypsum. The microstructure of mortars with the addition of methylcellulose consisted of smaller gypsum crystals, with the membranes spreading in the pores of the mortar formed from methyl cellulose. Although the described changes in microstructure could cause some changes in the thermal properties of the material, these parameters were not investigated.

Paper [20] presents the results of investigations of pastes and mortars made of cement and gypsum with admixtures of silico-organic compounds. The effects of organosilicate addition on setting time, bending and compression strength, wetting angle, water absorption, porosity and frost resistance were investigated. The studies showed that a particularly beneficial effect on the microstructure of cement pastes and mortars was obtained by using an addition of polydimethylsiloxane.

In papers [18–20], no attempt was made to assess the influence of the applied additives on the thermal properties of modified cement mortars and plaster. Kamseu et al. [15] showed that experimental results and theoretical models are used to assess the effective thermal conductivity of porous inorganic polymer cements, which are often considered as geopolymers that have a porosity between 30 and 70 vol.%. It was shown that the bulk chemical composition affects the structure (grain size, pore size, spatial arrangement of pores, homogeneity, micro cracks, bleeding channels), and consequently the heat flow behaviour through the porous matrix. In particular, the introduction of controlled fine pores in a homogeneous matrix of inorganic polymer cements results in an increase of pore volume and an improvement of the thermal isolation. The problem of heat conduction in heterogenic materials is mathematically analogous to the problems of the electrical conductivity, permittivity and magnetic permeability of such materials. The study of these topics dates back to the early works of Maxwell [21]. Since the late XIX century, many models allowing the prediction of the effective thermal conductivity of various types of composite materials have been proposed. The variation of the effective thermal conductivity with the total porosity was found to be consistent with analytical models described by Maxwell–Eucken [22,23] and Landauer [24].

A few papers [1,17,25] related to the thermal properties of gypsum materials recommend further research. It has been noted that there is a significant lack of information on:

- the values of the thermal parameters of gypsum modified with micro-additives, including different types of polymers or aerogel,
- the influence of the amount of micro additives on the values of the thermal conductivity of modified gypsum,

The above listed gaps and niches in the research resulted in the need to conduct studies with the use of various micro additives in different amounts, which aims to recognise the thermal properties of modified gypsum materials.

This paper presents the results of measuring the thermal conductivity λ of gypsum, and also its modification with micro additives such as: polymer (hydroxymethyl ethyl cellulose - HEMC) and aerogel. The research is based on a designed measuring stand using the "hot wire" method. There is a constant tendency to search for new and improved thermal measurement methods of building materials, especially thermal conductivity. Scientific and industrial activities can be seen to be the reason for the search for precise and rapid measurement methods of the thermal properties of new building materials and composites. The duration of measuring thermal conductivity with commercial experimental equipment is in the range of 0.5÷4 h. In the presented research, a single measurement took only one minute. Measurements were recorded with the help of a computer measuring system, with a sampling time of 0.01s. During the 60-second-long test, 6000 measurements of the heating wire temperature were collected. In addition, the changes of structure of the modified materials were examined.

2. Experimental procedures

2.1. Materials

In the work the building gypsum from Dolina Nidy (Pińczów) was used and as micro additives served: methyl 2-hydroxyethylcellulose (HEMC) and aerogel. In order to present the structure of micro additives compared to structure of ground fired gypsum stone, images of the used substances were made by a laboratory stereoscopic microscope, with the use of 100x magnification and the Moticam camera, which allowed for observation of details with a size of up to 20 μm (Fig. 1).

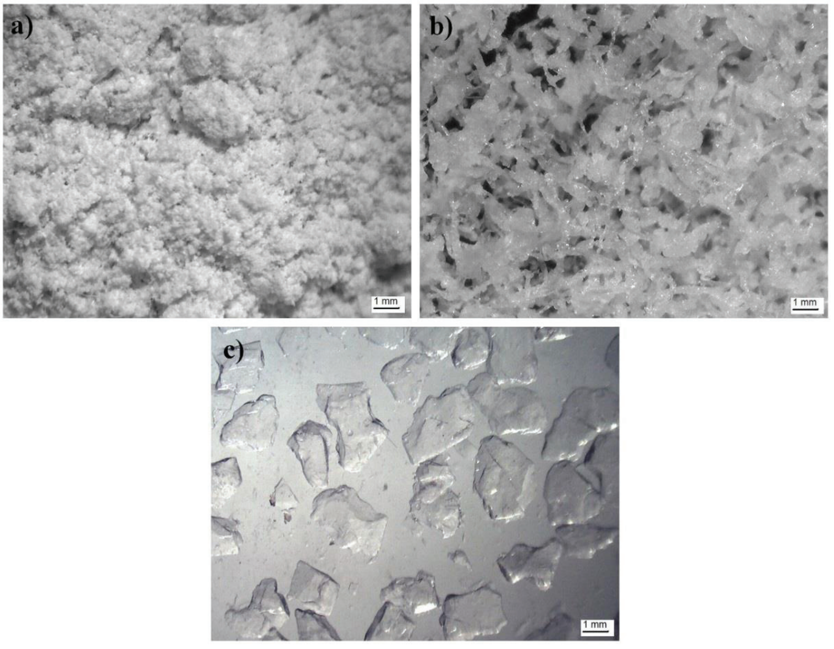

Fig. 1. Microscopic images (magnification x100) of: a) gypsum, b) polymer, c) aerogel

HEMC is a non-ionic polymer (cellulose ether) which is widely used in modifying materials made on the basis of cement or gypsum [17,26–28]. The polymer used in the research was produced by Sigma-Aldrich. Silica aerogel was used in the form of particles with a fraction of 0.7-4.0 mm [13]. It has hydrophobic properties and the specific density is from 120 to 150 kg/m^3. Such a low density reflects in thermal conductivity values of only 0.012-0.018 W/(m·K) which is achieved without the need for vacuum or gas sealed structure [29]. It results from the structure formed in the supercritical drying process in which nanostructured pores are created with a diameter of about 20 nm. The applied aerogel was a translucent material produced by Cabot Corporation.

Specimens of modified gypsum were made using building gypsum mixed with an aqueous solution of polymer or aerogel. The mixture was prepared with 2 kg of gypsum powder that was mixed with micro additives (HEMC or aerogel) dissolved in 1.5 litres of water so that the ratio of water to gypsum was constant and amounted to w/g = 0.75. The addition of HEMC or aerogel was 1% relative to the gypsum mass. The components were mixed with a low-speed rotary agitator for 1 minute at 20 °C. After mixing, the slurries were poured into a cuboid shaped mould. In addition, a specimen without the micro additives was also prepared.

2.2. Experimental setup

For the purpose of conductivity measurements, an improved “hot wire” method was used. The "hot wire" method, based on transient heat exchange, is one of the most interesting methods because of its simplicity, accuracy and ease of implementation. The idea of the experiment involves placing a source of heat in the material, usually a heating wire, and then measuring its temperature as a function of time. The wire is supplied with electric current in such a way that the heat flow emitted in time is constant. The measurement is taken during heating and the relationship between temperature and time in the *T-ln(t)* coordinates system takes on a linear character after the transitional period [30,31].

To measure the thermal conductivity λ, a computer-controlled experimental stand was used, which is described in detail in [25]. The basic element was a gypsum specimen measuring 50x50x305 mm containing an axially placed Kanthal resistance wire heater with a diameter of 0.2 mm. A miniature resistance temperature sensor type Pt100 was permanently attached to the heater. The temperature sensor was connected with the measuring module NI-9217 (National Instruments), which was then connected to the intermediary module cDAQ 9171 that transmitted the encoded temperature signal to the computer controlling the process (Fig. 2). The dedicated software in the LabView was written for the operation of the measurement procedure. The software made it possible to set the voltage and the current supply limitation, and indirectly the heating power, in order to read the actual parameters and to save them to a computer disk. The program also enabled the selection of the required power and data logging time sequences.

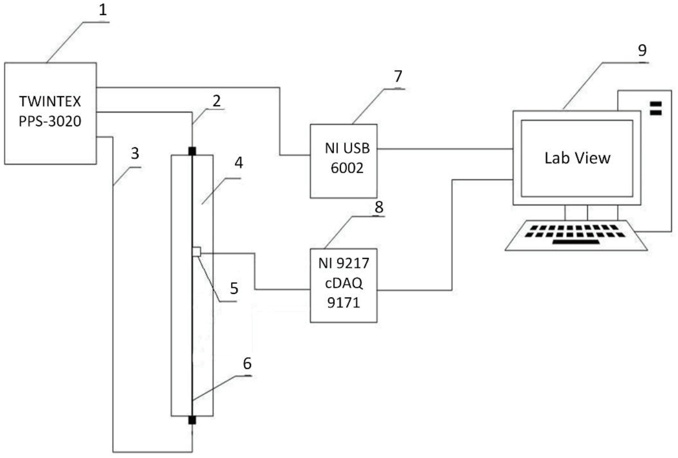

Fig. 2. Experimental set up for measurement of thermal conductivity using the "hot wire" method [25]: 1 – adjustable power supply of direct current, 2 and 3 – power cables, 4 – tested specimen, 5 – sensor Pt100, 6 – hot wire, 7 – data recorder, 8 – input module of the resistance temperature sensor, 9 – computer.

3. Results and discussion

3.1. Thermal conductivity

The gypsum after 35 days of maturing and drying was characterized by density of 1007 kg/m^3 and porosity of 57.1 %. The specimens were conditioned in temperature of 20 ÷ 22 °C and RH = 40 ± 1% for 28 days. After this time they were dried at a temperature of 50 °C for 7 days. During the experiment, thermal conductivity hardened gypsum mixes was determined successively at 1, 3, 7, 21, 28 and 35 days after preparing. At the same time, the density was determined. Bulk density was determined as a ratio of the mass and volume of the gypsum specimens. The specific density of gypsum was assumed as ρ_g = 2,350 kg/m^3 for all the specimens. Total porosity was calculated based on bulk density with reference to the density of the structure.

Figure 2 presents microscopic images after 35 days of maturation of the specimens. It was observed that the addition of the polymer to gypsum (Fig. 3b) caused an increase in porosity when compared to the gypsum without additions (Fig. 3a). The micro particles of the aerogel were built into the gypsum structure, and with it creating a non-uniform structure (Fig. 3c). The changed gypsum structure influences its thermal properties.

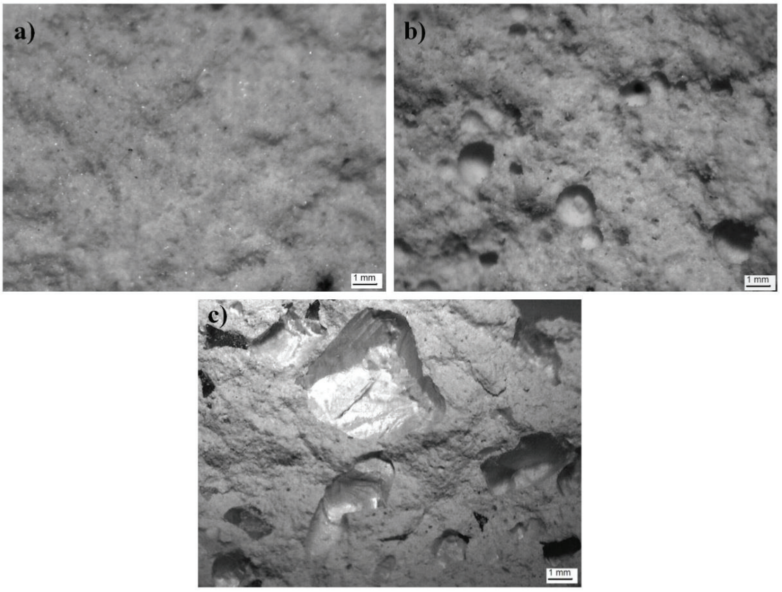

Fig. 3. Microscopic images (magnification x100) of hardened: a) gypsum, b) gypsum + polymer, c) gypsum + aerogel

For all the tested gypsum specimens, graphs of the dependencies: $T\text{-}T_0=f(t)$ and $T\text{-}T_0 = f(ln(t))$ were made and the slope S for the latter was determined. Figure 4 shows the graphs for the gypsum without micro additives.

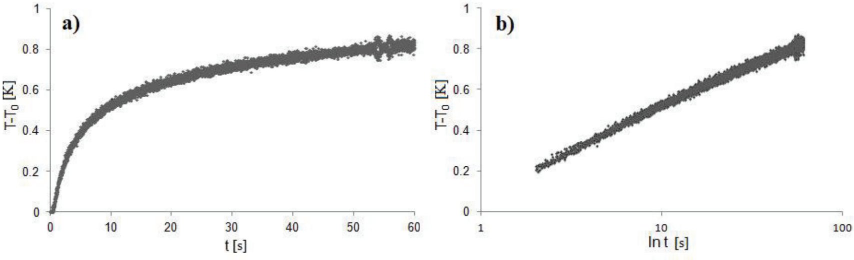

Fig. 4. Graph of the temperature change of the heating wire of the gypsum specimen: a) as a function of time, b) as a function of the natural logarithm of time

The thermal conductivity of the specimens was calculated using equation (3.1).

$$\lambda = \frac{Q}{4\pi L} \cdot \frac{1}{S} \qquad (3.1)$$

where:

λ – thermal conductivity coefficient (W m^{-1} K^{-1}), Q – heat emitted by the source of heat (W), L – length of heating element (m), S – slope of linear function

The heating power during measuring the thermal conductivity using "hot wire" method was 0.2 W. The values obtained experimentally after 35 days are shown in Table 1. The bulk density of specimen decreases and the porosity increases for gypsum with the content of micro additives. The thermal conductivity of the gypsum was compared with the literature data. The experimental value of λ differed from the literature value by less than 1.0 %. The thermal conductivity of the gypsum specimens with the addition of polymer or aerogel were respectively over 23% and 6% lower than for the reference specimen made of gypsum without additives.

Table 1. Bulk density, porosity and thermal conductivity of the gypsum mixes after 35 days of maturing

Material	Parameters		
	Density [kg/m^3]	Porosity [%]	Thermal conductivity [W/(m·K)]
Gypsum	1007	57.1	0.3465
Gypsum + polymer	989	58.9	0.2664
Gypsum + aerogel	939	60.1	0.3261

On the basis of results, diagrams of the thermal conductivity change in time (Fig. 5), and also in the function of varying density (Fig. 6), were made. It was observed that the thermal conductivity stabilized after the 14th day of the specimens' maturation.

For all three specimens, the general dependences of the change in thermal conductivity with the time and aging of specimens $\lambda=f(d)$ (3.2), and also with varying density $\lambda=f(\rho)$ (3.3), were proposed:

$$\lambda = A \cdot d^{-B} \qquad (3.2)$$

$$\lambda = C \cdot \rho - D \qquad (3.3)$$

where:

A, B, C, D - the experimental constants (presented in Table 2).

The constants in Eq. (3.2) and (3.3) were determined by the least squares method.

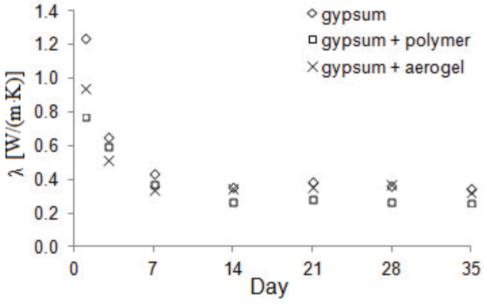

Fig. 5. Changes in thermal conductivity of gypsum specimens during the aging process of the pure gypsum and the gypsum with micro additives

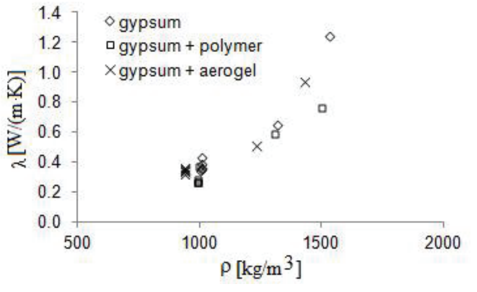

Fig. 6. Graph of the dependence of $\lambda = f(\rho)$ of the gypsum specimens with different moisture contents

Table 2. Values of constants A, B, C, D in equations (3.2) and (3.3)

Material	Values of constants			
	A	B	C	D
Gypsum	1.042	0.342	0.0015	1.129
Gypsum + polymer	0.768	0.325	0.0009	0.644
Gypsum + aerogel	0.762	0.261	0.0011	0.657

For the applied measurement system, the relation that allows to quickly determine the thermal conductivity of the tested specimens knowing only the registered temperature increase of the heating wire $\lambda(\Delta T)$, would be useful. For all the gypsum specimens, and on the basis of measurements, a generalized equation (3.4) was proposed (the estimation of the given measurement error is presented later in the article):

$$\lambda(\Delta T) = 0.3819 \cdot \Delta T^{-1.151} \pm 8\% \tag{3.4}$$

The results obtained and the observations made can be compared with the results of other researchers.

The increase in porosity should lead to a reduction in thermal conductivity, as confirmed by experiments conducted by Heim [17]. Gypsum with a HEMC content of 1.0% obtained a thermal conductivity of more than 20% lower in comparison to the material without the HEMC admixture [17]. Mathematically, the change of effective thermal conductivity was described as a function of total porosity by Kamseu in [15].

The effective use of aerogel to reduce the thermal conductivity of a material is not only limited to gypsum mixtures. Strzałkowski and Garbalińska [13] applied aerogel to concrete in order to improve its thermal properties. The authors obtained relatively low values of thermal conductivity coefficient λ in the range of 0.36 – 0.53 W/(m·K), which was low when compared with the control sample without aerogel, where λ equal to 1.198 W/(m·K) was obtained.

Sanz-Pont et al. [32] stated that highly efficient thermal insulation materials can be produced with mineral binders and hydrophobic aerogel particles after hydrophilisation with surfactants. The results of tests in which aerogel was mixed with anhydrite binder showed that composites with a volume content of about 60% of aerogel can achieve a thermal conductivity lower than 0.030 W/(m·K).

3.2. Estimation of the measurement error of thermal conductivity

The estimation of the measurement error of thermal conductivity was calculated for the gypsum specimens after 35 days of maturation and drying. It was assumed that random and methodical errors are very small and can be neglected. Therefore, the uncertainty of the final measurement will be affected by the measurement errors of the instruments. Based on this assumption the relative uncertainty of the measurement of the thermal conductivity of gypsum was calculated using Eq (3.5):

$$\frac{u(\lambda)}{\lambda} = \sqrt{\delta(U)^2+\delta(I)^2+\delta(l)^2+\left[\frac{\delta(t_1)}{\ln t_1}\right]^2+\left[\frac{\delta(t_2)}{\ln t_2}\right]^2+\delta(T_1)^2+\delta(T_2)^2} = 0.861\% \tag{3.5}$$

where: $\delta(U)$ – uncertainty of voltage measurement on the heating wire, $\delta(I)$ – uncertainty in measuring the intensity of the current flowing through the heating wire, $\delta(l)$ – uncertainty in measuring the length of the heating wire, $\delta(t_1)$ and $\delta(t_2)$ – uncertainties of time measurements of t_1 and t_2, $\delta(T_1)$ and $\delta(T_2)$ – uncertainties of temperature measurements T_1 and T_2.

As a result it can be stated that that an unknown value of thermal conductivity is in interval: $0.99139\cdot\lambda \leq \lambda \leq 1.00861\cdot\lambda$.

4. Conclusions

An additive in the form of polymer and aerogel changes the morphological structure of composite gypsum, which is reflected in the density and thermal conductivity of the final product. Modification of the pore structure and its total volume by micro additives leads to an observed decrease of thermal conductivity.

The gypsum with a polymer content results in more than 23% lower thermal conductivity in comparison to the specimen made of only gypsum. This is due to the different density and total porosity of the material. Experimental values of the thermal conductivity of the modified gypsum specimen with the addition of aerogel was 6% lower than that made of only gypsum. The analysed gypsum composites with micro additives are thus environmentally friendly materials due to their improved insulating performance.

The relative uncertainty of the measurement of the thermal conductivity of gypsum was lower than 0.9%. The experimental value of λ=0.3465 W/(m·K) of the specimen with the content of pure gypsum differed by 1.0% from the literature value of λ=0.3537 W/(m·K) [33]. This confirmed that the experimental set up that was based on the "hot wire" method was designed and made properly.

A promising direction for further research seems to be the combined use of aerogel with gypsum setting retardants and the study of the influence of such a combination on the formation of the thermal properties of the composite and its changes in time. Research on the use of other polymers, including other cellulose ethers, may also be a source of interesting results. Another issue that is worth considering would be an attempt to combine in one material both aerogel and HEMC in different proportions, together with an attempt to find an optimal ratio of both additives with regards to their thermal properties and technological conditions. All these studies should be supplemented with the observation of changes in thermal and physical properties over time, because in such studies the time factor is usually omitted.

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ANALIZA WŁAŚCIWOŚCI CIEPLNYCH I STRUKTURY GIPSU MODYFIKOWANEGO MIKRODODATKAMI

Keywords: przewodność cieplna, mikrododatki, struktura, metoda „gorącego drutu"

SUMMARY:

WPROWADZENIE

W pracy przedstawiono wyniki pomiarów przewodności cieplnej λ czystego gipsu, a także jego modyfikacji mikrododatkami takimi jak: polimer (hydroksymetylo etylo celuloza - HEMC) i aerożel. Dodatkowo zbadano zmiany struktury modyfikowanych materiałów. Badania przewodności cieplnej oparte są na zaprojektowanym i nowatorskim stanowisku pomiarowym wykorzystującym metodę "gorącego drutu". Czas trwania pomiaru przewodności cieplnej za pomocą komercyjnych urządzeń doświadczalnych mieści się w zakresie 0,5÷4 h, tymczasem w prezentowanych badaniach pojedynczy pomiar trwał minutę. Wyniki uzyskiwane podczas pomiaru rejestrowano za pomocą komputerowego systemu pomiarowego, z czasem próbkowania wynoszącym 0,01s. Podczas 60-sekundowego testu zebrano 6000 pomiarów temperatury drutu grzejnego.

MATERIAŁY I METODY

Próbki gipsu modyfikowanego wykonano przy użyciu gipsu budowlanego zmieszanego z wodnym roztworem polimeru lub aerożelu. Wykonano również próbki samego gipsu. Stosunek wody do gipsu był stały i wynosił w/g = 0,75. Dodatek HEMC lub aerożelu wynosił 1% w stosunku do masy gipsu. Do pomiaru przewodności cieplnej λ wykorzystano sterowane komputerowo stanowisko doświadczalne. Pomiar temperatury drutu grzejnego wykonano czujnikiem Pt100. Do obsługi procedury pomiarowej napisano dedykowane oprogramowanie w systemie LabView. Oprogramowanie umożliwiło ustawienie ograniczenia napięcia i prądu zasilania, a pośrednio mocy grzewczej, a także odczyt parametrów oraz ich zapis na dysku komputera.

WYNIKI

Analiza mikroskopowa wykonanych mieszanek gipsowych wykazała różnice w strukturze stwardniałego materiału w poszczególnych wariantach. Zmiany w strukturze polegały przede wszystkim na zróżnicowaniu ilości i wielkości porów. Zróżnicowanie to przełożyło się na uzyskane wyniki. Przewodność cieplna próbek gipsu z dodatkiem polimeru lub aerożelu była odpowiednio ponad 23% i 6% niższa niż w przypadku próbki referencyjnej wykonanej z gipsu bez dodatków. Stwierdzono jednocześnie, że przewodność cieplna materiałów stabilizowała się po 14 dniu dojrzewania próbek.

W przypadku wszystkich trzech próbek zaproponowano ogólną zależność zmiany przewodnictwa cieplnego wraz z czasem liczonym od chwili przygotowania mieszanki gipsowej λ=f(d), i w zależności od zmiany gęstości λ=f(ρ):

$$\lambda = A \cdot d^{-B}$$

$$\lambda = C \cdot \rho - D$$

gdzie: A, B, C, D – stałe wyznaczane doświadczalnie (wartości podano w artykule)

W przypadku wszystkich próbek gipsu na podstawie pomiarów zaproponowano uogólnione równanie wiążące przewodność cieplną mieszanki gipsowej z zanotowanym przyrostem temperatury w czasie badania:

$$\lambda(\Delta T) = 0.3819 \cdot \Delta T^{-1.151} \pm 8\%$$

WNIOSKI

Dodatek, czy to w postaci polimeru, czy aerożelu zmienia strukturę tak zmodyfikowanego gipsu, co znajduje odzwierciedlenie w gęstości i przewodności cieplnej produktu końcowego. Modyfikacja struktury porów i ich całkowitej objętości za pomocą mikro dodatków prowadzi do obserwowanego zmniejszenia przewodności cieplnej.

Gips z zawartością polimeru powoduje ponad 23% niższą przewodność cieplną w porównaniu z próbką wykonaną wyłącznie z gipsu. Wynika to z różnej gęstości i całkowitej porowatości materiału. Wartości doświadczalne przewodnictwa cieplnego zmodyfikowanej próbki gipsu z dodatkiem aerożelu były o 6% niższe niż w przypadku próbki wykonanej wyłącznie z gipsu. Analizowane kompozyty gipsowe z mikro dodatkami można nazwać materiałami przyjaznymi dla środowiska ze względu na ich lepsze właściwości izolacyjne, co przekłada się na mniejsze zużycie energii w budynkach, gdzie zostaną zastosowane

Received: 18.02.2020, Revised: 03.09.2020