POSSIBILITIES OF DETERMINING THE AIR-PORE CONTENT IN CEMENT COMPOSITES USING COMPUTED TOMOGRAPHY AND OTHER METHODS

MOŽNOSTI DOLOČANJA VSEBNOSTI ZRAČNIH POR V CEMENTNIH KOMPOZITIH Z UPORABO RAČUNALNIŠKE TOMOGRAFIJE IN DRUGIH METOD

Bronislava Moravcová, Petr Pőssl, Petr Misák, Michal Blažek

Brno University of Technology, Faculty of Civil Engineering, Veveří 331/95, 602 00 Brno, Czech Republic
BlazekM7@study.fce.vutbr.cz

The aim of the paper is to outline the possibilities and predictive values of the selected methods for determining air-pore characteristics in concrete and cement composites in general. Four samples (mixtures) of cement concrete without additives or admixtures were chosen for the measurement, differing in cement content while maintaining the consistency of fresh concrete S3 according to EN 206. Both standardized and non-standardized methods are used in Europe, therefore providing a possibility of comparing their outcomes.

Keywords: concrete, durability, porosity, surface, covering layer, X-ray computed micro-tomography

Namen članka je prikazati možnosti za splošno napovedovanje izbranih metod za določanje značilnosti zračnih por v betonu in v kompozitih iz cementa. Za meritve so bili izbrani štirje vzorci (mešanice) betona brez dodatkov in primesi z različno vsebnostjo cementa ob zadržanju konsistence svežega betona S3, skladno z EN 206. Standardizirane in nestandardizirane metode se uporabljajo v Evropi in zato omogočajo primerjavo rezultatov.

Ključne besede: beton, zdržljivost, poroznost, površina, prekritje, rentgenska računalniška mikro-tomografija

1 INTRODUCTION

The durability of cement composites has been a topic of increasing importance. It is illustrated by the fact that the requirement for the durability of concrete structures has become part of CPR (Construction Products Regulation).[1] The content, size and distribution of air pores, i.e., porosity, influence not only on the durability but also the physical/mechanical properties of cement composites and thus concrete structures in general.[2]

Recently, there have been claims that concrete air entrainment in percent is not sufficiently informative as both the air-entrained concrete and the traditional concrete contain a broad spectrum of air voids (**Figure 1**). Not all the pores are beneficial for the required property. Recent investigations argue that an effective entrainment (a pore diameter of 1 μm–1000 μm) above 2 % can significantly increase the resistance to water and chemical thawing agents.[3–5]

The pores can be divided, according to their characters[3], into gel, capillary, entrained and entrapped ones, or by their size[4], into micropores (<1.25 nm), mesopores (1.25–25 nm), macropores (25–5000 nm) and other large pores (5000–50000 nm).

Figure 1 shows a detailed diagram of the division of pores and the possibilities of their detection by means of the methods discussed below.

In connection with what was mentioned above, four methods for determining various pore characteristics dealing with the air-void content in concrete (total air content, micropore air content, average pore diameter and specific surface of an air-void system) were chosen. Out of these, two are used for determining the properties of fresh concrete (the pressure method and the air-void analyser) and the remaining two for determining the properties of hardened concrete (a microscopic analysis and CT).

The goal is to determine the air-pore-system characteristics using the above-mentioned methods and to
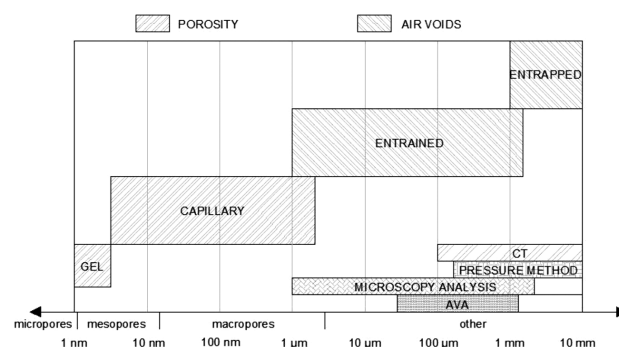

Figure 1: Example of pore size in concrete
Slika 1: Primer velikosti por v betonu

select the optimum method for determining the characteristics of specific types of the air-pore system in concrete.

2 METHODS USED IN THE EXPERIMENT

There is a number of situations where there is a need to have information about the pore distribution in concrete. This paper is concerned with the determination of concrete-cover porosity, which has a significant influence on the quality of the concrete and thus the durability of the whole structure.[2] This experiment determines the concrete cover to be 40–60 mm, taken from the surface of the specimen.

The following paragraphs describe standardised and non-standardised methods for the above-specified properties of fresh and hardened concrete, used in the experimental part of the research.

It must be emphasised that the results of the methods differ because of their different abilities to detect the pore size, due to the physical nature of the methods and differently sized specimens used for each method.

2.1 Pressure method

The method is applied for determining the air content in compacted fresh concrete made of dense or heavy-weight aggregate. It is based on the Boyle-Mariotte law, which says that the product of the pressure and volume of a gas is constant. This method uses an air meter according to EN 12350-7[5] (**Figure 2**).

Figure 2: Concrete pressure-air meter
Slika 2: Merilnik tlaka zraka v betonu

2.2 Air-void analyser (AVA)

For the analysis of the air-void structure in fresh concrete, AVA (an air-void analyser, **Figure 3**) was used.[6,7] Its advantages are the possibility of taking samples directly from the vibrated mixture without any further processing and, as in the case of the previous method, the possibility of performing a measurement in situ. The apparatus also detects the content of microscopic air pores A_{300}, but it is necessary to note that it cannot detect pores smaller than 50 μm.

It was discovered that, under certain conditions, the air pores in a cement mixture can be transported into a liquid without affecting their content or size.[8] Successful transport depends on the viscosity and hydrophilic character of the liquid. Once air bubbles have exited the cement paste, they start to rise through the liquid. The function of the air-bubble movement can be described with reversed Stokes' law, according to which larger bubbles rise faster than smaller ones. The total distribution of an air-void structure can be calculated by recording the amount of air that has risen up to a given level over the elapsed time. The number of pores escaping the cement paste is observed through the changes in the mass of an upside-down Petri dish placed just below the surface of the liquid.

The principle is that the apparatus determines the amount and size of air pores and thus enables an assessment of the spacing and the specific air-void surface. The method is primarily used for air-entrained concretes. However, even for non-entrained concretes, it is better at determining the air-void characteristics than method 2.1 as it can be used also for the determination of other characteristics, not just the total air content.

The following characteristics can be determined using this method:
- spacing factor,
- micro-air-void content (A_{300}),

Figure 3 Air Void Analyzer (AVA)
Slika 3: Analizator praznin (AVA)

- total air content,
- specific surface of an air-void system.

2.3 Determining air-void characteristics in hardened concrete according to EN 480-11

A stereoscopic microscope was used for the observation of the air-void diameter and distribution in hardened concrete according to the standard EN 480-11.[9] This device detects pores of 1–4000 μm and is able to identify microscopic air pores (A_{300}). Prior to its use, the specimens need to be prepared from a concrete cured for a minimum of 7 d. The measurement results can be recorded manually or automatically. Both procedures differ depending on human errors (the subjectivity of an evaluation) during pore detections (e.g., mistaking a hollow after a missing piece of the aggregate for an air pore).

The ability to detect air pores depends on the human ability to recognise a pore based on the contrast in the adapted specimen. During an automatic assessment, the factor is the resolution, at which a specimen is examined.

The following values can be determined using this method:
- spacing factor,
- micro-air-void content (A_{300}),
- total air content,
- specific surface of an air-void system,
- average pore diameter.

2.4 Computed tomography

Computed tomography (CT) is a method, which enables us to look inside the specimens non-destructively. CT detects differences in the material density. A 3D visualisation of a specimen can be constructed through the processing of several consecutive slices.[10]

Using special SW, the following characteristics can be determined on a 3D image of a specimen:
- spacing factor,
- micro-air-void content (A_{300}),
- total air content,
- specific surface of an air-void system,
- average pore diameter.

3 EXPERIMENTAL PART

The goal of this experiment was to compare the results of the selected methods used for the determination of air-pore characteristics in concrete and, at the same time, to ascertain whether the outputs of these methods correspond to one another. Four mixtures of cement concrete without additives or admixtures were chosen for the measurement, differing in the water/cement ratio while maintaining the consistency of fresh concrete S3 according to EN 206.[11] The components of the concretes identified as *R* (0/1, 0/2 and 0/3) are in **Table 1**. The

components in individual concretes were identical, i.e., of the same type of aggregate from the same location, one type of cement from the same mill.

The relevant properties of the concrete components were monitored according to EN 196-6[12], EN 933[13], and EN 1097[14] (**Table 2**). Samples were taken according to EN 12350-1.[15] Properties of fresh concrete were determined according to EN 12350-2, 5, 6 and 7.[5,16–18]

Although some of the methods are intended primarily for air-entrained concrete (2.2 and 2.3), it should be noted that, in terms of the entrained-air-void content, even non-entrained concretes contain voids corresponding, in size, to the entrained-air pores in air-entrained concrete.

Table 1: Composition of individual mixtures
Tabela 1: Sestava posameznih mešanic

Com-ponent	Aggregates (kg)			Cement 42,5 R, Mokrá (kg)	Water (kg)	Water ratio (-)
	0 – 4 Bratčice	4 – 8 Olbra-movice	8 – 16 Olbra-movice			
Concrete mixture R - C12/15 X0 S3 D16						
	978	177	693	255	206	0.81
Concrete mixture 0/1 - C20/25 X0 S3 D16						
	927	182	698	309	202	0.65
Concrete mixture 0/2 - C30/37 X0 S3 D16						
	892	175	695	358	192	0.54
Concrete mixture 0/3 - C35/45 (45/55) X0 S3 D16						
	888	202	692	402	203	0.50

Table 2: Characteristics of fresh concrete for individual mixtures
Tabela 2: Značilnosti svežega betona posameznih mešanic

Concrete	Characteristics			
	Flow table test (mm)	Density of fresh concrete (kg/m³)	Total air content (%) (pressure method)	Micro air content A_{300} (%) (AVA)
R	435	2250	2.7	0.2
				0.1
0/1	410	2315	2.7	0.0
0/2	385	2315	2.5	0.1
0/3	420	2290	2.5	0.1

For method 2.1, the specimen volume was 8 dm³. This method enables a detection of pores > 300 μm. It is the most commonly used method; however, its disadvantage is that it only allows a determination of the total air content and gives no information the on pore distribution and size.

In the case of method 2.2, the specimen volume was approximately 50 cm³. As it was a relatively small specimen, obtained with an impact drill, it was highly probable that it contained only smaller-sized pores (< 2 mm).

For the procedure according to 2.3, the specimen was a concrete slab of 100 mm × 100 mm. The evaluation was performed in two ways, i.e., manually and automatically, both of which have their drawbacks, see 2.3. Automatic evaluation took place at a resolution of 1 μm

Figure 4: Phoenix v|tome|x L 240[19]
Slika 4: Naprava Phoenix v|tome|x L 240[19]

per pixel, which corresponds to the minimum size of a detectable pore.

For the measurement with method 2.4, cylindrical specimens of 50 mm in diameter and 60 mm in height were used. All the cylinders were obtained from larger specimens, using a core drill. The drilling was performed perpendicular to the surface of the concrete and each specimen was cut to a length of 60 mm in order to represent the concrete cover. At this specimen size, the device was able to detect the pores of above 100 μm.

Each cylinder was scanned in consecutive slices using device Phoenix v|tome|x L 240 (**Figure 4**).[19] All the data was recorded and subsequently processed in the laboratory of the Central European Institute of Technology – Brno University of Technology (CEITEC BUT). A module of VG Studio MAX was used for the pore analysis.[20]

Table 3: Characteristics of air voids for concrete R
Tabela 3: Značilnosti zračnih por v betonu R

	Used methods				
	Fresh concrete		Hardened concrete		
	Pressure method	Air-void analyser	Microscopy analysis spacing		Computer tomo-graphy
			manual	automatic	
Total air content A (%)	2.7	$\bar{x} = 1.1$	$\bar{x} = 2.86$	$\bar{x} = 10.72$	1.69
		$s = 0.3$	$s = 0.21$	$s = 0.49$	
Micro air content A_{300} (%)	–	$\bar{x} = 0.15$	$\bar{x} = 1.20$	$\bar{x} = 5.58$	0.05
		$s = 0.05$	$s = 0.11$	$s = 0.67$	
Spacing factor L (mm)	–	1.13	$\bar{x} = 0.11$	$\bar{x} = 0.08$	0.70
			$s = 0.00$	$s = 0.01$	
Average pore diameter D (μm)	–	–	–	$\bar{x} = 114.50$	320.0
				$s = 6.50$	
Specific surface of air-void system α (mm^{-1})	–	7.5	$\bar{x} = 57.65$	$\bar{x} = 35.04$	11.48
			$s = 1.65$	$s = 1.95$	

4 RESULTS AND DISCUSSION

Tables 3 to **6** show the air-pore characteristics obtained with the selected methods for fresh and hardened concrete. **Figures 5** to **9** show the method outputs. In the cases, where the measurement was performed on more specimens, the tables include their average values and standard diversions. The number of decimal places in the tables is based on the output of individual methods.

Table 4: Characteristics of air voids for concrete 0/1
Tabela 4: Značilnosti zračnih por v betonu 0/1

	Used methods				
	Fresh concrete		Hardened concrete		
	Pressure method	Air-void analyser	Microscopy analysis spacing		Computer tomo-graphy
			manual	automatic	
Total air content A (%)	2.7	0.9	1.48	$\bar{x} = 4.44$	1.85
				$s = 0.15$	
Micro air content A_{300} (%)	–	0.0	0.27	$\bar{x} = 1.93$	0.02
				$s = 0.01$	
Spacing factor L (mm)	–	1.80	0.27	$\bar{x} = 0.13$	1.01
				$s = 0.01$	
Average pore diameter D (μm)	–	–	–	$\bar{x} = 99.00$	416.0
				$s = 12.00$	
Specific surface of air-void system α (mm^{-1})	–	6.0	31.80	$\bar{x} = 41.06$	7.82
				$s = 5.13$	

Table 5: Characteristics of air voids for concrete 0/2
Tabela 5: Značilnosti zračnih por v betonu 0/2

	Used methods				
	Fresh concrete		Hardened concrete		
	Pressure method	Air-void analyser	Microscopy analysis spacing		Computer tomo-graphy
			manual	automatic	
Total air content A (%)	2.5	1.0	1.76	$\bar{x} = 2.90$	1.95
				$s = 0.33$	
Micro air content A_{300} (%)	–	0.1	0.39	$\bar{x} = 1.33$	0.01
				$s = 0.36$	
Spacing factor L (mm)	–	1.28	0.28	$\bar{x} = 0.14$	1.16
				$s = 0.04$	
Average pore diameter D (μm)	–	–	–	$\bar{x} = 86.50$	406.0
				$s = 18.50$	
Specific surface of air-void system α (mm^{-1})	–	8.1	29.1	$\bar{x} = 48.68$	6.68
				$s = 10.47$	

Table 6: Characteristics of air voids for concrete 0/3
Tabela 6: Značilnosti zračnih por v betonu 0/3

	Used methods				
	Fresh concrete		Hardened concrete		
	Pressure method	Air-void analyser	Microscopy analysis spacing		Computer tomo-graphy
			manual	automatic	
Total air content A (%)	2.5	1.0	2.48	$\bar{x} = 3.51$	1.70
				$s = 0.78$	
Micro air content A_{300} (%)	–	0.1	0.26	$\bar{x} = 1.69$	0.01
				$s = 0.44$	
Spacing factor L (mm)	–	1.26	0.40	$\bar{x} = 0.13$	0.93
				$s = 0.03$	
Average pore diameter D (µm)	–	–	–	$\bar{x} = 88.5$	445.0
				$s = 10.5$	
Specific surface of air-void system α (mm^{-1})	–	8.4	18.20	$\bar{x} = 45.8$	9.20
				$s = 5.45$	

When determining the total air content in fresh concrete, the AVA method gave lower values than the pressure method (**Figure 5**), which could have been caused by the amount of fresh concrete being tested.

Different values of manual and automatic microscopy may have been caused by the ability/inability to distinguish between an air pore and a hollow created by a particle of the aggregate forced out during the cutting or grinding of the specimen. The differences may have also been caused by the operator during the manual measurement of the factor. During automatic measurement, the
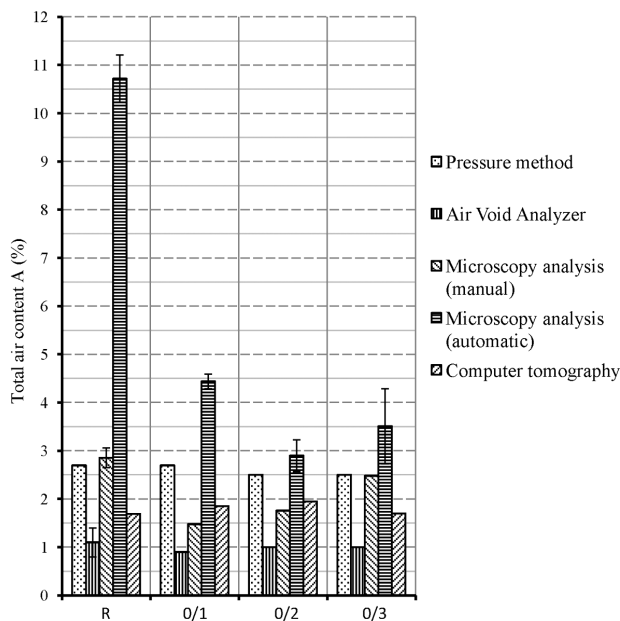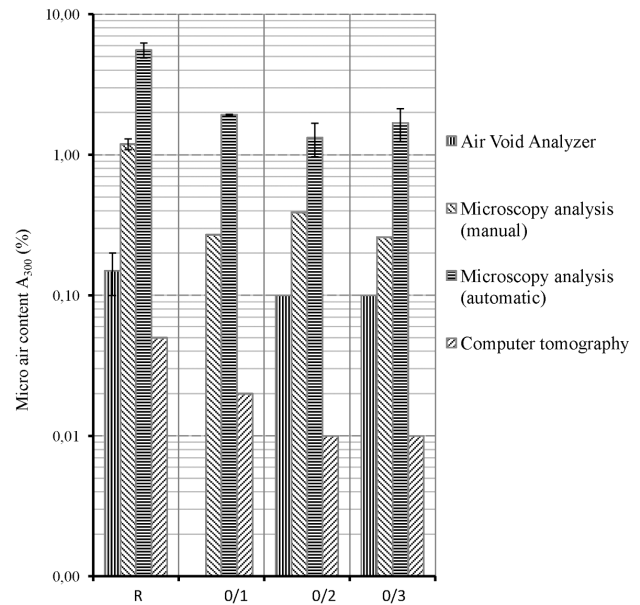

Figure 6: Results of micro-air-void content A_{300} for individual concretes
Slika 6: Rezultati vsebnosti zraka v mikroporah A_{300} pri posemeznih betonih

ability to detect a pore depends on the resolution (in our case, 1µm per pixel), while during manual measurement, this ability lies with the technician performing the test.

Figure 6 shows that out of all the methods used, the measurement results obtained with CT and AVA corresponded most closely to each other. A small difference between these results was probably caused by different abilities to detect air voids at the bottom threshold of the detection: AVA (at 50 µm), CT (at 100 µm).

Figure 5: Results of total air content for individual concretes
Slika 5: Rezultati vsebnosti vsega zraka v posameznem betonu
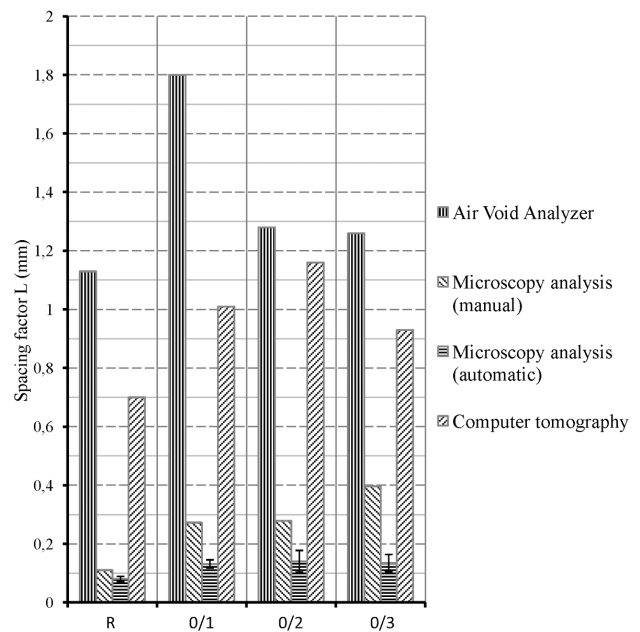

Figure 7: Results of spacing factor for individual concretes
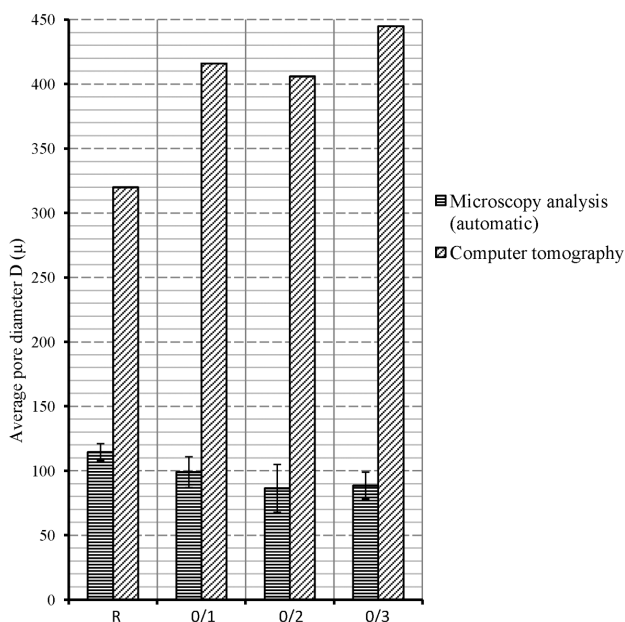**Slika 7:** Rezultati faktorja ločljivosti pri posamezznih betonih

Figure 8: Results of average pore diameter for individual concretes
Slika 8: Rezultati povprečne velikosti por pri posameznih betonih

Figure 10: Volume of air-void system – 3D
Slika 10: Volumen sistema zračnih por – 3D

In general terms, it can be stated that, in determining the A_{300} value, a comparison of the results of individual methods was very demanding. All the methods exhibited a trend of a decrease in the resulting values of A_{300} dependent on the increasing cement content in the individual concretes; however, each method displayed significantly different absolute values of A_{300}.
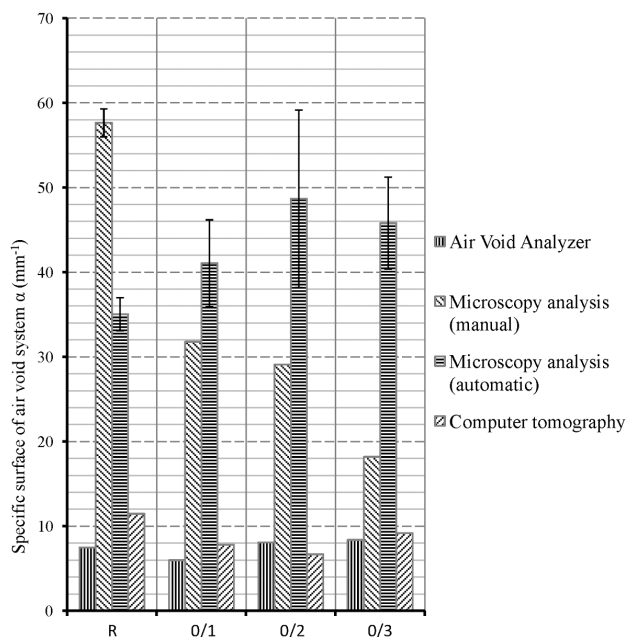
Based on the results shown in **Figure 7**, it could be stated that a comparison of the outcomes of the methods was again very demanding. The results that at least partially corresponded to each other were the values from the measurement using manual and automatic microscopy.

No conclusive proof was found for the premise that the spacing-factor results obtained with CT and the microscope would differ only minimally. This fact may have been caused by a difference in the mathematical model of the calculation between the microscope and CT. In the case of the microscope, the spacing factor was calculated from 2D scanning of the specimen, while in the case of CT, it was calculated from 3D scanning.

All the used methods displayed significantly different values of the average pore size for different concretes, as shown in **Figure 8**. The cause of the higher values in case of CT was probably its measurement range, where the bottom threshold was defined at 100 μm and the top threshold was not defined in this experiment; therefore,

Figure 9: Results of specific surface of air-void system for individual concretes
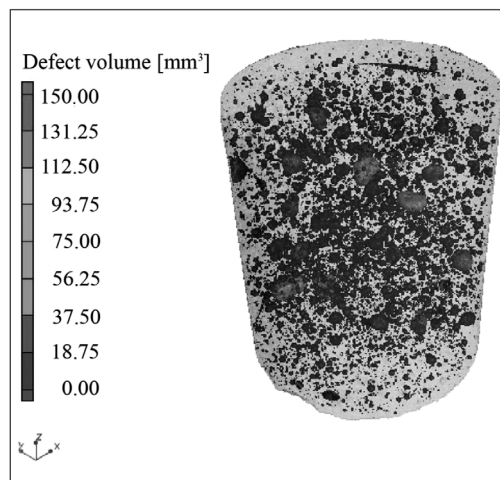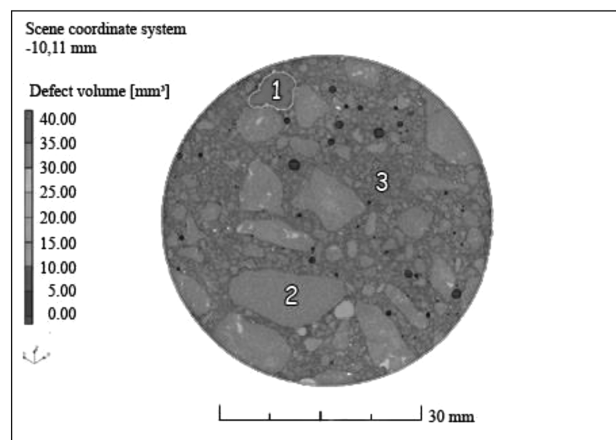**Slika 9:** Rezultati specifične površine sistema zračnih por pri posameznih betonih

Figure 11: Volume of air-void system – cross-section: 1 – air void, 2 – aggregate particle, 3 – cement paste
Slika 11: Volumen sistema zračnih por – presek: 1-zračna pora, 2 – delec agregata, 3 – cementna osnova

possible air voids may have also been included in the calculation. On the other hand, the microscope had its range limited to the maximum pore diameter of 4000 μm.

The measurements using CT and AVA displayed lower final values of the specific surface of the air-void system, while the results obtained with microscopy were significantly higher. It was nearly impossible to find any correlation between these results (**Figure 9**).

A possible cause for this great difference in the results may have been, much like in the case of most of the other properties, a different range of the diameter of the air pores being measured with the above methods and, in the case of CT, also the detection of possible air voids.

Some outcomes of the measurement with CT (the pore distribution and volume) are shown in **Figures 10** and **11**.

5 CONCLUSION

The performed measurements allowed the following conclusions:

- The results of the used methods are difficult to compare. The main causes for this difficulty are probably different air-pore measurement ranges and the measurement of different types of pores (**Figure 1**). The characteristics determined with the individual methods performed on the concretes hardly correspond to one another. If one method indicates a certain trend in the results based on the cement content in the concrete, the other methods do not generally follow the trend or they even contradict it.
- A conclusive comparison of the results of the methods for determining the air content in concrete would require us to include, in the calculation, only the pore diameters within the intersection of the measurement ranges of the all the methods used (**Figure 1**).
- When measuring according to EN 480-11, differences are found in the results for the pores recorded manually and automatically. Possible causes are the inability of automatic recording to differentiate between an air pore and a missing particle of the aggregate and different abilities of detecting a pore at the bottom resolution threshold during each method of evaluation.
- AVA and stereoscopic microscope are primarily intended for determining the pore characteristics of aerated concretes. Based on the obtained results, it can be concluded that these methods probably give lower predictive values when used for non-aerated concretes.
- CT is a modern method for determining the concrete porosity with a great potential lying in the possibility of rendering the complete pore structure of a concrete specimen in 3D. Given the fact that it is demanding

to set up the device along with the evaluation software, it appears that a correct interpretation of results is rather difficult. The results include possible voids, which would have to be filtered out of the chosen algorithm, thus increasing the accuracy of the predictive values.

- The results of this experiment may have been influenced by the small number of the specimens used, e.g., in the case of CT, each type of concrete was represented by one specimen only.
- CT appears to be a very suitable method for measuring air-pore characteristics in concrete; it is, however, necessary to tackle the above-mentioned flaws (filtering possible voids, a correct set-up of the device). When these problems are removed, the outputs of CT can be used as a benchmark for the results of the other methods measuring the pore structure in concrete, mainly thanks to detailed 3D mapping.
- During the determination of concrete-pore characteristics, it is necessary to consider the types and sizes of the pores being examined, which are the key factors for the purpose and characteristics of a pore system. It is then possible to choose the most suitable method of their determination in line with **Figure 1**.

Acknowledgement

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