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To cite this article: Beata Lazniewska-Piekarczyk and Jacek Goaszewski 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **471** 032044

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# Relationship Between Air-Content in Fresh Cement Paste, Mortar, Mix and Hardened Concrete Acc. to PN-EN 480-1 with Air-Entraining CEM II/B-V

Beata Lazniewska-Piekarczyk<sup>1</sup>, Jacek Gołaszewski<sup>1</sup>

<sup>1</sup> Silesian Technical University, Faculty of Civil Engineering, Department of Building Materials and Processes Engineering, Akademicka 5 Str., 44-100 Gliwice, Poland

beata.lazniewska@polsl.pl

**Abstract** Research results of the relationships between air-volume in air-entrained cement paste, mortar and concrete, all designed according to PN-EN 480-1 guidelines are presented in the paper. The cement paste, mortar and concrete, with w/c=0,5 ratio, were prepared using innovative air-entraining cement CEM II/B-V. The air-entraining cement CEM II/B-V was produced using two methods: mixed together with natural or synthetic aerated admixture. The air volume test of the volumetric method was carried out in case of fresh cement paste, mortar and concrete mix. Fresh concretes were evaluated in terms of stability of air entrainment and consistency for 5, 20 and 40 min. The porosity structure parameters, like summarized air-content, specific surface of air voids, air-voids spacing factor and micropores content of hardened concrete, were estimated using computer tomography with a resolution of 2-5  $\mu\text{m}$ . The aim of the research was to determine the dependence between air-content of cement paste, mortar and concrete on the measurement of air-entrainment of cement paste or mortar with the same w/c ratio and type of cement, all designed according to PN-EN 480-1 guidelines. Test results proved that there is a good correlation between the measured air-content of the cement paste, mortar and concrete. Therefore, it is possible to predict the aeration of concrete on the air-entrainment of the mortar. Moreover, the porosity parameters research results indicated, that the air-entrainment of concrete is very stable. Research results indicated that the porosity of hardened concrete meets the European standards for frost-resistant concrete. Research results of the influence of air-entraining admixture type have proved, that a greater amount of micro pores in concrete is the effect of a synthetic air-entraining admixture. In concrete with synthetic entraining admixtures the air pores have smaller diameters than in concrete with natural air-entraining admixture, which are being transferred into their specific surface. The synthetic admixture is more recommended than the natural one also because of the value of the air-voids spacing factor of concrete.

## 1. Introduction

In order to maintain better durability performance and extended service life in freezing and thawing environments, concrete should have an adequate air-void. For this, a suitable amount of entrained air-voids with accurate specific surface and spacing factor should be ensured. Commonly, limits on the volume of air-voids or air content are specified although the role of spacing factor is important. This is due to the fact that the air content can be determined more effortlessly and immediately than spacing factor [[1]]. This type of the air entrainment is desired for freeze-thaw protection. To accomplish this, air entraining agents are used to entrain or better stabilize the required air.



The type of air-entraining admixture influences significantly the porosity values of concrete parameters. Research results presented in publication [[2]] proved that the natural admixture aeration is more powerful than the synthetic one because the natural admixture participation is much smaller than the synthetic in cement. Generally, the more water in the volume of concrete, the more air bubbles are formed [[3]]. It is not known which type of admixture entraining is less sensitive to changes in the quantities of water in the concrete.

Moreover, cement type is an important factor for achieving proper porosity of concrete. Most problems with appropriate air-entrainment take place in concretes with fly ash cement. The main reason is that the coke in fly ash with a developed specific surface (loss of ignition) may absorb on its surface the surface-active factor, thereby decreasing its effectiveness. Therefore, a higher amount of air-entrainment admixture may be required in order to achieve the same degree of air-entrainment. Furthermore, if the distribution of coke in concrete is irregular, areas of different air content may occur. Fly ash, even in small quantities reduces natural air content by about 1%. Although Fagerlund [[4]], [[5]] proposed that for concrete with air-entrainment the effect of fly ash is negligible, provided that its content does not exceed 25% (ACI 218-95 recommends the reduction of puzzolan to the value of 25%). However, if the distribution of unburned coke in concrete is uneven, then the areas of different air content may occur. Accordingly, he proposes a higher dosage of air-entrainment admixture. It is worth indicating the types of cement – apart from CEM I – recommended in a XF environment. For example, in environmental classes XF2 and XF4, the Austrian Standard qualifies cement CEM II/A-V as “recommended”, while the German Standard qualifies this cement as “not recommended”.

Thus, designing aeration of concrete is complicated, because it depends on the type of cement and, above all, on the amount of water relative to the cement. Determining the amount of air-entraining admixture on the concrete laboratory test is cost-intensive and time-consuming work. A certain simplification in this area would be the creation of the possibility of aerating the concrete on the basis of the air-entraining test of cement paste or mortar, made of the same materials as concrete. Of course, while maintaining the same other technological parameters, above all the mortar and concrete temperature. The present study is an experimental analysis of the relationship between air-volume in air-entrained cement paste, mortar and plastic and hardened concrete, all designed according to PN-EN 480-1 [[6]] is presented in the paper. The cement paste, mortar and concrete, with  $w/c=0,5$  ratio, were prepared using innovative air-entraining cement CEM II/B-S, incorporating natural or synthetic air-entraining admixture.

## 2. Experimental procedure

In the first stage of the research, fresh pastes and mortars with innovative air-entraining cement CEM II/B-S were evaluated. These cement mixtures were made of air-entraining innovative CEM II/B-V with natural or synthetic air-entraining admixture (AEA) (Tables 1, 2 and 3), normalized sand, distilled water (Table 4). With these materials, standardized cement mortar with  $w/c = 0.50$  was prepared, in accordance with the recommendations of PN-EN 480-1 [[6]]. The consistency of the mortars was determined according to PN-EN 1015-3 [[7]], while the air-content according to PN-EN 1015-7 [[8]].

In the second stage of the research, fresh concrete mix with innovative air-entraining cement were evaluated (Table 5) in terms of stability of air entrainment and consistency of mortar for 5, 20 and 40 min. The concrete mix was prepared acc. to PN-EN 480-1. Ambient temperature during fresh concrete testing was  $20^{\circ}\text{C} \pm 1^{\circ}\text{C}$ . The relative air humidity was about 50%. The air-content of fresh concrete mix according to PN-EN 480-1 [[6]] was investigated according to PN-EN 12350-7:2009 [[9]]. The consistency of fresh concrete was investigated according to PN-EN 12350-2 [[10]]. The stability of air entrainment and consistency of cement mortar was checked for 5, 20 and 40 min.

**Table 1.** The amount of AEA admixture with respect to cement mass

Type of cement	Symbol of cement	Type of AEA admixture	Amount of admixture, % mass of cement
Non-air-entraining	CEM II/B-V	-	0.00
Air-entraining	CEM II/B-V (S1)	synthetic	1.94
Air-entraining	CEM II/B-V (N1)	natural	0.13
Air-entraining	CEM II/B-V (S2)	synthetic	1.70
Air-entraining	CEM II/B-V (N2)	natural	0.12

**Table 2.** The properties of CEM II/B-V

Cement	Specific surface	Setting time		NR, %	SO <sub>3</sub> , %	LOI, %
		start	end			
CEM II/B-V	4180	199	274	23.1	2.89	2.82

**Table 3.** The properties air-entraining CEM II/B-V components

Properties	CEM I 52.5R, % mass of cement	Silica fly ash, % mass of cement
Loss on ignition	0.64	2.26
SiO <sub>2</sub>	20.8	54.20
Al <sub>2</sub> O <sub>3</sub>	5.18	26.81
Fe <sub>2</sub> O <sub>3</sub>	2.94	5.62
CaO	63.9	3.03
MgO	1.38	0.82
SO <sub>3</sub>	4.61	0.34
K <sub>2</sub> O	0.73	2.92
Na <sub>2</sub> O	0.12	0.61

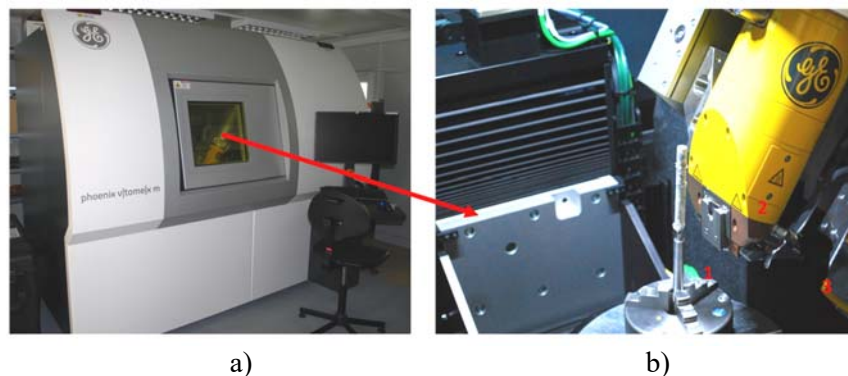
**Table 4.** Paste or mortar composition, gram

Type of cement mixture	Symbol of paste or mortar	Symbol of cement	Amount of cement	Amount of water	Standard sand acc. to PN EN 196-1
paste	n1	CEM II/B-V (N1)	1350.0	675.0	-
paste	s1	CEM II/B-V (S1)	1350.0	675.0	-
paste	n2	CEM II/B-V (N2)	1350.0	675.0	-
paste	s2	CEM II/B-V (S2)	1350.0	675.0	-
mortar	vn1	CEM II/B-V (N1)	450.0	225.0	1350.0
mortar	vs1	CEM II/B-V (S1)	450.0	225.0	1350.0
mortar	vn2	CEM II/B-V (N2)	450.0	225.0	1350.0
mortar	vs2	CEM II/B-V (S2)	450.0	225.0	1350.0

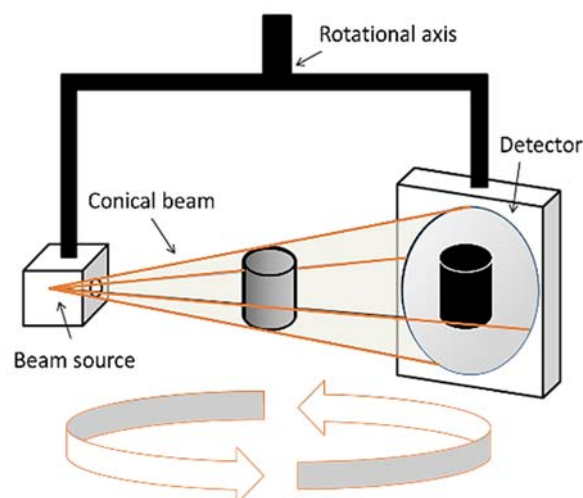
**Table 5.** The amount of concrete components; kg/m<sup>3</sup>

Symbol of concrete	Type of cement	Cement	Water	Sand 0-2 mm	Crushed gravel 2-8 mm	Crushed gravel 8-16 mm
VN1	CEM II/B-V (N1)	350	175.00	522.00	511.90	853.10
VS1	CEM II/B-V (S1)	350	175.00	522.00	511.90	853.10
VN2	CEM II/B-V (N2)	350	175.00	522.00	511.90	853.10
VS2	CEM II/B-V (S2)	350	175.00	522.00	511.90	853.10

In the third stage of the research the porosity of hardened concrete acc. PN-EN 480-1 with air-entraining CEM II/B-V (Table 5) was investigated. The porosity structure parameters of the air-entrained concrete according to PN-EN 480-11 [[11]] with air-entraining CEM II/B-V were estimated. Porosity parameters of concrete were obtained from processing of computed tomography (CT) scanned images. Tomographic studies of samples were performed using a CT scanner: v/tome/x m - firm's GE (Figure 1). The CT scanner was equipped with a detector panel and projection system using the X-ray beam which is formed into a cone. (Figure 2).



**Figure 1.** The view of computer tomograph V/Tome/x firm's GE a) general view b) view in the middle, 1 – a table with the sample, 2,3 – X-ray tubes [on the basis of materials of the Air Force Institute of Technology]

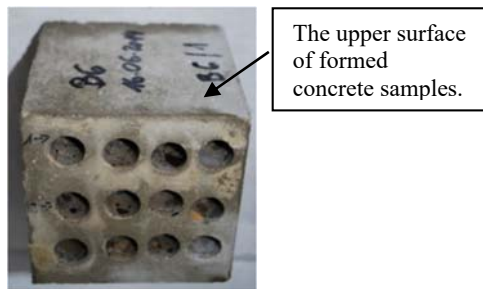


**Figure 2.** Diagram of the conical beam [on the basis of materials of the Air Force Institute of Technology]

Tomographic studies conducted on drilled core samples (with a diameter of 10 mm) which were taken from the cubic size 0,15x0,15x0,15 m (

Figure 3 and

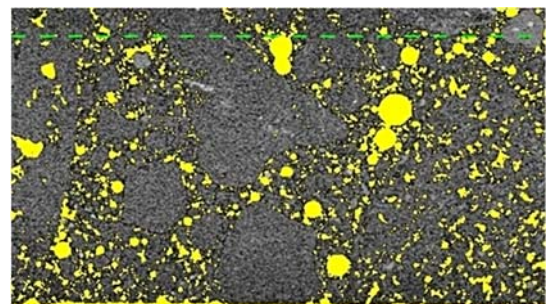
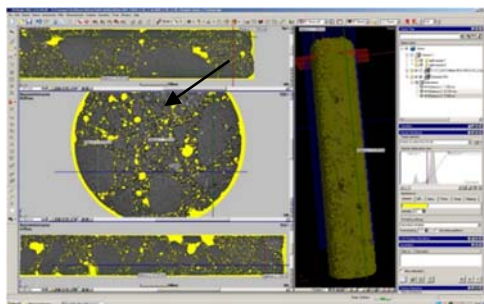
Figure 4). On the basis of digital image analysis of drilled concrete core the structure parameters of air voids were determined by the means of a computer program (Figure 5). The example of a cross-section of the sample for further analysis is shown in Figures 3 and 4.



**Figure 3.** Method of obtaining the drilling concrete [on the basis of materials of the Air Force Institute of Technology]



**Figure 4.** Example of drilling concrete [on the basis of materials of the Air Force Institute of Technology]



**Figure 5.** The image section of the concrete core obtained using CT software (air pores are marked in yellow – screenshot [on the basis of materials of the Military Academy of Sciences])

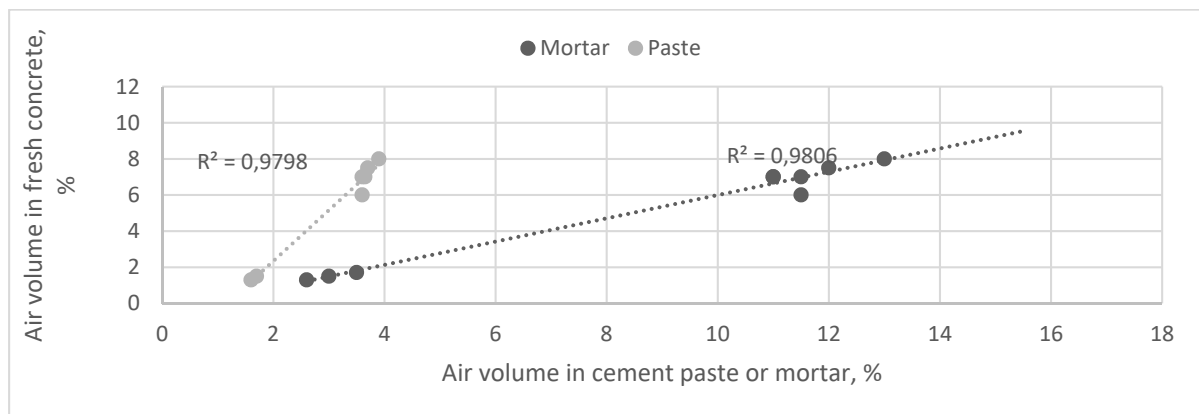
### 3. Results and discussions

In Figure 6 the relationship between the air content in cement paste, mortar and fresh concrete is presented. These results prove that there is a strong correlation between the air content in the cement paste, mortar and plastic concrete mix. The air content in the cement paste corresponds to about twice the air content in the concrete mix, while the air content in the mortar corresponds to about half of the air content in the concrete mix. Therefore, it can be assumed that air-content of the plastic concrete mix according to PN-EN 480-1 can be predicted on the basis of air-content test of mortar according to PN EN 480-1 (made of the same cement and w/c ratio and the same temperature as concrete).

Instead, the relationship between the air-content of plastic concrete and hardened concrete is described differently in the literature. A combined field and laboratory testing program was conducted to compare air content in plastic and hardened concrete and to study the relationship of air content to vibration and compressive strength [[12]]. There is essentially no change in the entrained air content between plastic and hardened concrete. The results [[13]] show that, at the ranges commonly used in the construction of pavements and bridges, the air content of fresh concrete measured by pressure meters and that determined by the microscopical method for essentially the same concrete after hardening are,

for practical purposes, the same. The air content obtained by a volumetric meter as normally run in the field is generally lower than that obtained for the same hardened concrete by the microscopical method. Whereas, research results [[14]] indicated that that the air content in the fresh concrete can be 0.5 to 1.5% greater than that present in the hardened concrete. The results of the tests presented in

Figure 9 show that the difference between the air content in the plastic mix and hardened concrete is up to about 1%, both up and down.



**Figure 6.** The relationship between the air content in cement paste, mortar and fresh concrete with CEM II/B-V, w/c=0.50, regardless of the type of air-entraining admixture

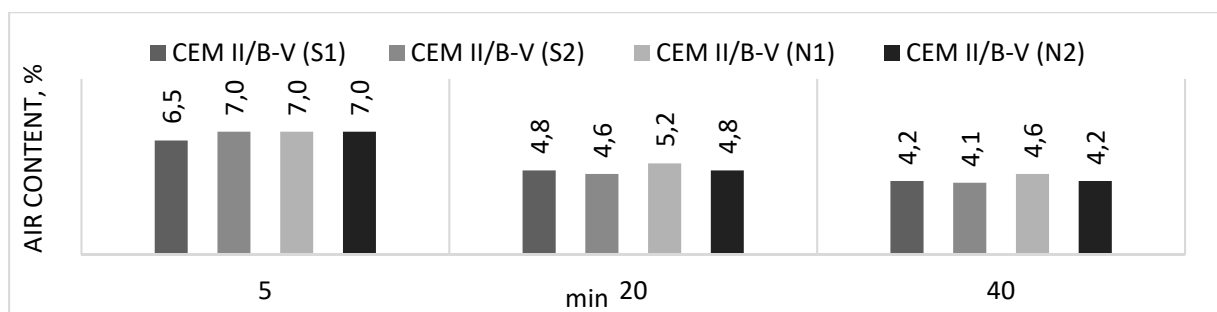
The change in the air content over time and the consistency of the concrete mix is shown in the Figure 7 and

Figure 8. The lower air content value of the concrete mix after 40 minutes (

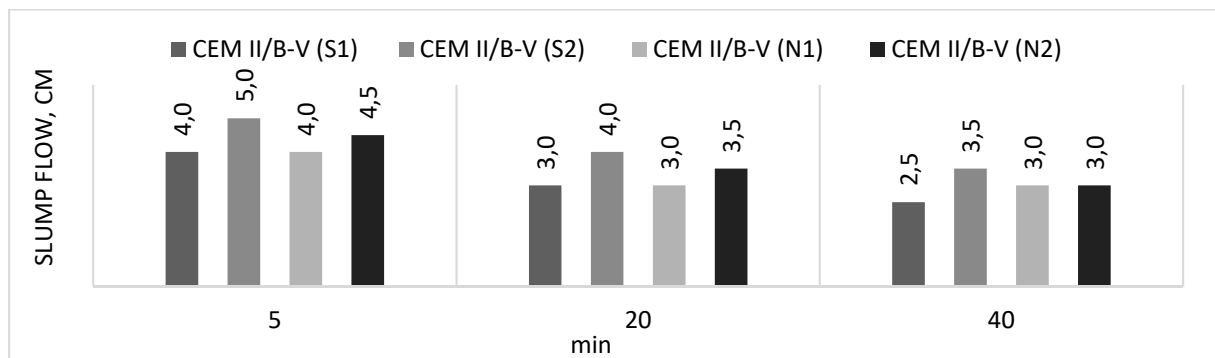
Figure 7) does not correspond to a lower air content in the hardened concrete (Figure 9), since this is in most cases even higher than was measured during the first measurement in concrete mix (Figure 7). This proves the high stability of aeration of concrete in spite of the presence of fly ash. The change of air-entrainment and consistency of concrete mix in time is not dependent on the type of air-entraining admixture (

Figure 7 and

Figure 8).



**Figure 7.** Air content in fresh concrete after 5, 20 and 40 minutes



**Figure 8.** Consistency of fresh concrete after 5, 20 and 40 minutes

Figures from

Figure 12 to

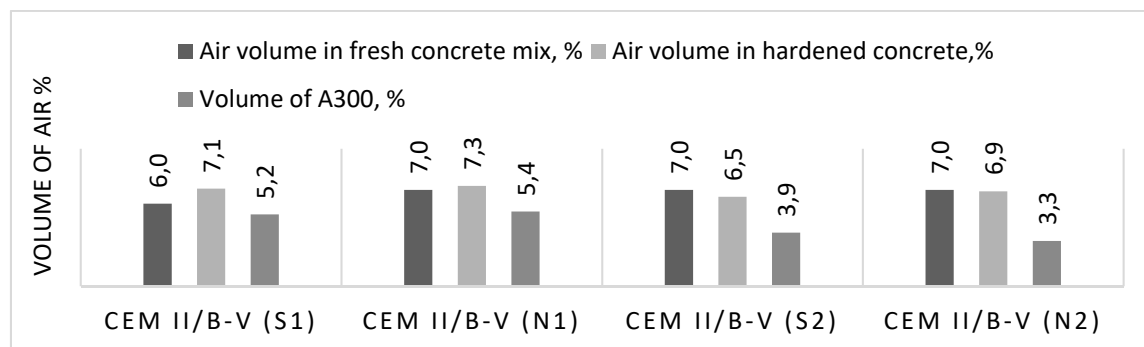
Figure 11 show the porosity research results of hardened concrete including natural or synthetic air-entraining admixture. Air entrainment of fresh concrete is more or less stable, depending on type of air-entraining admixture (

Figure 9). In case of synthetic admixture participation of these pores is by far the largest (

Figure 12 and

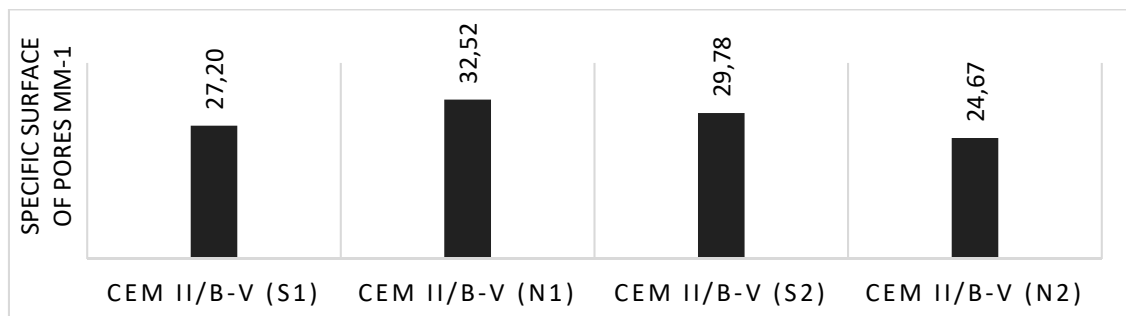
Figure 13). However, in case of synthetic admixture, the volume of micropores with diameters less than 300 microns' pore and specific surface of pores are bigger than in case natural admixture in case of N2 and S2 series, although in case of a natural admixture higher total air content was measured. (Figure 9 and

Figure 10). This relates to the fact that the pore spacing is smaller in case of concrete made with the use of synthetic air-entraining admixture S2 than in case of natural N2 use (Figure 11), again, despite the fact that in case of natural admixture there is a higher total air content.

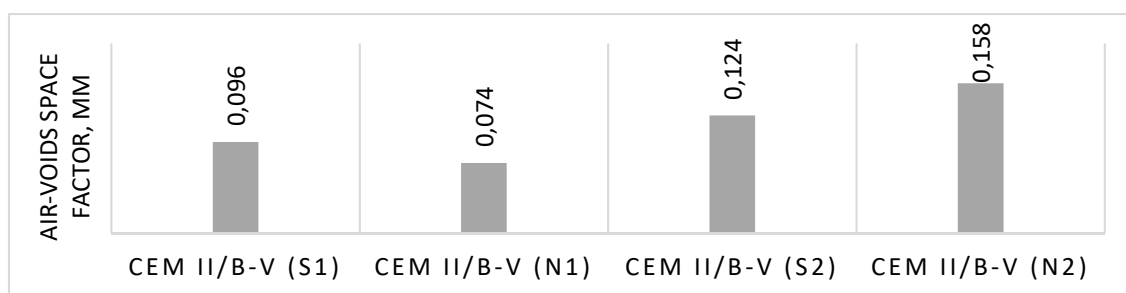


**Figure 9.** The comparison of air content of fresh concrete ( $A_c$ ), hardened concrete ( $A$ ) and content of air-voids with a diameter smaller than 0.300 mm ( $A_{300}$ ) research results





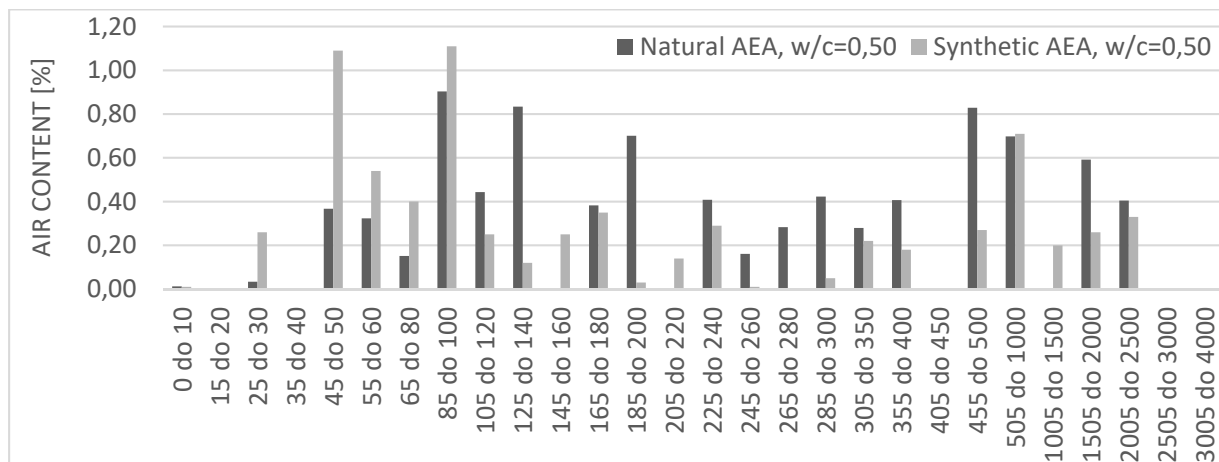
**Figure 10.** The research results of specific surface of the air voids of concrete ( $\alpha$ ) measurements



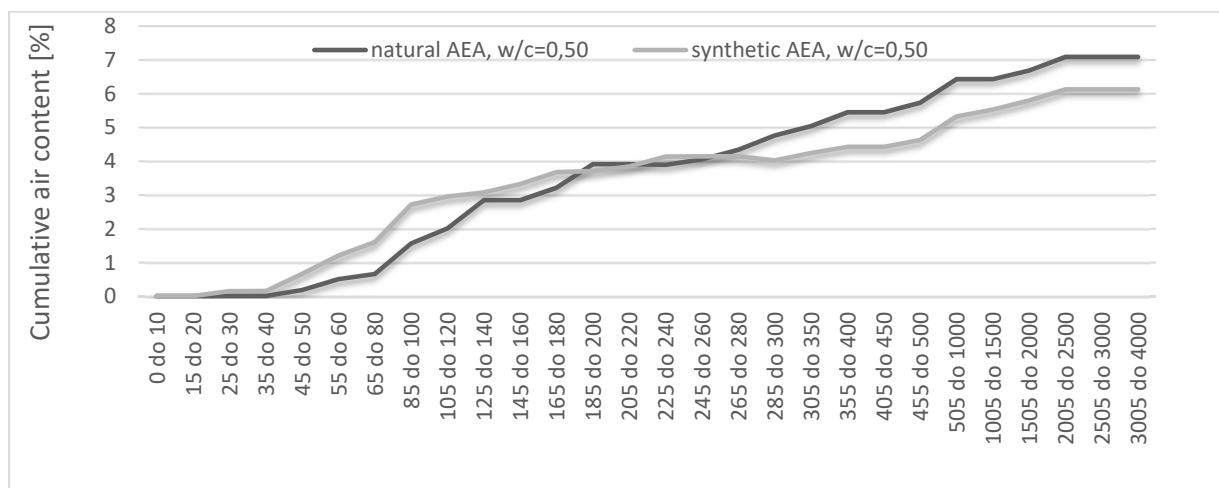
**Figure 11.** The research results of the air-voids space factor of concrete  $\bar{L}$  measurements

Research results presented in a publication [[2]] proved that the natural admixture aeration is more powerful than the synthetic one because the natural admixture participation is much smaller than the synthetic in cement. Commercially available air-entraining agents are generally manufactured from chemically complex raw materials, and the final products may consist of blends of these raw materials plus other raw materials or chemicals, hence it is challenging to define the air-entraining agents chemically except by a rather broad classification. Synthetic detergents allow the quick production of the air bubbles in concrete; these bubbles tend to be coarser than those produced using wood-derived materials [[15]], [[16]]. While their primary application has been for foaming agents, some are also used as the air-entraining agents. Generally speaking, the synthetic ones produce air quicker than the organic ones; yet, the organic ones have better compatibility with other admixtures than the synthetic ones [[17]]. The synthetic detergents have been blended with water-reducing agents to generate water-reducing/air-entraining agents. The synthetic agents were more active in lowering the surface tension of cement filtrate than their vinsol resin counterparts [[15]]. The results of the research presented in

Figure 12 and Figure 13 show that a greater amount of micro pores in concrete is the result of a synthetic air-entraining admixture. In concrete with synthetic entraining admixtures the air pores have smaller diameters than in concrete with natural air-entraining admixture, which are being transferred into their specific surface. As shown in Figure 11, the synthetic admixture is more favorable than natural one also because of the value of the air-voids spacing factor of concrete (compare S2 and N2 series – air voids space factor of S2 series has a smaller value despite of smaller volume of air in concrete (Figure 9)). The obtained results are consistent with the published results in articles.



**Figure 12.** The pore size distribution of concrete with CEM II/B-V (S1) or CEM II/B-V (N1)



**Figure 13.** The cumulative air content of concrete with CEM II/B-V (S1) or CEM II/B-V (N1)

It is considered that the air-voids structure of concrete constitutes its efficient protection against the damaging effects of cyclic freezing-thawing, if the total air content of concrete is between 4% and 7%, the air-voids space factor  $\bar{L}$  is smaller than 0,20 mm or 0,22 mm, specific surface area of air-voids  $\alpha$  is in the range 16-24 mm<sup>-1</sup>, and volume of micropores smaller than 0,300 mm ( $A_{300}$ ) is at least 1,5% [[18]], [[19]], [20]. The porosity characteristics of concrete (air content A (%), content of micro-voids  $A_{300}$  (%), void spacing factor  $\bar{L}$  (mm)) meet the requirements of the European standards for frost-resistant concrete:

- PN-EN 206-1 Concrete – Part 1: Specification, performance, production and conformity:  $A \geq 4\%$  (XF1-XF4),
- Austrian Standard ÖNORM. B 4710-1. Ausgabe: 2007-10-01. Beton. Teil 1: Festlegung, Herstellung, Verwendung und. Konformitätsnachweis:  $A \geq 2.5\%$  (XF2 and XF3),  $A \geq 4\%$  (XF4),  $A_{300} \geq 1.0\%$  (XF2 and XF3),  $A_{300} \geq 1.8\%$  (XF4),  $\bar{L} \leq 0.18\text{mm}$  (XF4),
- Danish Standard DS 2426, Concrete Materials – Rules for application of DS/EN 206-1 in Denmark, Annex U, May 2004:  $A \geq 4.5\%$  in mixture (XF2-XF4),  $A \geq 3.5\%$  concrete (XF2-XF4),  $\bar{L} \leq 0.18\text{mm}$  (XF2-XF4),

- German Standard DIN 1045-2: air content in mixture depends on maximum aggregate size:  $A \geq 3.5\%$  at  $d_{\max}=63$  mm,  $A \geq 4.0\%$  at  $d_{\max}=32$  mm,  $A \geq 4.5\%$  at  $d_{\max}=16$  mm,  $A \geq 5.5\%$  at  $d_{\max}=8$  mm (1 mm = 0.03937 in.).

Regarding the research results presented above (Figs 9-11) it can be concluded that innovative, air-entraining cement CEM II/B-V, regardless of the used air-entraining admixture, provides an adequate structure of concrete porosity (acc. PN-EN 480-1) with  $w/c = 0.50$ .

#### 4. Conclusions

Based on the research results conducted by the reference mortar and concrete according to PN-EN-480-1 with 0,50 ratio and CEM II/B-V it is concluded that:

- There is a strong correlation between the air content in the cement paste, mortar and plastic concrete mix. The air content in the cement paste corresponds to about twice the air content in the concrete mix, while the air content in the mortar corresponds to about half of the air content in the concrete mix. Therefore, it can be assumed that the air-content of the plastic concrete mix according to PN-EN 480-1 can be predicted on the basis of air-content test of mortar according to PN EN 480-1 (made of the same cement and  $w/c$  ratio and the same temperature as concrete).
- A greater amount of micro pores in concrete is the effect of a synthetic air-entraining admixture. In concrete with synthetic entraining admixtures the air pores have smaller diameters than in concrete with natural air-entraining admixture, which are being transferred to their specific surface. The synthetic admixture is more recommended than the natural one also because of the value of the air-voids spacing factor of concrete.
- The air-entraining cement CEM II/B-V, regardless of the air-entraining admixture used, provides the air-voids structure that meets the requirements of European standards and guidelines for frost-resistant concrete.

#### Acknowledgment

The present study was funded by the National Centre for Research and Development Project PBS1/A2/4/2012 "Innovative Cement Concrete Aerating".

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