TESTING THE FROST RESISTANCE OF CONCRETE WITH DIFFERENT CEMENT TYPES – EXPERIENCE FROM LABORATORY AND PRACTICE

Peter BOOS [a], Zbigniew GIERGICZNY [b]

[a] Dr.; Heidelberg Technology Center, Germany
E-mail address: *peter.booss@heidelbergtechnologycenter.com*

[b] Associate Prof.; Faculty of Civil Engineering, The Silesian University of Technology, Akademicka 5, 44-100 Gliwice, Poland; Górażdże Cement S.A.
E-mail address: *zbigniew.giergiczny@polsl.pl*

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Abstract
The paper presents the frost resistance test results of concretes, in accordance with CEN/TS 12390-9, produced on selected CEM I, CEM II and CEM III cements from different European countries. Concrete composition was designed in line with the concrete production specifications of individual country. It was proved that CEM II and CEM III concretes demonstrate more scaling comparing to CEM I concrete, while analyzing frost resistance in standard time. The hydration degree of CEM II and CEM III cements is increased by modification of storage conditions (better humidity access and limited carbonation process) thus the resistance of concretes to low external temperatures attack is also improved.

Streszczenie
W pracy przedstawiono wyniki badań mrozoodporności betonów zgodnie z CEN/TS 12390-9 na wybranych cementach CEM I, CEM II i CEM III pochodzących w różnych krajów europejskich. Skład betonu przyjęto zgodnie z zasadami wykonywania betonów mrozoodpornych w poszczególnych krajach. Stwierdzono, że badając mrozoodporność betonu po normowym okresie dojrzewania, beton na cementach CEM II i CEM III daje większą ilość złuszczeń w porównaniu do cementu portlandzkiego CEM I. Modyfikując warunki dojrzewania (lepszy dostęp wilgoci i ograniczenie karbonatyzacji) zwiększamy stopień hydratacji cementów CEM II i CEM III, a tym samym polepszamy odporność betonów na tych cementach na działanie niskich temperatur zewnętrznych.

Keywords: **Cement; Type of cement; Mineral additives; Prestorage; Concrete; Frost resistance.**

1. INTRODUCTION

Civil constructions are characterized to be "durable" if they show the required useful properties – under planned stress, over planned service life, at low maintenance costs [1]. A natural kind of stress is for example frost weathering. Temperature variations above and below the freezing point can lead to superficial weathering or internal destruction of the structure [2, 3] in concrete constructions as well as in natural rock. This process is a progressive phenomenon, i.e. weathering increases with the number of frost cycles. In connection with de-icing salt, the frost attack is considerably stronger. Therefore on long term, the frost attack can impair the usability or even the durability of civil constructions. In Europe, the frost resistance of concrete is defined by descriptive regulations. National concrete-technical measures are defined for different frost exposition classes (requirements for concrete composition and performance) [4, 5].

In addition, different laboratory frost test methods were developed for evaluation of the concrete frost resistance. With all methods the long natural frost weathering process can be accelerated by fast repetition of freeze-thaw-cycles. The test methods differ in sample form and sample size, degree of saturation of the test samples, temperature profile of the cycles, maximum and minimum temperature as well as duration and number of the cycles.

As the laboratory test methods have been mainly developed for Portland cement (CEM I) concrete, the question arose, if the methods realistically reflect both the frost resistance of CEM II and CEM III concrete. CEM II and CEM III concretes are used already in all fields of concrete construction. In the scope of a research project it was investigated, how CEM II and CEM III concretes, which were tested and have been approved in different countries, perform during test methods according to CEN/TS 12390-9 [6], compared to the corresponding Portland cement (CEM I) concretes.

2. FROST ATTACK – DESTRUCTING EFFECTS AND INFLUENCING VARIABLES

The concrete damaging effect of freeze-thaw-cycles is often explained by 9% volume increase during the phase of water transformation into ice and thus arising pressure. Considering a number of scientific investigations and the resulting models [7-14], it becomes clear that the reasons for the freeze-thaw damage are by far more complex and probably different mechanisms overlap. The "macroscopic" behavior of water can obviously not simply be transferred to the behavior of water in a nano- or microscopic pore room of concrete. The behavior of water is influenced by physico-chemical parameters;

– freezing point lowering by solved materials, for example salt,
– freezing point lowering by surface forces,
– super cooling effects,
– vapor pressure differences.

The microscopic and macroscopic destructing effects were concluded from the different parameters [15-18];

– theory of hydraulic pressure / pressure of ice,
– theory of osmotic pressure,
– diffusion theory, capillary effect,
– ice lens model,
– unequal temperature coefficients of expansion of concrete, ice and aggregates.

Important and damaging influencing parameters are summarized in Table 1.

3. DETERMINATION OF THE FROST RESISTANCE; CEN/TS 12390-9 [6]

For evaluation of the frost resistance of concrete in Europe, three test methods are described in the technical specification CEN/TS 12390-9 [6]. One reference method is the slab test.

Alternative methods are the cube test and the CF test (CF; **C**apillary Suction **F**rost – Test).

Table 2 schematically compares the most important parameters of the three test methods [19]. The production and prestorage conditions of the three methods are largely comparable (Table 2).

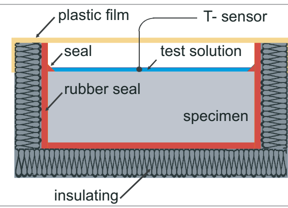
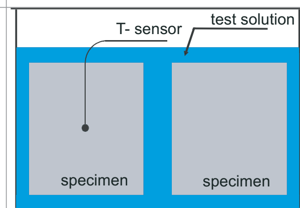
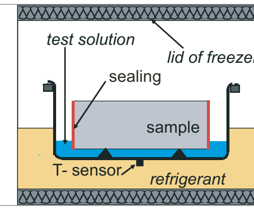
An internal destruction of the structure, which is described in the technical report CEN/TR 15177 "Testing the freeze-thaw resistance of concrete – Internal structural damage" [18], is not the subject of this study. The following short descriptions are only referring to the water-frost resistance investigated in this study.

In the Scandinavian slab test, the test pieces (150 x 150 x 50 mm^3), which are made by sawing concrete test cubes (edge length; 150 mm), are stressed by freeze-thaw cycle under a 3 mm layer of deionized water. The test surface is the sawed surface. All other surfaces of the test piece are sealed; see schematic drawing in table 2. The frost resistance in this slab test is evaluated by determination of the mass of

Table 1.
Effects influencing the frost resistance

Concrete composition	Technological influencing variables	Influencing parameter
Water / Cement ratio Porosity of hardened cement paste Admixtures Aggregates Cement	Curing Compaction Transport Protection measures	Moisture available Temperature conditions Deicing agents Carbonation

Table 2.
Overview of test methods according to specification CEN/TS 12390-9 [5, 17]

	Slab test	Cube method	CF – Test (CDF – Test)
Test parameters			
Prestorage	W (6d), L (21d), P (1d)	W (6d), L (20d), P (1d)	W (6d), L (21d), P (d)
Test piece in mm^3	150 × 150 × 50	100 × 100 × 100	150 × 150 × 70
Test age	minimum 31d	28d	minimum 35d
Test surface	sawed, centre of the cube	formed	formed
Test direction	one-way	all directions	one-way
T_{min}/T_{max}	20°C / +20°C in the test medium	-20°C / +20°C in the centre of the cube	-20°C / +20°C below test vessel
T	2° K	2° K	0.5° K
Cooling-/ Thawing speed	6.2° K/h / 1.8° K/h	6.2° K/h / 1.5° K/h	6.2° K/h / 10° K/h
Duration / Number of FT-cycle acc. to [8]	24h / 56 FTW*	24h / 56 FTW	24h / 56 FTW
Test criterion A	Surface destruction	Surface destruction	Surface destruction
Test criterion B		Internal destruction	Internal destruction
Suggested limit value; criterion A			
1. Frost test	-	< 10 M.-% / 100 FTW** < 5 M.-% / 100 FTW***	< 1.0 kg/m^2 / 28 FTW [20]
2. Frost de-icing salt	< 1.0 kg/m^2 after 56 FTW	< 5.0 M.-% / 56 FTW	< 1.5 kg/m^2 / 28 FTW (CDF)

* FTW; Freeze-thaw cycles
**XF1; Criterion acc. to [11, 17]
***XF3; Criterion acc. to [11, 17]
W; under water
L; in standard climate of 20°C/65% R. H.
P; in contact with the test liquid

material (in kg/m²), which is scaled (weathered) from the concrete slab after 56 freeze-thaw cycles (i.e. 56 days) [6].

In the cube test, the test cubes (edge length 100 mm), which are completely immersed in deionized water, are stressed by repeated freeze-thaw cycles. The freeze-thaw resistance is evaluated by determination of the percentage mass loss of the test cubes after 56 freeze-thaw cycles [6].

The test pieces for the CF test method are produced in form of a cube (edge length; 150 mm), which is bisected by a centrically arranged PTFE slab. After defined prestorage, the PTFE formed slab side is stressed in deionized water by repeated freeze-thaw cycle. The frost resistance is evaluated after determination of the material (in kg/m²) scaled from the test piece after 56 freeze-thaw cycles (28 days) [6].

While the temperature range (T_{max}; 20°C; T_{min}; -20°C) of all methods is identical, the methods differ for example regarding the permitted temperature variances, cycle length and temperature sensor arrangement.

The methods can be used in order to compare new starting materials or new concrete compositions with already known ones [6]. In addition, test results with limit values based on national experience can be compared and evaluated.

Regarding these national regulations, the number of cycles, the limit value is referring to, partly differs from the requirements in the technical specification CEN/TS 12390-9 [6] (Table 2). In the specification12390-9 [6] itself, there are no recommendations

Table 3.
Selected CEM II/III – CEM I pairs

Region/Plant	CEM II/III	Portland cement (CEM I)
A	CEM III/A 42.5 N	CEM I 52.5 N
B	CEM II/A-V 42.5 R	CEM I 42.5 R
C	CEM II/B-S 42.5 N	CEM I 42.5 R
D	CEM II/A-LL 32.5 R	CEM I 32.5 R

Table 4.
Concrete compositions

Concrete composition		CEM I 52.5 N	CEM III/A 42.5 N	CEM I 42.5 R	CEM II/A-V 42.5 R	CEM I 42.5 R	CEM II/B-S 42.5 N	CEM I 32.5 R	CEM II/A-LL 32.5 R
Region/Plant		A	A	B	B	C	C	D	D
Cement	kg/m^3	310		325		380		310	
Water	kg/m^3	160		179		167		171	
Sand 0/2	kg/m^3	690		547		606		705	
Gravel 2/8	kg/m^3	182		636		554		186	
Gravel 8/16	kg/m^3	492		558		644		503	
Gravel 16/32	kg/m^3	459		-		-		469	
w/c		0.52		0.55		0.44		0.55	
Plasticizer	%	0.44	0.50	0.30	0.30	1.00	1.00	1.20	1.20
AE agent	%	-	-	1.00	1.30	-	-	-	-
Consistency		F4	F4	F4	F4	F3	F3	F3	F4
Frost Exposition class [18]		XF1		XF3		XF3		XF1	

for limit values.

In Germany, the limit values for high frost resistance are for example defined in the technical bulletin of the Federal Waterways Engineering and Research Institute (BAW) [22]. In addition to the acceptance criterion for internal concrete destruction (acceptance of the relative dynamic young's modulus), the technical bulletin sets, as an additional acceptance criterion for a high water-frost resistance of concrete, a weathering degree of ≤ 1 kg/m^2 after 28 freeze-thaw cycles (14 days) in the CF method. Further suggested limit values are given in table 2.

4. MATERIALS AND TEST PROCEDURES

4.1. Materials

In the scope of the research project the frost resistance of four CEM I concretes was compared to the frost resistance of four CEM II/III concretes (Table 3). Each one Portland cement (CEM I) and one CEM II or III cement of four different European cement plants were used. By using the same clinker for production of the respective cement pairs, the influence parameter "clinker" could be eliminated during the comparable investigations afterwards.

Without exception, the four cement plants, which provided the respective cement pairs, are located in regions, which are characterized by heavy winter frost.

As plant A does not produce Portland cement of strength class 42.5, CEM I 52.5 N was chosen as reference Portland cement.

The composition of tested concrete mixtures is shown in Table 4.

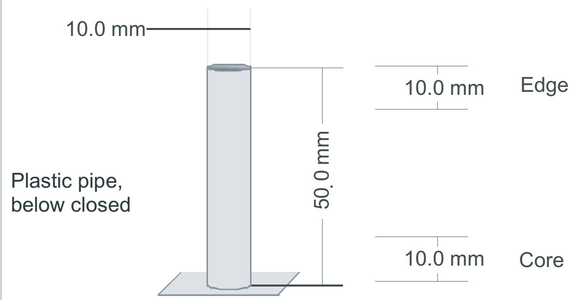


Figure 1.
Type of curing and test sample for determination of porosity (plastic tubes ø = 10.0 mm)

4.2. Test procedures

The test was subdivided into three subprojects.

In the first part of the project the porosity of mortars for all cements types was described. As porosity has a considerable influence on the frost resistance of the hardened cement paste, the porosity of the different cement types was investigated in detail on 28 days old mortar by means of the mercury penetration porosimetry.

The produced mortar had the following composition;

– 450 ± 2 g cement
– 1350 ± 5 g sand (1 mm)
– 250 ± 1 g water
– w/c = 0.55

The fresh mortar samples were filled in one-sided closed plastic tubes (Ø = 1 cm; Fig. 1).

According to the storage conditions of CEN/TS 12390-9 [6] the samples were stored for 24 hours in the sheltered formwork, 6 days under water and afterwards for 20 days in the standard climate (20°C, 65% relative humidity). On the 28th day, the samples were gently dried at a temperature of 40°C. Afterwards, the porosity of one core and one edge sample was determined (Fig 1). The test simulates the hydration conditions in the centre of the concrete sample cube and in the area close to the surface. While the porosity development under optimum prestorage conditions is determined by considering the core sample, consideration of the surface sample can help to understand the influence of the ambient conditions during prestorage on the results.

In the second part of the project the water frost resistance of the CEM II/III – CEM I pairs was investigated with all test methods according to the CEN/TS 12390-9 [6].

Regionally experienced concrete technologists provided concrete recipes for the cements, which are customary in practice (Table 4). Precondition for the recipe suggestion was that the concretes have proven to be frost resistant in practice on the local market already for a long time.

Mixture B is a recipe containing air-entraining agents corresponding to the local requirements. According to the German rules of application (DIN 1045-2 [4]), mixtures A and D fulfil the requirements of exposition class XF1 and mixtures B and C fulfil the requirements of the exposition class XF3. Although it was clear that the frost tests were actually only planned for exposition class XF3, the recipe suggestions of the countries were exactly adopted, as the main focus of the study was the comparison of the cement types.

The decision to use country-specific recipes limits the direct comparison of the concrete pairs among each other.

In the third part of the project the findings of the two previous subprojects were used in order to modify the prestorage conditions in such way that the CEM II/III cements showed comparable hydration degrees (or porosity of hardened cement paste) as the respectively corresponding Portland cements at the beginning of the frost resistance test.

The storage time was increased in two steps. As carbonation can have a negative effect on the surfaces of CEM II/III concretes and as the carbonation speed at standard climate (20°C, 65% relative humidity) is extremely high, the samples were additionally stored under film until beginning of the test. The following modified storage types were chosen;

– 1day in the form, 6 days under water, 21 days under film at 20°C (LA),
– 1day in the form, 6 days under water, 49 days under film at 20°C (LB),
– 1day in the form, 6 days under water, 77 days under film at 20°C (LC).

In the third part of the project, the frost resistance was only determined by the CF test, which proved to be the severest test. For these tests, the same concrete recipes as for the second subproject were used again.

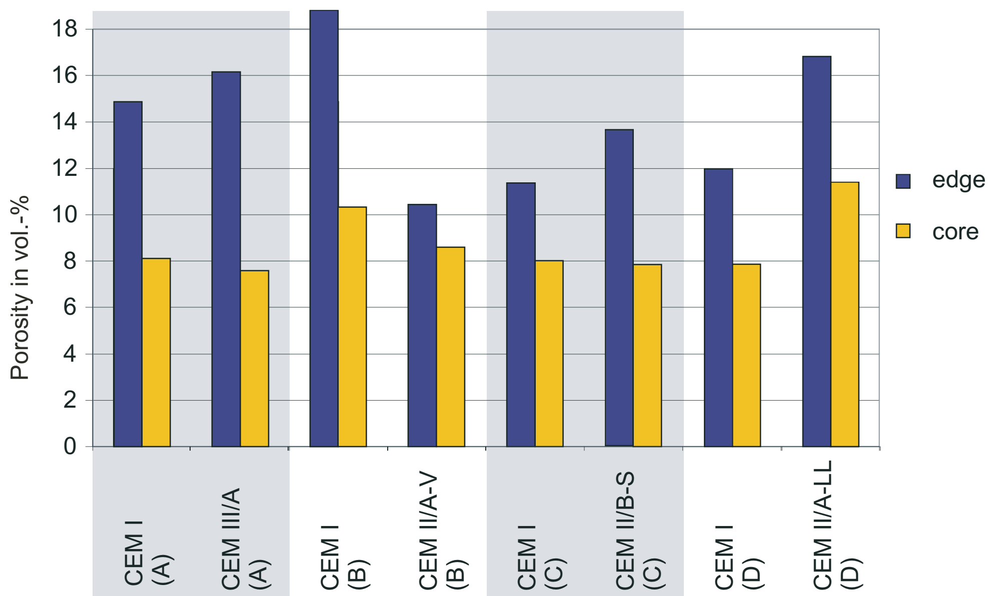


Figure 2.
Mortar porosities of the surface and core areas. The samples were stored 1 day in the form, 6 days under water and 21 days at standard climate (20°C/65% rel. moisture)

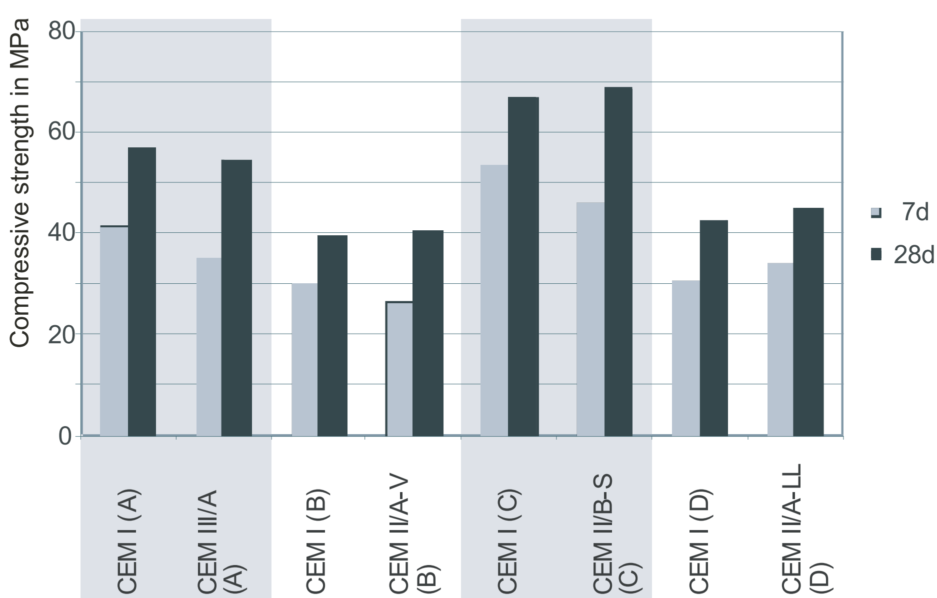


Figure 3.
Strength development of the chosen test concretes

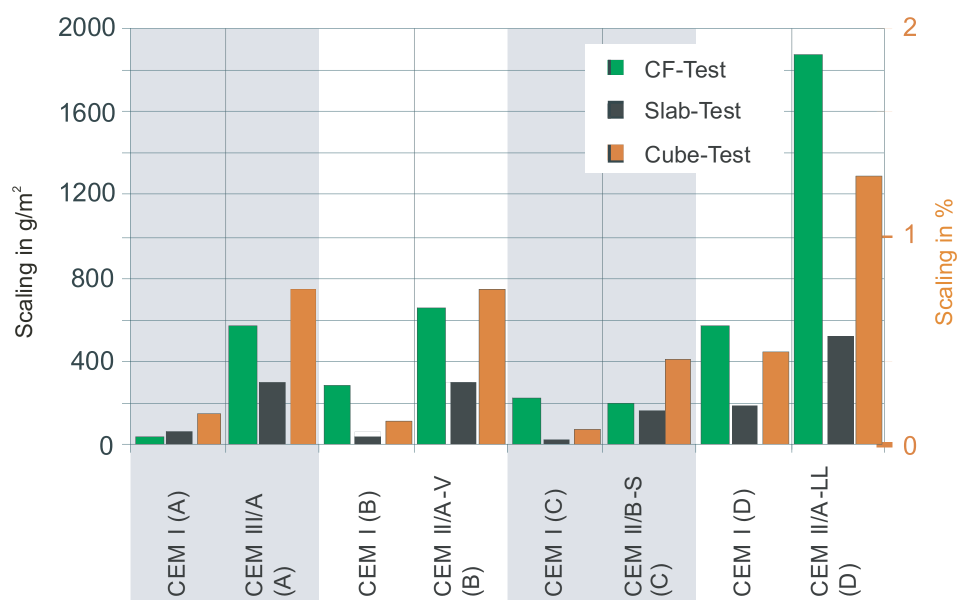


Figure 4.
Results of the CEN/TS 12390-9 frost tests. Weathering of the CF Test and slab test is given in g/m^2; weathering values of the cube method are given in mass-%

5. TEST RESULTS AND DISCUSSION

The results of the porosity investigations of the first project part are shown in Fig. 2. The sample age and the prestorage conditions met the requirements of CEN/TS 12390-9 [6].

By making an average comparison, considering all 28 days old mortar samples, it is noticeable that the medium CEM II/III core porosities ($Ø_{\text{CEM II/III}}$; 8.9 Vol.-%) are not different from the medium CEM I core porosities ($Ø_{\text{CEM I}}$; 8.6 Vol.-%). As expected, the porosities of the surfaces of all cements are considerably higher than of the core samples (Fig 2).

In the differentiated view, it becomes clear that the 28 days porosities of the core samples of the individual cement pairs can be compared. Apparently, the same strength cement categories (independent from the cement type) lead to comparable structures under optimum conditions. Only the CEM II/A-LL mortar shows higher core porosity (+3.5%) than the corresponding CEM I sample. This higher porosity is probably caused by the inherent porosity of the limestone.

Compared with this, the porosities of CEM II/III mortar on the surface are normally approximately 2-4 Vol. % higher than those of the corresponding CEM I mortar porosities. Only the CEM II/A-V-(B) sample shows a lower surface porosity than the CEM I sample. When summarizing all samples, CEM I and CEM II/III samples show an identical medium value of surface porosity (each 14.3 Vol.-%).

The surface porosities of the mortars are directly influenced by the prestorage conditions and time. Storage at standard climate, which leads to an increased carbonation and dehydration, has especially strongly influence the resulting surface porosity of most of the CEM II/III samples, compared to corresponding CEM I samples [9]. From experience, the hydration of blast furnace slag or fly ash containing cements after 28 days is not at all finished, so that an additional considerable reduction of porosity can be expected at later age of the samples. This has a positive effect on the durability parameters. That means the efficiency of those cements still increases with later age.

Fig. 3 shows a comparison of the 7 and 28 days compressive strength of the concrete pairs.

The determined 28 days compressive strength of the CEM II/III concretes is absolutely comparable with the strength of CEM I samples. Except for cement pair D, all CEM II/III concretes show a lower early strength compared to the respective Portland cement CEM I concretes. This is caused by the slower hydration speed.

Similarly cements from plant A, which differ in their strength class (Table 1) show similar 28 days compressive strength in concrete. Thus it is clear that the concrete pairs are comparable with each other in the frost tests.

The results prove that the influence of the prestorage conditions determined by the mercury penetration porosimetry can only be a superficial influence on the porosity. Otherwise, the 28 days strength of the CEM II/III concretes would have to be considerably lower than the strength of the CEM I concretes. Due to the proceeding hydration of blast furnace slag and fly ash containing cements after 28 days, the CEM II/III concretes will exceed the strength of the CEM I concretes on longer terms.

In the second part of the project the frost resistance of CEM II/III – CEM I concrete pairs was investigated with all test methods according to CEN/TS 12930-9 [6].

The results are shown in Fig. 4. As there is no definition for European uniform limit values for the frost test, the scaling is evaluated according to national limits suggested by Germany or Scandinavia.

Slab test; The chosen suggested limit value in the slab test comes from the Swedish standard [24] and amounts to 1000 g/m^2. This value is the actually suggested limit for the frost-de-icing salt resistance. However, as there is no other limit value defined the test results refer to this value.

The weathering values of all investigated concrete samples were significantly below the Swedish limit value. As during this method ground samples were tested, the determined absolute weathering values were – as expected – considerably lower than in both other used methods. The absolute weathering values of the CEM II/III concretes are indeed low, however slightly higher than the values of the corresponding Portland cement concretes (Fig. 4).

Cube method; The recommended limit value for the cube method is 5 mass-% after 100 cycles for exposition class XF3 or 10 mass-% after 100 cycles for exposition class XF1 [12, 19]. The weathering values shown in the picture are related to the right y-axis.

Also during this method, the measured weathering values were considerably below the suggested limit value for exposition class XF3. Thus, the absolute weathering values were in a range of 0.1-1.3%. When comparing the CEM II/III concrete results with those of the CEM I concretes, it appeared that the weathering rates were lower.

CF-Test; The recommended limit value at the beginning of the project was 2000 g/m^2 after 56 cycles [25]. In the end of 2004, the recommended limit value was changed to 1000 g/m^2 after 28 cycles (14 days) [22]. The shown weathering values refer to the old limit, i.e. 2000 g/m^2 after 56 cycles.

During this project, all CEM II/III and CEM I concretes showed weathering values below this suggested old limit value. As well, the new suggested limit value is not exceeded by any of these samples. Therefore it can be said that all tested concretes are frost resistant.

Comparing the absolute weathering values of CEM II/III – CEM I pairs among each other it becomes clear that all CEM II/III concretes showed higher weathering than the corresponding CEM I concretes (Fig. 4). CEM II/B-S concrete (sample B) is an exception; its absolute weathering value is in the same range as of the corresponding CEM I concrete.

As a conclusion it can be said that all investigated CEM II/III – CEM I concretes showed only low weathering after all three test methods and thus can be defined to be frost resistant. Even the concretes fulfil suggested strict XF3 limit values, which only fulfill recipe criteria of exposition class XF1 according to the German codes of practice [4]. Concretes of exposition class XF1 are normally not evaluated according to the CEN/TS 12390-9 [6] method.

It is noticeable that the measured weathering of CEM II/III concretes is normally higher than of the respectively corresponding CEM I concretes. As during this project specifically such concrete recipes were used, which according to the responsible plant technical adviser of the plants have been used without exception for years in practice, and which show the same behavior regarding frost resistance, the comparison of the absolute scaling quantities obviously leads to a wrong conclusion.

The porosity results of the first part of the project provide clear information that the discrepancy between practical experience and the absolute test results is connected with the hardened cement paste porosity of the surface.

In the third part of the project it was investigated, if the measured weathering of the CEM II/III concretes is more comparable with the weathering of the corresponding CEM I concretes, if the initial situation (hydration degree or matrix porosity) is identical. The porosity results show that an optimum prestorage, as it is given for example for the sample cores, also leads to comparable porosities of the investigated cements. In addition, it is known that for example slowly hardening concretes only at later age show performance comparable to CEM I concretes. For this reason, modified prestorage conditions were

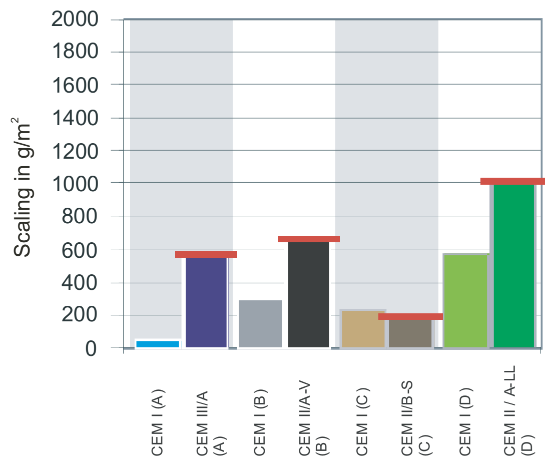


Figure 5.
CF test results after the standard type of storage; 1d in the form, 6 d under water, 21 d 20°C/65% R.H.; the red bar marks the level of standard storage

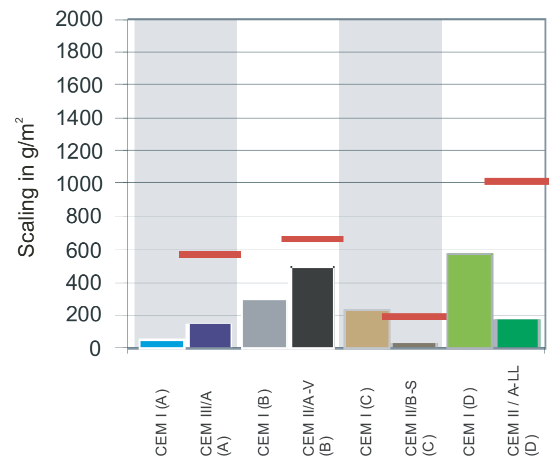


Figure 6.
CF test results after the modified type of storage LA; 1d in the form, 6 d under water, 21 d under film; the red bar marks the level of standard storage

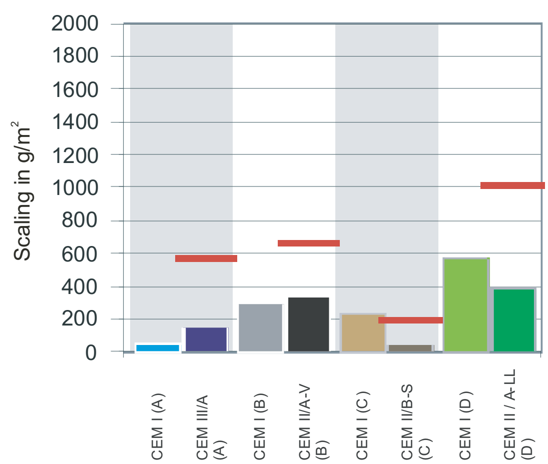


Figure 7.
CF test results after the modified type of storage LB; 1d in the form, 6 d under water, 49 d under film; the red bar marks the level of standard storage

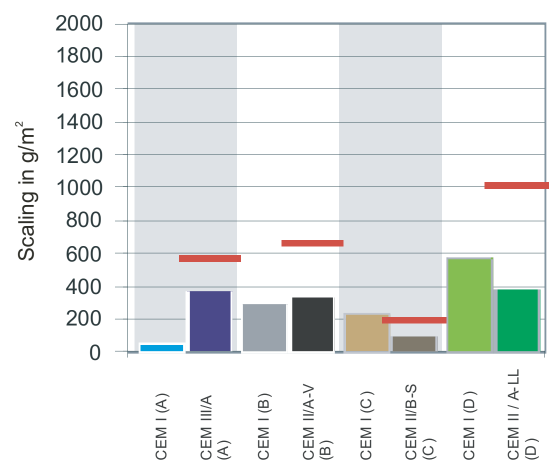


Figure 8.
CF test results after the modified type of storage LC; 1d in the form, 6 d under water, 77 d under film; the red bar marks the level of standard storage

chosen for the last part of the project;

- LA; 1day form, 6 days under water, 21 days under film at 20°C,
- LB; 1day form, 6 days under water, 49 days under film at 20°C,
- LC; 1day form, 6 days under water, 77 days under film at 20°C.

As in the second part of the project the CF test was the severest method, the frost resistance tests of the last part of the project were only carried out with the CF test. The tests with CEM I concretes were not repeated.

All results of the investigation are shown in Fig. from 5 to 10. The weathering of all samples are, as already determined during the first test series, far below the used recommended limit value (< 2000 g/m^2 after 56 FTW according to [25]).

At all modified storage types, (LA to LC) superficial weathering of CEM II/III concretes considerably decreases. The weathering determined for concretes C and D is now below the weathering of the respective Portland cement concretes. The weathering of concretes A and B is in the range of the corresponding CEM I concretes. Accordingly, the modified storage types lead to harder and more resistant structures, as expected. It is interesting that already storage type LA, where the storage time was not changed but where the samples were stored under film until the test date, leads to a considerably lower weathering during the test. Compared to storage type LA, the extended storage times (LB, LC) only lead to reduction of the degree of weathering regarding the CEM II/A-V sample (B). The result can be explained with the connection between the degree of weathering in the CF test and the variation coefficient [26]. The lower the absolute degree of weathering, the higher is the spread during the frost tests.

At this point, it has to be especially pointed out again that it does not make sense to compare measured absolute weathering degrees of different concrete samples with each other.

The test precision of the methods for such a comparison becomes too imprecise with decreasing degree of weathering [26]. The study illustrates that small changes of the prestorage conditions already lead to considerably modified degrees of weathering. Thus, the requirements of CEN/TS 12390-9 [6] are to be strictly fulfilled and the results should be evaluated with approved suggested limit values.

6. CONCLUSIONS

In the scope of a comprehensive study it was investigated how accurately the methods of CEN/TS 12390-9 [6] reflect the frost resistance of CEM I, CEM II and CEM III concretes. For this purpose, each one CEM I and one CEM II or CEM III cement from four European cement plants were used. Regionally usual concrete recipes were chosen for these cements. The regions and countries have been producing concrete according to these recipes for years and the concretes proved to be frost resistant in practice.

All concrete test series of the study did not exceed the different proposed limits and may therefore be considered to be frost resistant.

After standard storage, the degree of superficial scaling of CEM II or CEM III concretes was higher compared to CEM I concretes. The reason for this slightly increased superficial scaling could be related to the slightly increased surface porosities of CEM II or CEM III samples at the time the freeze-thaw test started.

By modification of the prestorage conditions before the frost test, CEM II/III concretes were given the possibility to develop a comparably dense and comparably hard surface structure as of the CEM I concretes. The worse carbonation conditions of standard climate storage were prevented by storage under film. Due to these modifications, the degrees of weathering were within the range of CEM I concretes, partly even below.

This is confirmed in practice. For example, the technical bulletin “Frostprüfung von Beton” of the Federal Waterways Engineering and Research Institute (BAW) [22] recommends prestorage of 14 days (instead of 7 days) under water for slowly hardening concretes and to carry out the test at a later date, for example after 56 or 90 days in prestorage conditions. It also proved well in Polish environment on the civil engineering objects [27].

The evaluation of the frost resistance of concretes of different composition is to be made according to approved limit value criteria. Gradual gradation of the determined degrees of weathering does not make sense because of the precision of the tests and different concrete characteristics.

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