Frost Resistance of Concrete from Innovative Air-Entraining Cements

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Abstract. The results of concrete tests on cements with mineral additives indicate that, despite better tightness and higher strength, there are problems with frost resistance of non-aerated concretes, even under conditions of moderate exposure to frost. In order to minimize problems with obtaining frost-resistant concrete, an attempt was made to create air-entraining cement. In this paper are presented frost resistance of concrete with air-entraining cement. The researches were carried for concretes made of multicomponent Portland, metallurgical and multi-component cements additionally differentiated by the type of air-entraining admixture (synthetic and natural) and the method of cement preparation (cement mixed together, common ground cement). There were tested internal frost resistance of concrete and a salt frost scaling of concrete.

1. Introduction

The durability of a cementitious composite is the most important criterion for the evaluation of this material. However, due to the durability of the cementitious composite, its frost resistance is an important property. In the assessment of freeze resistance of concrete, methods were sought to enable reliable and quick assurance of resistance to periodical freeze-thawing. Among the methods of material and structural protection of concrete against the harmful effects of frost, two basic actions can be distinguished aiming at obtaining a tight, non-absorbent structure, ie by lowering the w/c ratio, using mineral additives and plasticizing admixtures and by proper air-entraining. The term "air-entraining" refers to air intentionally produced in a concrete mix as a result of the air-entraining admixture. Frost resistance is promoted by increased air content and reduced pore size in concrete, as both factors reduce the distance between pores. A commonly used action to ensure material and structural protection of concrete is its proper air-entraining. In order to ensure frost resistance of concrete, the European standard PN-EN 206 [1] requires its air-entraining at a level of 4 - 7%. Recently introduced by GDDKiA, General Technical Specifications [2], as a model in new communication investments, introduce new and extended requirements for the air-entraining of the mixture and concrete, in particular in relation to the air-entraining structure of concrete. The Committee of the American Concrete Institute (ACI) 201 also requires the use of an aerated admixture in concretes exposed to frost destruction [3].

2. Factor affecting frost resistance of concrete

The amount of applied air-entraining admixture is significantly affected by the composition of the cement used. In general, it is recommended to increase the air-entraining admixture dose over the
recommended one in cases where the cement has an increased surface area or is characterized by low alkali content. However, it is difficult to clearly determine the effect of the cement type on the air-entraining stability of the mixture and the frost resistance of concrete due to the lack of ordered research in this area. It may happen that the change of cement type while maintaining all other technological and material parameters may reduce the frost resistance of concrete [4]. According to the Committee 225 (Guide to the Selection and Use of Hydraulic Cements) and Committee 201 (Guide to Durable Concrete) of the American Concrete Institute (ACI), it was stated that: "different varieties of Portland cements and multicomponent cements allow achieving the same level of frost resistance of concrete, provided the correct proportions of ingredients and correct air-entraining of the mixture" [5].

In case of the use of a mineral additive as a substitute for a part of cement is observed to reduce the air content in the fresh concrete in relation to the mixture containing only Portland cement with similar workability. The results of concrete tests on cements with mineral additives indicate that despite better tightness and higher strength, there are problems with frost resistance of non-aerated concretes, even in moderate frost conditions [6]. In order to minimize problems with obtaining frost-resistant concrete, an attempt was made to create air-entraining innovative cement. Air-entraining innovative cements are those that in their composition contain an air-entraining admixture in the form of a powder in an amount ensuring air-entraining of the concrete required by the standard PN-EN 206 [1]. Air-entraining innovative cements were produced using two manufacturing technologies: as cements co-mixed or co-milled together with an air-entraining admixture.

### 3. Experimental procedure

The tests were performed on the reference concrete, whose composition is shown in table 1. Two air-entraining admixtures were used for the tests: synthetic (A) and natural (D). Types of tested cements are given in table 2. The concrete mix preparation procedure was in accordance with PN-EN 480-1 [7].

| Component       | Amount of component/ 1000 dm³ |
|-----------------|-------------------------------|
| Water, kg       | 175,00                        |
| Cement, kg      | 350,00                        |
| w/c, -          | 0,50                          |
| Sand, kg        | 522,50                        |
| Gravel 2-8 mm, kg | 511,90                       |
| Gravel 8-16 mm, kg | 853,10                       |

After mixing, the air content in the concrete was measured. This test was in accordance with EN 12350-7 [8]. The cubic samples for the tests with a nominal dimension of 150 mm and 100 mm (for tomographic examinations of samples using a computer tomograph) were made in watertight forms and resistant to the absorption of water from fresh concrete mix. The shape, dimensions and dimensional tolerances between the surfaces obtained in the mold, between the upper surface and the lower surface obtained from the mold, flatness tolerance of the surface to which the load from the testing machine is passed and the cubical tolerance of the cube side planes in relation to the foundation at the concreting meet the requirements PN-EN 12390-1 [9] standards. Concrete immediately after laying in the mold was compacted, in accordance with the requirements of PN-EN 12390-2 [10], by means of a vibrating table with a minimum frequency of 40 Hz (2,400 cycles per minute) in two layers, in such a way that excessive segregation would not occur ingredients and the appearance of a coating of cement milk. The test samples remained in the molds for two days at 20 °C ± 5 °C, protected against shocks, vibrations and water loss. After removing from the molds until the moment of testing, the samples were stored in water at 20°C ± 2°C.
Table 2. List of air-entraining innovative cement and type of air-entraining admixture.

| Symbol of air-entraining cement | Production method: co-mixed (m) / co-milled (w) | Type of air-entraining admixture: A (synthetic) lub D (natural) |
|---------------------------------|-----------------------------------------------|---------------------------------------------------------------|
| CEM II/B-V A m                  | m                                             | A                                                             |
| CEM II/B-V D m                  | m                                             | D                                                             |
| CEM II/B-V A w                  | w                                             | A                                                             |
| CEM II/B-V D w                  | w                                             | D                                                             |
| CEM II/B-S A m                  | m                                             | A                                                             |
| CEM II/B-S D m                  | m                                             | D                                                             |
| CEM II/B-S A w                  | w                                             | A                                                             |
| CEM II/B-S D w                  | w                                             | D                                                             |
| CEM III/A A m                   | m                                             | A                                                             |
| CEM III/A D m                   | m                                             | D                                                             |
| CEM III/A A w                   | w                                             | A                                                             |
| CEM III/A D w                   | w                                             | D                                                             |
| CEM III/A-NA A m                | m                                             | A                                                             |
| CEM III/A-NA D m                | m                                             | D                                                             |
| CEM III/A-NA A w                | w                                             | A                                                             |
| CEM III/A-NA D w                | w                                             | D                                                             |
| CEM V/A (S-V) A m               | m                                             | A                                                             |
| CEM V/A (S-V) D m               | m                                             | D                                                             |
| CEM V/A (S-V) A w               | w                                             | A                                                             |
| CEM V/A (S-V) D w               | w                                             | D                                                             |
| CEM V/B (S-V) A m               | m                                             | A                                                             |
| CEM V/B (S-V) D m               | m                                             | D                                                             |
| CEM V/B (S-V) A w               | w                                             | A                                                             |
| CEM V/B (S-V) D w               | w                                             | D                                                             |

Internal frost resistance of concrete and salt frost scaling resistance of concrete. The frost resistance test using the ordinary concrete method was carried out in accordance with the PN-88/B-06250 [11] standard. This procedure simulates the working conditions of concrete exposed to water against freezing. In each test series, 6 samples were subjected to freezing / thawing cycles and 6 samples were stored in water as reference samples. The frost resistance requirements determined by the usual method in the standard are defined according to the N index equal to the number of expected years of construction usage (from N25 to N200 and N>200). The number of years required increases if the concrete is subject to capillary rising of the water, the action of de-icing agents or when the concrete is in the zone of changing water level. N index correspond to frost resistance F (from F25 to F300). This is the number of freezing and thawing cycles, made according to the mode specified in the standard, which the concrete should withstand without cracking, mass losses greater than 5%, or strength reduction greater than 20% in relation to the strength of non-frozen samples. To determine the salt frost scaling resistance of concrete, the "slab test" method was used (modeled on the Swedish standard SS 13 72 44, so-called Borås method) as the reference method [12]. This test consists in determining the mass of scaled-off material from the upper surface of the sample after 28 and 56 freezing and thawing cycles in the presence of 3% NaCl solution. Standard PN-EN 13877-2 [12] distinguishes three categories of concretes (table 3) depending on the amount of scaled-off material. The test was carried out after 28 days of concrete hardening under standard conditions. From cubic samples (150 mm), a 150 × 150 × 50 mm sample was cut perpendicular to the floated surface. All surfaces of the sample, except
the tested, were covered with silicone and placed in a polystyrene form, the edges of which protruded above the surface to a height of 20 mm. A 3% NaCl solution was poured onto the sample surface to be tested, and the whole was placed in the freezer chamber for 56 cycles. After the determination, the amount of scaled-of material was weighed.

| Table 3. Criteria for assessing frost resistance according to PN-EN 13877-2 [12]. |
|----------------------------------|------------------|------------------|--------------------|
| Category | Decrease of mass after 28 cyklach m28 | Decrease of mass after 56 cyklach m56 | Degree of decrease of mass m56/m28 |
| FT0 | No requirements | No requirements | No requirements |
| FT1 | average < 1,0 kg/m² (no single result > 1,5 kg/m²) | No requirements | No requirements |
| FT2 | average ≤ 0,5 kg/m² | average < 1,0 kg/m² (no single result > 1,5 kg/m²) | ≤ 2,0 kg/m² |

4. Results and discussions

Figures 1-3 show the air-content in fresh concrete mix. The air-content of the concrete meets the requirements for the environmental exposure class XF according to EN 206. The type of cement and the type of air-entraining admixture significantly affect the need for air-content in concrete. The amount of air-entraining admixture for the cement type was given to a cement plant producing aerated cement.

| Air content in fresh concrete, % |
|----------------------------------|------------------|------------------|--------------------|
| CEM II/B-S D m | 5,8 | 6,5 | 5,8 |
| CEM II/B-S A m | 5,8 | 5,8 | 5,4 |
| CEM II/B-S A w | 6,5 | 6,0 | 5,8 |

**Figure 1.** The air-content in fresh concrete with air-entraining CEM II/B-V or CEM II/B-S.

| Air content in fresh concrete, % |
|----------------------------------|------------------|------------------|--------------------|
| CEM III/AÁ D m | 6,5 | 6,5 | 7,0 |
| CEM III/AÁ w | 7,0 | 7,0 | 6,5 |
| CEM III/AÁ A w | 5,0 | 5,4 | 4,9 |

**Figure 2.** The air-content in fresh concrete with air-entraining CEM III/A or CEM III/A-NA.
Figure 3. The air-content in fresh concrete with air-entraining CEM V/A (S-V) or CEM V/B (S-V).

The analysis of the test results shown in Figures 4-15 show that the air-entraining cement production technology, by co-milling or co-mixing, and also the type of air-entraining admixture, natural or synthetic, do not affect the internal and surface frost-resistance of concrete. However, the percentage of mineral supplement relative to Portland clinker, as well as the type of clinker itself, i.e. the normal or reduced amount of alkali, is important.

The destruction of concrete in a water-saturated state at the alternate freezing and thawing is caused by complex physical processes that result in the deformation and mechanical destruction of concrete. The behaviour of concretes after 150 freezing/thawing cycles were studied and the reduction of compressive strength is shown in Figures 4-9 show. To obtain the internal resistance to concrete with air-entraining cements, it is recommended to use air-entraining concrete cement CEM II / B-V and CEM II / B-S and CEM III / A. In the case of CEM III / A-NA cement special attention should be paid to surface flaking, even in no presence of de-icing salts, although its strength has not been reduced more than is acceptable. Problem with internal frost-resistance of concrete, as unacceptable decrease of compressive strength and mass of concrete, is in case of concrete with CEM V / A (S-V) and CEM V / B (S-V). It is interesting that the strength of concrete after frost resistance test increased. This fact explains the further progressing hydration of pozzolanic additives in concrete, even during freezing-thawing of concrete. Particularly, when microcracks provide for water penetration into the interior of the concrete, as well as occurring capillary pores at w/c = 0.5, and still overgrown with hydration products of pozzolana additions over time. For this reason, the freezing resistance of concrete with cements with pozzolana additions should be tested at least after 90 days, not after 28 [13].

Figure 4. Change of compressive strength of concrete with air-entraining CEM II/B-V or CEM II/B-S after 150 cycles of freezing-thawing.
Figure 5. Change of compressive strength of concrete with air-entraining CEM III/A or CEM III/A-NA after 150 cycles of freezing-thawing cycles.

Figure 6. Change of compressive strength of concrete with air-entraining CEM V/A (S-V) or CEM V/B (S-V) after 150 cycles of freezing-thawing cycles.

Figure 7. Change of mass of concrete with air-entraining CEM II/B-V or CEM II/B-S after 150 cycles of freezing-thawing cycles.
Figures 10 to 15 present the results of the frost resistance test in the presence of de-icing salts. As the research results prove, that in cease of the air-entraining cements CEM II/B-V, CEM II/B-S, CEM III/A, and the majority of CEM III/A-NA concretes obtained the frost resistance class FT2 in the presence of de-icing salts. While almost all concretes with CEM V/A (S-V) and with CEM V/B (S-V) cement achieved the FT1 and FT0 respectively. The amount of exfoliated concrete in the case of CEM V/B slightly depends on the type of air-entraining admixture. A smaller amount of exfoliated concrete is the case of a synthetic admixture. All concrete test series with CEM III and CEM V of the study did not exceed the different proposed limits to FT2 and may therefore be considered to be frost resistant. After standard storage, the degree of superficial scaling of CEM II/A or CEM V/A and CEM V/B concretes was higher compared to CEM II/B concretes. The reason for this slightly increased superficial scaling could be related to the slightly increased surface porosities of CEM III/A or CEM V/A and CEM V/B samples at the time the freeze-thaw test started [13]. It was cited earlier, that "different varieties of Portland cements and multicomponent cements allow achieving the same level of frost resistance of concrete, provided the correct proportions of ingredients and correct air-entraining of the mixture" [6], should by added that, very important to frost-resistance concrete with CEM III/A, CEM III/A-NA, CEM V/A and CEM V/B is time of start frost-resistance laboratory tests. Frost resistance of concrete these concretes should by indicated after 90 days its hardening. Moreover, in addition to the type of air-entraining cement, very important for frost-resistance of concrete is w/c ratio, compressive strength and volume of air and capillary pores of hardened concrete.
Figure 10. The proportion of peeled concrete with air-entraining CEM II/B-V or CEM II/B-S after 56 and 28 cycles of freezing-thawing in presence of 3% NaCl solution.

Figure 11. The proportion of peeled concrete with air-entraining CEM III/A or CEM III/A-NA after 56 and 28 cycles of freezing-thawing in presence of 3% NaCl solution.

Figure 12. The proportion of peeled concrete after 56 and 28 cycles of freezing-thawing in presence of 3% NaCl solution.
Figure 13. The sum of mass of peeled concrete after 56 cycles of freezing-thawing in presence of 3% NaCl solution.

Figure 14. The sum of mass of peeled concrete after 56 cycles of freezing-thawing in presence of 3% NaCl solution.

Figure 15. The sum of mass of peeled concrete with air-entraining CEM V/A (S-V) or CEM V/B (S-V) after 56 cycles of freezing-thawing in presence of 3% NaCl solution.

5. Conclusions

Within the limits of the tests carried out, it is concluded that:

- The air-entraining cement production technology, by co-milling or co-mixing, and also the type of air-entraining admixture, natural or synthetic, do not affect the internal and surface frost-
resistance of concrete. However, the percentage of mineral supplement relative to Portland clinker, as well as the type of clinker itself, i.e. the normal or reduced amount of alkali, is important. Only in case of CEM V/B (S-V) type air-entraining admixture is important. The amount of exfoliated concrete in the case of this type of cement slightly depends on the type of air-entraining admixture. A smaller amount of exfoliated concrete is the case of a synthetic admixture.

- To obtain the internal resistance to concrete, it is recommended to use air-entraining concrete cement CEM II / B-V and CEM II / B-S and CEM III / A. In the case of CEM III / A-NA cement special attention should be paid to surface flaking, even in no presence of de-icing salts, although its strength has not been reduced more than is acceptable. Problem with internal frost-resistance of concrete, as unacceptable decrease of compressive strength and mass of concrete, is in case of concrete with CEM V/A (S-V) and CEM V / B (S-V).

- In case of the air-entraining cements CEM II /B-V, CEM II/B-S, CEM III / A, and the majority of CEM III / A-NA concretes obtained the frost resistance FT2 class in the presence of de-icing salts. While almost all concretes with CEM V /A (S-V) and with CEM V / B (S-V) cement achieved the FT1 and FT0 classes respectively.

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