Stability investigations of the thermal insulating performance of aerogel blanket

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Abstract

Nowadays fibre reinforced silica aerogels receive special attention due to their application in the building construction sector. Aerogel, besides the Vacuum Insulation Panels are one of the so-called super/advanced/nano-technological insulation materials due to their low declared thermal conductivity. However, these days their high manufacturing cost goes against their common use. Nonetheless, the investigations of their thermal and material properties are highlighted research topics in the last few years. The testing as well as the development of these insulation materials are very important both for the manufacturers and for the designers. Therefore investigations of the thermal properties (e.g.: thermal conductivity) are extremely important. In order to show the stability of the thermal insulation performance of glass fibre reinforced aerogel blankets, firstly, moisture related investigations will be presented taking special look at the sorption isotherm and the wetting kinetic curves of the samples carried out by climatic chamber method. Then thermal conductivity measurements will be presented after humidity and thermal annealing as climatic ageing and exposures. Two types of measurement series were accomplished for thermal annealing experiments: a) time kinetic (isothermal) investigations at 70 ºC for 1-42 days and b) isochronal (1 day) annealing experiments were done, where the temperature was changed from 70 ºC to 210 ºC. After both experiment series the thermal conductivities were registered by Holometrix 2000 apparatus. The measurement results will be presented as graphs, too. Afterwards, images taken with optical microscope for exploring the changes on the surface and in the hydrophobicity of the samples will be also presented. These investigations as well as the results can serve as proof of the good thermal stability and the excellent performance of this insulation.

Keywords: Aerogel, ageing, thermal conductivity, sorption

1. Introduction

Nowadays it is known that, the building sector is one of the key sectors for cost-efficient savings; furthermore it was previously manifested that at least 88–91% reduction of the energy loss is necessary in this field. In Hungary most of the buildings especially of the high rise buildings and multi-family (panel) block should be refurbished and renovated. To reach this goal the use of thermal insulation is a primary tool. These days, the commonly used insulation materials are the plastic foams and the fibre reinforced materials, while the use of the nano-technological/advanced/super insulation materials is (aerogel, vacuum insulation panel, graphite enhanced polystyrene) is widespread, too. [1-3] By using these materials as additional or supplementary insulation one can reduce heat losses and can make his house more comfortable at the same time. [4-6] Building thermal insulation materials are subjected to different temperature and humidity conditions and their thermal performance can change during a controlled environmental exposure. [7] Aerogel is an open cell (porous) material with low density, furthermore with cells on the nanoscale. It is prepared from supercritical drying of various types of gels, and is most commonly made from silica gel. It is said by Chal and his co-authors in Ref. [8] that aerogel is one of the most important super-insulation materials with lower than 0,025 W/mK, and more specifically lower than 0.018 W/mK thermal conductivity. [8] It is reported in several papers that aerogel is one of the most promising insulation materials. There are numerous papers in the scientific literature presenting aerogel related contents, but unfortunately only some of them can be covered, and included in the literature review of this paper, because the main aim of this paper is to present and understand measurement results carried out on glass fibre-enhanced aerogel samples. [9-13] Most of them are reporting the perfect insulation property of aerogel with low thermal conductivity (0.018 W/mK) measured at room temperature. [14-16] It was showed by Lakatos [17], by Hoseini and Bahrami [18] as well as by Nosrati and Berardi [19], that aerogel blankets/panels can be used at all parts of the building envelopes except for the moisture loaded parts. Ng et al. presented the effect of curing conditions at elevated temperatures on aerogel-incorporated mortar and ultra-high performance concrete. [20] Firstly, in this article thermal conductivity measurements will be presented after humidity and thermal annealing as climatic ageing and exposures. Sorption kinetic curves, sorption isotherm as well as, moisture related thermal conductivity changes are shown. Furthermore, two types of measurement series were accomplished for thermal annealing experiments: a) time kinetic (isothermal) investigations at 70 ^{o}C for 42 days (6 weeks) and b) isochronal (1 day) annealing experiments were done, where the temperature was changed from

70 ºC to 210 ºC. After both experiment series the thermal conductivities and their changes were followed by Holometrix 2000 apparatus. The measurement results will be presented as graphs, too. In addition, images taken with optical microscope for presenting the changes on the surface and in the hydrophobicity of the samples will be also provided. These investigations as well as the results can serve as proof of the good thermal stability and the long-term performance of this insulation.

2. The tested aerogel sample and its theoretical background

2.1. Properties of the investigated aerogel insulation:

This is a fibrous insulation blanket. In its data sheet the following characterization was written about the material: it is a flexible, nano-porous aerogel blanket insulation designed to meet the demanding energy conservation requirements of residential and commercial building applications. It's unique properties – extremely low thermal conductivity, superior flexibility, compression resistance, hydrophobicity, and ease of use – make it essential for those seeking the ultimate in thermal protection. Using patented technology, this insulation combines a silica aerogel with reinforcing fibers to deliver industry-leading thermal performance in an easy-to handle and environmentally friendly product. [21]

Table 1. The declared performance of the insulation is [21]

Thermal Conductivity via Guarded Hot Plate	0.0131 W/m*K @ 10°C
Thermal Conductivity declared	λ90,90 = 0.014 W/m*K
Flame and Smoke Spread	Class A: FSI <5, SDI 20
Reaction to Fire Performance	Passed Euroclass C-s1,d0
Compressive Stress / Strain	80 kPa at 10% strain, 305 kPa @ 25% strain
Specific Heat	1.000 J/g*K at 40°C
Water Vapor Transmission Rate	1877 ng/Pa*s*m^2 (dry cup method)
Linear Coefficient of Thermal Expansion (@ 10°C)	x: 1.06 x 10-5 K-1, y: 1.90 x 10-5 K-1
Water Vapor Sorption	Mass Gain = 1.08%
Porosity	92%

2.2.Thermal conductivity

The total thermal conductivity of a fibrous (insulation) material can be written by the following equation:

$$\lambda_T=\lambda_{c,s}+\lambda_{c,g}+\lambda_r+\lambda_{conv}+\lambda_{coupling}+\lambda_{leak} \quad \text{(Eq. 1)}$$

where ($\lambda_{c,g}$), (λ_r), ($\lambda_{c,s}$) and (λ_{conv}) are the conductive part of the gas filling ($\lambda_{c,g}$), and the solid skeleton ($\lambda_{c,s}$), the radiative part (λ_r), and the convective part of the gas filling (λ_{conv}). Moreover, $\lambda_{coupling}$ and λ_{leak} can be defined as: $\lambda_{coupling}$ = thermal conductivity term accounting for second order effects between the various thermal conductivities; λ_{leak} = leakage thermal conductivity. In order to reach a thermal conductivity as low as possible, each of the above thermal contributions has to be minimized. From Jelle et al. we know that normally, the leakage thermal conductivity λ_{leak}, representing an air and moisture leakage driven by a pressure difference, is not considered as a solid insulation materials and solutions are supposed to be without any holes enabling such a thermal leakage transport. The coupling term $\lambda_{coupling}$ can be included to account for second order effects between the various thermal conductivities. This λ_T is called to total/effective thermal conductivity. [22, 23]

2.3. The sorption isotherms

The connection between relative the humidity and the equilibrium moisture content of a material at constant temperature can be displayed graphically by a curve, which is the so-called moisture sorption isotherm. For each humidity value, a sorption isotherm indicates the corresponding water content value at a given, constant temperature (23±1 ^{o}C), regarding the 12571 standard. If the composition, contents or quality of the material changes its sorption properties are also changing. Due to the complexity of sorption processes isotherms cannot be determined by calculation, but must be taken up experimentally for every material. It is well known, that water can have harmful effects in the properties of building materials. Moisture in buildings as well as in building materials can appear in several ways e.g.: remainder from the construction. [24-26] A slight amount of water can be infiltrated in the material by the manufacturing process. Furthermore, materials can absorb moisture caused by outer weather conditions. Buildings under constructions are particularly exposed to moisture stress. Precipitation has also numerous impacts on the structure. [27] Large moisture load can occur in the internal side due to the function of the room; moreover, the humidity of the indoor air can also increase as a result of the activities of the inhabitants in the buildings. [28]

The sorption characteristics of the building materials are key points from the aspects of rating and from performance durability. As above mentioned water may cause unwanted changes in the physical, mechanical and chemical properties of the materials.

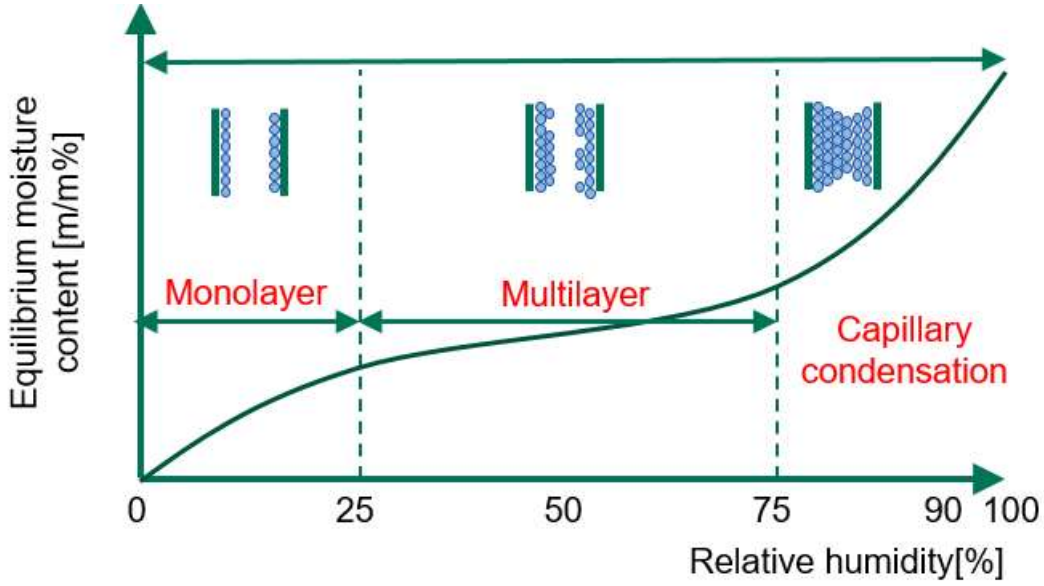

Figure 1.: A typical sorption isotherm graph

On the picture a typical sorption isotherm graph can be found: the BET type isotherm. This sorption characteristic is the most important and typical one. By analysing the BET isotherm on Figure 1 we can see that during the water up-taking process, at first the accumulation of the water particles on the surface can be expected as monolayers then the accumulation of the particles as multilayers takes place. At high humidity levels the capillary condensation and saturation happens. At the first 40 % of the relative humidity the curve shows a square root type function, this means that firstly, during the beginning of the water up-taking process the water molecule's sorption on the surface befalls, however, after this with increasing relative humidity value this isotherm shows a continuously increase. Through the BET isotherms one can follow the sorption phenomena. [17, 24-26, 29]

The moisture content ω (m/m%) of the material is the following:

$$\omega\ (m/m\%) = ((m_{wet}-m_{dry})/m_{dry})\times 100 \qquad \text{(Eq. 2)}$$

where m_{wet} and m_{dry} are the mass of the wet and dry samples respectively.

2.4.Wetting kinetic curves

If one registers the intermediate water contents during the sorption measurements, the moisture kinetic curves can be registered too. By applying long time wetting to the materials at a given humidity value and at a certain temperature, from the figure showed below one can reach the time kinetic curve.

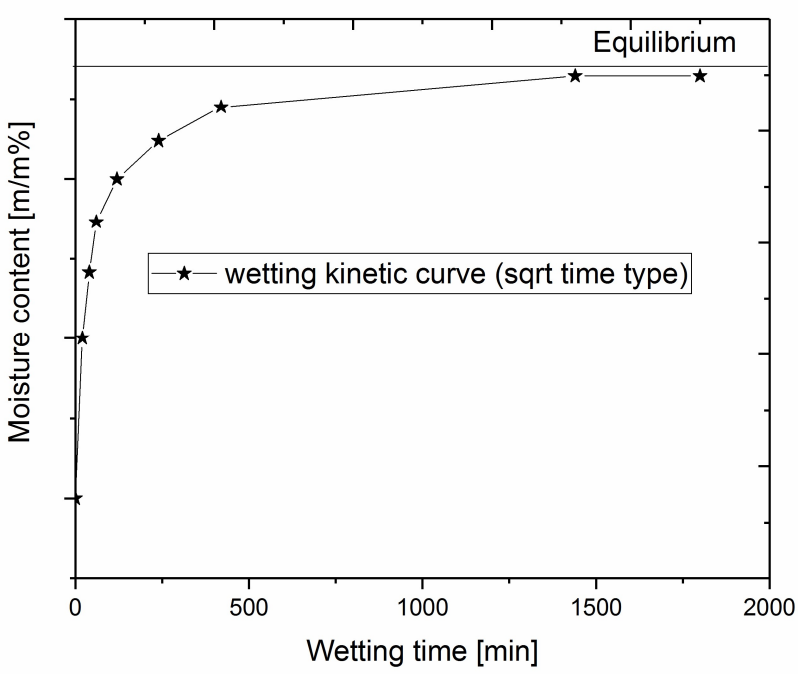

Figure 2.: The kinetic curve

The moisture kinetic curves were taken up by using the ISO 15148:2002. Hygrothermal performance of building materials and products -- Determination of water absorption coefficient by partial immersion standard, applied on the climatic chamber method.

2.5. The influence of moisture and temperature

The investigated aerogel sample has fibrous structure, the gaps can be present in up to 90%-95% of the total volume of the material. These holes/spaces are filled with air. The above mentioned (in Eq. 1) total/effective thermal conductivity mainly depends on the specific properties of the materials: on the density of the material (ρ), the temperature (T), the pressure (P), the moisture content (ω) and its age (t_s=service time). During service life the thermal conductivity of the materials changes. On microscopic level it depends on the size and distribution of the fibers. Therefore the total/effective thermal conductivity can be given by a function:

$$\lambda_{eff}=f(\omega,\rho,T,p,t_s) \qquad \text{(Eq.3)}$$

In a general temperature range (-10 ºC - 25ºC) it can be easily showed that the temperature of the ambient air has only slight effect on the thermal conductivity of a material, however at unusual temperature ranges (T>100 ºC) significant effects are expected. Moreover, in moist materials with great pores (fibrous materials) the equation of the thermal conductivity is different. Furthermore, if the air in the pores is substituted with water taken up from the ambient air, the thermal conductivity can change, too, because the thermal conductivity value of the water is greater than the air's thermal conductivity. At high temperatures (T> 100 ºC) (thermal annealing) the suffered physical and chemical changes can also increase the thermal conductivity of the sample.

2.6.Thermal annealing as accelerated ageing

High temperatures can increase the kinetic reaction rates within the molecules of the materials and enhance the chemical, moreover the physical degradation phenomenon. The effect of the change in the temperature of the environment (ageing) can be modelled with thermal annealing at not-general temperatures. Certainly, thermal annealing of the samples at high temperatures would never be experienced in typical outdoor climate conditions; it can happen that it will generate unrealistic aging issues. For these reasons, thermal annealing processes for modelling the ageing phenomena of the building materials are usually executed near 70 ºC. [30] An ISO standard 11561: Ageing of thermal insulation materials -- Determination of the long-term change in thermal resistance of closed-cell plastics (accelerated laboratory test methods) is available to have some information related to the ageing of materials, however this standard belongs to plastic foams. The purpose of that standard is to determine the ageing (long-term decrease in thermal resistance) of closed-cell cellular plastic materials and products which have properties that are due to the out-diffusion of the trapped gases (e.g.: pentane in expanded polystyrene). The long-term thermal resistance is one property required for establishing design thermal performance under service condition and for determining life-time energy requirements. Ageing is a process by which the physical, mechanical and thermal properties of a material change with time.

The ageing of materials can be done by irradiation (infrared or ultra-violet) as well as by thermal annealing. However, it is well known that moisture has unwanted effects too, but it is not taken as ageing process. In our opinion it should be included as one type of accelerated ageing, because water can also cause chemical changes in the material.

As one model the accelerated ageing can be well approximated with the Arrhenius' 10 degree law:

$t_a=t_s/(2^{(Ta-T_s)/10})$ (Eq. 4)

where t_a is the test time, t_s is the service time, T_a is the test temperature and T_s is the service temperature.

The declaration of an aged thermal conductivity value by trying to estimate the lambda changes and the acceptable service time during the expected economic lifetime, makes a lot of sense. It is well known how to thermally age the plastic cell materials due to their low melting point, however the accelerated ageing methods of the glass fibre reinforced blankets are unknown, and therefore researchers have greater freedom in the modelling. Due to its high melting point general temperature ranges (10 ºC-60 ºC) should not have a significant effect, but the higher temperatures should. Jelle in his articles in Ref. [23, 30] reports about climatic exposures as ageing factors e.g.: thermal annealing. However, this paper mainly focuses on the effects of the UV irradiation. Jedediah et al. report about the moisture and temperature combined ageing of aerogel samples, but it has to be mentioned that they followed the change in the thermal properties under extreme climatic conditions (e.g.: 65 ºC and 90 % relative humidity). [7] Koru et al. in Ref [31] investigated the thermal ageing effects in the thermal resistance of plastic foam materials. He reached exponential relationships for polyurethane and extruded polystyrene between the thermal conductivity and time (in days). Miros in his article presented thermal aging effects on thermal conductivity properties of mineral wool samples at high temperature (100-600 ºC). [32] It is noticeable that while the degradation of the thermal resistance for the plastic cell materials happens by the diffusion of low thermal conductivity cellular gases out of the cells within the insulation, only to be replaced by “air” with higher thermal conductivity. Moreover, for materials e.g.: mineral wool/glass fibre reinforced aerogel the degradation in the thermal performance can happen through chemical reactions as structural changes. In Ref. [8] very long-term and extreme conditioning of aerogel samples was presented. It has to be mentioned that they were measuring the thermal conductivity of aged aerogel samples conditioned at 70 ºC and 90% relative humidity. Similarly to this paper they followed once the microstructural changes due to the ageing as well as the thermal conductivities by heat flow meter. Moreover, they registered a sorption isotherm under these un-common conditions. It was further reported by them that, the aerogel hydrophobicity significantly increased under this hot and very humid ageing while only slight microstructural changes could be observed.

3. Measurement methods

3.1. Details of the thermal conductivity measurements

The thermal conductivity measurements during this research were executed according to the EN ISO 12664:2001 standard (Thermal performance of building materials and products. Determination of thermal resistance by means of guarded hot plate and heat flow meter methods. Dry and moist products of medium and low thermal resistance). [17, 22, 33, 34] For analysing the thermal conductivity of the aerogel sample a Lambda 2000 type Heat flow meter (HFM) was used. This apparatus is designed to determine the thermal conductivity of materials in accordance with standard ASTM C518 and ISO 8301 protocols. Aerogel samples were tested with 30 cm x 30 cm x 1 cm geometry and were positioned in the measurement area of the instrument. The samples were tested between the heating and cooling plates which were maintained at different temperatures (T_1=20 ºC and T_2=40 ºC, with T_{mean}=30 ºC). After reaching thermal equilibrium and evolving a homogenous temperature gradient throughout the sample, its "lambda value" can be found. To reach the exact measured thermal conductivity of a sample, five independent measurements were executed. The measured thermal conductivity of the tested sample is the mean value of the five measurement results.

In order to understand the measurement method of the equipment the following explanation has to be given. The value of the heat flow (Q) depends on some factors:

a) the thermal conductivity of the samples (λ=k)

b) the thickness of the specimen (d=x)

c) the temperature difference across the specimen (ΔT)

d) the area through which the heat flows (A).

The Fourier's equation gives the relationship between these parameters when the test section reaches thermal equilibrium.

$$Q=\lambda \times A \times \partial T/\partial x \qquad \text{(Eq. 5)}$$

The transducer measures the heat flow through the sample. The signal of a heat flow transducer (in Volts (V)) depends on the heat flow through itself. From the measurement of the equipment the heat flow will be:

$$Q=NV \qquad \text{(Eq. 6)}$$

Here N is the calibration factor, which relates the voltage signal of the heat flow transducer to the heat flux through the sample. For the calibration of instrument a fibre glass board standard sample with λ= 0.035 W/mK is used. Using the above mentioned equations the heat conductivity can be found:

$$\lambda = N \times V \times d / A \times \Delta T \quad \text{(Eq. 7)}$$

If the thermal resistance of the test specimen is commensurable with the reference standard's ± 5% or better accuracy can be obtained. The measurement orders can further be found in Ref. [17, 22, 33, 34]

Figure 3.: The Holometrix apparatus

3.2.Details of the moisture related measurements

In order to investigate the sorption/water up-taking properties of solid materials three different kinds of equipment should be combined: a drying oven, a climatic chamber and a weighting balance. Firstly, the samples should be dried to changeless weight in a drying oven, in our case in a Venticell 111 apparatus. It works between 10ºC and 250 ºC. With this device materials can be dried as well as can be thermally (heat) annealed by setting different air temperatures. It works with hot air circulation using an inbuilt ventilator. [17, 29]

Figure 4.: The Venticell 111 and Climacell 111 equipments and the milligram preciseness balance

After drying the materials in the Venticell apparatus to changeless weight, their masses should be measured with the balance. Then the materials should be placed in a climatic chamber in this example it is a Climacell 111 equipment. This apparatus is a laboratory incubator where homogenous environment can be created. The apparatus works in a 0 ºC to 100 ºC temperature range with humidities from 10 % to 90%. The wetting measurement should be separated to two parts. The first part is the determination of the sorption isotherms of building materials the other one is the determination of the kinetic curves.

For taking up the moisture kinetic curves, thus to create the sorption isotherms four aerogel samples with 10 × 10 cm base are were tested. The measurements were done by following the rules of ISO 12571: 2013 standard (Hygrothermal performance of building materials and products -- Determination of hygroscopic sorption properties, Part B- climatic chamber method). In the humidity chamber the temperature was fixed at 23 ºC, while the relative humidities were varied to 30, 50, 65, 80 and 90%. Before and after wetting the samples for 20, 40, 60, 120, 240, 420 and 1440 minutes under the above mentioned relative humidities their mass was registered. From the wet and dry masses of each samples of the four test materials the moisture contents were calculated by using (Eq. 2) and they were averaged. From these measurements the so-called square-root of the time type kinetic curves (see Figure 2) can be found, from the initially increasing moisture content to reaching the equilibrium. Moreover, taking the equilibrium moisture content from these investigations, the sorption isotherm of the sample was plotted too. After treating the samples with 30 cm x 30 cm base area and 1 cm

thickness under the above mentioned temperatures and relative humidities for 24 hours their thermal conductivities were registered, too. [17, 29]

4. Results

The measurement results will be presented on graphs with indicating the standard deviations too.

4.1.Moisture related investigations

As it was presented in section 3.3. for the moisture sorption investigations four pieces of fibre reinforced aerogel samples were tested. The samples were humidity treated in the climatic chamber at 23 ºC under different relative humidity for between 20 and 1440 minutes. In Figure 5 one can see the so-called wetting kinetic curves.

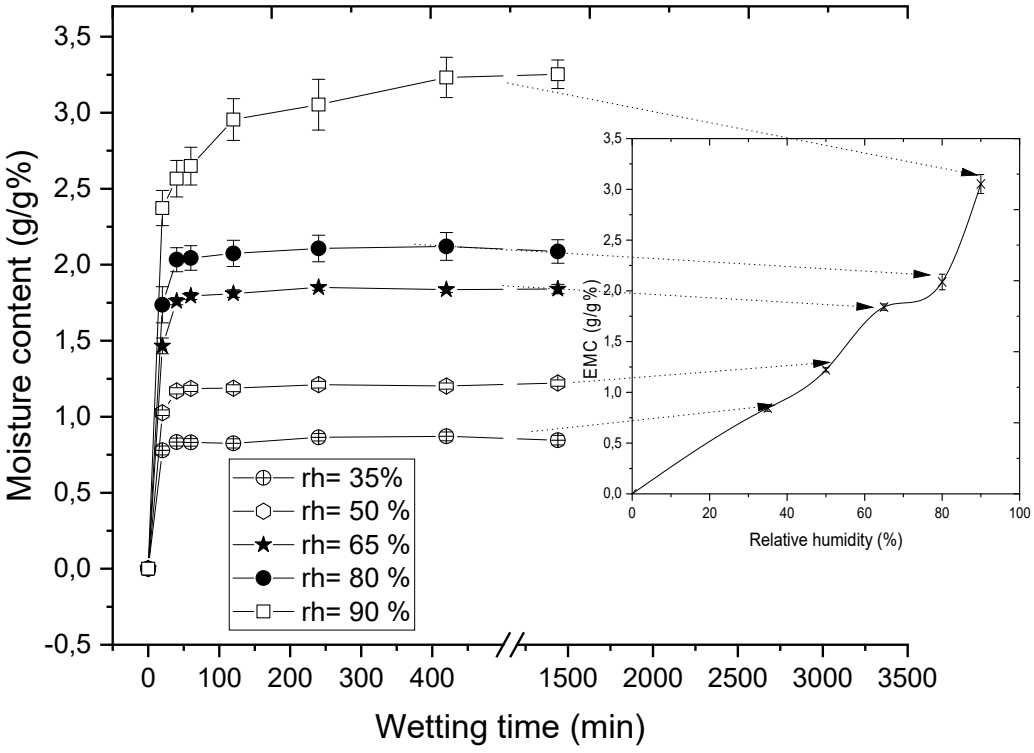

Figure 5.: The wetting kinetic curves at 23 ºC

From Fig. 5 it is observable that the moisture up-taking process of the aerogel samples can be well approximated by the curve presented in Figure 2. At first, the continuously increasing part, then the equilibrium state takes place belonging to each relative humidity. It is further observable that the amount of the water taken up is increasing with the increasing relative humidity. It is further observable that, for each relative humidity the increase of the moisture

content is completed after 420 minutes the latest and the wetting process reaches the equilibrium (steady state stage).

From the equilibrium moisture contents (EMC) presented in Figure 5 reached at a certain temperature, in function of the relative humidity one can create the sorption isotherm curve of the sample. In our case it is plotted on Figure 5, too.

In contrast to the previous paper of the author Ref. [17], where the sorption isotherms of the samples were taken up at 10, 20, and 30 ºC after 24 h wetting at each relative humidity, without registering the intermediate stages, here the whole sorption process was investigated (see Figure 5-7. Till 65 % a continuously increasing water up-taking can be observed: the mono- and multilayer stages; then after 80% the capillary condensation, (the filling of the free places) takes place. The shape of the isotherm graph can be identified as BET isotherm type IV. Ihara et al. presented sorption measurements on aerogel granules and proposed that the water entered the nano-pores of the aerogel granulate due to the pressure difference and broken the aerogel structure. They also concluded that the water was trapped in the pores and needed very long time to be released. The main aim of their research was to simulate the ageing of the granules by long-term wetting. [28] In order to compare our sorption isotherms with the available in the literature we have to mention the results of Ibrahim and his co-authors. They registered the sorption isotherms, where the water content was given in kg/m^3, as well as the thermal conductivity of the aerogel samples in function of the moisture. Similarly to this research they were applied the directions of the same standards: EN ISO 12571 and 12667 in order to reach the water content values and the thermal conductivity change. [35]

4.2. Thermal conductivity measurements in function of the moisture

At first, similarly to the research of Ibrahim et al. and Hoseini et al., the thermal conductivities were measured after wetting the aerogel samples of the equilibrium moisture content. [18, 35] The thermal conductivities of the samples were measured after wetting them at 23 ºC temperature and 30, 50, 65, 80 and 90% relative humidity for 24 hours. In Figures 6 a, and b the author plotted the thermal conductivities in function of the relative humidity and the moisture content respectively. In Figure 6a and b the moisture contents and the relative humidities are indicated on the graph next to the result points. From the graphs increasing thermal conductivities with increasing moisture content and relative humidities are observable.

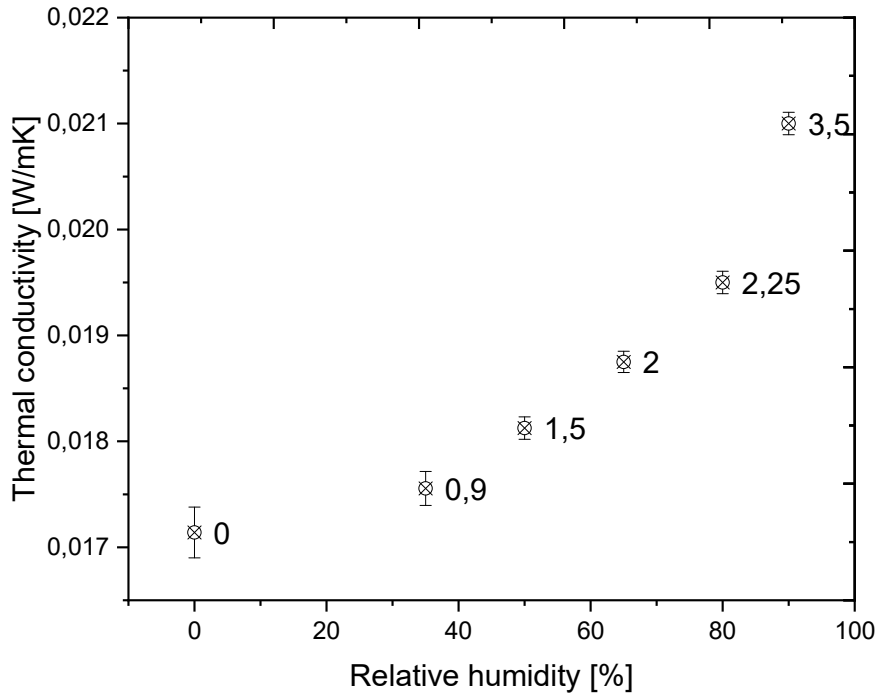

Figure 6a.: The thermal conductivity in function of the relative humidity (with the moisture contents)

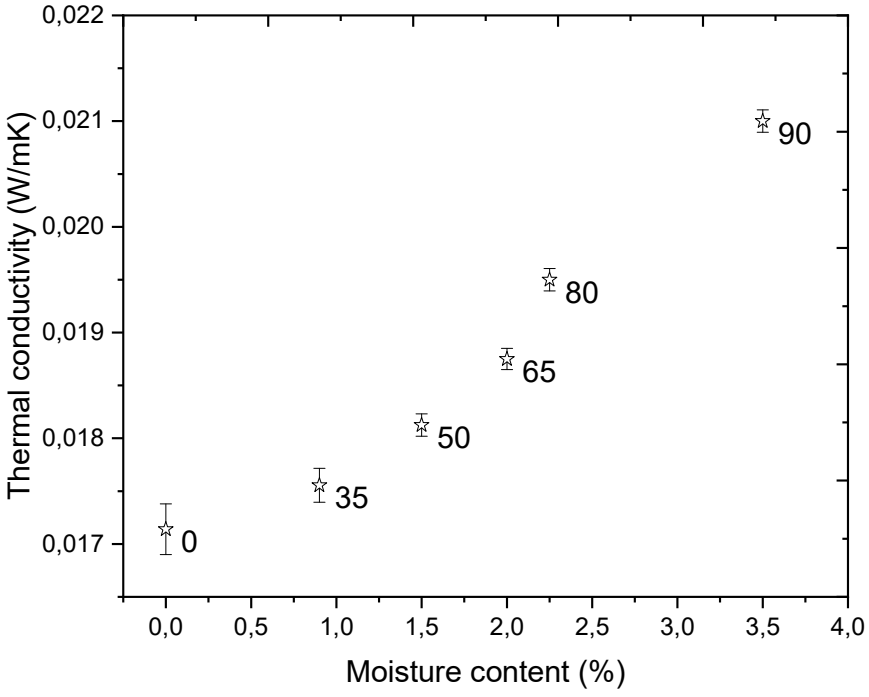

Figure 6b.: The thermal conductivity in function of the moisture content (with the relative humidities)

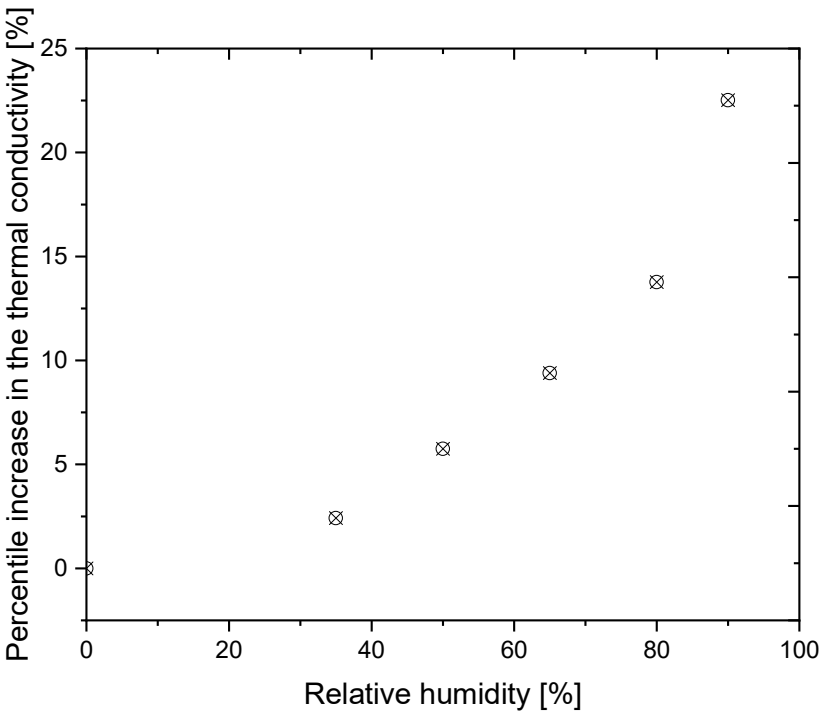

Figure 7.: The percentile change of the thermal conductivity

In Figure 7 the calculated percentile thermal conductivity increase can be found at the certain relative humidity values. After 65 % relative humidity significant (>10%) increase in the thermal conductivity can be manifested. While at 90% the change is about 20%.

4.3.Thermal conductivity measurements after heat treating

4.3.1. Time kinetic, isothermal investigations

During these measurements the thermal conductivity of the aerogel samples were measured after heat treating them in the drying oven at 70 ^{o}C temperature for about 42 days. In Figure 8 one cannot find any relevant changes in the thermal conductivity after heat treating the samples for about 6 weeks. As it is observable the results are rounding near the certain value: 0.017 W/mK.

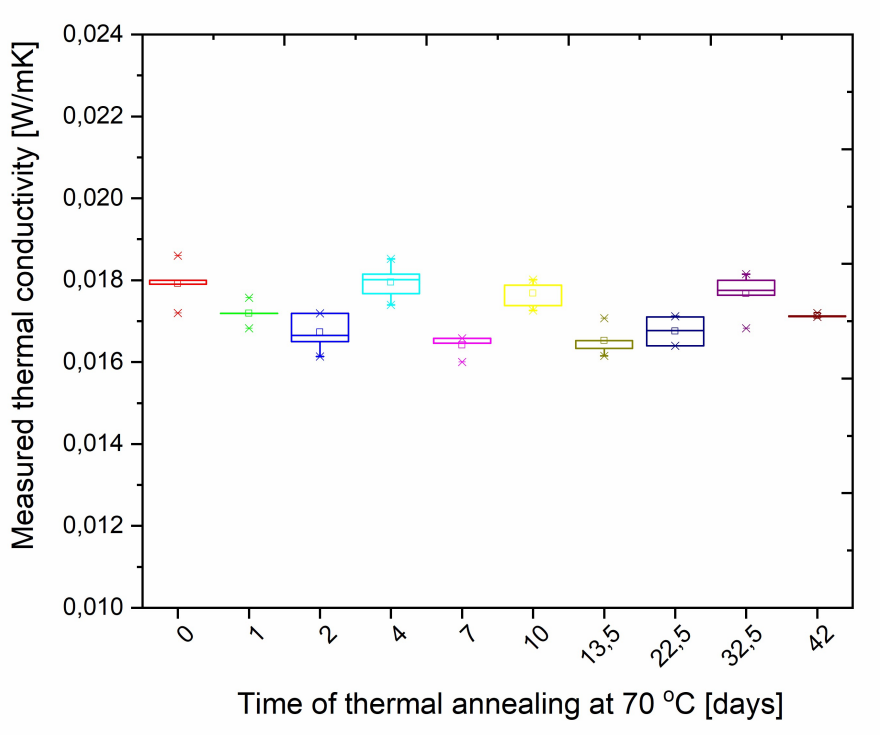

Figure 8: The thermal conductivities after thermal annealing (x-scale is non-linear)

By using Eq. 4 (the 10 degree Arrhenius' law) the test time (time/days of thermal annealing at a higher temperature) can be converted to a service time/years at a general temperature (e.g.: 10 °C). This connection in our context is represented in Figure 9.

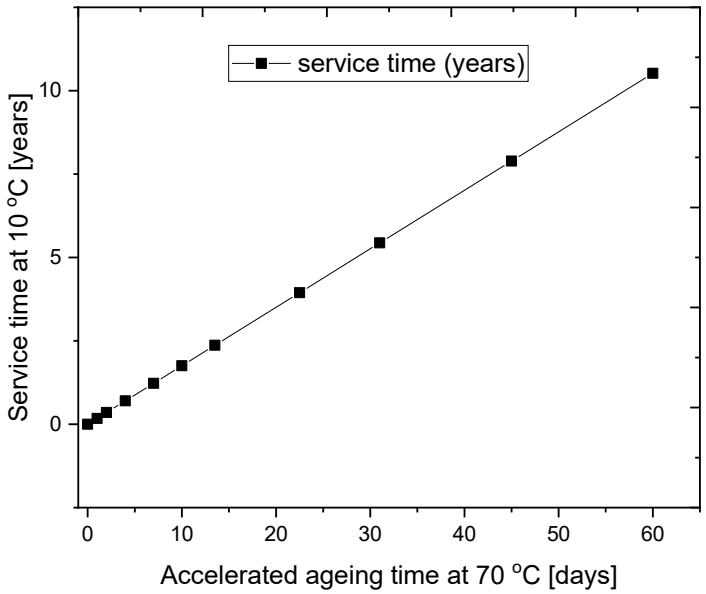

Figure 9. The plot by using Eq. 4 between the service time and the accelerated ageing time

From the combination of Figures 8 and 9, Figure 10 was created. From the graph we can observe that no significant changes were observable till almost 10 years.

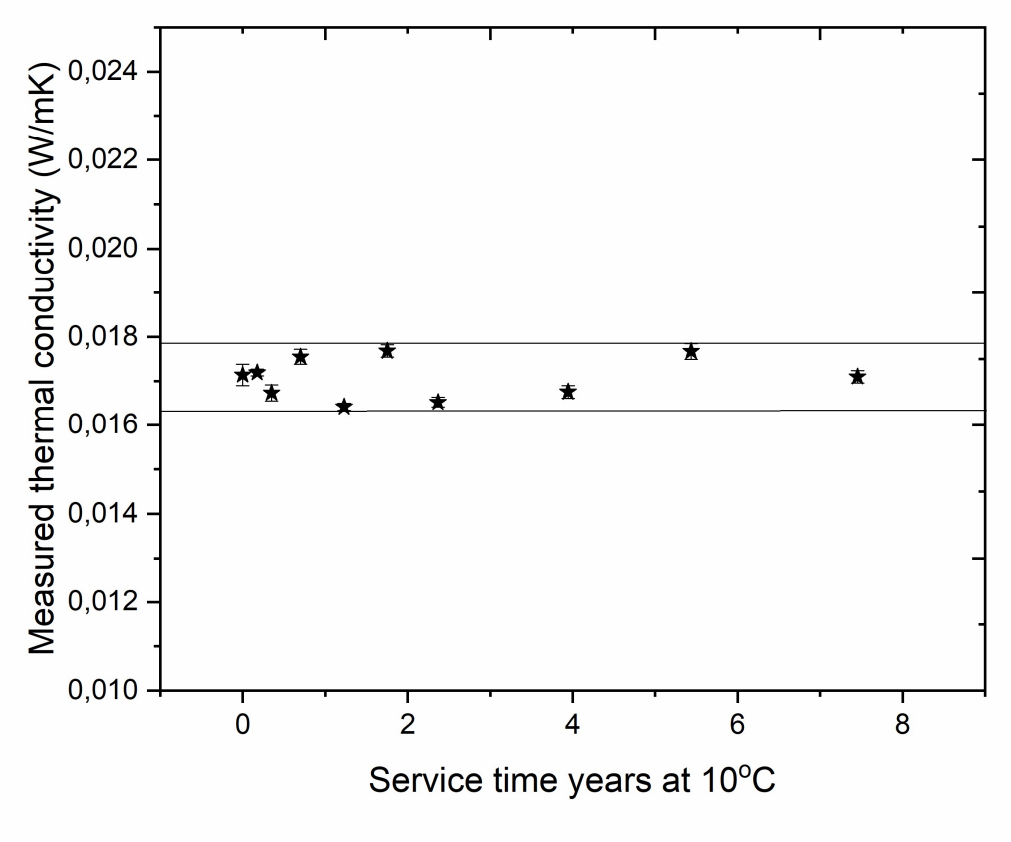

Figure 10. The thermal conductivity after years at 10 ºC service time

It has to be emphasized that all the measurement results plotted on Figure 10 are rounding near their average 0.017 W/mK thermal conductivity value, which is in the accuracy range (±5%) of the equipment.

We can make a comparison between our results and the literature especially with Ref. [36], where similar investigations were executed, but their test temperature was 90 ºC. However, it was previously compromised with Ref. [23, 30] that the measurements with elevated temperatures for thermal insulation materials should be done at 70 ºC. This temperature should be used in order to be able to do comparison with similar tests carried out on other materials e.g.: on plastic foams, where the melting points can be found at about 90-100 ºC. In Ref. [36] no changes in the thermal conductivities were presented. From our measurement series we can report the stability of the thermal conductivity at this temperature.

4.3.2. Temperature induced changes, isochronal investigations

Due to the above mentioned stability of thermal conductivity after annealing at 70 ºC for weeks, we put the samples under thermal annealing at higher temperatures till 210 ºC for 1 day. From Figure 11 we can conclude that the thermal conductivity of the aerogel sample significantly changes after thermal annealing at 180 ºC for 24 hours. The samples were further annealed at 210 ºC and the thermal conductivity increased with about 17 % (over 0.02 W/mK). The measurements were achieved on samples as the followings: Firstly the samples were heat treated at 70 ºC for 1 day, then its thermal conductivity was measured. Because no changes in thermal conductivity was observed the samples were further annealed at 100 ºC for 1 day and its thermal conductivity was followed again. This steps were repeated on the samples at 150, 180 and 210 ºC.

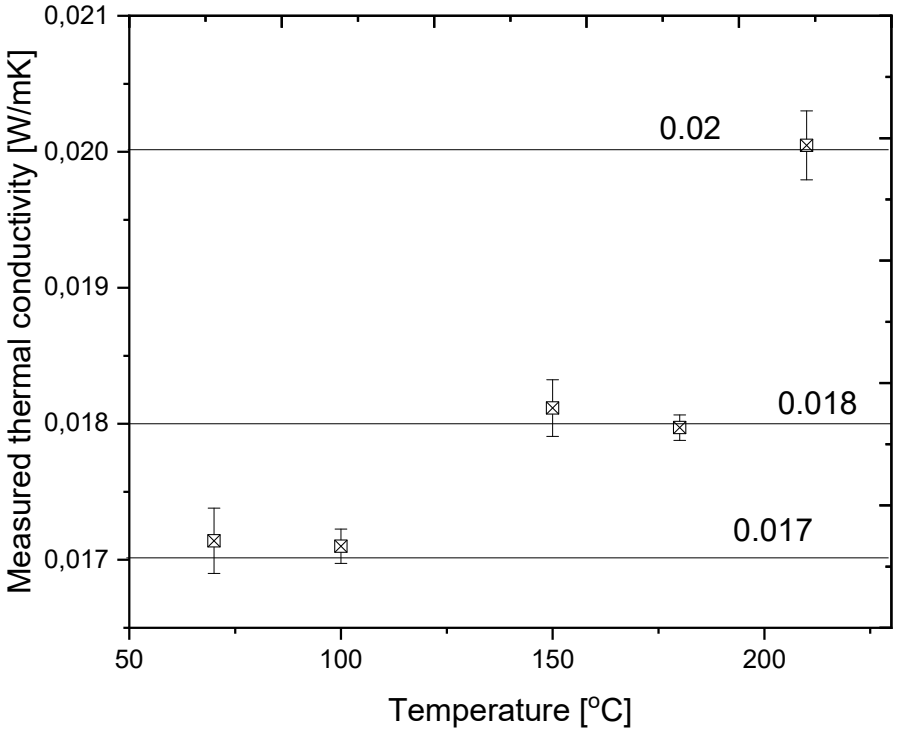

Figure 11. Thermal conductivities after thermal annealing

In order to see the changes photographs were taken of the annealed (70 ºC for 6 weeks and 210 ºC for 1 day) moreover from the not annealed (as-received) samples. Figure 12 shows significant changes in the outlook of the samples. First one can find changes in the colour of the samples. The article written by Miros et al. Ref. [37] presents possible industrial applications of aerogels. They presented the change of the thermal properties in a wide range of temperatures (+ 10–600°C). They stated that aerogels has perfect thermal stability. Similarly

to this paper they followed the thermal conductivity changes of the aerogel after thermal annealing. They reported research results on one other type of aerogel (Pyrogel). They presented that the thermal conductivity measured between 100 and 250 °C is in the same range as we found (<0.03 W/mK). They stated that the applicability limit of their sample is about 200 °C. [37] It has to be mentioned that the author observed only a small change both in the thermal conductivity and in the colour of the blanket till 180 °C. However, after annealing the samples at 210 °C for 1 day besides the notable change of the colour, their structures changed, too. The changes in the structure were observable with the naked eye (as inspection).

Figure 12. The photographs of the un-annealed sample (left), the annealed sample at 70 °C for 6 weeks (middle) and the annealed sample at 210 °C for 1 day (right)

It is observable that from the left to the right the colour of the blankets is getting darker.

It was also observable in higher zoom (see Figure 13), that aerogel granules among the fibres in the samples appeared. Further noticeable is that these granule precipitates appeared on the sample annealed at 70 °C for 30 and 42 days, but forcing no change in the thermal conductivity in contrast to the result after annealing at 210 °C for 1 day. It is supposed that after annealing at 70 °C for 42 days the change in the structure was not so advanced to significantly change the thermal conductivity. The reason for this can be that, this aerogel blanket is a silica aerogel embedded to the fiberglass mesh, and after annealing the granules may precipitate.

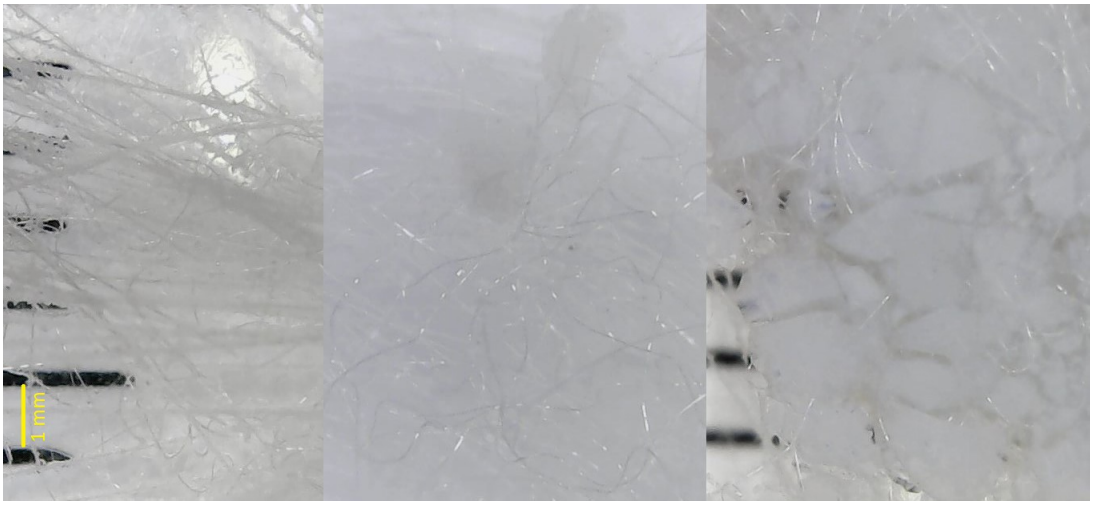

Figure 13. A microscopic image of the samples: from left to right: the as-received, annealed at 70 ºC for 42 days, annealed at 210 ºC for 1 day

4.3.3. Hydrophobicity tests

Hydrophobicity measurements were carried out on the samples, with a simple and rough method. Images were taken with an optical microscope after putting a water droplet on the surface of the aerogel sample. By photo analysing software the contact angle was measured. If the contact angle (θ) is smaller than 90º the sample is hydrophilic, if the contact angle is greater than 90º degree it is hydrophobic. In the following figure (from the left to the right) one can see the test results, once of the as-received sample, secondly the annealed sample's image at 70 ºC after 6 weeks as well as the result of the thermally annealed sample at 210 ºC for 1 day. The contact angles were estimated too, and they were found to be greater than 90º (110-120 º) for all the samples. One can conclude that, no significant change in the hydrophobicity is observable.

Figure 14.: The result of the hydrophobicity tests of the samples, from left to right: the as-received, annealed at 70 ºC for 42 days, annealed at 210 ºC for 1 day

Based on these measurements it can be stated that besides the use of the aerogel as thermal insulation of buildings, it can also be applied in industrial cases, too instead of the conventional polystyrene ones. The applicability temperature limit is about 180 ºC. In a recent article Ref. [38] He et al. present thermal stability tests in a wide 200 - 800 ºC temperature range carried out on hydrophobic aerogel samples (granules). They stated that the thermal conductivity of the aerogel samples at over 200 ºC was over 0.02 W/mK, similarly to the results presented in this article.

5. Conclusions

The studies conducted on aerogel samples allow us to reach interesting conclusions. The thermal conductivity of aerogel samples was investigated in various conditions. Aerogels are very diverse porous materials with high potential for the versatile applications. Already existing aerogels enable a very effective thermal insulation, supercapacitors, and carriers for different active agents. The goal of this investigation was first to find the change in the thermal conductivity of fibre aerogel samples after temperature treatment/thermal aging and wetting and then to reveal the application limit of this type of material.

The article pointed out the following objectives:

- At 23 ºC the moisture up-taking process finishes after 24 hours the latest, independently from the relative humidity. The sorption isotherm of the fibre reinforced aerogel at 23 ºC can be identified as BET IV type isotherm.
- Significant change in the thermal conductivity was found after wetting the samples for 24 hours at 23 ºC and over 65% relative humidity; showing greater than 10 % percent change. At lower humidities the thermal conductivity meets the applicability limit. Near 90 % it changes with about 20%.
- It was showed that during a time dependent (isothermal) annealing the thermal conductivity is stable at 70 ºC for at least 6 weeks. However some structural changes were observed. Aerogel precipitates were detected.
- Concluding from the results of the thermal conductivity measurements and from the microscopic images, besides the structure the thermal conductivity of the sample annealed over 180 ºC significantly changes (with about 20 %).
- Hydrophobicity tests showed no changes.

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Nomenclature

T: temperature (°C)

Q: heat flow (W)

A: area (m^2)

t: time (h)

λ=k: thermal conductivity (W/mK)

Δx=d: thickness (m)

R: resistance (m^2K/W)

ω: moisture content (g/g%)

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