Effect of various curing methods and addition of silica aerogel on mortar properties

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Research Paper

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Mechanical and thermal properties and porosity of aerogel-incorporated mortars exposed to various curing conditions (curing by wetting-drying, curing by magnesium sulphate ($MgSO_4$), and water curing) are experimentally investigated in this study. Maximum compressive strengths at 0.5 % aerogel content under the effects of wetting-drying and $MgSO_4$ curing conditions amounted to 60.8 MPa and 44.3 MPa, respectively. In addition, compared to the other curing methods, the gel pores formation in mortars exposed to $MgSO_4$ effects increased with an increase in aerogel content.

Key words:

aerogel, wetting–drying effect, magnesium sulphate effect, thermal conductivity, pore structure

Prethodno priopćenje

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Utjecaj raznih postupaka njege i dodavanja silicijskoga aerogela na svojstva morta

U ovome se radu eksperimentalno istražuju poroznost te mehanička i toplinska svojstva morta s dodatkom aerogela u raznim uvjetima njege (njega vlaženjem i sušenjem, magnezijevim sulfatom [$MgSO_4$] i njega vodom). Maksimalna tlačna čvrstoća morta sa sadržajem aerogela od 0,5 % iznosila je 60,8 MPa nakon njege vlaženjem i sušenjem te 44,3 MPa nakon njege magnezijevim sulfatom. Osim toga, u usporedbi s ostalim metodama njege, udio formiranih gel pora u mortovima njegovanima magnezijevim sulfatom raste s količinom dodanoga aerogela.

Ključne riječi:

aerogel, utjecaj vlaženja i sušenja, utjecaj magnezijeva sulfata, toplinska provodljivost, struktura pora

Vorherige Mitteilung

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Einfluss verschiedener Pflegeprozesse und Zugabe von Silica-Aerogel auf die Mörteleigenschaften

In dieser Arbeit werden die Porosität sowie die mechanischen und thermischen Eigenschaften von Mörtel unter Zusatz von Aerogel unter verschiedenen Pflegebedingungen (Behandlung mit Benetzung und Trocknung, Magnesiumsulfat [MgSO4] und Wasser) experimentell untersucht. Die maximale Druckfestigkeit des Mörtels mit einem Aerogelgehalt von 0,5 % betrug nach der Behandlung mit Benetzung und Trocknung 60,8 MPa und nach der Behandlung mit Magnesiumsulfat 44,3 MPa. Darüber hinaus steigt im Vergleich zu anderen Pflegemethoden der Anteil mit der Menge des zugesetzten Aerogels der gebildeten Gelporen in Mörteln, die mit Magnesiumsulfat behandelt werden.

Schlüsselwörter:

Aerogel, Einfluss der Benetzung und Trocknung, Einfluss von Magnesiumsulfat, Wärmeleitfähigkeit, Porenstruktur

1. Introduction

With the high rate of scientific and technological changes in the construction materials sector and in all other sectors, the production of technological materials with superior features, and the development of existing materials, has become quite inevitable. In this respect, the idea of enhanced thermal insulation through the developed pore structure in the cement based materials has an important place in innovation strategies as a means to limit energy consumption considering the decrease in energy resources. Numerous experimental studies on additive materials such as mineral wool, expanded polystyrene, extruded polystyrene, polyurethane, aerogels, nano insulation materials etc., are currently conducted for this purpose [1].

The improvement of thermal insulation through contribution of silica aerogels to C-S-H structure of materials has become an interesting and attractive subject in recent years. Silica aerogels, first produced by Kistler in 1931, and considered as one of the world's lightest solid materials, have become scientifically attractive materials that have a wide range of applications due to their high surface area, total porosity values that can be found in 99 % and low density [2, 3].

Aerogel is generally used as a thermal insulator additive in mortar and concrete applications. Experimental studies carried out with silica aerogel admixtures have generally focused on large volume scale (50-90 %) aerogel replacement with sand in mortar or concrete mixtures. The scope of these studies, conducted with regard to the decrease in thermal conductivity coefficient, is often restricted to the production of non-load-bearing elements having low mechanical strengths and the use of insulating plastics. A meaningful correlation between the thermal insulation target and the mechanical strength exists, and this relation limits the structural use of the produced cementitious materials [1, 4, 5]. The first experimental study in which silica aerogels with granular form were placed in cement matrix was carried out by Ratke in 2008 [6].

Ratke preferred, in his study, the cement types of CEM II 32.5 R, CEM I 42.5R and CEM I 52.5R in mixtures containing 50-70 % of aerogel by volume. When silica aerogel was used at 70 % by the volume, compressive strengths ranged from 0.6 to 1.5 MPa corresponding to a thermal conductivity coefficient of 0.010 W/mK [7].

Hub et al. obtained the compressive strengths in the range of 1.4 – 2.5 MPa from the mixtures including 65-75 % aerogel by the volume corresponding to a thermal conductivity coefficient interval of 0.10-0.14 W/mK [8].

Gao et al. investigated the change of mechanical strength and thermal conductivity coefficient by producing aerogel-incorporated concrete mixtures at the change interval of 0-60 % in volume. They determined that the thermal conductivity coefficients decline as the density and mechanical strengths decrease, while the aerogel content increases. They found a 8.3 MPa compressive strength value corresponding to the thermal conductivity of 0.26 W/mK at 60 % aerogel content rate by volume [4].

Fickler et al. prepared various mixtures in which aerogel ratio varied from 60 % and above by volume for high-performance aerogel concrete. They determined a compressive strength of 10.0 MPa corresponding to the thermal conductivity coefficient value of 0.17 W/mK in the mixture that gives optimum results [6].

Serina et al. determined a 20 MPa compressive strength corresponding to the thermal conductivity of 0.55 W/mK by testing mortar mixtures including 50 % aerogel rate by volume. However, the cases in which aerogel ratio exceeds the range of 50-60 % by volume are not recommended in terms of mechanical strength [9].

In their experimental work, Julio et al. prepared lightweight aggregated cementitious aerogel-based renders composed of 60 % aerogel and 40 % granular expanded cork, expanded clay, and perlite mixtures. A 0.92 MPa value of compressive strength was found corresponding to the thermal conductivity of 0.084 W/mK and 60 % total porosity values of the samples [10].

Kim et al. prepared gel-typed aerogels through methanol to reduce the difficulties encountered in mixing the aerogels in granular form and the capillary cracks during hydration. In the mortar mixtures, they used aerogels in gel-form at the rates of 0.5 %, 1.0 %, 1.5 % and 2.0 % by weight and obtained compressive strengths of 13.1 MPa, 8.0 MPa, and 5.9 MPa, respectively, while the compressive strength of the reference sample amounted to 26.3 MPa [11].

It can be deduced from these studies that the pore structures of mortar must also change in addition to an increase in thermal insulation properties resulting from aerogel effect. Generally, a porous structure is needed in the cement matrix for high thermal insulation properties. However, an exact opposite pore structure is required for high mechanical strength [1, 4, 5].

Similarly, when the effect of aerogel contribution on the concrete pore structure was investigated, it was found that the aerogel used at 20 % by volume can increase the total porosity value by 8.63 % through an additional pore volume created, especially at the porosity range of 10 - 30 nm [12].

Studies that examine the change of pore structure in aerogel-incorporated mortars under durability conditions are currently quite limited. It was established in [13] that the freezing-thaw cycles exert a significant negative effect on the thermal conductivity coefficient of aerogel-incorporated plaster mortars.

In the study conducted by Ng et al., 2016, it was established that curing conditions exert a significant effect on thermal conductivity coefficient and compressive strength of aerogel-incorporated samples. In samples with aerogel admixtures subjected to the first 24-hour moulding process at around 80 °C, followed by 28 days of curing at 80 °C in water, covered with aluminium foil, the compressive strength increased by 10 % compared to the reference sample, and a 35 % decrease in thermal conductivity coefficient was observed [14].

Magnesium sulphate, which is found in groundwater, sea water, or industrial liquid wastes, affects the hydration process and formation of hydration products in calcium-silicate-hydrate (C-S-H) based materials by Mg and SO_4 ions existing in its structure. Considering its negative effect on the hydration process, $MgSO_4$ is one of the dangerous salts that are effective in reducing service life of structural elements. In previous experimental studies, significant losses were observed in the modulus of elasticity, stiffness, compressive and flexural strengths of the structural elements exposed to $MgSO_4$ effect. As a result of hydration process, the formation of the C-S-H bond structure, which has quite a vital role in the gain of mechanical strength, is reduced by Mg salts and the formation of C-S-H is partially replaced by magnesium-silicate-hydrate (M-S-H) structure which is very fragile and does not have the capability of binding [15]. In order to reduce the negative effect of $MgSO_4$ on C-S-H structure, low porosity material designs are frequently examined through alternatives such as designs with high cement dosage, low water/binder ratio, and various salt-resistant mineral added binders. Porosity-controlled designs, which are provided with additional additives in mortar mixtures in order to improve their durability properties under the effect of $MgSO_4$, generally cause losses in mechanical strength of mortar despite the change in total porosity of mortar.

The originality of this investigation lies in revealing significant mechanical, thermal conductivity and porosimetric properties of silica aerogel - incorporated mortars under durability curing conditions such as wetting-drying and $MgSO_4$.

2. Materials and methods

2.1. Materials

CEM I 42.5 R type Portland type cement produced by Limak Cement Co. and standard CEN sand were used in mortar mixtures produced for the experimental works. Chemical composition and physical properties of the cement are presented in Table 1. In experimental studies, silica aerogel, which is preferred as additive material for improving thermal insulation properties of mortars, was provided by Alison Aerojel Co. The aerogel was produced according to technical specifications given in Table 2. Physical properties of aerogel used in the study are presented in Table 2.

Table 1. Chemical and physical properties of CEM I 42.5

Chemical composition (wt / %wt)		
SiO_2 (solute)		18.99
Al_2O_3		4.77
Fe_2O_3		3.15
CaO		63.69
MgO		1.12
SO_3		2.96
K_2O		0.64
Na2O		0.17
Cl -		0.0099
Loss on ignition		3.70
Insoluble residue		0.38
Total alkali		0.59
C_3A		7.32
Physical properties		
Setting time [min]	Initial	150
	Final	195
Specific gravity		3.11
Specific surface area [cm²/g]		3769
Volume stability [mm]		1

Table 2. Physical properties of silica aerogel

Properties	Aerogel
Pore diameter	8 – 10 nm
Apparent density	90 – 100 kg/m³
Surface area	790 – 840 m²/g
Porosity	> 94 %
Surface groups	OH-

2.2. Mixing and preparing specimens

In order to investigate mechanical, thermal and porosimetric properties of silica aerogel-incorporated mortars, aerogel added mortars mixtures for five content ratios in total, and three

Table 3. Mixture proportions of aerogel-incorporated mortars

Mixture	Aerogel content [%]	Cement content [%]	Aerogel [g]	Cement [g]	W/C	Water [ml]	Sand [g]
A1	0.1		0.45	450			
A2	0.3		1.36	450			
A3	0.5	100	2.25	450	0.50	225	1350
A4	0.7		3.16	450			
A5	1.0		4.50	450			

Table 4. Experimental test programme

Test	Number of specimens	Dimension [mm]	Standarts
Compressive strength	45	40 x 40 x 160	TS EN 196-1
Flexural strength	45	40 x 40 x 160	TS EN 196-2
Thermal conductivity	15 x 5 times	40 x 40 x 160	ASTM C-518
Mercury intrusion porosimetry	15	40 x 40 x 160	ASTM D-7984

samples from each mixture, were prepared for three different curing methods. Aerogel-incorporated cement mortars were formed selecting the content ratio as 0.1 %, 0.3 %, 0.5 %, 0.7 % and 1.0 % by weight of cement. Mix proportions of aerogel-incorporated mortars are shown in Table 3.

In the process of mixture preparation, a dry mixture containing cement, standard sand, and aerogel particles was initially formed. Water was subsequently gradually added to the dry mixture in order to provide for a uniform distribution of aerogel particles. A 0.50 water/cement ratio was adjusted in the mixtures. The total amount of cement and aerogel in the mixtures was considered as the total binder material.

After the end of the mixing process, cement mortar was placed in steel casts in which standard flexural specimens of 40 mm x 40 mm x 160 mm are produced and an effective compacting operation was carried out in order to prevent separation of mix components. Mixture samples were kept at 21 ± 2 °C for 24 hours and then removed from the casts. Samples were subjected to three different curing conditions, i.e. the wetting-drying effect, $MgSO_4$ effect, and water-curing.

2.3. Curing methods

Curing conditions involving the wetting-drying effect and $MgSO_4$ effect were provided in addition to conventional water-curing. The samples are prepared separately for each mixtures and three samples were produced in order to obtain the arithmetic mean of mixture samples for three different curing methods.

The samples produced for water curing were immersed in the curing tank at a temperature of 21 ± 2 °C and the curing was carried out continuously for 14 weeks.

Unlike the conventional water curing process, an alternative curing method was prepared to test the wetting-drying effect. Aerogel-incorporated mortar samples, which completed the wetting-drying curing process in the first week, were removed from the curing tank and kept at room temperature of 21 ± 2°C for the following week. The curing process for 14 weeks in total was performed in seven consecutive cycles. As a second alternative to the conventional water curing process, the curing method involving $MgSO_4$ was conducted. The specimens were kept in a curing tank, which contained 13 % $MgSO_4$ by weight, and kept for one week at a temperature of 21 °C ± 2 °C. Then in the following week they were oven-dried at 105°C. The curing process performed as seven consecutive cycles were completed in 14 weeks.

2.4. Testing

The samples, which completed the 14-week curing process for water curing, wetting-drying, and $MgSO_4$ effects, were subjected to flexural and compressive strength tests, thermal conductivity coefficient measurement, and mercury porosimetry (MIP) tests, respectively. The experimental test programme is shown in Table 4.

A three-point bending test was performed on standard mortar samples measuring 40 mm x 40 mm x 160 mm, and a 50 ± 10 N/s loading speed was applied. Flexural and compressive strength tests were carried out according to TS EN 196-1 and TS EN 196-2, respectively. The flexural strength of the mixture was determined by calculating the arithmetic mean of sample strengths, determined during strength tests conducted on all three samples from each mixture.

Compressive strength tests were carried out following the flexural strength tests by means of a contact area of 40 mm x 40 mm utilizing the two parts obtained from the flexural strength tests. The samples were subjected to a 2400±100 N/s vertical compression load, and the compressive strength value of the mixture was obtained calculating the arithmetic mean of the compressive strengths of the total of 6 samples.

Following the flexural and compressive strength tests, the thermal conductivity coefficient measurement and the MIP test were carried out on the appropriate sample pieces obtained from the mechanical tests.

The thermal conductivity coefficient measurement of aerogel-incorporated mortar samples under different curing conditions was carried out with the C-Therm device developed by TCi. This device measures the thermal conductivity coefficient of small samples using the Modified Transient Plane Source (MTPS) method. TCi can practically measure the thermal conductivity coefficient of the materials existing in solid, liquid, powder and mixed form in a short period of time differently from the other devices. In addition, the thermal conductivity coefficient can be determined by means of the device using only one side of the sample. The thermal conductivity coefficient measurement for each of the prismatic mortar samples was performed five times at different regions of the samples. Benefitting from this quite practical method, the conductivity coefficient can be measured within 1-3 seconds.

The MIP test was implemented using a Micromeritics device. The MIP is utilized to calculate the total porosity and pore size distribution of materials. The device operates at a pressure

range of 0.35-414 MPa and allows determination of pore diameters in the range of 3 nm to 360 micrometers.

For each curing method, 0.1 % aerogel-incorporated samples are considered as reference samples of their respective curing conditions.

3. Results and discussion

3.1. Compressive strength results

Compressive strengths of aerogel-incorporated mortars, associated with the curing process in water, wetting-drying, and $MgSO_4$ testing, are presented in Figure 1. Regarding the compressive strengths, the strengths in the sample groups, which are cured in water at the aerogel content rates increasing from 0.1 % to 1.0 %, changed in the range of 37.4 - 57 MPa, while the strengths varied in the 36.3 - 60.8 MPa range under the wetting-drying effect. In the case of $MgSO_4$ curing, partial compressive strength changes were determined in the range of 42.3-44.3 MPa for all content rates.
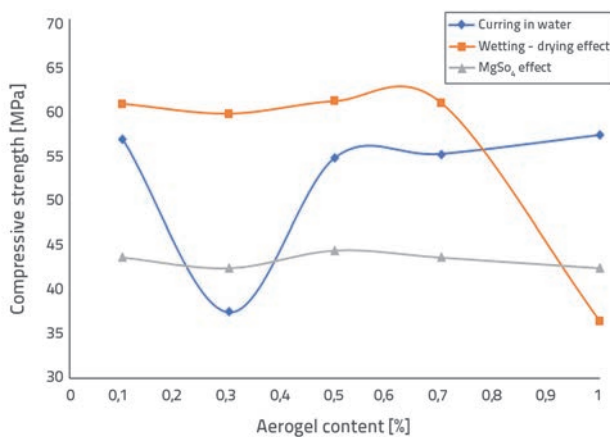

Figure 1. Relation between aerogel content and compressive strength test results of aerogel - incorporated mortars

The highest compressive strength results are generally obtained from the curing with the wetting-drying effect. In this curing method, similar compressive strengths, which are around 60 MPa, were obtained at 0.1-0.7 % aerogel content ratios, and the compressive strength decreased to 36.3 MPa, which means 40 % strength loss at 1.0 % content rate.

In the sample group that completed the curing process in water, the compressive strength was determined as 56.5 MPa at the lowest content rate (0.1 %). As the content rate was increased up to 0,5 %, 0,7 %, and 1,0 %, the compressive strengths were not adversely affected by the increased aerogel additive and the strengths for the corresponding content rates amounted to 54.5, 54.9 and 57 MPa, respectively. The minimum compressive strength of 37.4 MPa was obtained at the 0.3 % aerogel content ratio, which means that the strength loss was 33.8 % compared to the reference sample. The strength values are generally close to those that were obtained from the wetting-drying effect.

However, they were found to be lower. Although a significant decrease in strength (max. 2.75 %) does not appear for the samples which completed the curing process under $MgSO_4$ effect, the strengths are lower than the ones obtained by the remaining two curing methods.

3.2. Flexural strength results

Flexural strengths of aerogel-incorporated mortar samples, which completed the curing process in water and under the wetting-drying and $MgSO_4$ effects, are shown in Figure 2. Examining the flexural strength results, in the sample group which is cured in water at the aerogel content rates increasing from 0.1 % to 1.0 %, the strengths varied in the range of 6.6 – 8.1 MPa, while the strengths varied in the 8.1 – 9.1 MPa range under the wetting-drying effect. For the $MgSO_4$ effect, the flexural strengths were observed to vary from 7.4 to 8.5 MPa throughout the increase in additive content.
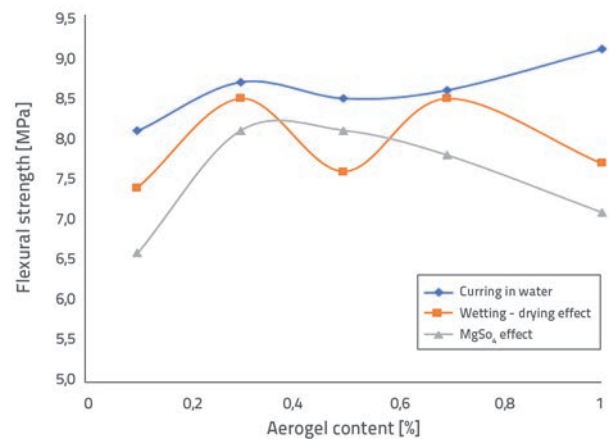

Figure 2. Relation between aerogel content and flexural strength test results of aerogel - incorporated mortars

The highest flexural strengths in individual curing groups were determined for the samples cured under the effect of wetting-drying. With an increase in content rate to 0.3 %, 0.5 %, and 0.7 %, flexural strengths values increased to 8.7, 8.5, and 8.6 MPa, respectively. The maximum flexural strength was found to be 9.1 MPa at the highest aerogel content rate (1.0 %). The improvement in flexural strength of up to 12.3 % was detected with the rise in the quantity of additive.

In the sample group which completed the curing process in water, the flexural strength of 6.6 MPa was found at the lowest content rate (0.1 %) and this value represents the lowest strength value obtained by the samples cured in water. By increasing the content rate to 0.3 %, 0.5 %, and 0.7 %, the flexural strengths amounted to 8.1, 8.1 and 7.8 MPa, respectively. The strength of the 1.0 % aerogel content ratio was reduced to 7.1 MPa, but the value is 7.5 % higher than that of the reference specimen.

In the sample group that completed the curing process under the $MgSO_4$ effect, the flexural strengths are higher than the ones

obtained by the curing in water experiments except for 0.5 % aerogel content ratio. While the lowest flexural strength was 7.4 MPa at the 0.1 % additive ratio, the strength reached 8.5 MPa, which means an increase of 14.8 % compared to the reference sample, with the increase in content rate of 0.3 % and 0.7 %.

3.3. Thermal conductivity test results

The thermal conductivity coefficients of aerogel-incorporated mortar samples that completed the curing process in water, under the wetting-drying, and $MgSO_4$ effects, are presented in Figure 3. The lowest thermal conductivity coefficient for curing in water amounted to 1.56 W/mK at the 0.3 % aerogel content. The minimum thermal conductivity coefficient resulting from the wetting-drying effect amounted to 1.80 W/mK at the 0.3 % aerogel content, and the lowest thermal conductivity coefficient for samples cured in $MgSO_4$ was found to be 1.61 W/mK at the 0.1 % aerogel content.
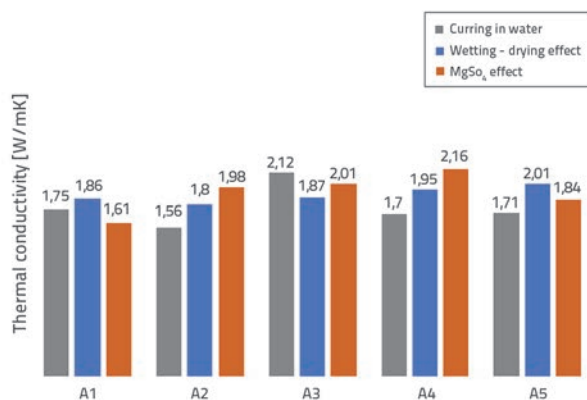

Figure 3. Thermal conductivity test results for aerogel-incorporated mortars

In the sample group that completed the curing process in water, the thermal conductivity coefficient amounted to 1.75 W/mK at the 0.1 % aerogel content. As a result of using 0.3 % of aerogel additive, a decrease of 10.8 % in thermal conductivity coefficient was determined compared to the reference sample, and the conductivity was 1.56 W/mK. With an increase in aerogel content to 0.7 % and 1.0 %, the conductivity coefficients amounted to 1.70 W/mK and 1.71 W/mK, respectively, and these values, which are

quite close to each other, imply a limited conductivity decrease of 2-3 % compared to the reference sample. The conductivity coefficient, established as 2.13 W/mK at the 0.5 % aerogel content, is the highest conductivity coefficient in the water curing group. The aerogel content of 0.3 % seems to be the optimum point of aerogel contribution in terms of the thermal conductivity coefficient for the water curing group.

In the sample group that completed the curing process involving the wetting-drying effect, the conductivity coefficient at 0.1 % aerogel content ratio was 1.86 W/mK. For the sample group which was cured in water, the maximum conductivity reduction occurred at the 0.3 % aerogel content. However, the conductivity coefficient decreased only by 3.3 % at the same content rate under the wetting–drying curing effect. The conductivity coefficient (1.87 W/mK) determined at 0.5 % aerogel content ratio was almost the same as the one obtained at the 0.1 % content and the decrease was not observed at relatively high aerogel contents.

In the samples which completed the curing process involving treatment with $MgSO_4$, higher thermal conductivity coefficient values were determined compared to the ones obtained at 0.1 % aerogel additive ratio depending on the increase in aerogel content. Even though an increase in aerogel content causes the increment in thermal conductivity under the effect of $MgSO_4$, the thermal conductivity coefficient of 1.61 W/mK observed at 0.1 % aerogel content is remarkable since the coefficient is the second lowest among the values obtained from different curing methods.

3.4. MIP test results

The results of porosimetry analysis of aerogel-incorporated mortar samples which completed the curing process in water, under the wetting-drying and $MgSO_4$ effects are presented in Table 5.

According to porosimetry results for samples cured in water, the highest total porosity value (15.29 %) of the curing group was determined for A2 specimen with 0.3 % aerogel content where the lowest compressive strength (37.4 MPa) was obtained. The compressive strengths for samples A1 (56.5 MPa), A4 (54.9 MPa) and A5 (57.0 MPa), which are close to each other, correspond to total porosity values of 14.77 %, 14.46 % and 13.07 %, respectively. Thus, similar total porosity values in the range of 13-14 % were determined for those samples. The lowest median pore diameter-volume values of 93.4 and 79.9

Table 5. MIP test results of aerogel - incorporated mortars

Mixture	Water curing group		Wetting – drying curing group		MgSO₄ curing group	
	Total porosity [%]	Median pore diameter-volume [nm]	Total porosity [%]	Median pore diameter-volume [nm]	Total porosity [%]	Median pore diameter-volume [nm]
A1	14..77	93.4	13.64	117	2.63	163.8
A2	15..29	103.4	14.96	117.4	2.68	173.3
A3	2..4	113.7	14.46	109.6	14.49	168.2
A4	14.46	121.4	12.17	47.2	2.2	156.3
A5	13.07	79.9	14.15	109.2	13.58	164

nm were obtained for A1 (6.6 MPa) and A5 (7.1 MPa) samples, respectively, which had the lowest flexural strengths in the water curing group.

In the sample group cured by the wetting-drying procedure, compressive strengths in the range of 59.4– 60.8 MPa were obtained at the 0.1-0.7 % aerogel content, as a result of total porosity values which varied from 12.17 to 14.96 %. The lowest compressive strength in the curing group amounted to 36.3 MPa at the highest aerogel content (A5).

It was observed that compressive strengths of the samples cured by $MgSO_4$ increased under the influence of the median pore diameter-volume with an increase in the aerogel content. The change in compressive strength varies between 42.3 and 44.3 MPa and provide the maximum value of 2.75 % depending on the limited changes in median pore diameter-volume in the range of 156.3 – 173.3 nm.

The cumulative pore volume - pore size distribution, and the relation between content of pores - thermal conductivity - compressive strength of the samples cured in water, are shown in Figure 4a and Figure 4b, respectively.
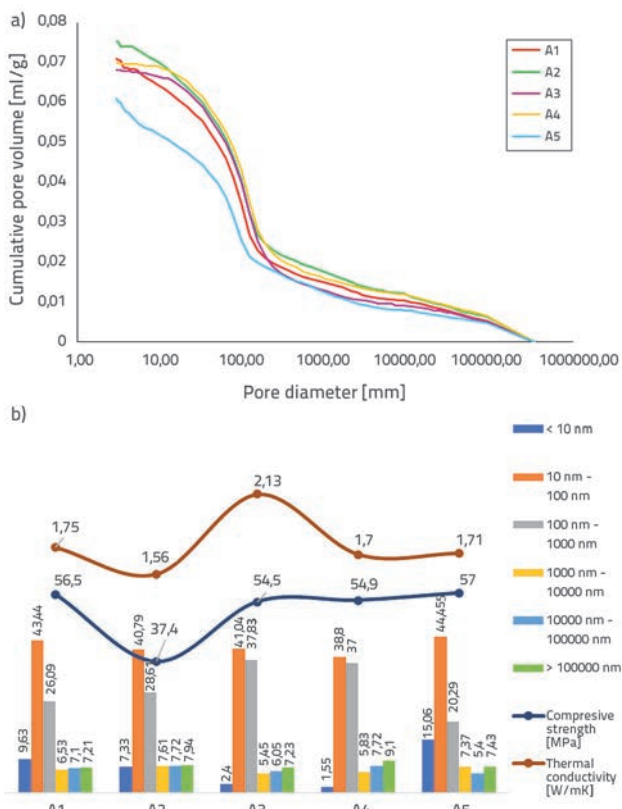

Figure 4. a) Pore size distribution for all samples cured in water obtained from MIP data; b) Relation between content of pores, thermal conductivity and compressive strength for the group cured in water

Among the samples that completed the curing process in water, the samples A2 (1.56 W/mK) and A4 (1.70 W/mK), which have the lowest thermal conductivity coefficient, exhibited the highest cumulative pore volume compared to other samples in the diameter range of 3 and 340000 nm throughout the chart.

The A1 sample (1.75 W/mK) exhibited a cumulative pore volume higher than that of the A4 sample only for a diameter range of 3 - 3.50 nm. However, this behaviour ended at around 3.50 nm. Because of this behaviour throughout the graph, the lowest thermal conductivity coefficients were determined in samples A2 and A4. In the capillary diameter range from 225 to 9000 nm, the A2 sample exhibited a higher cumulative pore volume than the A4 sample but, at higher diameters, the A4 sample exhibited a higher cumulative pore volume compared to the A2 sample. Therefore, the total porosity value of A2 was higher (15.29 %) than that of A4 and other samples in the curing group within the capillary diameter range of 225 - 9000 nm. The median pore diameter-volume (121.4 nm) of the A4 sample was higher than that of A2 and other samples in the cure group due to behaviour in the range from 9000 nm to 340000 nm. In particular, when the diameter distribution at 10000 nm and above is examined, macro pores formation of the highest volumetric ratio (15.6 % and 16.8 %) of the sample group cured in water was observed in A2 and A4 samples due to cumulative pore volume in the upper region of the graph. The formation of macro pores at high levels exerts a considerable influence on the thermal conductivity coefficient.

The highest thermal conductivity coefficient (2.13 W/mK) in the water-cured group was found in A3 sample. Therefore, the lowest total porosity value of the curing group (2.4 %) is observed for the same sample. Considering the highest thermal conductivity coefficient and the lowest total porosity value, the results are thought to be consistent.

Although the total porosity value of the A3 sample is quite different from that of the other samples, the compressive strength of the sample (54.5 MPa) is similar to the compressive strengths of the samples A1, A4 and A5 (54.9 - 57 MPa), since the distribution of pore diameters is similar to that of other samples in the curing group.

The lowest total thermal conductivity coefficient (1.56 W/mK) and the lowest compressive strength (37.4 MPa) of the curing group for the sample A2 having the highest total porosity value of the curing group is quite reasonable and consistent. Since the total porosity value of the A1 sample (14.77 %) was lower than that of the A2 sample, it was also very consistent to determine the higher compressive strength (56.5 MPa) and thermal conductivity coefficient (1.75 W/mK) in the A1 sample compared to the A2. However, examining the mixture content ratios of the samples A1 and A2, it was observed that there is only a difference in the amount of aerogel content that increases from 0.1 % to 0.3 % by weight of cement. Therefore, the influence of aerogel content from 0.1 % to 0.3 % on the compressive strength and thermal conductivity coefficient of mortar is remarkable.

Previous experimental studies have shown that the aerogel additive can provide thermal insulation without significant loss in compressive strength. Similarly, in this study, thermal conductivity coefficient decreased from 2.13 W/mK to 1.7 W/mK with an increase in aerogel content from 0.5 % to 0.7 % in A3 and A4 samples. The decrease in thermal conductivity did not cause a strength loss and, expectedly, a strength gain was observed by means of aerogel content.

It was determined that a similar effect cannot be achieved with the A1 - A2 content rate. At this transition rate, the thermal insulation provided with aerogel content and loss of compressive strength are related to a greater macro pores formation (15.6 %) in the pore structure at 0.3 aerogel content, compared to other samples. Although it is known that aerogel particles are stable during hydration and that they maintain their high mechanical properties under high total porosity - high macro-pore structure with low aerogel content, it cannot contribute to the compressive strength of mortar due to poor adhesion.

The cumulative pore volume - pore size distribution and the relation between content of pores - thermal conductivity - compressive strength of the samples cured by wetting-drying, are presented in Figure 5a and Figure 5b, respectively.
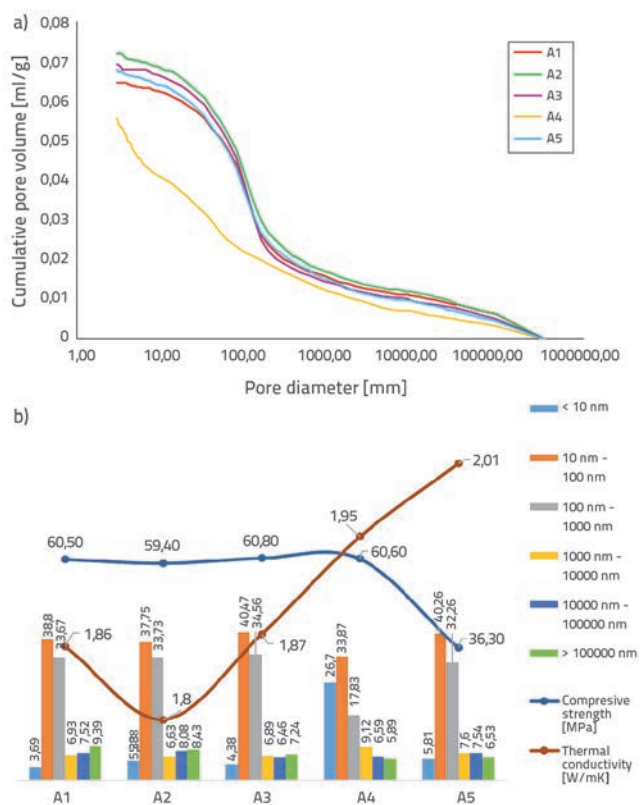

Figure 5. a) Pore size distribution for all samples subjected to wetting – drying cycles obtained from MIP data; b) Relation between content of pores, thermal conductivity and compressive strength for wetting-drying curing group

Among the samples which completed the curing process under the wetting-drying effect, A2 sample with the lowest coefficient of thermal conductivity (1.80 W/mK) showed the highest cumulative pore volume in the range of 3 – 340000 nm diameters along the graph, compared to other samples. The A2 sample also does not show deviation in any region throughout the graph and, therefore, the highest total porosity (14.96 %) and the highest median pore diameter-volume (117.4 nm) values in the group are exhibited by this sample. As a result of these porosimetric findings, the lowest thermal conductivity

coefficient was obtained at sample A2. If sample behaviour in the diameter range of 10000-340000 nm is examined, it can easily be observed that samples A4 and A5 always exhibit the lowest cumulative pore volume. In particular, median pore diameter-volume (47.2 nm) and the total porosity value (12.17 %) of the A4 sample, which exhibits the lowest cumulative pore volume at all diameters throughout the graph, are the lowest porosimetric values in their curing group. As a result of these porosimetry analysis findings, both samples present the highest thermal conductivity values in their curing groups (1.95 W/mK and 2.01 W/mK).

The cumulative pore volume - pore size distribution, and the relation between content of pores - thermal conductivity - compressive strength of the samples that completed the curing process under the MgSO$_4$ effect, are presented in Figures 6a and 6b.
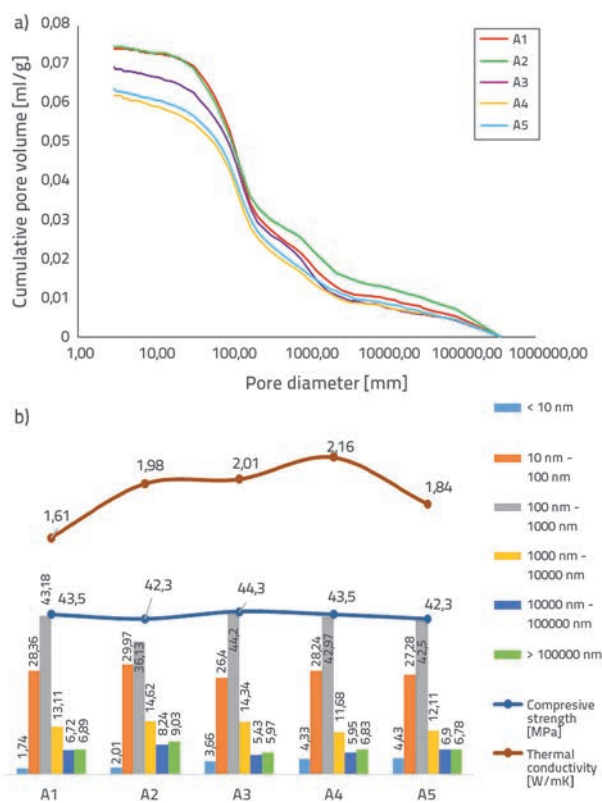

Figure 6. a) Pore size distribution for all samples subjected to MgSO$_4$ exposure obtained from MIP data; b) Relation between content of pores, thermal conductivity and compressive strength for MgSO$_4$ curing group

The A1 (1.61 W/mK) sample, which has the lowest thermal conductivity coefficient among the MgSO$_4$–cured samples, exhibited the highest cumulative pore volume in the diameter range of 3 - 120 nm compared to other samples. The A1 sample showed the second highest cumulative pore volume behaviour after the A2 sample in the diameter range of 120-340000 nm. The A1 sample has a higher rate of capillary pores distribution (84.6 %) and lower gel pores content (1.74 %) in the mortar pore structure compared to sample A2.

The total porosity values of the samples belonging to the MgSO$_4$ curing group (2.63 %, 2.68 %, 14.49 %, 2.2 %, 13.58 %) varied in a wide range compared to each other. However, this variability is not reflected in the compressive strength results (43.5 MPa, 42.3 MPa, 44.3 MPa, 43.5 MPa, 42.3 MPa, respectively). The highly uniform distribution of the compressive strength results is related to the relatively close median pore diameter – volume values of mortar samples (163.8 nm, 173.3 nm, 168.2 nm, 156.3 nm, 164 nm, respectively). The increase in gel - pore formation having a high correlation with an increased aerogel content in the MgSO$_4$ curing group indicates that representation of pore structure would be more accurate using median pore diameter – volume than employing the total porosity.

The determination of quite different total porosity values in spite of uniform median pore diameter – volume and compressive strength values under the effect of MgSO$_4$ shows that the effect of MgSO$_4$ on the pore structure of the mortar involves formation of pore geometry and connectivity that cannot be explained with pore diameters only. Nevertheless, for the MgSO$_4$ curing group, highly correlated and controlled effect created by silica aerogel, which is used in experimental studies in diameter range of 8 – 10 nm, for a diameter of not more than 10 nm in the gel formation, could provide uniform distribution of compressive strengths although total porosity value was varying.

Volumetric pore levels in pore structures of aerogel-incorporated mortars under the effect of different curing conditions are shown in Figure 7.
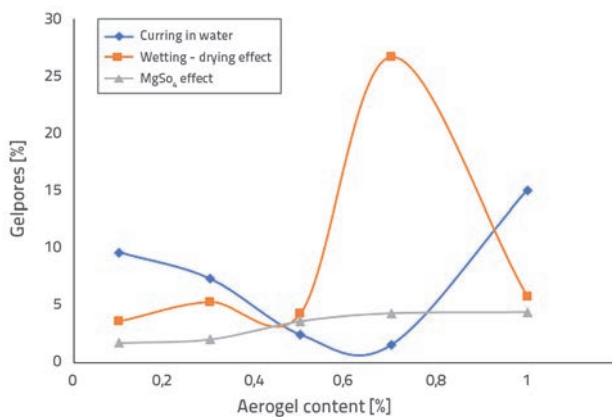

Figure 7. Relation between aerogel content and distribution of gel pores of aerogel-incorporated mortars

The gel pores formation at low content rates of 0.1 % and 0.3 % for the samples that completed the curing process in water are 9.6 % and 7.3 %, respectively. The formation of gel pores decreases to minimum levels of 2.4 % and 1.5 % with an increase in aerogel content to moderate levels of 0.5 and 0.7 %, respectively. The gel pores formation reaches the maximum value of 15 % at the high aerogel content of 1.0 %. At the moderate level content of 0.5 and 0.7 % (samples A3 and A4), the gel pores formation decreases to a minimum level, which is accompanied by stabilisation of compressive strengths at 54.5 and 54.9 MPa, and flexural ones at 8.1 and 7.8 MPa. The A5 sample where the gel pores formation

reaches the maximum level (15 %) exhibited the maximum compressive strength (57.0 MPa) in the curing group.

Gel pores formation is at the level of 3.6 - 5.8 % at other content rates excluding the rate of 0.7 % for samples which completed the curing process under the effect of wetting-drying. The gel pores formation at the rate of 0.7 % aerogel content reaches up to 26.7 %, which is the maximum value in the curing sample group. Due to the high gel pores formation at this content ratio, the median pore diameter-volume of A4 sample at the aerogel content rate of 0.7 % decreases to 47.2 nm, which is the minimum of the curing group, unlike the other samples. Therefore, the lowest total porosity value (12.17 %) and high compressive strength (60.6 MPa) of the curing group could be achieved at the A4 sample.

Due to an increase in aerogel rate in the MgSO$_4$ curing group, the gel pores formation also increased steadily and finitely. The gel pores formation regularly increased from 1.7 % to 4.4 % with an increase in aerogel content from 0.1 to 1.0 %.

Figure 8 shows volumetric capillary porosities of pore structure in aerogel-incorporated mortars under various curing conditions.
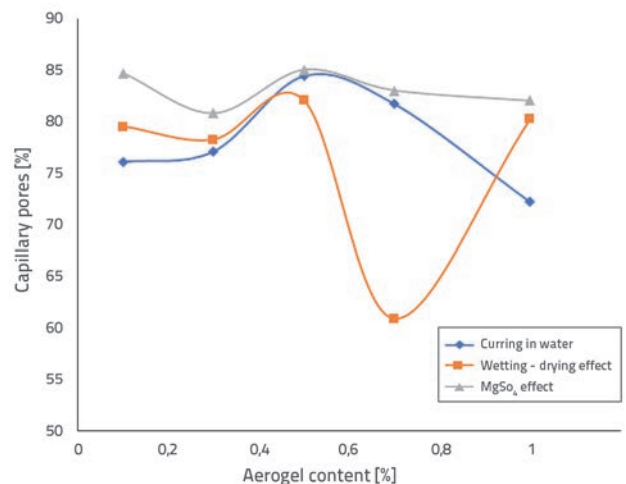

Figure 8. Relation between aerogel content and distribution of capillary pores in aerogel-incorporated mortars

Capillary pores formation varies from 72.2 to 85.0 % with an increase in aerogel content in water-cured samples. The highest capillary pores formation was observed at the 0.5 % aerogel content in sample A3.

The lowest total porosity value (2.4 %) and the highest thermal conductivity coefficient (2.13 W / mK) of the curing group were observed in sample A3 where the capillary pores formation was observed at the highest level (85 %) regarding the results of the total porosity and thermal conductivity coefficient of the group. For the same reason, although the compressive strengths (54.5 and 54.9 MPa) were similar for A3 and A4 samples, thermal conductivity coefficients (2.13 and 1.7 W/mK) are quite different due to dissimilar capillary pores distributions (85 % and 81.7 %). The difference in the capillary pores levels of the samples, as well as the total porosity effect, had an impact on the conductivity results. Capillary pores formation varied from 60.9 % to 82.0 % due to an increased aerogel content in the samples

cured by wetting-drying. The lowest capillary pores formation was observed at sample A4 with the 0.7 % aerogel content. The lowest total porosity (12.17 %) and median pore diameter-volume (47.2 nm) values of the curing group were obtained at sample A4 where the lowest formation level of capillary pores (60.9 %) was established, when evaluating the total porosity and thermal conductivity coefficients results of the group. Due to this reason, the high compressive strength (60.6 MPa) and flexural strength (8.6 MPa) of the curing group was observed at sample A4. The thermal conductivity coefficient of sample A4 is quite high (1.95 W/mK) compared to other samples in the curing group.

The formation of capillary pores in pore structure due to an increased aerogel content in the $MgSO_4$ curing group varied from 80.8 to 85 %. The change in capillary pores depending on the content rate is rather limited compared to other curing methods. As previously mentioned, since the median pore diameter - volume changes in a limited range of 156.3 nm-173.3 nm in the samples exposed to $MgSO_4$ effect, the change in compressive strengths also varies in the 42.3 – 44.3 MPa range and provides a maximum value of 2.75 %. The volume of macro pores in pore structure in aerogel-incorporated mortars under different curing conditions is shown in Figure 9.
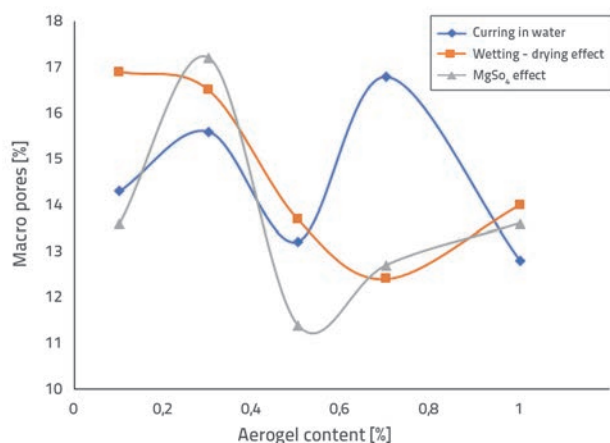

Figure 9. Relation between aerogel content and distribution of macro pores in aerogel-incorporated mortars

The macro pores formation varies from 12.8 to 16.8 % due to an increased aerogel content in the samples that completed the curing process in water. The lowest thermal conductivity coefficients in the curing group of 1.56 W/mK and 1.70 W/mK were registered for samples A2 (15.6 %) at 0.3 % and A4 (16.8 %) at 0.7 % content, which also exhibited the highest macro pores formation. The highest total porosity (15.29 %) and the lowest compressive strength (37.4 MPa) of the cure group were determined at sample A2 due to high proportion of macro pores exceeding 15 %. The median pore diameter-volume of sample A4 reached 121.4 nm, which is the highest value of the curing group due to the maximum pore formation of 16.8 %.

The macro pores formation varies in the range of 12.4 – 16.9 % depending on the increase in aerogel content of the samples which completed the curing process under the effect of wetting-drying. Their lowest thermal conductivity coefficients amounted to 1.86 W/m.K and 1.80 W/mK, which showed the highest macro pores formation: sample A1 (16.9 %) at 0.1 % aerogel content and sample A2 with 0.3 % content (16.5 %). The highest median pore diameter-volume of the curing group amounted to 117 and 117.4 nm for A1 and A2 samples with a high macro pores formation.

The macro pores formation changes in a range of 11.4 – 17.2 % in the curing group under the effect of $MgSO_4$ relying on the increase in the aerogel content. The highest median pore diameter-volume (173.3 nm) of the curing group was found at sample A2 (17.2 %), which demonstrated the highest macro pores formation of 0.3 %.

4. Conclusions

The mechanical and thermal conductivity coefficients and porosimetric properties of cement mortars under different curing conditions for the 0.1 - 1.0 % aerogel content range are investigated experimentally in this study.

In-water curing group
- Compressive strength results are lower than the ones obtained from the wetting-drying experiments even though they are quite close to each other.
- Despite the increased aerogel content, the maximum change in compressive strength is around 3.5 % excluding the 0.3 % content ratio. Total porosity values have an influence in compressive strength changes.
- The flexural strength of 0.3 % and 0.5 % aerogel content reaches the maximum value of the curing group.
- The lowest thermal conductivity coefficient of the curing group was found as 1.56 W/mK at 0.3 % content ratio.
- The maximum capillary pores formation (85 %) was observed at 0.5 % aerogel content. The lowest total porosity value (2.4 %) and the highest thermal conductivity coefficient (2.13 W/mK) were observed in this sample.
- 0.3 % and 0.7 % aerogel-incorporated samples, which demonstrate the highest cumulative pore volume, have the lowest thermal conductivity coefficient (1.56 W/mK) and (1.70 W/mK) of the curing group for the diameter of 10000 nm and above.

Wetting-drying curing group
- The highest compressive strengths are usually found in samples that completed the curing process under the wetting-drying effect.
- Despite the increased aerogel content, the maximum change in compressive strength is 1.8 % except for the 1.0 % content. The total porosity values have an impact on changes in compressive strengths.

- The highest flexural strengths were determined in this curing group compared to the other groups.
- The lowest thermal conductivity coefficient measured in this curing group amounted to 1.80 W/mK for 0.3 % aerogel content.
- The minimum capillary pores formation in the mortar pore structure was determined at 0.7 % aerogel added sample. The lowest total porosity value (12.17 %) and the lowest median pore diameter - volume (47.2 nm) of the curing group were determined in this sample. A high compressive strength (60.6 MPa) was also obtained from the sample by means of the aforementioned porosimetric results.

$MgSO_4$ curing group
- The lowest compressive strengths were determined on samples that completed the curing process under $MgSO_4$ effect. Median pore diameter – volume values have an impact on the changes in compressive strength.
- Despite an increase in aerogel content, the maximum change in compressive strength is 2.75 %. In the limited change amongst the compressive strengths, median pore diameter-volume varying in the range of 156.3 – 173.3 nm were effective.
- Even though flexural strengths are lower compared to the wetting - drying curing method, the strength values are higher than the ones obtained from the curing in water experiments except for the 0.5 % aerogel content.
- Formation of gel pores in mortar pore structure increases consistently depending on an increase in aerogel content.

The lowest thermal conductivity coefficient of the curing group amounted to 1.61 W/mK at the 0.1 % aerogel content. This value is the second lowest coefficient of thermal conductivity amongst all mixture samples exposed to different curing conditions.

It can be observed that higher flexural and compressive strengths can be obtained for all aerogel contents in the wetting-drying curing method compared to the water curing method. For this reason, in the production of prefabricated structural elements with high mechanical strength expectations, the wetting-drying method seems to be an attractive curing process compared to the traditional curing in water method. The compressive and flexural strength gains that were obtained even at room temperature point to larger mechanical strength gains for the prefabricated structural elements to be produced under higher curing temperature.

Similarly, flexural strengths in the $MgSO_4$ curing method are generally higher than in the conventional water-curing method. Especially under the $MgSO_4$ effect, it should be noted that the mechanical strength of mortar remains stable despite the change of total porosity value of mortar samples in the range of 2.2-14.49 % with aerogel content. Therefore, aerogel incorporation is an attractive idea in the design of structural elements exposed to the effect of $MgSO_4$ compared to other traditional additives used in improving durability characteristics of the elements under the influence of sulphate that lead to a loss of mechanical strength.

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