Evaluation of frost resistance of various blended binders

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Abstract. The use of blended binders using alternative raw materials is one of the key areas of sustainable development in terms of reducing CO$_2$ in the atmosphere. This approach reduces the proportion of energy-intensive clinker in cement when satisfying technical parameters. The paper presents a proposal of variants of blended binders with the application of fly ash, blast furnace slag and perlite for their use in building mixtures. The durability of the mixtures is assessed on the basis of testing the frost resistance of mortars. Test specimens were produced according to STN EN 196-1. The samples were subjected to 25 freeze-thaw cycles, and both the flexural and compressive strength were then determined, to express a durability factor. Except for slag as a standard component of blended binders, perlite appears to be a perspective material; it meets a limit for durability factor when used as a 30% substitution for cement, as well as when used in ternary composition with slag. Fly ash showed weak frost resistance, even when used with a suitable perlite.

1. Introduction

One of the most important properties of concrete that determines its durability is frost resistance. Freezing-thawing effect is one of the main problems that can lead to damage to materials like concrete or mortar. Durability problems of cement-based materials associated with cold weather conditions can take two main forms:

a) Internal cracking due to freeze-thaw cycles accompanied by loss of strength and stiffness.

b) Surface scaling due to freezing in the presence of de-icing salts.

Typically, damage occurs when the material is saturated and its pore structure is unable to withstand the stresses caused by ice formation. According to [1], concrete can be resistant to cyclic freezing and thawing provided it has sufficient strength and an adequate air-void system, and the aggregates are frost-resistant. The main factors affecting the above mentioned features of concrete, thus its frost resistance, are varieties of cement, water-cement ratio (directly linked to the porosity/permeability of matrix) and the density of aggregates [2]. In the case of mortars, the degree of saturation, total pore volume, pore size and distribution, frost velocity and tensile strength contribute to freeze-thaw resistance [3]. High porosity means higher water content and a higher frost force which reduces the resistance to freezing and thawing cycles.

According to [3], also aggregate size is a factor affecting frost resistance. Small grains are more favourable as they provide a larger specific surface area. As a consequence, there is more cement mortar between the grains. The bonding force is stronger and the resistance to freezing and thawing cycles is better.

Above mentioned varieties of cement are on its composition and on the possible application of mineral additives, including their type and quantity. A categorization of the effects from the mentioned additives is given by [4]; they can be by their type, their scale and the mechanism by which they improve the freeze-thaw performance of concrete.
Studies on application of ground granulated blast furnace slag (GGBFS) [5, 6] show 25%, 50%, 61% and 75% range of it usage in concrete presenting that GGBS has performed well under frost resistance tests [5, 6]. Tavasoli et al. point that the frost resistance of mixtures is highly influenced by percentage; by increasing slag replacement percentage the durability is decreasing. According to author, using 50% GGBS do not have impressive effect on the frost resistance concrete [7].

Perlite powder contains approximately from 70 to 75 % SiO$_2$ and from 12 to 18 % Al$_2$O$_3$. Due to its glassy structure and high SiO$_2$ and Al$_2$O$_3$ contents, perlite can be specified to be a pozzolan [8]. Some investigations report on the properties of cement composites with perlite powder [9,10,11,12], however there are limited results on the frost resistance.

On the other hand, there are quite lots of investigations on effect of fly ash on the frost resistance of cement-based mixtures [1, 13, 14, 15], however as mentioned by [14], there are often not compared on an equal footing. Generally can be summarized that concrete containing fly ash may be less resistant when subjected to freezing and thawing [1]. As presented by [15], frost resistance decreases with dosage of fly ash, while the rate of decline from reference (cement) sample is different with different kinds of fly ash.

The frost resistance of cement composites is affected also by external factors, like the number of freeze-thaw cycles [15]. Liu et al. state, that as the number of freeze-thaw cycles increased, the quality of the cement mortar gradually decreased. Compressive strength, flexural strength and modulus of elasticity of the mortar were also reduced [16]. Glukhovsky et al. concluded that slag concrete activated by sodium silicate usually has the lowest porous structure, the highest strength, and the best frost resistance. These concretes were found to withstand between 300 and 1300 freeze-thaw cycles [17].

The measures to improve frost resistance of cement mixtures are to enhance density, reduce water-cement ratio and apply an air-entraining agent. Bubbles are able to accommodate water being pushed forward by the ice in the process of being formed [2]. Pogorelov and Semenyak report that one of the effective ways to improve the strength and resistance of a material to cracking, including frost aggression, is to introduce fibre reinforcement [18].

The principle of the frost resistance test is the freezing and thawing of test beam which are soaked with water. The number of freezing cycles, as well as applied temperatures, depends on the relevant standard or project [19].

The aim of this work was to verify the influence of selected additive (F, S, P) on the freeze-thaw resistance of cement composites. Perlite, fly ash and ground granulated blast furnace slag, together with cement in different proportions, were used in this work to prepare different blended binders. A reference sample was made up of cement, standard sand and water. The samples were subjected to 25 freeze-thaw cycles. The effect of individual blended binders on the frost resistance of mortars was evaluated by durability factor based on both the flexural strength and the compressive strength.

2. Materials
For testing the frost resistance of mortars, following materials were used:
- Standard sand CEN according to STN EN 196-1 [20]
- Cement CEM I 42,5 R (C): CRH Turňa nad Bodvou
- Mineral additives (Ad):
  - Fly ash (F): US Steel Košice
  - Ground granulated blast furnace slag GGBFS (S): US Steel Košice
  - Perlite powder (P): Lehôtka pod Brehmi
- Water (W)

2. Methods
Preparation of samples
To verify the effect of selected additives (F, S, P) on the freeze-thaw resistance of mortars, six different variants of blended binders were prepared in following models:
- 70 % C + 30 % Ad: C70S30, C70F30 and C70P30
- 70 % C + 15 % Ad + 15 % Ad: C70S15F15, C70P15F15 and C70P15S15
- 100% C - reference sample
Then, mortars were prepared in accordance with STN EN 196-1 [20]. The mixtures contained 450 g ± 2 g of binder, 1350 g ± 5 g of standard sand and 225 g ± 1 g of water. After mixing by standard regime, testing beams of 40x40x160 mm were casted and cured in standard way (water curing, 28 days). Then, one series of samples was subjected to freezing-thawing testing, while the second one (comparative samples) was cured in the water under constant temperature of ± 20 °C.

Parameter evaluated

To express the frost resistance of cement composites, durability factor based on the flexural strength is usually used, as given by formula (1). Then, the samples are considered to be frost resistant if the freeze-thaw durability factor is not less than 0.85 [19].

Freeze-thaw durability factor is calculated as follows:

\[ x_f = \frac{f_{f,f}}{f_{f,0}} \]  

where,

- \( x_f \) freeze-thaw durability factor;
- \( f_{f,0} \) flexural strength of the comparative samples, in MPa (N/mm²);
- \( f_{f,f} \) flexural strength of samples after freezing-thawing cycles, in MPa (N/mm²).

In the paper, this model was also used for the evaluation of frost resistance in terms of compressive strength and relevant durability factor is marked as \( x_c \). Both of durability factors are compared in figure 1.

To find the strength characteristics for calculation of durability factors, one series of samples was subjected to 25 freezing-thawing cycles, according to Slovak standard STN 73 1322: Determination of frost resistance of concrete [19]. The cycles consisted of freezing under -20 °C (2 hours of temperature rise, 2 hours of temperature holding) and defrosting under +20 °C (0.5 an hour of temperature rise and 1.5 hours of temperature holding). One freezing-thawing cycle lasts 6 hours. After 25 freezing-thawing cycles, the flexural strength of both series of samples (frozen and comparative) was measured to calculate durability factor \( x_f \) following by testing the compressive strength using the halves of the beams broken, to calculate durability factor \( x_c \).

4. Results and discussion

The results of flexural strength of comparative samples \( (f_{f,0}) \) as well as samples after freezing-thawing cycles \( (f_{f,f}) \) are given in table 1. Table 2 shows the results of compressive strength of comparative samples \( (f_{c,0}) \) as well as samples after freezing-thawing cycles \( (f_{c,f}) \).

| Samples         | Before freezing \( f_{f,0} \) [MPa] | After freezing \( f_{f,f} \) [MPa] |
|-----------------|------------------------------------|----------------------------------|
| Reference sample| 7.0                                | 7.9                              |
| C70 S30         | 7.2                                | 7.2                              |
| C70 F30         | 5.9                                | 1.4                              |
| C70 P30         | 5.3                                | 5.0                              |
| C70 S15 F15     | 6.8                                | 2.2                              |
| C70 P15 F15     | 6.2                                | 1.9                              |
| C70 P15 S15     | 5.9                                | 5.6                              |

Table 1 shows that the flexural strength of samples after freezing-thawing cycles decreases, excepting for reference sample (the strength increased from 7 MPa to 7.9 MPa) and C70S30 sample (no any change after the freezing-thawing process).
Table 2. Compressive strength of samples before and after freezing-thawing cycles.

| Samples          | Before freezing $f_{c,0}$ [MPa] | After freezing $f_{c,f}$ [MPa] |
|------------------|-------------------------------|-------------------------------|
| Reference sample | 54.5                          | 50.5                          |
| C70 S30          | 41.5                          | 34.0                          |
| C70 F30          | 39.0                          | 25.0                          |
| C70 P30          | 31.0                          | 33.5                          |
| C70 S15 F15      | 44.5                          | 31.5                          |
| C70 P15 F15      | 38.5                          | 25.5                          |
| C70 P15 S15      | 38.0                          | 36.5                          |

Table 2 shows that the compressive strength of samples after freezing-thawing cycles decreases, excepting for C70P30 sample. The results are evaluated deeper by durability factors, presented in figure 1.

The samples are considered to be frost resistant when durability factor is bigger than 0.85. In addition to the reference sample CEM 100, this limit is achieved by mixtures C70S30, C70P30 and C70P15S15 in terms of $x_c$, indicating good performance of GGBFS and perlite. This is supported also by durability factor $x_f$, which runs in acceptable (even better) values.

On the other hand, the samples containing fly ash (C70F30, C70S15F15 and C70P15F15) do not meet limit for frost resistance. But it was also observed that the durability factor $x_c$ of these samples is more than twice as good as durability factor $x_f$.

The results are in line with [21], who states that samples containing GGBFS achieved better results than that of containing fly ash. The beneficial effect of GGBFS is also presented by [4], who associated this with the influence of slag dosage on the lower permeability.

Good performance of perlite is in line with results presented by [22]. They show that the frost resistance is improved when mixtures are prepared with higher concentration of perlite. Authors suggest this is partly attributed to the porous nature of perlite that helps reducing the disruptive expansive stresses caused by freezing-thawing process.
5. Conclusion
The results confirm both the perlite and GGBFS as satisfactory components of blended binders, either in combination with cement only (C70P30 and C70S30), or in ternary composition C70P15S15, while 30% of the dose seems to be optimal. Compared to GGBFS as a standard component of blended binders, perlite appears to be a perspective material – while keeping the limit for x, durability factor and when used in separate combination with cement (C70P30), it provides the best performance in compressive strength out of all samples. This finding may be useful in the production of blended cements in regions suffering from the unavailability of GGBFS.

6. References
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