# Laboratory Testing of Frost Resistance - Do these Tests Indicate the Real Performance of Blended Cements?

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## 1. Introduction

The technical term durability of a (concrete) structure defines the continued performance of the intended functions concerning the required strength and serviceability during the expected lifetime under conditions to which the structure is expected to be exposed [1]. Natural freeze-thaw (FT) cycles during winter time are one of those exposure conditions that attacks concrete regularly and can reduce the service life of a structure. Frost attack can lead to surface scaling or, if the worst comes to the worst, to cracks and internal damages.

The frost resistance of concrete depends on the concrete design, concrete mixture design and the used concrete constituents. To determine the frost resistance of different concrete types accelerated laboratory test methods have been developed all over the world. All of these frost tests simulate a certain number of freeze-thaw cycles. The tests vary in sample dimension, temperature maximum and minimum of a cycle as well as the time a cycle lasts and the recommended scaling limit or internal damage limit.

FT-tests were developed for ordinary Portland cement (OPC) concretes. Today, by discussing freeze-thaw tests, the trend to Blended cements has to be considered.

The objective of the research presented in this report is to explore the frost test performance of Blended cement concretes compared with associated OPC concretes. Therefore, 4 different Blended cement types were compared with 4 OPC types. Each "Blended cement - OPC couple" was produced with the same clinker in one cement plant in order to eliminate other influencing factors.

The concrete mixtures chosen within this exploration have been successfully used in construction under frost attack by constructors of different European countries. Thus it is possible to compare the lab-results with field experiences.

## 2.1 Background

Frost damages may appear as surface scaling or internal cracking. Scaling may occur on horizontal and vertical surfaces, but manly were water and snow can deposit and the surface remains wet for periods. Internal cracking is less commonly observed under field conditions. The phenomenon may be observed on structures in direct contact with water and subjected to capillary suction such as given with dams and lower parts of walls [1, 2, 3].

Ice expands by freezing by 9 vol.-%, which exceeds the facture strain of concrete. If a concrete was saturated with water it would be destroyed after the first frost cycle. But it is known that several cycles are necessary to damage a concrete. Because of that, frost damages do not suddenly appear, but they increase with freeze-thaw cycles. The main reason is that the increasing number of cycles makes the structure more permeable on the surface (i.e. porosity increases) and consequently more water is absorbed [4].

### 2.2 Process of a FT-attack

If temperature drops below 0°C and linear deformation of a mortar or concrete is measured during the freezing process, it is determined that the freezing goes along with temperature increase and sample expansion. During the process of water freezing, crystal formation heat is released, which not only results in temperature increase, but also considerably affects linear deformation [5, 6]. Warming and ice formation cause spontaneous expansion of the mortar with subsequent fast contraction. Deformation is caused by:

- the hydrodynamic pressure resulting from the water freezing process
- pressure suppression through escaping water into still empty pores
- thermal linear deformation due to temperature variation.

The damage process was described in detail by for example [4], who explained also the successively accumulating destroying frost effect as micro-ice-lens formation.

### 2.3 Test methods

In Europe commonly used FT- laboratory tests are laid down in the draft of the European standard CEN/TS 12390-9 [7]. This standard describes one reference test (slab test) and two alternative tests (cube test, CF/CDF), see table 1. For determination of internal damage a technical report is in process and will be published as CEN-report and RILEM- Recommendation [11]. The European test methods are more or less similar with ASTM methods:

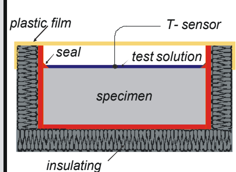
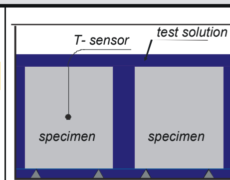
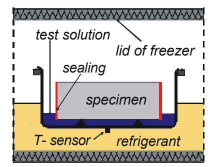
- ASTM C 666 (≈ beam-test)
- ASTM C 672 (≈ CF/CDF-test)

In table 1 schemes show the experiment set-up of the in this exploration used three different EN 12939-9 methods. While the temperature range is within all these methods between +20°C and -20°C, the cooling down and heating up velocity is different and the time one CF/CDF test cycle last is half as long as the cycles of the other two methods.

Within all these methods the accumulated scaled material is determined either as absolute mass loss per attacked area (kg/m$^2$) or percentage of mass loss (m.- %). There are only rarely scaling limits laid down in standards or directives. For example, the Swedish standard doesn't recommend a high FT resistance criterion for the slab test. Actually this standard prescribes only for the more aggressive test with deicing salt a limit [9] when scaling is the predominantly damage. The German FT criterion of the cube test is either less than 10 wt.-% after 100 cycles or less than 5 wt.-% after 50 cycles. If the scaling is lower than 5 wt.-% after 100 cycles and 3 wt.-% after 50 cycles the

concrete is rated to have a high frost resistance [8, 11]. The limit for the high frost resistance for the CF test is given as 1 kg/m$^2$ after 28 cycles in [13]. CEN/TS 12390-9 [7] prescribes 56 cycles for the CF-test. A scaling of 1 kg/m$^2$ is a surface damage of < 1 mm. Internal damage is usually measured with young's modulus loss per time or ultra sonic velocity loss.
As there are no internationally accepted scaling limits, the scaling results of this exploration are only used to compare the concretes with each other.

Table 1: Comparison of test methods of CEN/TS 12390-9: 2006 [7]

| | ***FT- test methods*** | | |
|---|---|---|---|
| | ***slab-test*** | ***cube test*** | **CF/CDF-*test*** |
| ***test parameters*** |  |  |  |
| Storage | W (6d) / L (21) / P (3d) | W (6d) / L (20d) / P (1d) | W (6d) / L (21d) / P (7d) |
| Shape of samples | 150 × 150 × 50 | 100 × 100 × 100 | 150 × 150 × 70 |
| Testing start | at least 31d | 28d | at least 35d |
| test surface | sawed, standing | moulded | moulded |
| testing direction | one-sided | all-sided | one-sided |
| $T_{min}$ / $T_{max}$ | -20°C / +20°C measured in solution | -20°C / +20°C measured in one cube | -20°C / +20°C measured under container |
| ΔT | ± 2°K | ± 2°K | ± 0,5°K |
| Cooling down velocity / Heating up velocity | 6.2°K/h / 1.8°K/h | 10°K/h / 1.5°K/h | 10°K/h / 10°K/h |
| Cycle duration/No of cyles | 24h / 56 FTC | 24h / 56 FTC | 12h / 28 FTC |
| Testing criteria | scaling/surface | % of water uptake % of mass loss | scaling/surface |
| Proposed limit | < 1.0 kg/m$^2$ after 56 d*** | < 10 m.-% / 100 FTC < 5 m.-% / 50 FTC | < 1 kg/m$^2$/ 28 FTC |

* Air: 20°C / 65% RH
** In contact with the test solution
*** with deicing salt

### 3.1 Materials of the exploration

The following 4 different Blended cement types were compared with 4 OPC types (reference specimen). Each "Blended cement – OPC couple" was produced in one cement plant, i.e. was produced with the same clinker, to eliminate other influencing factors.

Table 2: Cement couples and used plant code

| No. | Plant code | Reference cement | Blended cement |
|---|---|---|---|
| 1 | LB | CEM I 52.5 N | CEM III/A 42.5 N LA* |
| 2 | BN | CEM I 42.5 R | CEM II/A-V 42.5 R** |
| 3 | GP | CEM I 42.5 R | CEM II/B-S 42.5 N*** |
| 4 | GB | CEM I 32.5 R | CEM II/A-LL 32,5 R**** |

* composition: clinker 35–64 wt.-%, slag 36-65 wt.-%

** composition clinker 80–94 wt.-%, fly ash 6-20 wt.-%

*** composition: clinker 65–79 wt.-%, slag 21-35 wt.-%

**** composition: clinker 80–94 wt.-%, slag 36-65 wt.-%

All chosen Blended cement types have been successfully used in construction under frost attack, see Fig. 1a and 1b. As there is no CEM I 42.5 N produced by LB plant a reference specimen of cement grade 52.5 N was chosen.

Fig. 1a: 9 years old concrete road produced with CEM II/A-LL in a region hat is suffered by strong frost during winter time. The surface of the road is still in a very good condition. It shows neither cracks nor scaling or spalling.

Fig. 1b: Concrete Bridge, which was produced with CEM II/A-V concrete in Scandinavia in the year 2001. The bridge shows no scaling or cracks.

### 3.2 Methods of the exploration

The exploration was structured into 3 subprojects. In the 1st subproject all cement types were chemically and mineralogically characterized. As the porosity primarily impacts the frost resistance, porosity development of different cement type mortars were measured with Mercury Intrusion Porosimetry [12]. Therefore, mortars were stored according to the storage regime of the FT-test methods of CEN/TS 12390-9 (table 1 and Fig. 2). The porosity of hardened cement paste is influenced by:

- cement type, i.e. composition
- w/c-ratio
- finesses of cement
- hydration speed of cement components (fly ash reaction is slow compared to clinker reaction)
- curing regime
- curing sensitivity (parameter of hydration speed)
- carbonation behavior

To estimate the curing and carbonation impact on porosity of mortars depending on the used cement type a core sample, which wasn't influenced by environment, and an edge sample, which was influenced by environment condition, were determined. Because of methodical reasons the porosity research was only possible on mortars. For comparison causes, the following uniform mix design was used:

- 450 ± 2 g cement
- 1350 ± 5 g sand
- 225 ± 1 g water

The produced prisms were also used to determine the carbonation depth after 28 days and additionally after 56 days.

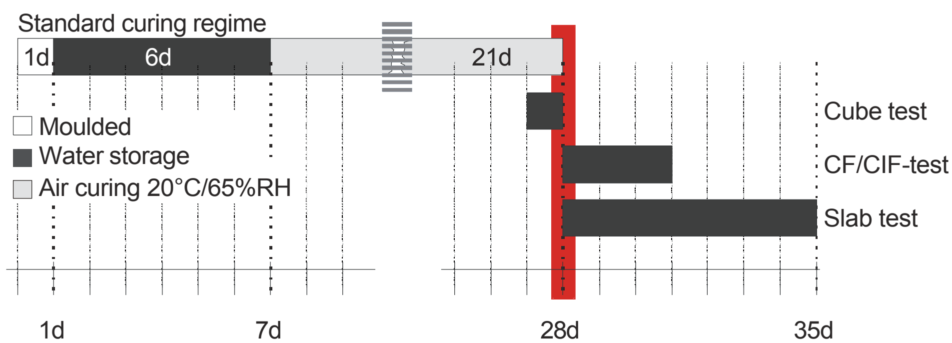



Fig. 2: Curing condition of the test methods of subproject 1.

In the 2nd subproject different concretes were prepared and explored. The concrete mix design was taken from ready mix concrete producers that use Blended cement types for their production. All used concrete mixtures have been proven a high frost resistance in the field. The chosen concrete mix design is shown in table 3. In this subproject the field proven FT-performance of the Blended cement-OPC couples was compared with all three EN standard frost tests. Furthermore, different fresh and hardened concrete properties were determined.

Table 3: Concrete mix design

| Concrete mixture | | CEM I 32.5 R GB) | CEM II/A-L 42.5 R (GB) | CEM I 52.5 N (BL) | CEM III/A 42.5 R (BL) | CEM I 42.5 R (PG) | CEM II/B-S 42.5 N (PG) | CEM I 42.5 R (NB) | CEM II/A-V 42.5 R (NB) |
|---|---|---|---|---|---|---|---|---|---|
| Plant code | | GB | | BL | | PG | | NB | |
| Cement | kg/m$^3$ | 310 | | 310 | | 380 | | 325 | |
| Water | kg/m$^3$ | 171 | | 160 | | 167 | | 179 | |
| Sand 0/2 | kg/m$^3$ | 705 | | 690 | | 606 | | 547 | |
| Gravel 2/8 | kg/m$^3$ | 186 | | 182 | | 554 | | 636 | |
| Gravel 8/16 | kg/m$^3$ | 503 | | 492 | | 644 | | 558 | |
| Gravel !6/32 | kg/m$^3$ | 469 | | 459 | | - | | - | |
| w/c | | 0.55 | | 0.50 | | 0.44 | | 0.55 | |
| Superplasticizer | kg/m$^3$ | 1.2 | | 2.5 | | 0 | | 0.3 | |
| Air entraining agents | kg/m$^3$ | - | | - | | - | | 1.0 | |

In the 3$^{rd}$ step, which was still ongoing by the time this report was written, the technical expertise were used to adjust the curing procedure to consider the different hydration behavior of Blended cement and OPC types by the FT-tests.

**4.1 Results of subproject 1**

The results of the porosity determinations of the mortars are shown in Fig. 3. The core porosities proves that the Blended cement types with alternative hydraulic-active constituents as slag and fly ash develop almost similar low porosities compared with the associated OPC types. This is mainly because of the better curing conditions in the middle of a cement bound product; i.e. with optimal curing conditions the hydration degree determined indirectly by the porosity is absolutely similar. Furthermore, there is no carbonation influence in the middle of the specimens. Only the sample CEM II/A-LL has a higher porosity (4 Vol.-%) in the core.

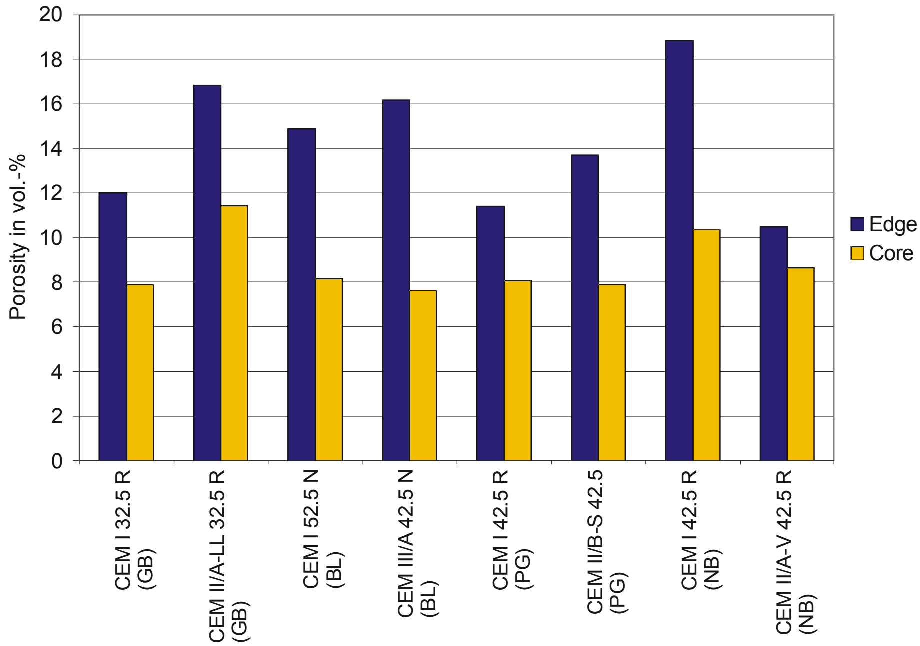



Fig. 3: Total porosity of OPC and Blended cement mortars after standard curing determined with Mercury Intrusion [12]

Furthermore, the results shown in Fig. 3 prove that the edge porosity of all mortar samples is higher than the core porosity. These results demonstrate the influence of the environment on the cement paste matrix surface. The long period in the standard climate (20°C, 65 RH) influences the density of both cement types sustainable because of drying as well as carbonation. The results prove that most of the Blended cement surfaces are more influenced by the ambient environment or curing conditions than the OPC samples; as the porosity of the edge-specimens of the Blended cement types are almost always higher. The results point out that the agreed conventions on curing and test start of CEN/TS 12390-9 at 28 days causes a lower degree of hydration and thus higher porosity of Blended cement sample surfaces.

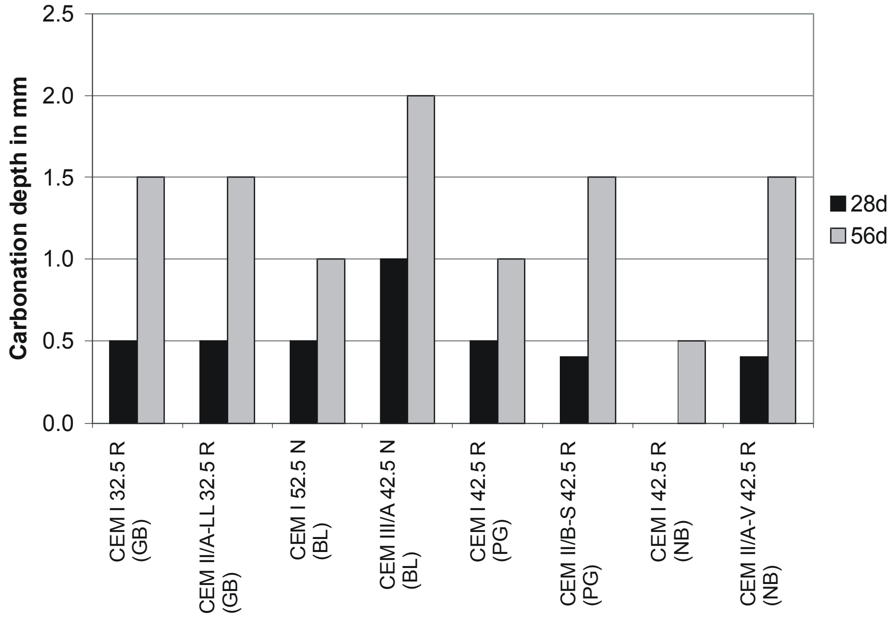


Fig. 4: Carbonation depth of the samples stored in the climate chamber.

Fig. 4 shows the development of the carbonation depth of the different OPC and Blended cement mortars stored in the climate chamber, measured after 28 and 56 days. While there is no difference between the carbonation depths of the OPC-Blended cement couples of GB plant, the Blended cement types of all other couples have a higher carbonation depth than the corresponding OP cements. Especially, the 56d-carbonation depth of the CEM III (BL) and CEM II (NB) were extremely high compared with the carbonation depth of the associated OPC sample. This is probably because carbonation of slag containing samples increases the porosity, which is contrary to OPC-sample behaviour.
As the carbonation of CEM III as well as CEM II cement produced with slag or fly ash leads to an increase of the pore volume the resistance against freeze-thaw attack is reduced by carbonation [2, 3].

### 4.2 Results of subproject 2

The comparison of the compressive strength shown in Fig. 5 demonstrates that there is no difference between the Blended cement and OPC concretes after 28 days. The “early strength” after 7 days shows slight differences, especially, with the slag containing CEM III/B 42.5 R (PG).

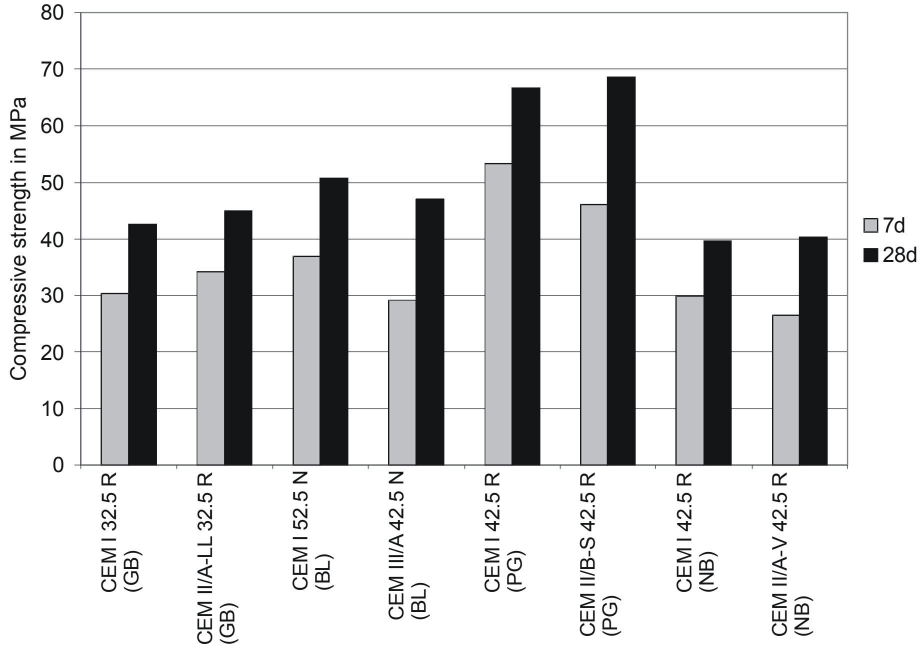



Fig. 5: Develop of compressive strength depending on time.

The FT-test results are illustrated in Fig. 6 to 8. In general all concretes showed a low scaling and, thus, have a high FT-resistance.

The results of the Cube test are summarized in Fig. 6. By comparing the single couples it could be seen that all OPC-concrete samples performed slightly better than the corresponding Blended cement concrete samples. The CEM II/A-V 42.5 R concrete, which was produced with air-entraining agent, neither performed as good as the OPC concretes without air-entraining agents nor showed lower scaling compared with the other Blended cement concretes. In which extend incomplete reaction of fly ash is responsible for this phenomenon will be investigated in further tests.

The results of the Slab test are shown in Fig. 7. Again, low level of scaling was determined with Blended cement concretes as well as OPC concretes. Compared with the corresponding Blended cement concrete samples all OPC concrete samples showed a slightly lower scaling. It is absolutely notable that neither the OPC concretes nor the Blended cement concretes exceeded the recommended limit prescribed in the Swedish standard [9] for FT tests with deicing salt. Furthermore it is astonishing that the scaling of the Blended cement and OPC concretes of NB plant showed a relatively high scaling, although, both concretes were produced with air entraining agents.

The CF-test results are shown in Fig. 8 (recommended scaling limit: 1000 g/m$^2$ at 28 days [13]).
Again, by comparing the OPC - Blended cement couples all concretes produced with Blended cement performed below the corresponding OPC reference. Especially the CEM II/A-LL of GB plant has a rather high scaling compared with the other concretes although this cement is

commonly and successfully used for concrete units and roads under frost load with and without deicing salts, see Fig. 1a.

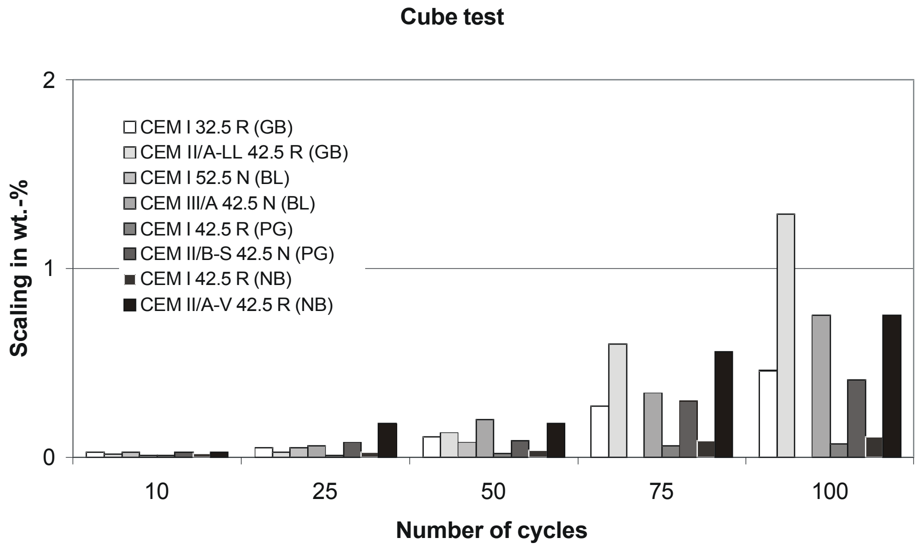



Fig. 6: Results of the cube tests.

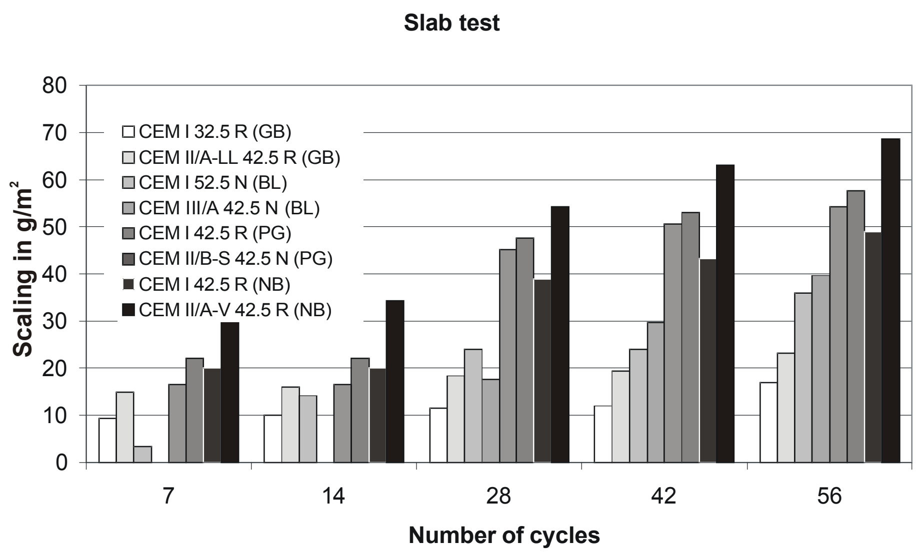



Fig. 7: Results of the slab test.

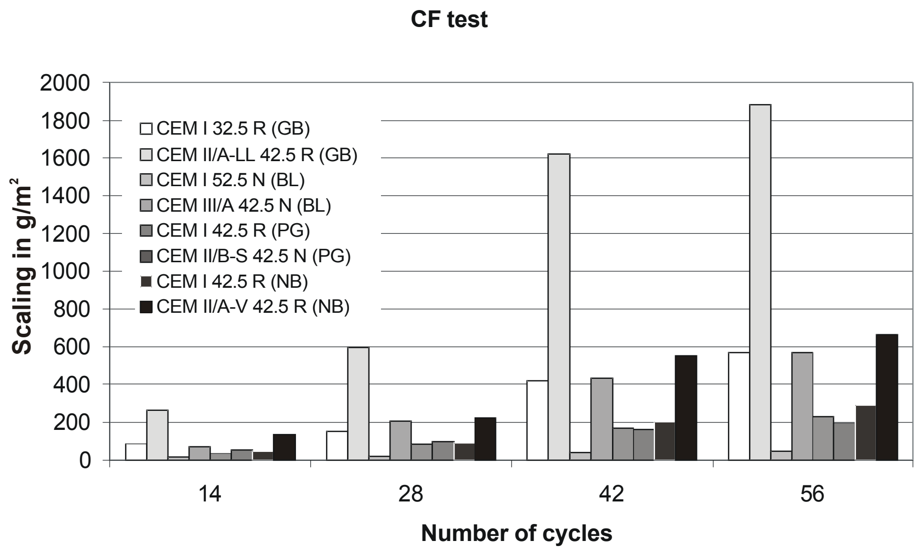



Fig. 8: Results of the CF test.

## 5.1 Summary

Comparing the different test methods, the ranking of the performance of the different concrete types varies. In general all concrete mixtures showed very low scaling in the tests, which confirms the well known positive practice experiences of all tested concretes in the field under frost attack.

But all FT-tests are reflecting a higher scaling of Blended cement concrete than the associated OPC concrete, if the concrete composition and the curing regime are similar.

The conclusion has to be drawn that the surface areas of the Blended cement samples are more negatively influenced by the curing regime of the test methods than OPC concrete surfaces. This thesis is supported by the porosity results.

Due to that fact, the laboratory tests do not reflect the correct basis for evaluating the frost resistance of concretes containing blended cements.

To produce more realistic results similar conditions in the concrete surface area at the beginning of the FT-tests are necessary. This could be approached by adjusting the curing conditions, which leads to a similar degree of hydration or pore structure of the hardened cement paste for the different cement types. The investigation of these questions should be the issue of the last part of the research program.

## 5.2 Outlook

As the results of all 3 different freeze-thaw tests proved that the Blended cement concretes showed higher scaling than the corresponding OPC concretes (which isn’t confirmed by field experiences) it is sufficient to concentrate on one test method, the CF-test. To achieve similar starting conditions for Blended cement concretes we adjusted the curing regime as follows:

- 1d mould, 6d under water, 21d covered with foil stored at 20°C
- 1d mould, 6d under water, 49 covered with foil stored in 20°C

- 1d mould, 6d under water, 77d stored at 20°C/65% R.H.
- 1d mould, 6d under water, 21d covered with foil, 28d stored at 20°C/65% R.H.
- 1d mould, 6d under water, 21d at 20°C/65% R.H.

To prove how the adjusted curing procedure impacts the matrix density, the porosity is again determined by Mercury Intrusion [12].

## 6. Literature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