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Porosimetric, Thermal and Strength Tests of Aerated and Nonaerated Concretes

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Abstract. The paper presents the results of porosimetry tests of lightweight concretes, obtained with three research methods. Impact of different porosity structures on the basic thermal and strength properties was also evaluated. Tests were performed, using the pressure gauge method on fresh concrete mixes, as well as using the mercury porosimetry test and optic RapidAir method on specimens prepared from mature composites. The study was conducted on lightweight concretes, based on expanded clay aggregate and fly ash aggregate, in two variants: with non-aerated and aerated cement matrix. In addition, two reference concretes, based on normal aggregate, were prepared, also in two variants of matrix aeration. Changes in thermal conductivity λ and volumetric specific heat c_v throughout the first three months of curing of the concretes were examined. Additionally, tests for compressive strength on cubic samples were performed during the first three months of curing. It was found that the pressure gauge method, performed on a fresh mix, gave lowered values of porosity, compared to the other methods. The mercury porosity tests showed high sensitivity in evaluation of pores smaller than 30µm. Unfortunately, this technique is not suitable for analysing pores greater than 300µm. On the other hand, the optical method proves good in evaluation of large pores, greater than 300µm. The paper also presents results of correlation of individual methods of porosity testing. A consolidated graph of the pore structure, derived from both mercury and optical methods, was presented, too. For the all of six tested concretes, differential graphs of porosity, prepared with both methods, show a very broad convergence. The thermal test results indicate usefulness of aeration of the cement matrix of the composites based on lightweight aggregates for the further reduction of the thermal conductivity coefficient λ of the materials. The lowest values of the λ coefficient were obtained for the aerated concretes based of fly ash aggregate. A diminishing influence of aeration on the volumetric heat capacity c_v is clearly seen. Simultaneous aeration of the matrix and use of lightweight aggregates brought about also a significant decrease in the average compressive strength f_{cm} of the tested composites.

1. Introduction

Development of modern lightweight concretes provides huge possibilities for their application in construction of various elements of buildings, starting from partition walls and floors, ending with structural elements. Nowadays the latter ones are not only required to be strong, but also their insulating and heat accumulating capacities must meet certain standards. Due to the fact that current requirements demand reduction of heat consumption in buildings, the emphasis is placed on specific designs of external building partitions, so that they presented appropriate thermal performance, with no compromise as to the strength parameters. Lightweight concretes can be used with success to build external partitions, as their both thermal and mechanical properties can be modelled easily. In the literature of the subject it is possible to find descriptions of e.g. works consisting in substituting natural aggregates with pumice or dune sand [1] and assessments of their impact on the strength parameters and the thermal conductivity. Other authors [2] tested the influence of ultra-lightweight concretes on energy-saving parameters of the entire building, as well as on the thermal comfort. It has been proven that structures built in this technology prevent over-heating better than lightweight wood structures. Moreover, lightweight concretes make it possible to utilize some industrial waste materials. In paper [3] influence of aggregates produced from blast furnace ashes was studied. Similarly, in paper [4] influence of rubber granulate on qualities of light concretes was analysed.

Because of high porosity of aggregates used in production of lightweight concretes, also the possibility of using the phase change materials, by putting it directly to the aggregate, is tested [5], [6]. This way it is possible to produce a material with very high capacities to store heat energy, with simultaneously high thermal insulation performance. Also, an aerogel additive is used in lightweight mixes [7], resulting in improved thermal insulation properties. This is caused by the increase of the porosity of the whole product, thanks to the porous structure of the aerogel. Moreover, impact of aerogel on thermal and strength parameters of lightweight concretes produced on the basis of fly ash aggregate was tested [8].

Porosity of lightweight concretes and mortars is the main factor that has an effect on their both thermal and strength properties [9]. Therefore, in this paper the authors decided to study the porosity structures of various concrete composites, using different porosity testing methods, and then to compare results obtained with different techniques. This paper presents also results of tests for thermal properties and compressive strength. The experiment covered three basic groups of concretes, where from each group aerated (marked as A) and non-aerated (marked as N) mixes were prepared.

2. Materials and methods

The tests were performed on lightweight concretes, prepared on the basis of expanded clay aggregate (EC) and fly ash aggregate (FA). Recipes for non-aerated cement matrix (N) and strongly aerated matrix with aerating admixture (A), applied as 1.1% of the cement mass were prepared. Additionally, two standard non-aerated concrete (R/N) and aerated concrete (R/A) mixes were prepared, where both were used for reference. Composition of each of the recipes is shown on table 1.

Table 1. Concrete recipes prepared for testing

Type	Type of aggregate	Initial moisture of coarse aggregate	Coarse aggregate	Sand	Cement	Water	Aerating admixture	Aerating admixture
		[%]	[kg/m^3]	[kg/m^3]	[kg/m^3]	[kg/m^3]	[kg/m^3]	[%]
R\N	Normal	0	1312.73	469.53	391.27	215.20	0.00	0.00
R\A		0	1030.86	370.52	308.76	169.82	3.40	1.10
FA\N	Fly ash	21	844.55	471.10	392.58	215.92	0.00	0.00
FA\A		21	632.78	352.95	294.13	161.77	3.24	1.10
EC/N	Expanded clay	28	338.38	486.87	405.73	223.15	0.00	0.00
EC/A		28	290.10	418.08	348.40	191.62	3.83	1.10

For each of the concrete mixes, six plate specimens, sized 16x14x4cm, were produced for the thermal tests. Additionally, 20 specimens sized 10x10x10cm were produced, to conduct the strength and porosimetric tests. The samples were first stored for 28 days in a high humidity climate box. Then the specimens were drying in dry-air laboratory conditions. Figure 1 presents sections of the concretes, magnified 10-fold.

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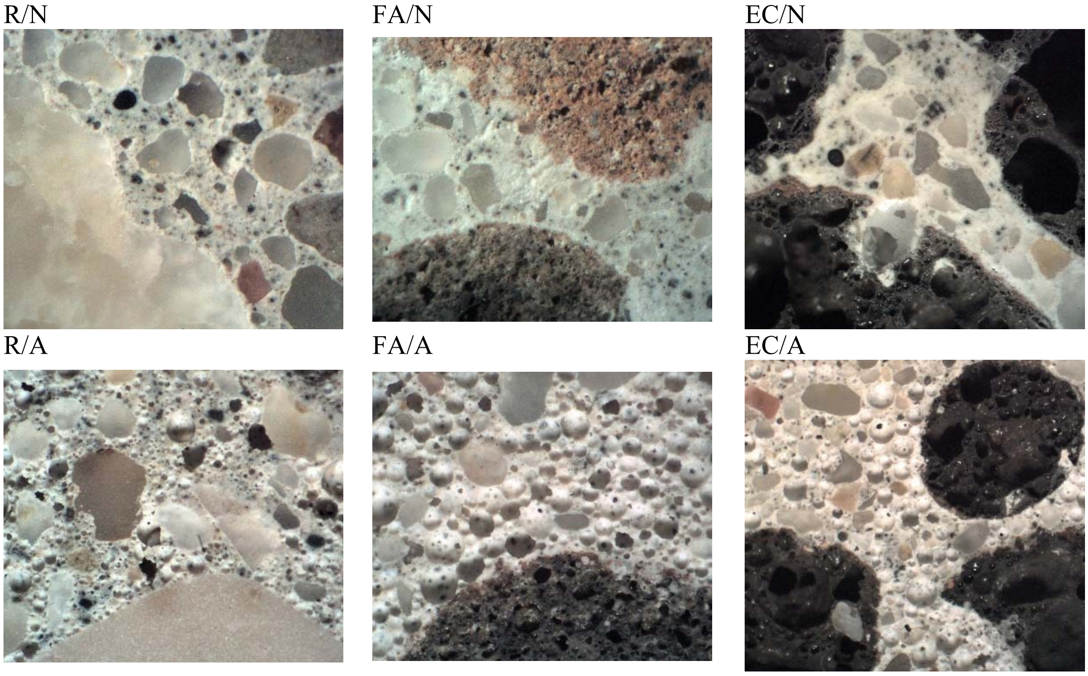

Figure 1. Cross-section of the tested concretes under 10x magnification

During preparation of the concrete mixes, the first test for air content in the fresh mix was conducted with the pressure gauge method, in a 5-liter tank after a proper preparation and consolidation of the mix in a standardised cylinder. By standard, this test is not recommended for concretes based on lightweight aggregates due to their high porosity. Despite that, the experiment was conducted on all concretes in order to compare the results to data obtained from other tests.

The second porosity test was performed, using the mercury intrusion porosimetry method. After three months of curing, from the cubical specimen central sections were cut out, and from these specimens sized 0.7x0.7x2.0cm were formed. The specimens were selected in such a way that they included both a piece of the cement matrix and some of coarse aggregates. Each concrete underwent two tests to verify repeatability of the results. The mercury surface tension was assumed as equal to 0.48N/m, and the contact angle was set to 140 degrees upon intrusion. As the first step, the specimens were made subject to low pressure (max. 0.34MPa), then the cells containing the specimens filled with mercury were weighed. Next, they were put into a pressure chamber and subjected to high pressure (up to ca. 413MPa).

On the next stage, an optical porosimetric tests were conducted, using the RapidAir 457 device. From the cured cubical samples, 1cm-thick middle sections sized 10x10cm on sides were cut out. Samples were ground and polished thoroughly, using the polishing powders. Then, on each, the surface to be tested was painted black, and pores on the surfaces of the specimens were filled with white zinc paste. For each concrete, two specimens were planned for testing. The surface in question had the area of 8x8cm, and the traverse from the reading was taken had the length of 1600mm. Each specimen was tested twice. The second reading was performed after the specimen was turned by 90 degrees in the device.

The variety of thermal parameters was tested by the means of apparatus Isomet 2104. This method is based on analysis of heat flow values in non-stationary conditions. Thermal conductivity λ and volumetric specific heat c_v were read out. The tests were performed in the first 3 months of the specimens curing: after 7, 14 and 28 days, and then after 2 and 3 months. On each of the six specimens the place of measuring was marked so that each time the parameters could be measured in the same spot on the particular specimen. Based on those measurements, average values, as well as standard deviations of the results were determined.

3. Results and discussion

Figure 2 presents the results for apparent total porosity of fresh concrete mixes. Upon determining the porosity value, the aggregate corrective coefficient was not taken into account. The assumption was to obtain the total porosity of the mix, with the aggregate porosity taken into account.

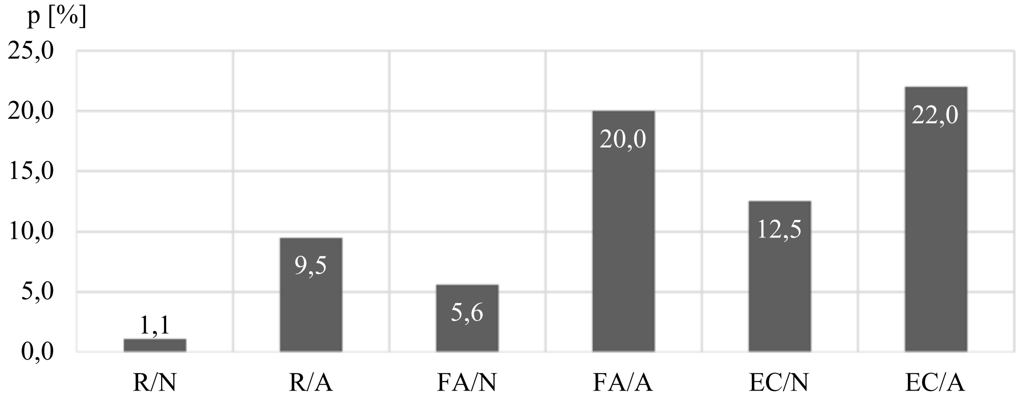

Figure 2. Apparent total porosity of fresh mixes.

A comparison of the concretes without any aerating admixtures (R/N, FA/N, EC/N) proved existence of an evident influence of the aggregate porosity on total porosity of fresh mixes. A similar situation is in the group of aerated concretes (R/A, FA/A, EC/A), where much higher values were obtained for the lightweight aggregate concretes. The highest porosity was observed for the composites based on the expanded clay aggregate (leca).

Figure 3 shows log-differential graphs, drawn out for the non-aerated concretes, using the mercury porosimetry method.

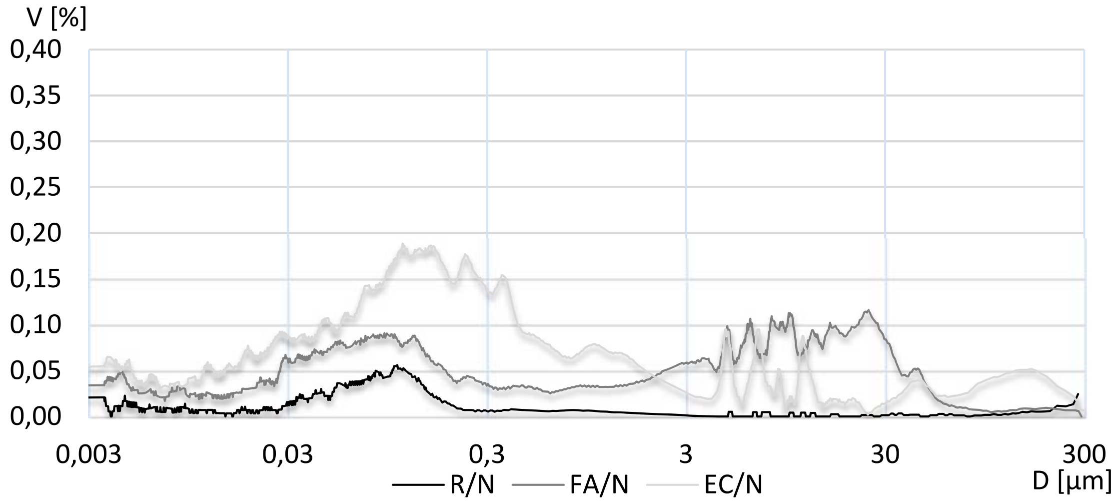

Figure 3. Log-differential pore structure graphs of composites without air-entraining admixture

In this case, pores occurring in the lightweight aggregates (4-30µm) and pores in the cement matrix (0.03-0.2µm) are dominating. In the expanded clay (EC) based concrete, a second dominating extreme range appears from 0.12 to 0.4µm.

Figure 4 presents pore structure graphs for composites with some air-entraining admixture. Comparing them to the non-aerated composites, one may observe that pores in the aerated cement matrix remain within the range from 1 to 30μm. As far as the value is concerned, the graph of the smaller diameter pores is similar to those of the non-aerated concretes.

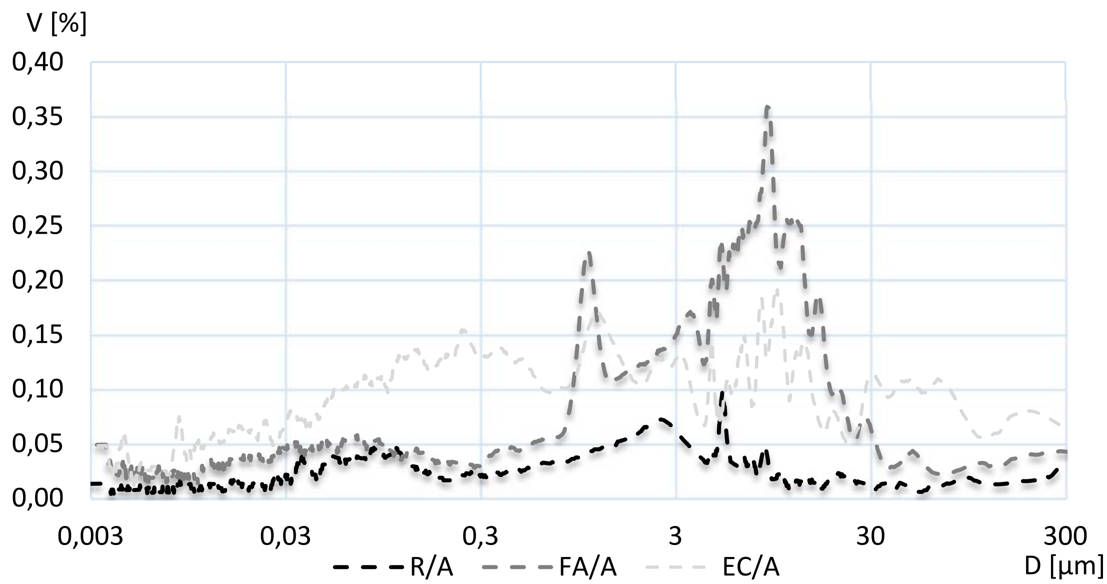

Figure 4. Log-differential pore structure graphs of composites with air-entraining admixture

Table 2 shows the basic properties of the composites, determined with the mercury porosimetry. The data for the absolute porosity have significantly higher values, comparing to the data obtained by the means of the pressure gauge method. With the porosimetric method it is possible to establish the air content within the range from 3nm to 300μm more precisely. The presented values of the total porosity are obtained from the specimen skeletal density and volumetric density, thanks to which this method is more reliable.

Table 2. Porosity properties derived from mercury porosimetry tests.

Type	R/N	R/A	FA/N	FA/A	EC/N	EC/A
Total specific surface [m^2/g]	5.20	5.58	17.25	14.19	27.86	22.65
Pore tortuosity [-]	2.12	1.98	1.83	1.72	1.78	1.66
Permeability[10^{-4} nm^2]	4.00	54.00	32.00	127.00	27.00	121.00
Porosity (3 nm-300 μm) [%]	**10.10**	**23.38**	**36.58**	**50.40**	**43.43**	**57.57**
Average pore volume [cm^3/g]	0.04	0.10	0.19	0.35	0.26	0.41
Median pore volume [cm^3/g]	0.02	0.06	0.12	0.20	0.16	0.25
Dominant pore volume [cm^3/(μm·g)]	2.26	1.14	4.82	4.97	7.62	5.32

In the lightweight concretes, there are also large pores and air voids of diameters exceeding 1mm which the mercury method does not cover. Therefore, tests using optical porosimetry were conducted. Figure 5 presents a differential graph of pore structure for the non-aerated composites.

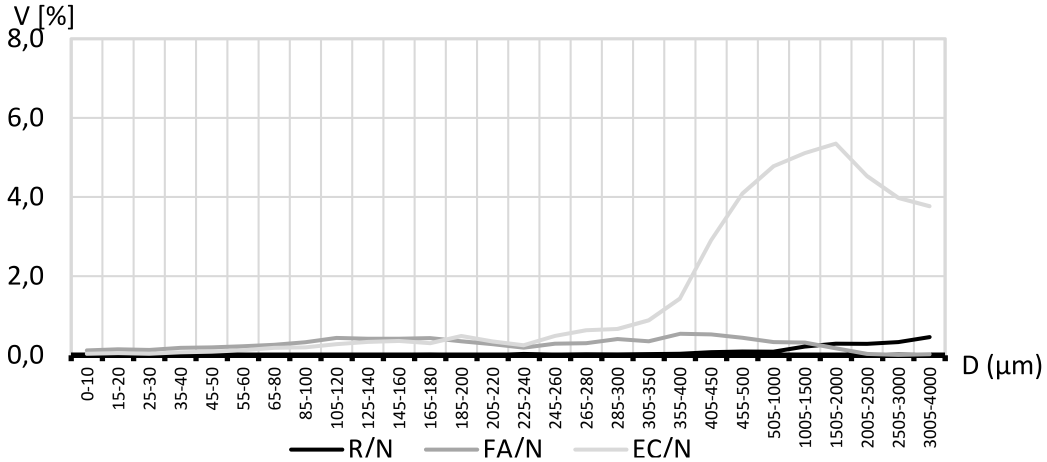

Figure 5. Differential pore structure graphs of composites without air-entraining admixture

The graphs shown in figure 5 indicate that in the cement matrix large airy pores (>300 μm) do not have a larger effect on the total porosity of the concrete. Pores occurring in the expanded clay aggregate (EC/N) within the range of 300 - 4000μm are clearly seen. In the other composites, due to the low content of large pores in the aggregate structure, the pore distributions are rather flat.

By contrast, the pore structure graphs presented in figure 6 prove that the air-entraining admixture brings about appearance of additional pores in the range from 350 to 2500μm, which do not occur in the non-aerated concretes. Compared to the mercury method, the optical method is distinctly less sensitive in the range from 10 to 300μm.

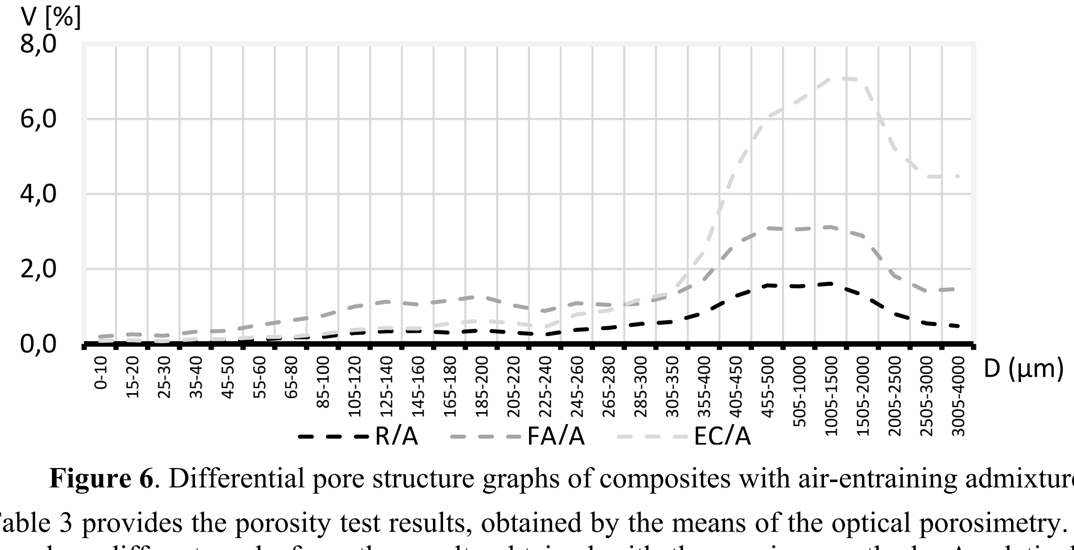

Figure 6. Differential pore structure graphs of composites with air-entraining admixture.

Table 3 provides the porosity test results, obtained by the means of the optical porosimetry. Again, these values differ strongly from the results obtained with the previous methods. A relatively high convergence with the mercury method test results was observed in regards of the expanded clay based composites.

Table 3. Porosity properties derived from optic porosimetry tests

Type:	R/N	R/A	FA/N	FA/A	EC/N	EC/A
Total porosity A [%]	**2.09**	**14.73**	**7.88**	**36.27**	**42.15**	**56.65**
Porosity (300-4000 μm) [%]	1.84	10.41	2.82	22.44	37.17	49.47
Micro porosity A_{300} [%]	0.25	4.33	5.06	13.84	4.98	7.18
Total specific surface [m^{-1}]	11.84	18.59	49.97	26.10	9.86	11.18

Figure 7 presents variation of the thermal conductivity coefficient of the composites during the first three months of their curing. An evident decrease of the thermal conductivity took place after a month of free drying in the dry-air conditions (t = 60 days). Application of both the lightweight aggregates and air-entraining additives resulted in strong reduction of the thermal conductivity coefficient. Combination of the two effects seems to be reasonable way of producing composites with thermal conductivity coefficients much lower than those of standard concretes or lightweight aggregate based non-aerated concretes. The lowest values were recorded for the aerated composite, based on fly ash aggregate FA/A, for which, after 3 months of curing, the thermal conductivity coefficient amounted to 0.333W/(m·K).

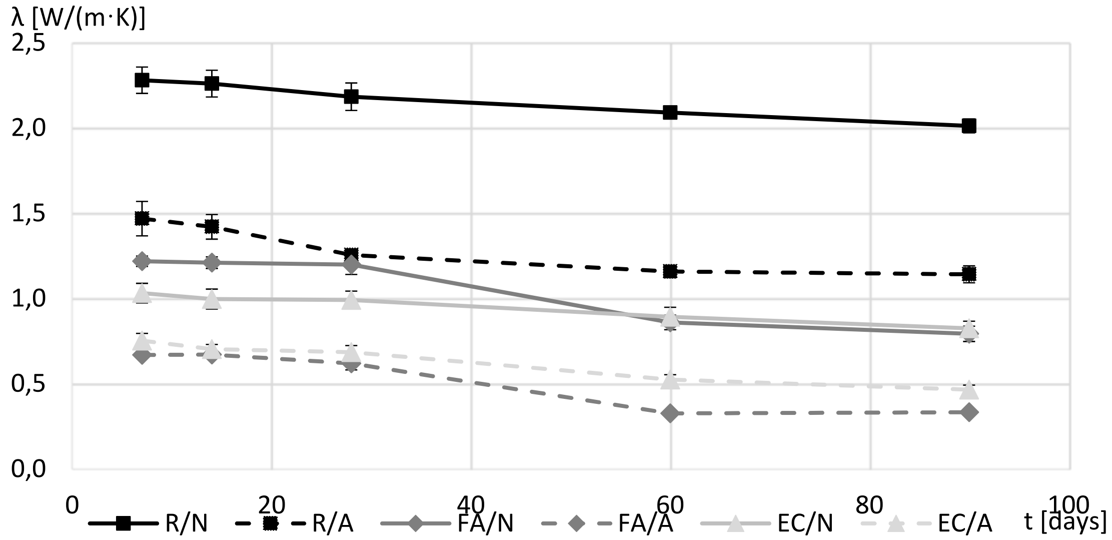

Figure 7. Thermal conductivity coefficients in the first 90 days of curing of composites

The volumetric specific heat values we recorded, as presented in figure 8, are highly diversified due to the high water content in the specimens on the first stage of their curing. In each subsequent period of measurements the standard deviations become lower and lower, along the free drying progress. The concretes with high porosity values retained their high specific heat even after 90 days of curing and simultaneous drying: 1,617 · 10^6J/(m^3·K) for EC/A and 1,465 · 10^6J/(m^3·K) for FA/A.

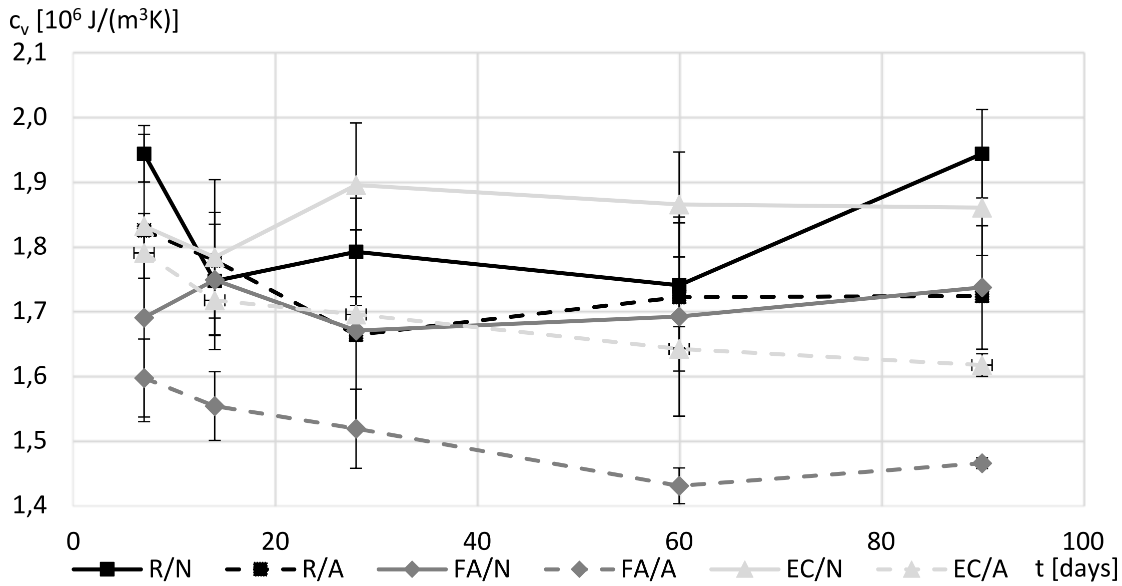

Figure 8. Volumetric specific heat in the first 90 days of curing of composites

Figure 9 shows compressive strength test results for each of the concretes. As expected, both strong aeration of the cement matrix with the air-entraining admixture, and the type of used lightweight aggregate, resulted in strong reduction of average compressive strength of the concretes in question. Nevertheless, even the most porous composites demonstrated satisfactory strength values: 9.32MPa for FA/A, and 7.84MPa for EC/A.

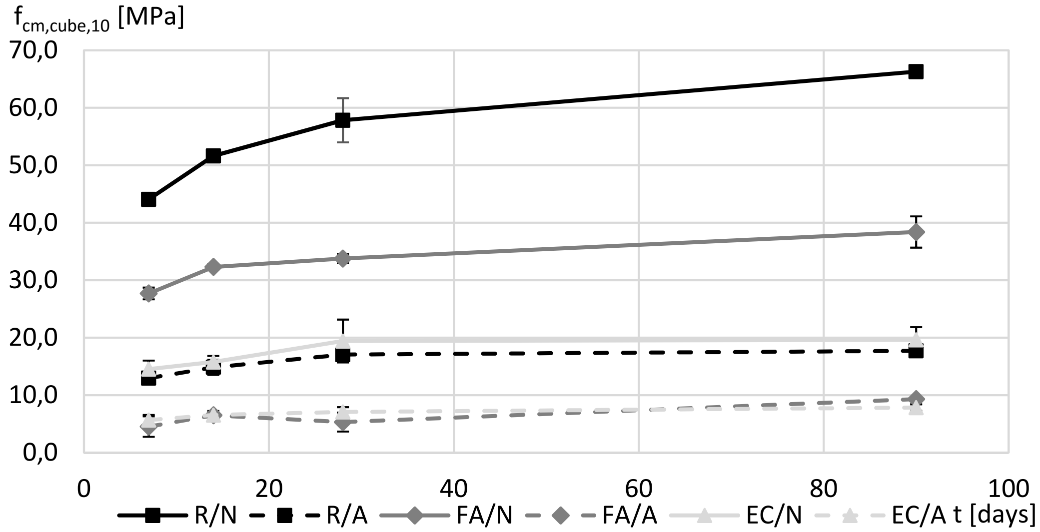

Figure 9. Average compressive strength in the first 90 days of composites curing

Figure 10 comprises of three comparative graphs, on which porosity results, obtained with the three methods, are juxtaposed. Despite the rather large absolute discrepancies in the results, it has to be admitted that the data gathered with all the three methods are coherent and represent a noticeably high level of correlation, as for this kind of studies, being of $r^2 \sim 0.8$.

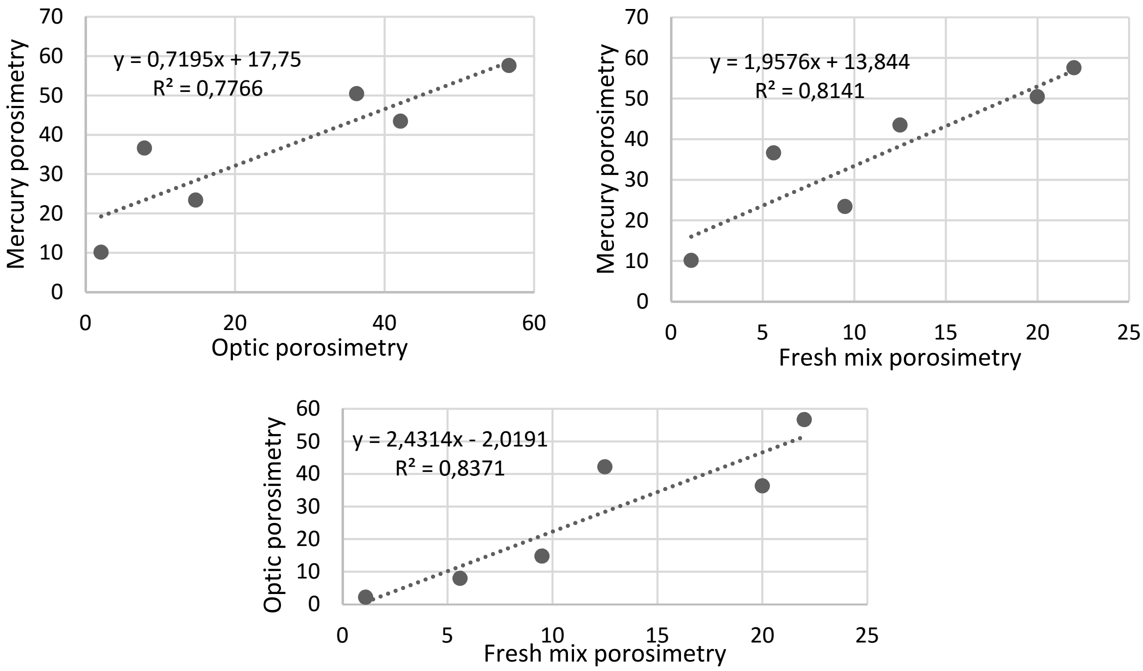

Figure 10. Comparison of porosity tests results

Figure 11 juxtaposes porosity graphs for an exemplary FA/A concrete, obtained with the mercury and optical methods. The authors presented results for the range up to 300µm, where formally the measurement capabilities offered by the mercury porosimetry end. In practice, this technique ceases to be useful even at much finer pores. This is attributed to the fact that mercury may penetrate larger pores of the ~ 30 – 300µm range, without a noticeable increase of pressure, and may also be a result of presence of closed pores, which mercury is not pressed into. Such drawbacks are not observed in the optical method, which perfectly fills up the data concerning the larger pores that exceed ~ 20µm. At the same time, both research techniques provide for relatively high result compliance for pores of ~10 – 20µm. Similar results were obtained for all six concretes in study. Therefore, the opinion that combination of the two methods helps build a fuller and truer picture of porosity of the material of our interest seems to be well grounded.

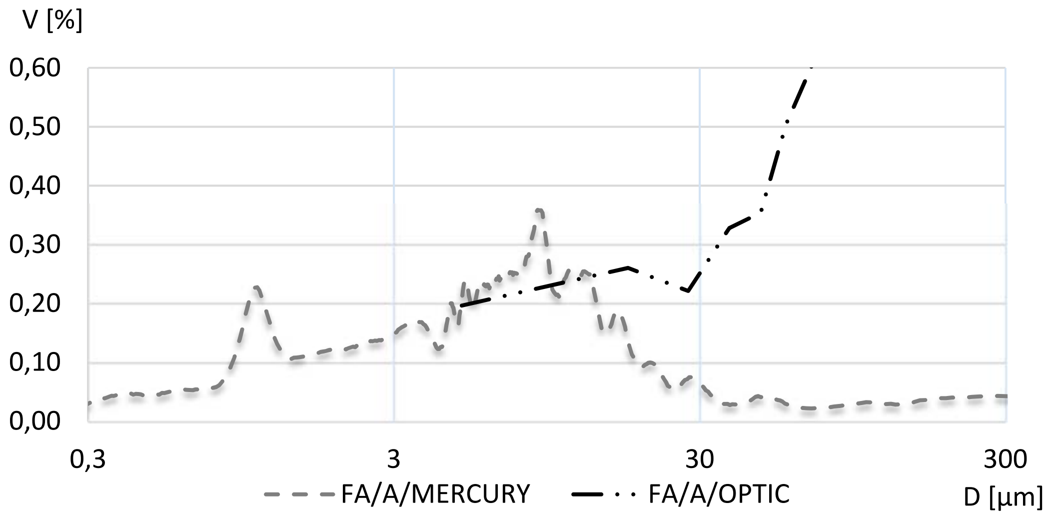

Figure 11. Log-differential pore structure graphs of composite based on fly ash aggregate

4. Conclusions

This paper presents results derived from porosimetry studies conducted with various methods, performed on concretes with diversified porosity of both their cement matrix and the applied aggregate. It was established that the pressure gauge method, performed on fresh mixes, provides underestimated porosity values, comparing to the other two methods. The mercury tests demonstrated their high sensitivity upon assessing the pores of less than 30µm diameter, whereas this method proved no good for studying pores larger than 300µm. This is caused by filling those spaces with mercury, without controlling the mercury pressure. On the other hand, the optical method is usable for assessing larger pores, those exceeding 300µm. In this method, smaller pores are either not counted, or, if located close one to another, can be qualified as pores of a much greater diameter. Thus, the authors find it recommendable to conduct combined tests, which will make it possible to build a comprehensive image of porosity of the materials in study.

The most precise value of total porosity was obtained with the mercury method. This is thanks to the fact that the results were derived from the specific and volumetric densities. Nevertheless, one has to remember that such values are flawed due to the conditions of the measurements (e.g. closed pores which mercury does not penetrate are not taken into account in this method).

The thermal research results that are presented in the paper indicate validity of aerating the lightweight aggregate based concretes in order to further reduce their thermal conductivity coefficients λ. The use of exclusively porous aggregate, or only an aerated mix with some normal aggregate provides results much worse than a combination of those two ways. The lowest thermal conductivity coefficients λ were observed in the aerated composite, produced on the basis of fly ash aggregate. The reducing effect of aeration on volumetric specific heat c_v is evident. Despite that, even the most aerated composite featured relatively high values of specific heat.

Aeration of the matrix and application of lightweight aggregates brought about a significant decrease of average compressive strength f_{cm} in the tested composites. The lowest strength was observed for the aerated composites, produced on the basis of lightweight aggregates. Regardless that, even the minimum value of 7.84MPa is still quite good, comparing to other load-bearing lightweight materials, such as autoclaved aerated concrete or perforated porous clay brick.

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