Scaling Resistance of Concretes Made with Fly Ash Cement

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Abstract. The paper presents test results of 11 series of concretes with three various levels of the water-binder ratio= 0.35, 0.40, 0.45, made with the CEM II/B-V cement. The aim of the testing was to determine the impact of the water-binder ratio and air-entrainment on the freezing and de-icing salt resistance of concrete made with fly ash cement. The tests were conducted on samples of non-air-entrained and air-entrained concretes using an air-entraining admixture and polymer microspheres. Regardless of the water-binder ratio (from 0.35 to 0.45), the samples of non-air-entrained concretes demonstrated no surface scaling resistance in the presence of 3% NaCl. The tests of air-entrained concretes demonstrated that concrete air-entrainment using polymer microspheres and an air-entraining admixture protect the concrete made with fly ash cement from surface scaling. The concrete air-entrainment using an air-entraining admixture causes a substantially higher drop in compressive strength than using polymer microspheres.

1. Introduction

The protection of concrete against the harmful effects of frost can be obtained in two ways [1]: by lowering the water-binder ratio and thus obtaining a non-permeable structure or by concrete mixture air-entrainment. The lowered water-binder ratio causes an increase in frost durability in both concretes with an air-entraining agent and without such admixtures [2]. Adding fly ash to concrete mixtures causes trouble with air-entrainment due to the varying properties, but mainly due to the presence of carbon. Testing concerning the dosage of air-entraining admixtures in concrete including fly ash was conducted, among others, by Ley, Harris, Folliard and Hover [3]. The tests [4-6] demonstrate that the addition of fly ash causes an increased demand for the air-entraining admixture. In general, the required dose of the air-entraining admixture is higher, the higher is the percentage share of organic particles in fly ash or the higher the calcination losses [7]. Adding ash in quantities higher than 30% to an air-entrained concrete negatively affects the creation of small air pores [4].

Concrete, in which a substantial amount of cement was replaced with fly ash, can demonstrate low frost durability [8]. The basic cause of lower frost durability is the slow pozzolanic reaction and slow increase in concrete strength [9-10]. The best method of improving the frost durability of concrete with the addition of fly ash is to change the microstructure by way of using plasticising admixtures which allow for lowering the water content of concrete and obtaining lower water-binder ratios as well as introducing air-entraining admixtures into the concrete [10].

The tests [11] demonstrate that in the case of the CEM I cements, the water-binder ratio of ≤ 0.37 allows for shaping the frost durability matrix of concrete and mortar without the need of air-entrainment. In the case of the CEM II/B-V fly ash cement, comparable features can be achieved only...
at the water-binder ratio of approx. 0.30. According to [12], concretes air-entrained at the level of 4%, with the addition of fly ash, demonstrate a frost durability of F150. On the other hand, in the case of non-air-entrained concretes with the addition of fly ash, the factor that determines the frost durability is the water-binder ratio. Concretes with the water-binder ratio of \( \leq 0.38 \), regardless of the type and amount of fly ash addition, meet the frost durability criteria of F150. It is more difficult to protect the concrete from surface scaling than from internal cracking [13, 14].

In construction engineering practice, the problem is obtaining a repeatable and stable air pore structure, both in terms of overall volume and the size of air pores [15]. The effects of air-entrainment can differ substantially due to the impact of many factors, such as mixture consistency and temperature, time of mixing, time of transport, method of concrete mixture placement and compacting [1, 16, 17]. Transport of a concrete mixture causes losses in air content, whereas substantial air losses occur during mixture pumping (approx. 1–1.5%). Air losses also occur during mixture vibration. The method that prevents most of the aforementioned problems is the use of microspheres, i.e. spherical particles with specific dimensions, which do not disappear, do not combine, and the pore system is precisely determined. The authors’ own tests [18-20] concerning the air-entrainment of concrete mixtures using polymer microspheres demonstrate a very high effectiveness of this method. The tests also demonstrate that the use of polymer microspheres allows for obtaining a frost durable concrete at a substantially lower air content than in the case of traditional air-entrainment (AEA).

The aim of the testing was to determine the impact of the water-binder ratio and air-entrainment on the scaling resistance of concrete made with fly ash cement. The tests featured preparation of a series of non-air-entrained and air-entrained concretes using an air-entraining admixture and polymer microspheres.

2. Materials and Methods
The tests were conducted on 11 series of concretes with three various levels of the water-binder ratio - 0.35, 0.40, 0.45. The experiment programme included manufacturing samples divided into three groups: three non-air-entrained concretes, three air-entrained concretes with an addition of microspheres and five air-entrained concretes with air-entraining admixture. The aim of the testing was to determine the impact of the water-binder ratio as well as two various methods of air-entrainment on the scaling resistance of concrete made with the CEM II/B-V cement.

The concretes were made up of the following components:
- cement CEM II/B-V 42,5N, S
- natural sand 0 - 2 mm, P
- coarse aggregate - basalt with the fraction of 4 - 8, 8 - 16 mm (50:50%), B
- polycarboxylate superplasticiser,
- polymer microspheres, MSP, D = 40 \( \mu \)m, (series C-MSP4-C-MSP6)
- air-entraining admixture AEA (series C-AEA7-C-AEA11).

The scope of tests for the concrete mixture embraced the determination of the consistency using the concrete slump method (SL), bulk density (\( \rho_b \)) and air content using the pressure method (\( \rho_p \)).

The scope of testing of hardened concrete included the determination of:
- compressive strength (\( f_{cm} \)),
- water absorption (\( n_w \)),
- freezing and de-icing salt resistance (surface scaling) (\( m_{56} \)),
- air-pores structure characteristics (\( A, A_{300}, \alpha, L \)).

The samples matured in water for 7 days, after which they were stored in air-dry conditions for 21 days.

Concrete cubes with the dimensions of 10x10x10 cm were used for the compressive strength and water absorption testing. The compressive strength of concrete was determined in accordance with the PN-EN 12390-3:2011 standard [21] and the water absorption according to the PN-88/B-06250 standard [22].
The surface scaling resistance was determined using the slab test method according to PKN-CEN/TS 12390-9:2007 [23]. The testing consisted of determining the mass of the scaled material from the sample’s top surface after 7, 14, 28, 42 and 56 freeze-thaw cycles in the presence of 3% NaCl solution. Four 50x150x150 mm slabs were tested in each of the eleven series. The tests were conducted on the natural finished surface of the concrete, perpendicular to the moulding direction. The samples were stored, prepared and tested following the standard methods. The mass of the scaled material after 56 cycles relative to the surface of the specimen, was taken as the test result.

The preparation of polished sections and the determination of air void characteristics by the traverse method were performed according to PN-EN 480-11: 2008 [24]. Automatic image analysis was made with the use of a setup consisting of a stereoscopic microscope, a CCD camera and a motorized stage. The composition and selected properties of the concretes are presented in table 1.

### Table 1. Basic information on concrete compositions and properties

| Series | W/S | S kg/m³ | P kg/m³ | B 4-8 kg/m³ | B 8-16 kg/m³ | MSP % m.s. | Zp % | SL cm | g₀ kg/m³ |
|--------|-----|---------|---------|-------------|--------------|------------|-------|-------|---------|
| C1     | 0.35| 430     | 626     | 666         | 666          | -          | -     | 20    | 2544    |
| C2     | 0.40| 387     | 615     | 653         | 653          | -          | -     | 18    | 2461    |
| C3     | 0.45| 370     | 635     | 674         | 674          | -          | -     | 19    | 2524    |
| C-MSP4 | 0.35| 418     | 608     | 646         | 646          | 0.8        | -     | 18    | 2471    |
| C-MSP5 | 0.40| 389     | 624     | 663         | 663          | 0.8        | -     | 19    | 2496    |
| C-MSP6 | 0.45| 363     | 623     | 662         | 662          | 0.8        | -     | 22    | 2476    |
| C-AEA7 | 0.35| 414     | 604     | 641         | 641          | -          | 6.0   | 18    | 2452    |
| C-AEA8 | 0.40| 371     | 595     | 632         | 632          | -          | 7.1   | 19    | 2380    |
| C-AEA9 | 0.45| 334     | 579     | 616         | 616          | -          | 1.0   | 15    | 2297    |
| C-AEA10| 0.45| 359     | 623     | 661         | 661          | -          | 3.9   | 18    | 2467    |
| C-AEA11| 0.45| 349     | 605     | 643         | 643          | -          | 6.5   | 18    | 2401    |

3. Results and discussions

The test results of hardened concretes are presented in tables 2 and 3. The compressive strength of concretes amounts from 26.3 to 85.0 MPa, which correspond to the C12/15-C55/67 strength classes. The highest strengths were obtained for non-air-entrained concretes (C1-C3), whereas the lowest - for air-entrained concretes (C-AEA7-C-AEA110). Air-entrainment using polymer microspheres caused a substantially lesser drop in compressive strength than traditional air-entrainment. The obtained water absorption amounts from 3.77 to 6.03%.

Figure 1 depicts the changes in the mass of non-air-entrained samples and samples air-entrained using the polymer microspheres MSP as well as using the air-entraining admixture AEA after 56 freeze-thaw cycles in the 3% NaCl solution. Figure 2 presents the comparisons of mass losses of samples with the given water-binder ratio.

Regardless of the water-binder ratio, the C1-C3 series of non-air-entrained samples showed to be non-frost resistant. The mass loss after 56 freeze-thaw cycles amounted from 5.593 to 4.731 kg/m². In the case of samples made with polymer microspheres, the water-binder ratio turned out to be the deciding factor. The C-MSP4 and C-MSP5 sample series with the water-binder ratio of 0.35 and 0.40, respectively, turned out to be sufficiently resistant to surface scaling in the presence of 3% NaCl solution. The mass loss after 56 freeze-thaw cycles amounted to 0.863 and 0.540 kg/m² (< 1.0 kg/m²). The C-MSP6 sample series with the water-binder ratio of 0.45 demonstrated a mass loss of 4.094 kg/m², which classifies them as non-frost resistant.

The traditionally air-entrained C-AEA7 and C-AEA8 series sample with the water-binder ratio of 0.30 and 0.40 demonstrated a mass loss of 0.188 and 0.614 kg/m², i.e. they achieved good and sufficient scaling resistance. Three air-entrainment levels (air content from approx. 4 to over 10%) were used for concrete with the water-binder ratio of 0.45. Air content was the deciding factor in terms of the surface scaling resistance of concretes. The concrete with the air content of above 10% (C-
AEA9) demonstrated a good scaling resistance (m56 = 0.344 kg/m²), but the high air content resulted in a substantial drop in strength (C12/15 class compressive strength). Good scaling resistance (m56 = 0.348 kg/m²) was also demonstrated by the C-AEA11 concrete with the air content of approx. 6%. The C-AEA10 concrete with the air content of 4% turned out to be no scaling resistant and its mass loss after 56 freeze-thaw cycles amounted to 3.475 kg/m².

Table 3 summarises the results of the air pore structure tests. The values of spacing factor L for air-entrained concretes (MSP and AEA) amounted from 0.088 to 0.285 mm. The content of pores with the diameter of up to 300 µm A300 in the tested concretes amounted from 0.66 to 7.13%. The air content A amounts from 2.71 to 13.19%.

Table 2. Properties of hardened concrete samples

| Series | f_cm MPa | n_w % | m56 kg/m² |
|--------|----------|-------|-----------|
| C1     | 85.0     | 3.77  | 5.593     |
| C2     | 71.7     | 4.41  | 4.731     |
| C3     | 63.3     | 4.95  | 5.555     |
| C-MSP4 | 80.8     | 4.22  | 0.863     |
| C-MSP5 | 56.0     | 4.65  | 0.540     |
| C-MSP6 | 55.0     | 5.16  | 4.094     |
| C-AEA7 | 70.8     | 4.30  | 0.188     |
| C-AEA8 | 54.3     | 5.18  | 0.614     |
| C-AEA9 | 26.3     | 6.03  | 0.344     |
| C-AEA10| 38.0     | 5.79  | 3.475     |
| C-AEA11| 32.0     | 5.91  | 0.348     |

Table 3. Air pore structure parameters

| Series | A % | A300 % | α mm⁻¹ | L mm |
|--------|-----|-------|--------|------|
| C3     | 1.69| 0.16  | 8.65   | 0.931|
| C-MSP4 | 2.71| 0.66  | 2.00   | 0.285|
| C-MSP5 | 3.15| 1.59  | 60.09  | 0.101|
| C-MSP6 | 3.20| 1.32  | 69.14  | 0.088|
| C-AEA7 | 5.29| 2.19  | 18.62  | 0.257|
| C-AEA8 | 5.17| 2.12  | 17.87  | 0.266|
| C-AEA9 | 1.19| 7.13  | 28.14  | 0.111|

Figure 1. Comparison of the changes in the mass of a) non-air-entrained samples, b) samples air-entrained using polymer microspheres, c) samples air-entrained using AEA after 56 freeze-thaw cycles in the 3% NaCl solution
Figure 2. Comparison of the changes in the mass of concrete samples with the water-binder ratio a) 0.35, b) 0.40, c) 0.45 after 56 freeze-thaw cycles in the 3% NaCl solution

4. Conclusions

Based on the test results of 11 series of concretes with the water-binder ratio of 0.35, 0.40 and 0.45, made with the CEM II/B-V cement the following conclusions were drawn:

1. Regardless of the water-binder ratio (from 0.35 to 0.45), the non-air-entrained concrete series samples demonstrated no scaling resistance in the presence of 3% NaCl.

2. The use of polymer microspheres is an effective method of concrete air-entrainment, which allows to obtain concrete with surface scaling resistance. All series, regardless of the water-binder ratio, were made using a constant addition of microspheres of 0.8% of the binder’s mass. This quantity turned out to be sufficient in the case of concretes with the water-binder ratio of 0.30 and 0.40, but insufficient in the case of the water-binder ratio of 0.45. Air-entrainment using polymer microspheres caused a substantially lesser drop in compressive strength than traditional air-entrainment.

3. The traditionally air-entrained concrete series samples with the water-binder ratio of 0.30, 0.40 and 0.45 turned out to be scaling resistant in the presence of 3% NaCl solution. Air content was the deciding factor in terms of the surface scaling resistance of the concrete with the water-binder ratio of 0.45. The air content of 4% turned out to be insufficient to protect the concrete from surface scaling. Air-entrainment at the level of 10% protected the concrete from surface scaling, but the high air content resulted in a substantial drop in compressive strength. The concrete with the air content of approx. 6% also demonstrated a surface scaling resistance with a smaller drop in compressive strength.

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