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RESEARCH REPORT 3

CLASSIFICATION OF CONCRETES IN FREEZE/THAW
RESISTANCE CLASSES

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CLASSIFICATION OF CONCRETES IN FREEZE/THAW RESISTANCE CLASSES

Introduction

Currently, in national regulations, ensuring sustainability is done, as in most of the national annexes for the implementation of EN 206 [1] in Europe, by a descriptive approach (concrete "designed to last") referring to mandatory) to a series of requirements of the concrete composition (water / cement ratio, minimum cement dosage, entrained air, frost-thaw-resistant aggregates, etc.) and of the compressive strength (concrete class) depending on the classification of the element in a certain exposure class "X".

The support of this descriptive national approach is based on a large number of results of a complex experimental program, containing "candidate" cements and "reference" cements as well as "in situ" tests / determinations on "reinforced concrete column" type construction elements. made with both categories of cements.

Based on the experimental research performed, performance criteria can be determined for the concrete resistance to freeze-thaw (performance approach).

The test methods used at European level are:

- CEN/TS 12390-9 - Tests on hardened concrete - Part 9: Freeze-thaw resistance of concrete. Scaling;
- CEN/TR 15177 - Testing the freeze/thaw resistance of concrete. Internal structural damages.

The European-accepted freeze/thaw resistance test methods are complemented by proposals for classification criteria, depending on the results obtained, in different XF exposure

classes. Their application largely reflects the real-time behavior of concrete subjected to freeze-thaw.

The provisions of the national annex SR 13510: 2006 [5] are correlated with the level of acceptance of certain types of cements in other national annexes (belonging to technologically developed countries and with a similar climate - continental temperate - to that of the country and even more restrictive in some respects than those in France and Germany, for example.

The provisions of our national annex stop, at the level of 2006, for CEM I cements, selectively for CEM II and punctually only for CEM III/A type, and as new types of cements are produced, they will be tested in order to define some domains of use, as otherwise provided by NE 012/1: 2007 (# 5.1.2.) mandatory. [2]

The types of cement covered by this research report can be considered as new, "candidate" cements for which there is no relevant national experience in use. In addition, CEM II/A-M cements with limestone in their composition represent cements for which at European level the level of knowledge and acceptance in regulations remains quite limited.

Ensuring the durability of concrete by establishing levels (criteria) of performance depending on the place of use of concrete (materialized by the class of resistance to actions of the environment "RX") is an absolutely necessary step forward given that, even at European level, reached a certain "degree of saturation" in terms of acceptance of new types of "candidate" cement in different exposure classes by the (current) descriptive method.

This research report aims to present the concept of freeze/thaw resistance class ("RXF") and proposals for the classification of concrete prepared with different types of cements in freeze/thaw resistance classes. Along with the environmental action on concrete on carbonation, there will be changes in the regulations for concrete production, but also in the design in terms of sustainability.

An analysis will be presented on the experimental results obtained in research on concrete prepared with different types of cements, carried out in collaboration with the laboratory of the Department of Reinforced Concrete Constructions, Technical University of

Constructions Bucharest. Freeze-thaw resistance classes will also be proposed for different types of cements.

1. Defining freeze/thaw resistance classes

The national annex of SR EN 206-1: 2002, SR 13510 [5], in force at the time of the experimental research, provides for the four classes of freeze/thaw exposure the limit values presented in table 1.

Table 1 - Recommended limit values for concrete composition and properties

	Exposure classes					
	Freeze-thaw attack					
	XF1	XF2		XF3		XF4
Maximum water / cement ratio	0,50	0,55 ^a	0,50	0,55 ^a	0,50	0,50 ^a
Minimum strength class	C25/30	C25/30	C35/45	C25/30	C35/45	C30/37
Minimum dosage of cement (kg/m³)	300	300	320	300	320	340
Minimum entrained air content (%)	-	a	-	a	-	a
Other conditions	Freeze-thaw resistant units SR EN 12620					d
a) The entrained air content is determined according to the maximum size of the granule. If the concrete does not contain intentionally entrained air, then the performance of the concrete must be measured according to an appropriate test method, compared to a concrete for which the freeze-thaw resistance has been established for the appropriate exposure class.						
d) In case of exposure in marine areas, cements resistant to the action of seawater will be used.						

There are also recommendations for choosing the limit values of the composition and properties of the concrete according to the exposure class according to SR EN 206 + A1: 2017 (table 2) [1].

Table 2 - Recommended limit values for concrete composition and properties

	Freeze-thaw attack			
	XF1	XF2	XF3	XF4
Maximum water / cement ratio	0,55	0,55	0,50	0,45
Minimum strength class	C30/37	C25/30	C30/37	C30/37
Minimum dosage of cement (kg/m³)	300	300	320	340
Minimum entrained air content (%)	-	4,0 ^a	4,0 ^a	4,0 ^a
Other conditions	Aggregates according to EN 12620 with sufficient freeze/thaw resistance			

^a If the concrete does not contain entrained air, then the performance of the concrete must be measured using an appropriate test method, by comparison with a concrete, for which the freeze/thaw resistance for the appropriate exposure class has been established.

^c When the concept of value k is applied, the maximum water / cement ratio and the minimum cement dosage are changed according to 5.2.5.2.

SR EN 206 [1] shows changes in W/C ratios, cement dosages and concrete grades compared to SR EN 206-1: 2002. XF2 is the only exposure class in which the composition is the same.

An example of the use of cements in XF1 exterior elements is given in Table 3 for several European countries [16]. The question is: do all these concretes have the same performance?

Table 3 - Comparison of cement applications in Europe.

Example: Concrete for exterior construction elements (XF1)

Stat	Max. A/C _{eq}	Min. C	CEM I	CEM II						CEM III		CEM IV		CEM V	
		S		L/LL		M									
		Kg/m ³		A	B	A	B	A	B	A	B	A	B	A	B
Austria	0,55	300	x	x	x	x	(x)	x	(x)	x	(x)				
Belgia	0,55	300	x	x	x	x	x	x	x	x	x			(x)	
Danemarca	0,55	150	(x)			(x)									
Finlanda	0,60	270	x	x	x	x		x		x	x				
Franța	0,60	280*	x	x	x	x	x	x	x	x	x	x	x	x	x
Germania	0,60	280	x	x	x	x	o	(x)	(x)	x	x	o	(x)	(x)	(x)
Irlanda	0,60	300	x			x									
Italia	0,60	320	x	x	x	x	x	x	x	x	x	x	x	x	x
Olanda	0,55	300	x	x	x	(x)	(x)	(x)	(x)	x	x	(x)	(x)	(x)	(x)
Norvegia	0,60	250	x	x		x									
Anglia	0,60	280	x	x	x	x				x	x	(x)	(x)		

Caption: Not mentioned, Permitted use, With limitations, Unauthorized use;

Obs.: * and x indicate that there are qualifications, e.g. types of main constituents.

In Romania, at present, for the exposure class XF1 the maximum W/C ratio is 0.5, the minimum cement dosage 300 kg / m³, concrete class C25 / 30. The fields of use for cements are presented in table F.2.1. and F.2.2. of CP012-1 [24]

There is a proposal to introduce in EN 206 [1] the RXF freeze-thaw resistance classes - Chapter 4.2, Table 3 and Table F.1 in Annex F, respectively.

Table 4 (Table 3 of pr EN 206) - Exposure resistance classes,
applicable limit values and standards [14]

RXF freeze-thaw resistance classes		
	RXF0,5	RXF1
Limit values, kg/m ²	0,5	1,0
Applicable standards	CEN/TS 12390-9 CEN/TR15177	

Table 5 (Table F.1 - partly from pr EN 206) - Freeze-thaw resistance classes; values considered satisfactory for different binder compositions (eg preliminary values)

Preliminary Values	RXF freeze-thaw resistance classes		
	RXF 0,2	RXF 0,5	RXF 1,0
Cement type or equivalent binder combination	Maximum water / binder ratio the binder sums up the amount of cement and concrete additives, within the limits defined according to EN 206-1 for cement		
CEM I	0,4	0,45	0,5
CEM II-A	?	?	?
CEM II-B	?	?	?
CEM III-A	?	?	?
CEM III-B	?	?	?
Minimum binder content [kg/m ³]	280	280	280
Minimum entrained air content	4%	4%	-

The more refined alternative method, which distinguishes between the different types of binders in Annex F of pr EN 206 is presented in Table 6.

Table 6 - Freeze-thaw resistance classes for different types of cement or binder combinations [19]

Preliminary Values		RXF freeze-thaw resistance classes		
		RXF0,2	RXF0,5	RXF1,0
Cement type or equivalent binder combination				
CEM I		0,40	0,45	0,50
CEM II/A-	V			
	S			
	D			
	L			
	LL			
	M			
CEM II/B	V			
	S			
	D			
	L			
	LL			
	M			
CEM III/A	S			
CEM III/B	S			
Minimum binder content [kg/m ³]		280	280	280

There are proposals to supplement EC2. Table 7 presents the initial proposal of EC2, including the notations regarding the freeze/thaw resistance classes.

Table 7 - Proposal to complete EC2

Concrete degradation	
Freeze/thaw resistance class	
RXF (Medium)	RXF (High)
The definition of the class is 50 years of exposure to XF4, with a 10% probability of exceeding the concrete loss at the surface [kg/m ²]	
10	2

Proposals can be made to supplement EC2 with the notions of freeze/thaw resistance classes, for example in Table 8 a proposal under discussion for the use of 2 climate classes is presented [19].

Table 8 - Concrete damage,
resistance classes allowed for XF exposure classes

Freezing - thawing action		
Exposure class EC	Minimum allowable freeze/thaw resistance classes	
	Severe frost climate	Light frost climate ¹
XF1	RXF12	RXF12
XF2	RXF12	RXF12
XF3	RXF0,5	RXF1,0
XF4	RXF0,5	RXF1,0
¹ The mild frost climate can be defined in locally valid provisions, based on the zonal climate, regarding frost cycles and extreme temperatures.		

- RXF12 is covered by DtS (Deemed to Satisfy) and the descriptive text from EN 206 for XF1 and XF2
 - RXF0.5 assumes that $m_{56} < 0.5 \text{ kg/m}^2$
 - RXF1.0 assumes that $m_{56} < 1.0 \text{ kg/m}^2$
- and condition valid for all RXF classes $m_{56} / m_{28} < 2$

RXF0.5 and RXF1.0 can be defined with an alternative DtS (Deemed to Satisfy) classification that completes the test requirement.

Another proposal for introduction into EC2 presented in CEN TC250 / SC2 / WG1 / TG10 N0045 [9] for the classification of freeze/thaw resistance according to exposure classes is presented in Table 9.

Adequate freeze/thaw resistance only for moderately saturated concretes (exposure classes XF1 and XF2) can be achieved by using a composite concrete according to EN 206. Adequate durability against the freeze-thaw action of concrete exposed to moisture (exposure

classes XF3 and XF4) can be assumed by selecting the appropriate RF class according to Table 9 regarding the climatic conditions at the location and the projected service life (L) of the structure.

Table 9 - Freeze-thaw resistance classes
(proposal for tables 4.2 and 4.5 of EC2, only the freeze-thaw part)

Concrete damage		
Freeze / thaw resistance		
RFW H ² (High resistance)	RFW M ² (Medium resistance)	RFW L ² (Low resistance)
RFD H ³ (High resistance)	RFD M ³ (Medium resistance)	RFD L ³ (Low resistance)
² Tested according to the slab Test method CEN / TS 12390-9 with water		
³ Tested according to the slab Test method CEN / TS 12390 9 with salt solution		
	Exposure class	
	XF3	XF4
Mild winters ¹	RFW L	RFD L
Moderate winters ² and L<100		
Moderate winters ² and L≥100	RFW M	RFD M
Severe winters ³ and L<100		
Severe winters ³ and L≥100	RFW H	RFD H
¹ Few frost cycles per year, temperatures rarely below -5 °C. ² A few cycles of frost per year, temperatures rarely below -10 °C. ³ Many frost cycles per year, temperatures occasionally below -20 °C. L = projected service life		

Definitions of mild, moderate and severe winters can be discussed and adjusted.

It would be better to use the maximum real value of exfoliation in the designation of the resistance class, as there may be further discussions on the corresponding values for the different winter climates.

Proposal for criteria:

- RFW L and RFD L: $m_{56} < 2,0 \text{ kg/m}^2$, $m_{56}/m_{28} < 2$ or $m_{110} < 2,0 \text{ kg/m}^2$
- RFW M and RFD M: $m_{56} < 1,0 \text{ kg/m}^2$ or $m_{110} < 1,0 \text{ kg/m}^2$
- RFW H and RFD H: $m_{56} < 0,5 \text{ kg/m}^2$ or $m_{110} < 0,5 \text{ kg/m}^2$

The above criteria are valid for the initial testing of a concrete composition. When tested for control or in existing structures, the maximum exfoliation values may be increased by 20%.

Table 10 presents a variant of classification of freeze/thaw resistance, including classification into specific exposure classes.

Table 10 - Classification of concrete in freeze-thaw exposure classes

Exposure class	Freeze/thaw resistance class	
	Moderate frost climate	Severe frost climate
XF1	RF1 ¹	RF1
XF2	RF1	RF0,2
XF3	RF1	RF0,2
XF4	RF0,2	RF0,2
¹ In the moderate frost climate the test environment may be fresh water		

Table 11 presents another proposal, less restrictive, related to the freeze-thaw action.

Table 11 - Proposal for the classification of concrete in freeze/thaw resistance classes

Exposure class	Clasa de rezistență minim acceptată	
	Severe frost climate	Moderate frost climate
XF1	RF1 ¹	RF1 ¹
XF2	RF0,5	RF1
XF3	RF0,5	RF1
XF4	RF0,2	RF0,5
¹ - In the moderate frost climate the test environment may be fresh water		

A problem with the proper classification for the four XF classes is that there are two different scenarios, one with salt (chlorides) and one without and possibly different degradation mechanisms.

According to the Swedish regulations [17] regarding the requirements for the four existing XF classes (freeze-thaw test according to the slab test method CEN / TS 12390-9 [3]) we have:

XF1 - Only the composition requirement ($A / C < 0.6$) will be used, salt-free method, no testing required,

XF2 - Initial test according to CEN / TS 12390-9 [3] with salt, exfoliation $< 0.5 \text{ kg} / \text{m}^2$ after 56 cycles or $< 0.5 \text{ kg} / \text{m}^2$ after 112 cycles) or the requirement for minimum air content in depending on the size of the aggregates.

XF3 - Initial testing according to CEN / TS 12390-9 [3] (without salt, exfoliation $< 0.5 \text{ kg} / \text{m}^2$ after 56 cycles or $< 0.5 \text{ kg} / \text{m}^2$ after 112 cycles) or minimum content requirement air depending on the size of the aggregates.

XF4 - Initial testing according to CEN / TS 12390-9 [3] (with salt, exfoliation $< 0.5 \text{ kg} / \text{m}^2$ after 56 cycles or $< 0.5 \text{ kg} / \text{m}^2$ after 112 cycles) is mandatory.

In XF4 and if the test criterion is also used in XF3, the tests must be performed continuously, in which case the exfoliation after 56 cycles will be less than $1.0 \text{ kg} / \text{m}^2$.

The minimum air content requirement is normally used in XF2 and XF3 in Sweden. It should also be noted that the test requirements in XF2 and XF3 are not as well investigated as the requirements in XF4 and must be subsequently validated and calibrated based on in-situ experience.

However, there may be an adaptation of the method based on the concrete composition to realistically reflect the long-term frost resistance of the concrete for different binders. For example, the freeze-thaw cycle with the existing method begins at the age of 28 days, which is clearly a disadvantage for fly ash concrete, in which the development of resistance is slower at first than in Portland CEM I cement concrete. It can also be a disadvantage for slag concrete for the same reason. For ash / slag concrete, long-term freeze/thaw resistance can be better reflected when a curing time of 91 days is used. For

concretes with slag of 30% or more, the test procedure may involve some exposure to carbon dioxide, in order to take into account, the reduction in frost resistance due to carbonation. In the presence of larger amounts of silica powder, for example > 5% of the binder, it would be appropriate to extend the period to 112 cycles, as a sudden increase in exfoliation may occur after 56 cycles. Such changes to the binder composition should be provided in the EN 206 proposal and applied throughout Europe.

2. Experimental research

The research program presented in this research report consists in determining the freeze/thaw resistance on concrete samples, in accordance with the Romanian standard SR 3518 [10], the norm NE012-1 [2] and the European standard CEN / TS 12390- 9 [3], the slab test method and the use of the results obtained in the classification of concretes into freeze/thaw resistance classes.

2.1. Determination of freeze/thaw resistance in accordance with Romanian regulations

2.1.1. Characteristics of concretes prepared with CEM II/A-M 32.5R and CEM II/A-S 32.5R

This research report presents the results obtained for the prepared concretes CEM II/A-M (S-LL) 32,5R and CEM II/A-S 32,5R, after maintaining the samples at freeze-thaw cycles. Cements have the following percentages of additives:

1. CEM II/A-M (S-LL) 32.5R - slag 10%, limestone 7% - CEM 1;
2. CEM II/A-M (S-LL) 32.5R - slag 10%, limestone 6% - CEM 2;
3. CEM II/A-S 32.5R - slag 17%, limestone 3% - CEM 3.

Concrete with different cement dosages was prepared with these types of cements, sorted aggregates 0-4mm (35%), 4-8mm (15%), 8-16mm (21%) and 16-32mm (29%), hyperplasticizing additive (polycarboxylate base substance)/superplasticizer (dinaftilmethane-sulfonate base substance) in combination with various air entraining additives.

The preparation and the experimental researches carried out on the concretes were made in accordance with the regulations in force at the time of the researches.

I mention the fact that these tests were made in collaboration with the laboratory staff of the Department of Reinforced Concrete Constructions, Faculty of Civil, Industrial and Agricultural Constructions, Technical University of Constructions of Bucharest.

2.1.1.1. Characteristics of fresh concrete

The concretes were prepared with hyperplasticizer / superplasticizer additive and in combination with air entraining additive, at different cement dosages, according to tables 12 ... 14, for settlements between 100 - 130 mm.

Table 12 - Characteristics of fresh concrete prepared with CEM 1

Cement dosage (kg/m ³)	Settlement (mm)	W/C ratio	Density (kg/m ³)	Trained air (%)
0,6% hyperplasticizing additive (polycarboxylates)				
370	120	0,43	2405	-
470	115	0,37	2366	-
0.6% hyperplasticizer additive + 0.06% air entrainer (sulfonated hydrocarbon)				
450	130	0,36	2379	4,0
550	120	0,33	2348	5,1

Table 13 - Characteristics of fresh concrete prepared with CEM 2

Cement dosage (kg/m ³)	Settlement (mm)	W/C ratio	Density (kg/m ³)	Trained air (%)
0,6% hyperplasticizing additive (polycarboxylates)				
370	125	0,43	2423	-
470	125	0,36	2415	-
0.6% hyperplasticizer additive + 0.06% air entrainer (sulfonated hydrocarbon)				
450	130	0,36	2389,6	4,0
550	120	0,32	2397,0	3,8

Tabelul 14 – Caracteristicile betoanelor proaspete preparate cu CEM 3

Dozaj ciment (kg/m ³)	Tasare (mm)	Raport A/C	Densitate (kg/m ³)	Aer antrenat (%)
1% hyperplasticizing additive (polycarboxylates)				
320	105	0,5	2364	-
1,5% hyperplasticizer additive + 0.06% air entrainer (sulfonated hydrocarbon)				
430	100	0,40	2308	4,9
500	100	0,37	2298	4,9
570	100	0,34	2327	4,8
0,4% air entraining additive (tenside sintetice)				
570	100	0,41	2280	6,0

2.1.1.2. Characteristics of strengthened concrete

The results obtained for the compressive strengths at 2 and 28 days, for the concretes prepared with CEM 1 are presented in table 15.

Table 15 – Compressive strengths for the concretes prepared with CEM 1

Cement Dosage (kg/m³)	W/C ratio	Compressive strength (N/mm²)			
		f _{ci} 2 days	f _{cm} 2 days	f _{ci} 28 days	f _{cm} 28 days
hyperplasticizing additive					
370	0,43	19,44	19,00	43,71	42,81
		19,17		41,6	
		18,38		43,11	
470	0,37	24,92	24,84	50,04	49,66
		24,68		49,18	
		24,91		49,76	
hyperplasticizing additive + air entrainer					
450	0,36	21,83	21,57	53,72	53,74
		21,47		54,28	
		21,40		53,23	
550	0,33	26,59	26,78	55,29	56,56
		26,64		57,07	
		27,10		57,32	

In the case of a continuous production, the classes obtained are C40/50 (dosage 450 and 550 kg/m³), the supplementation of the cement dosage with 100 kg/m³ not remarkably influencing the value of resistance.

In the case of concretes prepared with CEM 2, the results obtained for the compressive strengths at 2 and 28 days are presented in table 16.

Table 16 - Compressive strengths of concrete prepared with CEM 2

Cement Dosage (kg/m³)	W/C ratio	Compressive strength (N/mm²)			
		f _{ci} 2 days	f _{cm} 2 days	f _{ci} 28 days	f _{cm} 28 days
hyperplasticizing additive					
370	0,43	28,89	28,83	44,94	44,07
		29,15		43,28	
		28,44		44,00	
470	0,36	34,19	33,17	52,44	52,51
		33,21		53,16	
		32,12		51,94	
hyperplasticizing additive + air entrainer					
450	0,36	28,93	28,33	50,55	51,07
		26,79		52,22	
		29,26		50,45	
550	0,32	32,68	34,31	52,43	53,38
		35,30		54,39	
		34,96		53,31	

In case of a continuous production, the classes obtained are C35/45 (dosage 450 kg/m³) and C40/50 (dosage 550 kg/m³).

The results obtained for the compressive strengths at 2 and 28 days, for concretes prepared with CEM II/A-S 32,5R (CEM 3) are presented in table 17.

Table 17 - Compressive strengths of concrete prepared with CEM 3

Cement Dosage (kg/m³)	W/C ratio	Compressive strength (N/mm²)			
		f _{ci} 2 days	f _{cm} 2 days	f _{ci} 28 days	f _{cm} 28 days
hyperplasticizing additive					
320	0,50	21,92	21,00	31,32	31,90
		20,90		31,71	
		20,19		32,67	
hyperplasticizing additive + air entrainer					
430	0,40	21,20	21,93	41,82	41,89
		22,28		41,70	
		22,32		42,15	
570	0,34	30,16	30,38	52,36	53,74
		30,72		54,68	
		30,25		54,19	
air entrainer additive					
570	0,41	19,49	19,52	42,58	43,63
		19,20		44,38	
		19,87		43,93	

The influence of the air entraining additive with superplasticizer effect in reducing the W/C ratio and implicitly in increasing the compressive strength can be observed.

2.1.2. Determination of freeze/thaw resistance on concrete samples made in accordance with NE012-1 [2]

During the research program, concrete of resistance classes, cement dosages and W/C ratios were tested for freeze-thaw in accordance with NE012-1 [2]. The determination of freeze/thaw resistance was made in accordance with the Romanian standard SR 3518 [10].

The destructive method according to SR 3518 [10] determines the decrease of the compressive strength of the test specimens tested in freeze/thaw compared to the control specimens (made at the same time, of the same concrete and preserved until the test under the same conditions as the specimens to be tested).

The tests were performed on cubic specimens with a side of 150 mm, made according to SR EN 12390-2 [20] with a minimum age of 28 days.

During the first day after casting, the specimens are kept in molds at an air temperature of $(20 \pm 2) ^\circ\text{C}$, being protected against drying by the use of polyethylene films. After (24 ± 2) hours, the samples are stripped and immersed in water at a temperature of $(20 \pm 2) ^\circ\text{C}$. At the age of 7 days, the specimens are removed from the water and kept in the air at a temperature of $(20 \pm 2) ^\circ\text{C}$ and a humidity of $(65 \pm 5) \%$ for 21 days. The specimens at least 28 days old are immersed in water at a temperature $(20 \pm 5) ^\circ\text{C}$ for saturation, 4 days before the start of the test.

The test specimens shall be placed in the cold room and the test specimens shall be kept under water or at high humidity in accordance with SR EN 12390-2 [20]. Saturated specimens placed in the cold room at $(-17 \pm 2) ^\circ\text{C}$ are kept for 4 hours.

The specimens are then removed from the cold room and immediately immersed in either water or continuously sprayed with water at $(20 \pm 5) ^\circ\text{C}$ for 4 hours.

After the specimens have been subjected to freeze-thaw cycles, the loss of compressive strength is determined, subjecting to the compression test, according to SR EN 12390-3, three of the test specimens and the same number of control specimens.

The maximum number of successive freeze-thaw cycles that concrete specimens can withstand without suffering a reduction in compressive strength greater than 25% compared to the control specimens is considered as freeze/thaw resistance.

The resistance losses in the case of concretes prepared with CEM 1 determined in accordance with SR 3518 [10] after 100 and 150 freeze-thaw cycles, respectively, are shown in Table 18.



Table 18 - Loss of strength of concretes prepared with CEM 1 and hyperplasticizing additive, after 100/150 freeze-thaw cycles

Nr. cycles	Cement dosage (kg/m ³)	W/C ratio	Compressive strength mator (N/mm ²)		Compressive strength after 100/150 freeze-thaw cycles (N/mm ²)		Loss of compressive strength (%)
			individual value	average value	individual value	average value	
G100	370	0,43	57,05	56,61	53,20	51,39	9,21
					52,38		
			55,34		51,28		
					50,58		
			57,43		49,62		
G150	470	0,37	68,25	67,35	62,04	62,29	7,52
					62,01		
			67,40		61,18		
					62,80		
			66,39		63,39		
					62,29		

Resistance reductions after 100/150 freeze-thaw cycles were below 10%.

The determination of the freeze/thaw resistance was also performed on the concretes prepared with air entraining additive, using the method of appreciation / evaluation of the decrease of the compressive strength of the concrete after performing a certain number of freeze-thaw cycles.

Thus, Table 19 shows, first of all, the low values of the W/C ratios, below 0.4, which led to the realization of high-strength concrete.



Table 19 - Loss of strength of concretes with entrained air prepared with CEM 1, after 100/150 freeze-thaw cycles and de-icing agents

Nr. cycles	Cement dosage (kg/m ³)	W/C ratio	Compressive strength mortar (N/mm ²)		Compressive strength after 100/150 freeze-thaw cycles (N/mm ²)		Loss of compressive strength (%)
			individual value	average value	individual value	average value	
G100	450	0,36	55,36	55,94	50,51	50,49	9,74
					51,68		
			56,00		50,28		
					49,66		
			56,45		51,58		
G150	550	0,33	58,12	58,15	52,95	53,00	8,86
					53,78		
			57,41		51,28		
					54,67		
			58,93		51,72		
					53,59		

Resistance reductions after 100/150 freeze-thaw cycles and de-icing agents were below 10%.

The resistance losses for concretes prepared with CEM 2, determined in accordance with SR 3518 [10], after 100 and 150 freeze-thaw cycles, respectively, are shown in Table 20.



Table 20 - Loss of strength of concretes prepared with CEM 2 and hyperplasticizing additive, after 100/150 freeze-thaw cycles

Nr. cycles	Cement dosage (kg/m ³)	W/C ratio	Compressive strength mortar (N/mm ²)		Compressive strength after 100/150 freeze-thaw cycles (N/mm ²)		Loss of compressive strength (%)
			individual value	average value	individual value	average value	
G100	370	0,43	52,28	53,62	47,56	46,67	12,96
					46,67		
			55,35		45,32		
					47,34		
			53,23		46,11		
G150	470	0,36	60,24	60,32	54,05	55,03	8,77
					53,53		
			61,66		55,4		
					55,89		
			59,05		56,72		
					54,56		

Reducerile de rezistență după 100/ 150 de cicluri de îngheț-dezgheț au fost sub 13%.

In the case of concretes prepared with CEM 2, higher values for loss of strength were obtained compared to the values obtained for concretes prepared with CEM 1.

The determination of the freeze/thaw resistance was also performed on the concretes prepared with air entraining additive, using the method of appreciation / evaluation of the decrease of the compressive strength of the concrete after performing a certain number of freeze-thaw cycles.

Thus, Table 21 shows first the low values of the W/C ratios, below 0.4, which led to the realization of high-strength concrete.

Table 21 - Loss of strength of concretes with entrained air prepared with CEM 2, after 100/150 freeze-thaw cycles and de-icing agents

Nr. cycles	Cement dosage (kg/m ³)	W/C ratio	Compressive strength mator (N/mm ²)		Compressive strength after 100/150 freeze-thaw cycles (N/mm ²)		Loss of compressive strength (%)
			individual value	average value	individual value	average value	
G100	450	0,36	56,69	57,95	53,47	52,16	9,99
					52,72		
			59,12		52,08		
					51,51		
			58,03		52,16		
					51,01		
G150	550	0,32	64,31	63,4	57,09	56,13	11,48
					56,71		
			63,78		55,98		
					56,25		
			62,12		56,39		
					54,34		

Resistance reductions after 100/150 freeze-thaw cycles and thawing agents were below 12%.

In the case of concretes prepared with CEM 3, the resistance losses after 100 freeze-thaw cycles are shown in Table 22.

Table 22 - Loss of strength of concretes prepared with CEM 3 and superplasticizer after 100 and 150 freeze-thaw cycles

Nr. cycles	Cement dosage (kg/m ³)	W/C ratio	Compressive strength mator (N/mm ²)		Compressive strength after 100/150 freeze-thaw cycles (N/mm ²)		Loss of compressive strength (%)
			individual value	average value	individual value	average value	
G100	320	0,50	45,25	44,55	41,19	41,52	6,79
					41,68		
			44,31		40,65		
					41,60		
			44,08		42,14		
					41,86		
G150	320	0,50	45,71	46,02	42,41	42,30	8,09
					42,15		
			46,64		42,84		
					42,97		
			45,71		41,87		
					41,55		

Table 23 - Loss of strength of concretes prepared with CEM 3 and air entraining additive, after 100 and 150 freeze-thaw cycles and de-icing agents

Nr. cycles	Cement dosage (kg/m ³)	W/C ratio	Compressive strength mator (N/mm ²)		Compressive strength after 100/150 freeze-thaw cycles (N/mm ²)		Loss of compressive strength (%)
			individual value	average value	individual value	average value	
G100	430	0,40	53,79	53,28	49,01	48,65	8,69
					48,32		
			53,36		49,16		
					48,15		
			52,69		48,00		
G150	430	0,40		53,10	49,25	48,17	9,27
			53,35		48,25		
					48,56		
			52,73		48,81		
					47,88		
			53,21		47,50		
					48,04		

Table 24 - Loss of strength of concretes prepared with CEM 3 and air entraining additive, after 100 and 150 freeze-thaw cycles and de-icing agents

Nr. cycles	Cement dosage (kg/m ³)	W/C ratio	Compressive strength mator (N/mm ²)		Compressive strength after 100/150 freeze-thaw cycles (N/mm ²)		Loss of compressive strength (%)
			individual value	average value	individual value	average value	
G100	570	0,34	60,04	60,68	55,90	56,14	7,49
					56,75		
			61,07		56,68		
					56,45		
			60,93		55,70		
G150	570	0,34		61,10	55,34	56,08	8,21
			61,07		55,73		
					56,80		
			61,72		56,70		
					54,87		
			60,50		55,93		
					56,46		

Concretes prepared with CEM 1 and CEM 3 falling into the same concrete class C40 / 50 have similar values after 150 freeze-thaw cycles with de-icing agents (Table 25).

Table 25 - Loss of compressive strength of concrete after 150 cycles of freeze-thaw and de-icing agents

Cement type	Cement dosage (kg/m ³)	W/C ratio	Loss of compressive strength of concrete after 150 cycles (%)
CEM 3	570	0,34	8,21
CEM 1	550	0,33	8,86
CEM 2	550	0,32	11,48

Concretes prepared with the three types of cements had lower strength losses than the value imposed by SR 3518 [10] (25%).

It is also possible to emphasize the importance of the hyperplasticizing additive used in the preparation of concrete with CEM 1 and CEM 2 to reduce the W/C ratio and thus increase the compressive strength.



2.2 Determination of freeze/thaw resistance on concrete samples made in accordance with European standards

In Europe the most common test methods are performed according to the CEN/TS 12390-9 standard [3]. This standard describes a reference method (slab test) and two alternative test methods (cube test and CF/CDF test). The introduction of the CEN / TS 12390-9 standard states that when new component materials or compositions are used, they must be tested in accordance with the methods presented.

The application of the methods cannot completely reproduce the real conditions, but in any case, the methods must be correlated with the practical situations in order to provide credible results.

Also, the application of some limit values requires the establishment of a correlation with the results obtained in the laboratory and the experience of behavior in real environments. Given the nature of the freeze-thaw action of concrete, this correlation must take into account local conditions.

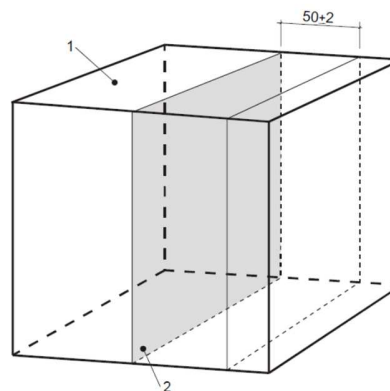
The standard describes test methods for freeze/thaw resistance to determine the amount of scaled material.

The methods are also used in the case of freeze-thaw action and de-icing agents to compare new component materials or compositions to the material, with concrete materials or compositions that have provided adequate in-situ performance, or to compare the results obtained experimentally with absolute limit values established based on local experience.

No correlations are established between the results obtained by applying these three methods.

→ **Slab test (reference method)**

The samples are obtained by cutting (figure 1) and are subjected to the freeze/thaw attack in the presence of water 3 mm deep, deionized water or 3% NaCl solution.



1. Casting surface
2. Test surface

Fig. 1 - Orientation of the test surface

Freeze/thaw resistance is assessed by measuring the mass of scaled concrete on the exposed surface after 56 freeze-thaw cycles.

The test requires four samples, one in four cubes. The first day after casting the cubes are kept in molds and protected against drying by using a polyethylene film at an air temperature of $(20 \pm 2) ^\circ\text{C}$.

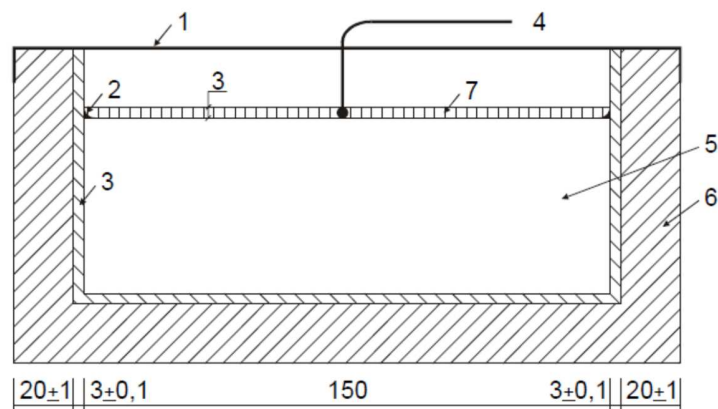
After this time the cubes are stripped and placed in water at a temperature of $(20 \pm 2) ^\circ\text{C}$.

At the age of 7 days, the cubes are removed from the water and placed in a climatic chamber until the beginning of the test.

On day 21, a (50 ± 2) mm thick sample is cut from the middle third of the cube.

After cutting, the sample is washed and reintroduced into the climatic chamber.

At the age of (25 ± 1) days of the concrete, the support of the sample is made (figure 2).

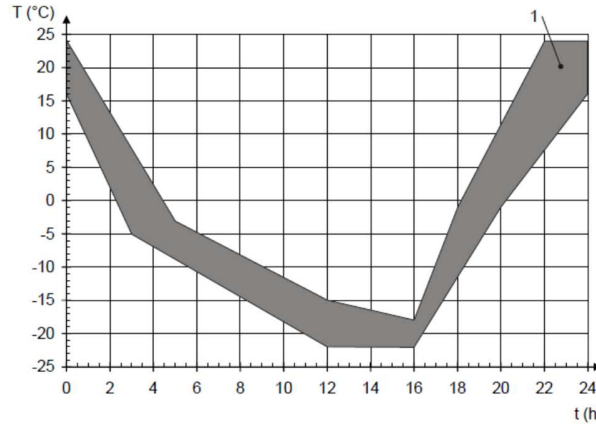


1. Polyethylene film
2. Glue
3. Rubber band
4. Temperature recording device
5. Sample
6. Thermal insulation material
7. Freezing environment

Fig. 2 - Preparation of the test sample

At the age of 28 days, water is poured, 3 mm deep, at a temperature of $(20 \pm 2)^\circ\text{C}$ on the surface of the concrete sample, and is maintained at this level for (72 ± 2) h.

The test starts at the age of 31 days of the concrete, respecting the freeze-thaw cycles shown in figure 3.



1. Temperature in the center of the sample

Fig. 3 - Cycle time (t) - temperature (T) in the center of the test sample

- After (7 ± 1) (14 ± 1) (28 ± 1) (42 ± 1) and 56 cycles the following procedure is applied:
- Collect the scaled material from the surface, brush the exposed concrete surface;
- Pour enough water to a depth of 3 mm (67 ml)
- Place the sample in the refrigerator
- Then determine the mass of scaled material, dried at a constant mass $(110 \pm 10)^\circ\text{C}$.
- Round off the amount of scaled material to 0.1 g.

$$m_{S,n} = m_{S,\text{before}} + (m_{V+S} - m_{V(+f)}) \quad (1)$$

where:

$m_{S,n}$ represents the mass of the scaled material after n cycles of freeze-thaw, rounded to 0.1 g

$m_{S,\text{before}}$ represents the mass of the scaled material previously determined;

m_{V+S} represents the mass of the vessel containing the scaled material (by brushing) filtered (from water) and of the filter;

$m_{V(+f)}$ represents the mass of the empty vessel and the dry filter.

The results are expressed using the expression:

$$S_n = \frac{m_{s,n}}{A} \cdot 10^3 \quad (2)$$

S_n represents the mass of the scaled material relative to the surface, after n freeze-thaw cycles

$m_{s,n}$ in accordance with relation (1)

A = the total area of the tested area calculated before the preparation of the test device.

This method may also be used in cases where other conditions relating to:

- a) the geometry of the sample may be different, for example if it is processed from a core extracted from an existing work, but its thickness must always be 50 ± 2 mm.
- b) Use of a test surface other than that indicated in the method related to the casting surface.
- c) Provision of other treatment conditions and the start of the test at an age other than 31 days.
- d) Use of other defrosting agents.
- e) The number of cycles may be more than 56. In some cases, for example, when testing pavers or curbs, 28 cycles may be used.

If these alternative applications are used, the samples are cut to a thickness of 50 ± 2 mm 10 days before the start of the test. During these 10 days the samples are exposed in a climatic chamber for 7 days and then saturated 3 days before the start of the test.

→ European evaluation criteria for exfoliation, slab test method:

1. Exposure class XF4 (cement dosage 320 kg / m³ and W/C ratio = 0.5, entrained air)

The amount of scaled material must be less than 1Kg / m² after 56 freeze-thaw cycles.

→ Criteria proposed by UTCB and accepted by MDRAP, slab test method:

2. Exposure class XF1 (cement dosage 320 kg / m³ and W/C ratio = 0.5)

The amount of scaled material must be less than 1.3 kg / m² after 56 cycles.

3. XF3 exposure class (cement dosage 320 kg / m³ and W/C ratio = 0.5)

The amount of scaled material must be less than 1Kg / m² after 56 cycles.

4. XF2 exposure class (cement dosage 320 kg / m³ and W/C ratio = 0.5, entrained air)

The amount of scaled material must be less than 1.3 kg / m² after 56 cycles.

2.2.1. CEM II/A-M(S-LL) 32.5R - CEM 1

The experimental program consisted in determining the freeze/thaw resistance of the samples by measuring the amount of scaled material in accordance with the CEN / TS 12390-9 standard. [3]

Concrete compositions were prepared using a cement dosage of 320 kg/m³, according to the data shown in Table 26.

Table 26 – Characteristics of fresh concrete

Additive dosage	W/C ratio	Cement dosage (kg/m ³)	Settlement (mm)	Density (kg/m ³)
1% hyperplasticizer	0,50	320	180	2430
1% hyperplasticizer + 0.2% air trainer	0,50	320	185	2425

Table 27 shows the results obtained for the compressive strength of concrete kept in water until the test period.

Table 27 - Strength characteristics of reinforced concrete

W/C ratio	Cement dosage (kg/m³)	Compressive strength (N/mm²)				Concrete class
		2 days		28 days		
0,50	320	19,23	19,47	38,94	39,12	C25/30
		20,04		39,39		
		19,14		39,02		
0,50	320	17,00	17,37	37,68	38,73	C25/30a
		16,81		39,69		
		18,29		38,82		

■ **For exposure classes XF1, XF3:**

The results on the exfoliation of the concrete performed with the slab test method are presented in table 28.

Table 28 - Amount of scaled concrete after n freeze-thaw cycles, 50x150x150 mm concrete strips

Cement dosage (kg/m ³)	W/C	Series	7 cycles	14 cycles	28 cycles	Total amount of concrete scaled after 28 cycles	Sn, kg/m ²	Sn, average, kg/m ²	56 cycles	Total amount of concrete scaled after 56 cycles	Sn, kg/m ²	Sn, average, kg/m ²
			amount of scaled concrete, g	amount of scaled concrete, g	amount of scaled concrete, g				amount of scaled concrete, g			
320	0,5	834	2,12	6,88	3,46	12,46	0,55	0,39	10,30	22,76	1,01	0,90
			1,64	4,00	2,94	8,58	0,38		12,26	20,84	0,93	
			1,68	1,96	1,98	5,62	0,25		11,82	17,44	0,78	
		total	5,44	12,84	8,38	26,66	1,18		34,38	61,04	2,71	

According to the criteria accepted by the MRDPA, for a dosage of 320 kg/m³ and W/C = 0.5, it can be seen that the concretes meet the criterion for classification in exposure classes XF1 and XF3.

■ **For exposure classes XF2, XF4:**

The amount of scaled material was determined on concrete strips of 50x150x150 mm, for a cement dosage of 320 kg/m³ and a W/C ratio = 0.5, concretes prepared with air entraining additive.

The results on the exfoliation of the concrete performed with the slab test method are presented in table 29.

Table 29 - Quantity of scaled concrete after n cycles of freeze-thaw with thawing agents, concrete strips of 50x150x150 mm

Series	7 cycles	14 cycles	28 cycles	Total amount of concrete scaled after 28 cycles	Sn, kg/m ²	Sn, average, kg/m ²	56 cycles	Total amount of concrete scaled after 56 cycles	Sn, kg/m ²	Sn, average, kg/m ²
	amount of scaled concrete, g	amount of scaled concrete, g	amount of scaled concrete, g				amount of scaled concrete, g			
835	1,60	1,64	1,86	5,10	0,23	0,23	11,74	16,84	0,75	0,78
	1,66	1,62	1,92	5,20	0,23		13,44	18,64	0,83	
	1,72	1,64	1,82	5,18	0,23		11,86	17,04	0,76	
total	4,98	4,90	5,60	15,48	0,69		37,04	52,52	2,33	

In accordance with the criteria accepted by the MDRAP for XF2, the concretes meet the criterion for classification in the XF2 exposure class.

In the case of exposure class XF4, the amount of scaled material must be less than 1Kg / m² after 56 freeze-thaw cycles. It is observed that the value obtained after 56 cycles is lower than that corresponding to the European criterion.

Exposure	XF1	XF2	XF3	XF4
Method	Slab test**	Slab test**	Slab test**	Slab test*
CEM III/A-M (CEM 1)	X	X	X	X

X = criterion met,

*) the European criterion

**) criteria proposed by UTCB and endorsed by MDRAP

2.2.2. CEM II/A-M(S-LL) 32.5R - CEM 2

Concrete compositions were prepared using a cement dosage of 320 kg/m³, according to the data shown in Table 30.

Table 30 – Characteristics of fresh concrete

Additive dosage	W/C ratio	Cement dosage (kg/m ³)	Settlement (mm)	Density (kg/m ³)
1% hyperplasticizer	0.50	320	160	2415
1% hyperplasticizer + 0.2% air trainer	0.50	320	170	2402

Table 31 shows the results obtained for the compressive strength of concrete kept in water until the test period.

Table 31 - Strength characteristics of reinforced concrete

W/C ratio	Cement dosage (kg/m³)	Compressive strength (N/mm²)				Concrete class
		2 days		28 days		
0.50	320	22.47	22.61	41.78	41.11	C25/30
		22.62		41.38		
		22.74		40.16		
0.50	320	18.98	18.77	38.12	39.18	C25/30a
		18.55		39.94		
		18.77		39.48		

■ **For exposure classes XF1, XF3:**

The results on the exfoliation of the concrete performed with the slab test method are presented in table 32.

Table 32 - Quantity of scaled concrete after n freeze-thaw cycles, 50x150x150 mm concrete strips

Cement dosage (kg/m³)	W/C	7 cycles	14 cycles	28 cycles	Total amount of concrete scaled after 28 cycles	Sn, kg/m²	Sn, average, kg/m²	56 cycles	Total amount of concrete scaled after 56 cycles	Sn, kg/m²	Sn, average, kg/m²
		amount of scaled concrete, g	amount of scaled concrete, g	amount of scaled concrete, g				amount of scaled concrete, g			
320	0.5	5.64	2.86	1.86	10.36	0.46	0.38	9.74	20.10	0.89	0.89
		5.26	1.86	2.14	9.26	0.41		17.80	27.06	1.20	
		2.02	2.18	1.88	6.08	0.27		6.80	12.88	0.57	
total		12.92	6.90	5.88	25.70	1.14		34.34	60.04	2.67	

According to the criteria proposed by UTCB and accepted by MDRAP, for a dosage of 320 kg/m³ and W/C = 0.5, the concretes meet the criterion for classification in exposure classes XF1 and XF3.

■ **For exposure classes XF2, XF4:**

The amount of scaled material was determined on concrete strips of 50x150x150 mm, for a cement dosage of 320 kg/m³ and a W/C ratio = 0.5, concretes prepared with air entraining additive.

The results on the exfoliation of the concrete performed with the slab test method are presented in table 33.

Table 33 - Quantity of scaled concrete after n cycles of freeze-thaw with thawing agents, concrete strips of 50x150x150 mm

Cement dosage (kg/m³)	W/C	7 cycles	14 cycles	28 cycles	Total amount of concrete scaled after 28 cycles	Sn, kg/m²	Sn, average, kg/m²	56 cycles	Total amount of concrete scaled after 56 cycles	Sn, kg/m²	Sn, average, kg/m²
		amount of scaled concrete, g	amount of scaled concrete, g	amount of scaled concrete, g				amount of scaled concrete, g			
320	0.5	2.86	1.80	1.94	6.60	0.29	0.28	4.20	10.80	0.48	0.61
		3.10	2.04	1.98	7.12	0.32		5.80	12.92	0.57	
		1.76	1.90	1.84	5.50	0.24		11.90	17.40	0.77	
total		7.72	5.74	5.76	19.22	0.85		21.90	41.12	1.83	

Concretes prepared with CEM 2 meet the criteria for classification in classes XF2 and XF4.

Exposure	XF1	XF2	XF3	XF4
Method	Slab test**	Slab test**	Slab test**	Slab test*
CEM II/A-M (CEM 1)	X	X	X	X

X = criterion met,

*) the European criterion

***) criteria proposed by UTCB and endorsed by MDRAP

2.2.3. CEM II/A-S 32,5R – CEM 3

This subchapter presents the results obtained on the performance of concretes prepared with cement type CEM II/A-S 32,5R.

The experimental program consisted in determining the freeze/thaw resistance by determining the amount of scaled material.

Concrete compositions were prepared using 320 kg/m³ cement dosages and two types of additives (superplasticizer additive, base substance: dinaphthylmethane sulfonate and air entraining additive with superplasticizer effect, base substance: sulfonated hydrocarbon), in according to the data presented in Tables 34 and 35.

Table 34 – Concrete compositions prepared with CEM 3

Cement dosage (kg/m ³)	Water (l)	Additive (l)	Aggregate (kg)	Sort 0-4 mm	Sort 4-8 mm	Sort 8-16 mm	Sort 16-32 mm	W/C
1% superplasticizer additive								
320	157,30	2,71	1890,30	661,60	283,54	396,96	548,19	0,50
1,5% air entraining additive with superplasticizer effect								
320	155,57	4,44	1890,30	661,60	283,54	396,96	548,19	0,50

Table 35 – Characteristics of fresh concrete prepared with CEM 3

Cement dosage (kg/m ³)	Additive	W/C	Settlement (mm)	Density (kg/m ³)
320	1% superplasticizer	0,5	190	2352
320	1,5% air entraining	0,5	170	2329

Table 36 shows the results obtained for the compressive strength of concretes prepared with CEM 3 with superplasticizing additive at 2 and 28 days, samples kept in water until the test period.

Table 36 - Strength characteristics of concrete prepared with CEM 3, cement dosage 320 kg/m³, W/C = 0.5

Additive	Compressive strength (N/mm²)				Concrete class obtained
	2 days		28 days		
1% superplasticizer	17,58	17,05	44,34	41,93	C25/30
	16,88		40,29		
	16,70		41,16		

Table 37 presents the results obtained for the compressive strength of concretes prepared with CEM 3 with air entraining additive, at 2 and 28 days, samples kept in water until the test term.

Table 37 - Strength characteristics of concretes with entrained air prepared with CEM 3, cement dosage 320 kg/m³, W/C = 0.5

Additive	Compressive strength (N/mm ²)				Concrete class obtained
	2 days		28 days		
1,5% air entraining additive with superplasticizer effect	11,15	11,29	27,80	27,22	C16/20a
	11,43		25,81		
	11,29		28,06		

■ **For exposure classes XF1, XF3:**

The results on the exfoliation of the concrete performed with the slab test method are presented in table 38.

Table 38 – The amount of concrete scaled after 56 cycles of freeze-thaw

C15	7 cycles	14 cycles	28 cycles	Total amount of concrete scaled after 28 cycles	Sn, kg/m ²	Sn, average, kg/m ²	56 cycles	Total amount of concrete scaled after 56 cycles	Sn, kg/m ²	Sn, average, kg/m ²
Additive	amount of scaled concrete, g	amount of scaled concrete, g	amount of scaled concrete, g				amount of scaled concrete, g			
1% superplastifiant	0,12	0,08	0,20	0,40	0,02	0,03	0,34	0,74	0,03	0,04
	0,28	0,36	0,50	1,14	0,05		0,28	1,42	0,06	
	0,20	0,22	0,12	0,54	0,02		0,18	0,72	0,03	
total	0,60	0,66	0,82	2,08	0,09		0,80	2,88	0,13	

Concretes prepared with CEM 3 meet the criteria for classification in exposure classes XF1 and XF3.

■ **For exposure classes XF2, XF4:**

The results on the exfoliation of the concrete obtained by the slab test method are presented in Table 39.

Table 39 – Quantity of scaled concrete after n cycles of freeze-thaw with thawing agents, concrete strips of 50x150x150 mm

C15	7 cycles	14 cycles	28 cycles	Total amount of concrete scaled after 28 cycles	Sn, kg/m ²	Sn, average, kg/m ²	56 cycles	Total amount of concrete scaled after 56 cycles	Sn, kg/m ²	Sn, average, kg/m ²
Additive	amount of scaled concrete, g	amount of scaled concrete, g	amount of scaled concrete, g				amount of scaled concrete, g			
1,5% air entraining additive with superplasticizer effect	0,58	0,36	0,12	1,06	0,05	0,04	0,42	1,48	0,07	0,06
	0,44	0,36	0,14	0,94	0,04		0,60	1,54	0,07	
	0,18	0,26	0,10	0,54	0,02		0,32	0,86	0,04	
total	1,20	0,98	0,36	2,54	0,11		1,34	3,88	0,17	

Concretes prepared with CEM 3 meet the criteria for classification in classes XF2 and XF4.

Exposure	XF1	XF2	XF3	XF4
Method	Slab test**	Slab test**	Slab test**	Slab test*
CEM II/A-M (CEM 1)	X	X	X	X

X = criterion met,

*) the European criterion

**) criteria proposed by UTCB and endorsed by MDRAP

The Sn values obtained in the case of concretes prepared with Portland cement with CEM II/A-S 32,5R slag are lower than those obtained for concretes prepared with the two Portland cementitious cements CEM II/A-M(S-LL) 32,5R, all falling within evaluation criteria for freeze/thaw resistance by exfoliation.

The results obtained for scaled concrete for the three types of concrete will be further used to propose classifications in freeze/thaw resistance classes.

3. Freeze-thaw resistance classes

3.1. Results obtained in the laboratory

Tests for the determination of freeze/thaw resistance were carried out in accordance with the CEN/TS 12390-9 standard. [3]

Tables 40 (freeze-thaw without de-icing agents) and 41 (freeze-thaw with de-icing agents) show the results recorded for freeze/thaw resistance and classification into freeze/thaw resistance classes by types of cement.

Table 40 - Results recorded for freeze/thaw resistance and classification in freeze/thaw resistance classes by types of cement

Cement type	Cement dosage (kg/m ³)	W/C	Compressive strength (N/mm ²)	Sn, kg/m ² (56 cycles)	Freeze/thaw resistance classes
CEM II/A-S 32,5R - CEM 3	320	0,5	41,93	0,04	RXF0,2
CEM II/A-M (S-LL) 32,5R - CEM 1	320	0,5	39,12	0,90	RXF1
CEM II/A-M (S-LL) 32,5R - CEM 2	320	0,5	41,11	0,89	RXF1

Similar behaviors of concrete prepared with CEM 1 and CEM 2 are observed.

Table 41 - Results recorded for freeze/thaw resistance and de-icing agents and classification of freeze/thaw resistance classes by type of cement

Cement type	Cement dosage (kg/m ³)	W/C	Compressive strength (N/mm ²)	Sn, kg/m ² (56 cycles)	Freeze/thaw resistance classes
CEM II/A-S 32,5R - CEM 3	320	0,5	27,22	0,06	RXF0,2
CEM II/A-M (S-LL) 32,5R - CEM 1	320	0,5	38,73	0,78	RXF1
CEM II/A-M (S-LL) 32,5R - CEM 2	320	0,5	39,18	0,61	RXF1

Even until the current introduction of environmental resistance classes, these tests are useful to be able to recommend / promote certain cements with different applications.

Existing variants for the classification of freeze/thaw resistance according to exposure classes are presented below.

3.2. Framing results obtained in existing evaluation criteria in the EU

The criteria for assessing the exfoliation strength of concrete after freeze-thaw cycles in the presence of 3% NaCl, according to the Boras method presented in the Swedish standard SS 137244 [11] are:

- Very good concrete: $m_{56} < 0,1 \text{ kg/m}^2$
- Good concrete: $m_{56} < 0,2 \text{ kg/m}^2$ or
 $m_{56} < 0,5 \text{ kg/m}^2$ and $m_{56}/m_{28} < 2$ sau
 $m_{112} < 0,5 \text{ kg/m}^2$
- Acceptable concrete: $m_{56} < 1 \text{ kg/m}^2$ and $m_{56}/m_{28} < 2$ sau
 $m_{112} < 1 \text{ kg/m}^2$
- Unacceptable concrete: $m_{56} > 1 \text{ kg/m}^2$ and $m_{56}/m_{28} > 2$ sau
 $m_{112} > 1 \text{ kg/m}^2$

In accordance with the criteria set out above, Tables 42 and 43 show the classification of the concrete according to the results obtained experimentally.

Table 42 - Evaluation of the strength of concrete after freeze-thaw cycles

Cement type	m ₂₈ , kg/m ²	m ₅₆ , kg/m ²	Evaluation criteria
CEM II/A-M (S-LL) 32,5R - CEM 1	0,39	0,90	Acceptable concrete
CEM II/A-M (S-LL) 32,5R - CEM 2	0,38	0,89	Acceptable concrete
CEM II/A-S 32,5R - CEM 3	0,03	0,04	Very good concrete

Table 43 - Evaluation of concrete strength after freeze-thaw cycles with de-icing agents

Cement type	m ₂₈ , kg/m ²	m ₅₆ , kg/m ²	Evaluation criteria
CEM II/A-M (S-LL) 32,5R - CEM 1	0,23	0,78	Acceptable concrete
CEM II/A-M (S-LL) 32,5R - CEM 2	0,28	0,61	Acceptable concrete
CEM II/A-S 32,5R - CEM 3	0,04	0,06	Very good concrete

The ratio m_{56} / m_{28} is higher than 2 for concretes prepared with CEM II/A-M, with or without thawing agents, this aspect leads to the idea that for concretes prepared with CEM II/A-M a higher quantity is scaled between 28 and 56 cycles compared to CEM II/A-S.

Table 44 presents a variant of the freeze/thaw resistance classification, including the classification in the specific exposure classes, according to the classification presented in Table 10. The definitions of moderate / severe climates are still under discussion.

Table 44 - Classification of concrete in freeze-thaw exposure classes

Exposure Class	Freeze/thaw resistance classes	
	Moderate frost climate	Severe frost climate
XF1	RF1	RF1
	II/A-S 32,5R - CEM 3	II/A-S 32,5R - CEM 3
	II/A-M (S-LL) 32,5R - CEM 1	II/A-M (S-LL) 32,5R - CEM 1
	II/A-M (S-LL) 32,5R - CEM 2	II/A-M (S-LL) 32,5R - CEM 2
XF2	RF1	RF0,2
	II/A-S 32,5R - CEM 3	II/A-S 32,5R - CEM 3
	II/A-M (S-LL) 32,5R - CEM 1	
	II/A-M (S-LL) 32,5R - CEM 2	
XF3	RF1	RF0,2
	II/A-S 32,5R - CEM 3	II/A-S 32,5R - CEM 3
	II/A-M (S-LL) 32,5R - CEM 1	
	II/A-M (S-LL) 32,5R - CEM 2	
XF4	RF0,2	RF0,2
	II/A-S 32,5R - CEM 3	II/A-S 32,5R - CEM 3

The concretes prepared with the studied cements can be classified according to table 11 in the classes presented in table 45.

Table 45 - Classification of concretes in freeze/thaw resistance classes

Exposure Class	Minimum accepted resistance class	
	Severe frost climate	Moderate frost climate
XF1	RF1	RF1
	II/A-S 32,5R - CEM 3	II/A-S 32,5R - CEM 3
	II/A-M (S-LL) 32,5R - CEM 1	II/A-M (S-LL) 32,5R - CEM 1
	II/A-M (S-LL) 32,5R - CEM 2	II/A-M (S-LL) 32,5R - CEM 2
XF2	RF0,5	RF1
	II/A-S 32,5R - CEM 3	II/A-S 32,5R - CEM 3
		II/A-M (S-LL) 32,5R - CEM 1
		II/A-M (S-LL) 32,5R - CEM 2
XF3	RF0,5	RF1
	II/A-S 32,5R - CEM 3	II/A-S 32,5R - CEM 3
		II/A-M (S-LL) 32,5R - CEM 1
		II/A-M (S-LL) 32,5R - CEM 2
XF4	RF0,2	RF0,5
	II/A-S 32,5R - CEM 3	II/A-S 32,5R - CEM 3

Using the proposal to complete EC2 in Table 9 we can formulate evaluation criteria for concrete exposed to freeze/thaw presented in Tables 46 and 47.

Table 46 - Evaluation criteria for concrete exposed to freeze-thaw,
samples kept in water

	Exposure Class	
	XF3	XF4
Mild winters ¹ Moderate winters and L<100	RFW L*	RFD L*
	$m_{56} < 2,0 \text{ kg / m}^2$	$m_{56} < 2,0 \text{ kg / m}^2$
	II/A-M (S-LL) 32,5R - CEM 1 II/A-M (S-LL) 32,5R - CEM 2 II/A-S 32,5R - CEM 3	II/A-M (S-LL) 32,5R - CEM 1 II/A-M (S-LL) 32,5R - CEM 2 II/A-S 32,5R - CEM 3
Moderate winters ² and L≥100 Severe winters ³ and L<100	RFW M	RFD M
	$m_{56} < 1,0 \text{ kg / m}^2$	$m_{56} < 1,0 \text{ kg / m}^2$
	II/A-M (S-LL) 32,5R - CEM 1 II/A-M (S-LL) 32,5R - CEM 2 II/A-S 32,5R - CEM 3	II/A-M (S-LL) 32,5R - CEM 1 II/A-M (S-LL) 32,5R - CEM 2 II/A-S 32,5R - CEM 3
Severe winters ³ and L≥100	RFW H	RFD H
	$m_{56} < 0,5 \text{ kg / m}^2$	$m_{56} < 0,5 \text{ kg / m}^2$
	II/A-S 32,5R - CEM 3	II/A-S 32,5R - CEM 3
¹ Few frost cycles per year, temperatures rarely below -5 ° C. ² Several cycles of frost per year, temperatures rarely below -10 ° C. ³ Many frost cycles per year, temperatures occasionally below -20 ° C. L = projected service life		

*) The ratio $m_{56}/m_{28} < 2$ is an additional proposal for RFW L and RFD L. and in this case, $m_{56}/m_{28} < 2$ for CEM II/A-S and $m_{56}/m_{28} > 2$ for CEM II/A-M.

Table 47 - Evaluation criteria for concrete exposed to freeze-thaw
with defrosting agents

Cement type	m_{56} , kg/m ²	Evaluation criteria
CEM II/A-M (S-LL) 32,5R - CEM 1	0,78	RFW L, RFD L RFW M, RFD M
CEM II/A-M (S-LL) 32,5R - CEM 2	0,61	RFW L, RFD L RFW M, RFD M
CEM II/A-S 32,5R - CEM 3	0,06	RFW H, RFD H

Exposure Class	Amount of scaled concrete, kg/m ²		
	Projected lifetime, years		
	50	100	200
XF1	$m_{56} \leq 0,5$	$m_{56} \leq 0,2$	$m_{56} \leq 0,1$
XF3	$m_{56} \leq 0,2$	$m_{56} \leq 0,1$	$m_{112} \leq 0,1$
XF2, agenti	$m_{56} \leq 0,5$	$m_{56} \leq 0,2$	$m_{56} \leq 0,1$
XF4, agenti	$m_{56} \leq 0,2$	$m_{56} \leq 0,1$	$m_{112} \leq 0,1$

According to the proposal to supplement EC2 presented in Table 8, concretes prepared with the three types of cements can be classified into freeze/thaw resistance classes depending on the exposure classes.

Table 49 - Resistance classes allowed for XF exposure classes

Exposure Class	Minimum freeze/thaw resistance classes allowed	
	Severe frost climate	Light frost climate ¹
XF1	CEM II/A-M (S-LL) 32,5R - CEM 1 CEM II/A-M (S-LL) 32,5R - CEM 2 CEM II/A-S 32,5R - CEM 3	CEM II/A-M (S-LL) 32,5R - CEM 1 CEM II/A-M (S-LL) 32,5R - CEM 2 CEM II/A-S 32,5R - CEM 3
XF2	CEM II/A-S 32,5R - CEM 3	CEM II/A-M (S-LL) 32,5R - CEM 1 CEM II/A-M (S-LL) 32,5R - CEM 2 CEM II/A-S 32,5R - CEM 3
XF3	CEM II/A-S 32,5R - CEM 3	CEM II/A-M (S-LL) 32,5R - CEM 1 CEM II/A-M (S-LL) 32,5R - CEM 2 CEM II/A-S 32,5R - CEM 3
XF4	CEM II/A-S 32,5R - CEM 3	CEM II/A-M (S-LL) 32,5R - CEM 1 CEM II/A-M (S-LL) 32,5R - CEM 2 CEM II/A-S 32,5R - CEM 3
¹ The mild frost climate can be defined in locally valid provisions, based on the zonal climate, regarding frost cycles and extreme temperatures		

Proposals can be made for life depending on the exposure class and the amount of scaled concrete.[22]

Using the experimental results obtained, CEM II/A-S can be used in all exposure classes for lifetimes of 50 and 100 years. For a service life of 200 years, determinations of the amount of scaled concrete after 112 freeze-thaw cycles are required for exposure classes XF3 and XF4.

The test results allow to obtain recommendations regarding the correct design of an exfoliation-resistant concrete. The durability of concrete exposed to freeze-thaw in the presence of defrost salts can be ensured by a low water/cement ratio.

The test results confirm that the freeze-thaw resistance of concrete depends on several factors, which determine the structure of the concrete. Exfoliation of the concrete surface is a very complicated phenomenon due to the large number of independent factors that produce it.

4. Conclusions

The inclusion of concrete in freeze/thaw resistance classes using the results of European test methods and the criteria proposed in the technical committees for the development of European standards in the field, is the novelty of this research report.

The fact that the freeze/thaw resistance decreases as the percentage of cement additives increases is known and accepted, this aspect being reflected in various national rules for the application of EN 206 [1]. Part of this decrease in strength can be compensated by the appropriate choice of concrete composition (a high grade of concrete, the use of air entraining additives and ensuring a low water/cement ratio), as well as by a correct installation associated with an efficient and sufficient treatment.

The resistance to freeze/thaw attack is substantially improved if the following generally valid conditions are met: the use of a low water/cement ratio, the use of an appropriate cement dosage, the choice of a cement class according to the strength of the concrete, the use of a addition of air trainer to the preparation of the concrete, efficient and sufficient treatment of the

concrete, maintenance of the concrete for as long as possible in dry air before being exposed to freeze-thaw.

Certain researches carried out "in situ" and in the laboratory have shown a behavior, in general, corresponding to the attack given by the freeze-thaw of the cements with added slag, in different classes of exposure to this attack. Important are the correct choice of the concrete composition (grade, water/cement ratio, etc.), a good application and treatment of the concrete, as well as the type and dosage of cement additives.

The negative effects of freeze-thaw on Portland cement composites CEM II/A-M composites (of course depending on the M components) may be similar or even more pronounced than the effects produced on Portland cement composites with CEM II/A-S slag, for example.

The experiments performed comparatively show the behavior of CEM II/A-M composite Portland cements (S-LL) compared to a CEM II/A-S "reference" cement. In this situation, of the testing of some "candidate" cements for freeze-thaw, the comparative approach, using relative criteria, must be associated with the approach based on performance criteria. Comparison with the candidate cement is part of the performance approach. The existence of assessment methods / criteria leads to the need to introduce durability classes as a unitary assessment system.

Laboratory tests were performed in accordance with the standards in force at the time of the determinations. The application of European methods and the interpretation of results based on the criteria specified in the proposals for improving European standards is a study direction for defining areas of use of cements studied at different levels of intensity of the freeze-thaw action.

The research program consisted in determining the freeze/thaw resistance on concrete samples, in accordance with the Romanian standard SR 3518 [10], of the norm NE012-1 [2] and respectively of the European standard CEN/TS 12390-9 [3] by the slab test method and the use of the results obtained in the classification of concretes into freeze/thaw resistance classes.

Determination of freeze/thaw resistance by Romanian norme

Concretes prepared with CEM 1 and CEM 3 falling into the same concrete class C40 / 50 have similar values after 150 freeze-thaw cycles with de-icing agents (Table 25).

Concrete prepared with the three types of cements had lower strength losses than the value required by SR 3518 [10] (25%).

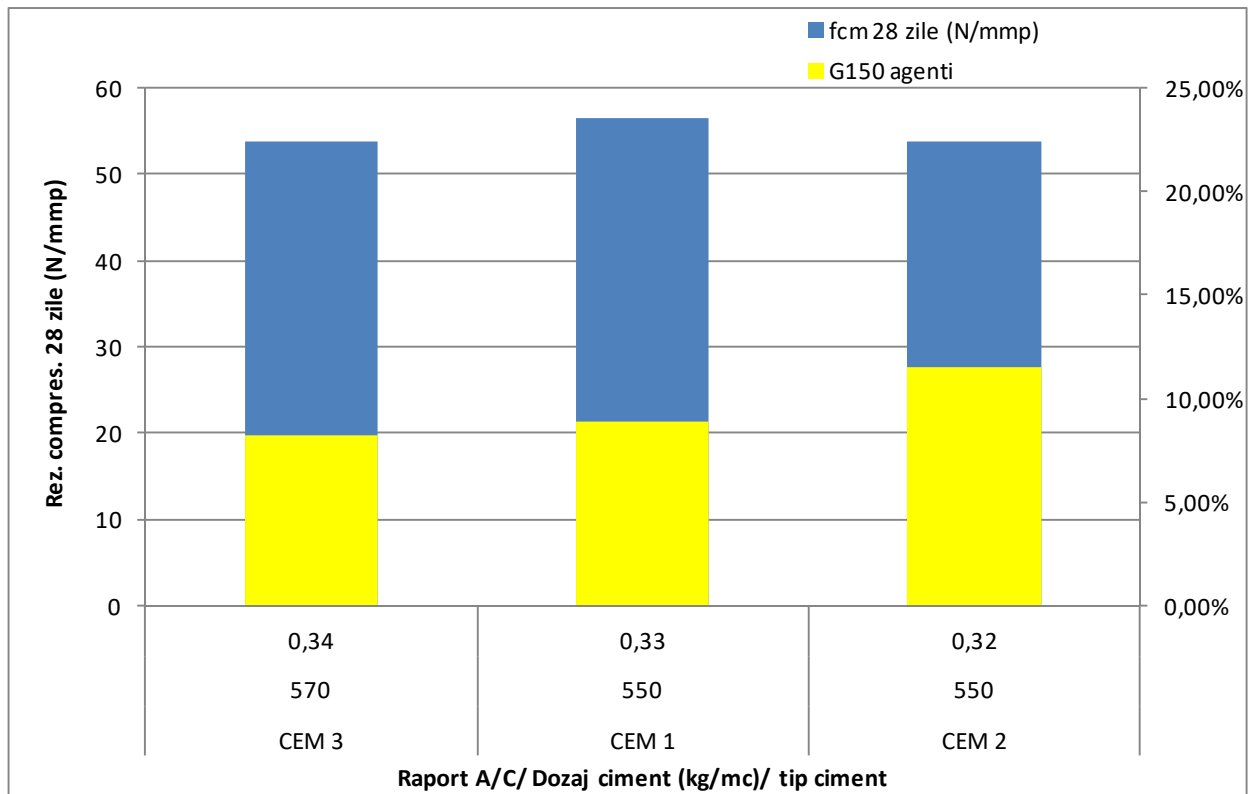


Fig. 4 – Comparison of resistance losses fcm 28 days / G150 with defrosting agents

It is also possible to emphasize the importance of the hyperplasticizing additive used in the preparation of concretes with CEM 1 and CEM 2 for the reduction of the water / cement ratio and implicitly the increase of the compressive strength.

Determination of freeze/thaw resistance by European standards

In order to be able to compare more effectively the prepared concretes with the three types of cements, the same compositions were used, cement dosage 320 kg/m³ and W/C = 0.5, the tests being carried out in accordance with CEN / TS 12390-9. [3]

The compressive strengths obtained for the three types of concrete are different. The strengths of concretes prepared with CEM II/A-S (CEM 3) are higher than those obtained for concretes prepared with CEM II/A-M cements (CEM 1 and CEM 2). The values obtained for CEM 1 and CEM 2 are approximately equal, which is reflected in the values of the amount of scaled concrete, after performing the cycles without thawing agents.

The Sn values obtained in the case of concretes prepared with CEM II/A-S are lower than those obtained for concretes prepared with the two CEM II/A-M cements, all falling within the evaluation criteria for freeze/thaw resistance by exfoliation.

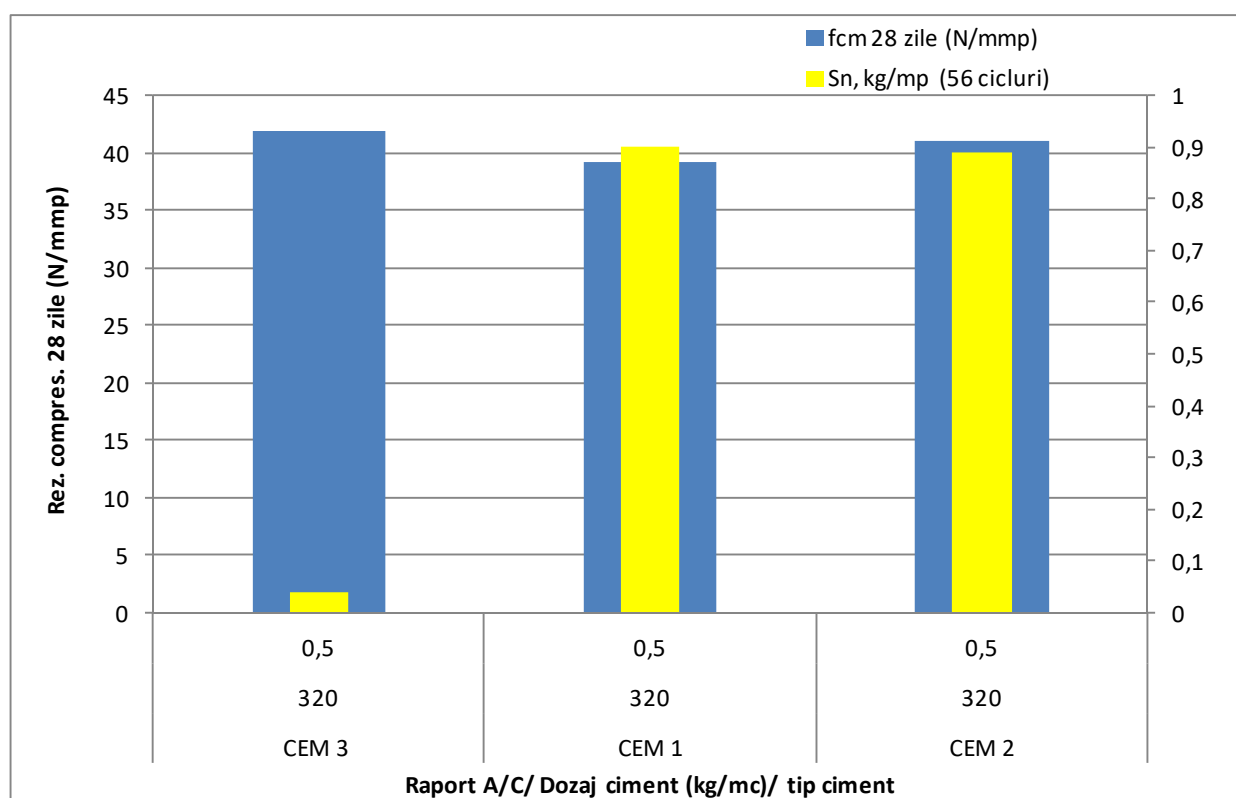


Fig. 5 – Comparison of the masses of the scaled material after 56 freeze-thaw cycles, without thawing agents

For concretes exposed to freeze-thaw with thawing agents, the values for the amount of scaled concrete obtained for CEM 3 are lower than those obtained for CEM 1 and CEM 2 (Tables 38 and 39).

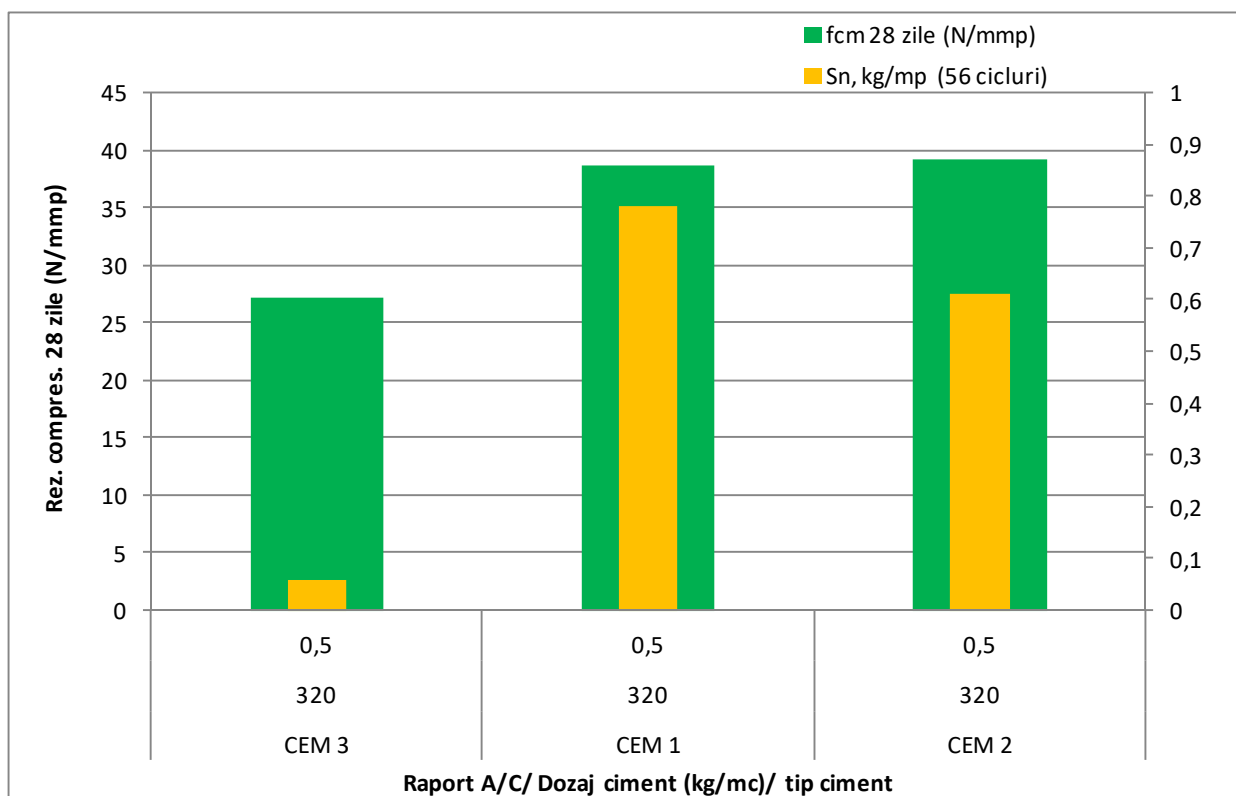


Fig. 6 - Comparison of the masses of the scaled material after 56 cycles of freeze-thaw, with thawing agents

Concretes prepared with the three types of cements meet the criteria for classification in XF exposure classes.

Exposure	XF1	XF2	XF3	XF4
Metod	Slab test**	Slab test**	Slab test**	Slab test*
CEM II/A-M (CEM 1)	X	X	X	X
CEM II/A-M (CEM 2)	X	X	X	X
CEM II/A-S (CEM 3)	X	X	X	X

X = criterion met,

*) the European criterion

**) criteria proposed by UTCB and endorsed by MDRAP

The results obtained for the amount of scaled concrete for the three types of concrete can still be used to propose classifications in freeze/thaw resistance classes.

Regarding the ratio $m_{56}/m_{28} < 2$ which appears in several proposals for completing the rules, in the case of concrete prepared with CEM II/A-M it is higher than 2, this aspect leads to the idea that concrete prepared with CEM II/A-M it exfoliates more between 28 and 56 cycles compared to CEM II/A-S. In this case it is necessary to supplement the tests to determine the amount of scaled concrete of the concrete prepared with CEM II/A-M.

In order to be able to complete the proposals for EN 206 presented in tables 5 and 6, tests must be performed to determine the amount of scaled concrete by the slab test method for concretes with different cement dosages, higher than 280 kg/m^3 , with at least 4% entrained air. The water/cement ratios obtained for different classes of freeze/thaw resistance can also be associated with each type of cement.

Using the experimental results obtained and the performance criteria presented, CEM type II/A-S cement can be used in all exposure classes for lifetimes of 50 and 100 years. For a service life of 200 years, determinations of the amount of scaled concrete after 112 freeze-thaw cycles are required for exposure classes XF3 and XF4.

The test results allow to obtain recommendations regarding the correct design of an exfoliation-resistant concrete.

As a direction of future research, it is necessary to carry out several tests on different concrete mixtures with water/cement ratios between 0.6 and 0.4, in order to observe the classification of concretes prepared with different types of cements in the freeze/thaw resistance classes.

In the absence of the practical existence of the concept of resistance to environmental action (for the time being), these tests are useful in order to promote certain varieties of cement, recommended for use in various special applications.

BIBLIOGRAPHY

1. SR EN 206 +A1:2017, Beton. Specificație, performanță, producție și conformitate;
2. NE 012/1-2007 Normativ pentru producerea betonului and executarea lucrărilor din beton, beton armat and beton precomprimat. Partea 1: Producerea betonului;
3. CEN/TS 12390-9:2016 Testing hardened concrete - Part 9: freeze-thaw resistance with de-icing salts;
4. SR EN 197-1:2011 Ciment - Compoziție, specificații and criterii de conformitate;
5. SR 13510:2006 - Beton. Partea 1: Specificație, performanță, producție and conformitate;
6. Dan Georgescu, Radu Gavrilescu, Abordări moderne pentru asigurarea performanțelor betonului în ceea ce privește durabilitatea – Revista Româna de Materiale 2013;
7. CEN-TC104-SC1-WG1_N0110 - Performance criteria for resistance;
8. CEN/TC 250/SC 2/WG 1 - PT1prEN1992-1-1 2017-10-30 D2;
9. SR EN 1992-1-1:2004 Eurocod 2: Proiectarea structurilor de beton; Partea 1-1: Reguli generale and reguli pentru clădiri;
10. SR 3518:2009, Încercări pe betoane. Determinarea rezistenței la îngheț-dezghet prin măsurarea variației rezistenței la compresiune și/sau modulului de elasticitate dinamic relativ;
11. SS 137244:2005 - Concrete testing - Hardened concrete - Scaling atfreezing (Standard Swedish);
12. Daria Jozwiak-Niedzwiedzka - Scaling resistance of different concretes tested with Boras method;
13. EN 1992-1-1 (2004): Eurocode 2: Design of concrete structures - Part 1-1: General rules and rules for buildings;
14. Steinar Leivestad – Durability, Concrete, Environment and Sustainability în the Eurocodes, 02.05.2019, Standard Norge;
15. CEN 104/SC1/WG1 N55 - European experience with performance testing for durability and the specification of durability by performance;
16. Christoph Müller, VDZ, Latest developments în the standardization of concrete, JRC Side-event to the Standardization Summit - Construction Standards, 3 June Riga;
17. CEN TC250/SC2/WG1/TG10 N0045, Freeze thaw Resistance on concrete – Classification, februarie 2017;
18. Andrei Shpak, Stefan Jacobsen - Requirements and recomandations for frost durable concrete. Test methods. Overview of national and international standards, codes,

- committees, representative projects. WP2 Production and documentation of frost durable concrete: air entrainment, cracking and scaling în performance testing, 2019;
19. CEN/TC250/SC2/WG1 N479;
 20. SR EN 12390-2:2009. Încercare pe beton întărit. Partea 2: Pregătirea and păstrarea epruvetelor pentru încercări de rezistență;
 21. SR EN 12390-3:2009. Încercare pe beton întărit. Partea 3: Rezistența la compresiune a epruvetelor;
 22. Erika Holt, Tuula Råman, Mika Tulimaa, Implementing Environmentally friendly and Durable Concrete to Finnish Practice;
 23. CP 012/1- 2007: Cod de practica pentru producerea betonului;
 24. SR EN 12390-9:2009. Încercare pe beton întărit. Partea 9: Rezistență la îngheț-dezghet. Exfoliere;
 25. UTCB: 2012 - Stabilirea, în funcție de domeniul de utilizare, a cerințelor pentru caracteristicile betonului determinate prin aplicarea standardelor europene armonizate. Metode bazate pe încercări - cercetare (prenormativă) – Preambul;
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