DURABILITATEA UNOR BETOANE PE BAZĂ DE DIFERITE TIPURI DE CIMENT
DURABILITY OF CONCRETE WITH DIFFERENT TYPES OF CEMENT

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Adequate measures must be taken during the determination of the concrete composition for ensuring the durability of concrete exposed to an aggressive environment. The influence of the cement type on the freeze/thaw resistance with and without de-icing salt of concrete was tested. Samples were made using five types of cement. All types of concrete made according to the recommendations of the Serbian and European norms had the required frost resistance. Besides the respected recommendations, concrete made using cement with 35% mineral additions of fly ash and lime was not freeze/thaw resistant with de-icing salt.

Weathering resistance for paving units is determined according to EN 1338 and EN 1339 for freeze/thaw resistance with de-icing salt or water absorption. Taking into account previous investigations, only samples which were made in accordance with the recommendations of the Serbian Building code requirements for concrete and reinforced concrete - BAB 87 and European norms EN 206-1 were tested. Regarding water absorption, it is expected that in the case of established production all types of concrete would be integrated into class 2 according to EN.

Also, for two representative types of concrete with different air content and degree of damage done by destructive test, non-destructive test by device RapidAir 457 was done (specific surface, spacing factor, void frequency and average chord length).

Keywords:*types of cement, concrete durability, freeze/thaw resistance, scaling, spacing factor, weathering resistance*

1. Introduction

Concrete structures deteriorate more rapidly in northern regions because of the cold weather during the winter. It is well known that one of the major causes of the rapid degradation of concrete pavements, bridge decks, parking structures, and similar structures is the widespread use of de-icing salts during the winter. De-icing salt is usually used by spreading it on the surface of concrete roads in the winter for driver safety because it lowers water's freezing point. In these cold weather environments, concrete deteriorates more rapidly because it is subjected to supplementary action of de-icing salts under freezing and thawing cycles. When a de-icing salt is applied on concrete structure under freezing and thawing cycles, scaling, cracking, or other erosive effects are seen on the concrete structures' surface layer [1]. De-icing salts' erosive effect subjects the concrete to more rapid deterioration than the destructive effect of normal freezing and thawing process in concrete. In addition to reducing the serviceability of the structure, surface scaling makes it easier for harmful chemicals ingress into the concrete which increases the risk of deterioration by other phenomena such as corrosion of reinforcing steel [2].

The mechanisms of the implied processes for the scaling of concrete surfaces due to freezing in the presence of de-icing salts are still not fully understood [1, 3]. Possible factors that influence resistance of concrete include: air-void structure, water cementitious materials ratio (W/CM), compressive strength, type and amount of mineral additives (fly ash, slag, etc.), type and amount of chemical admixtures, finishing and curing procedures, aggregate type and granulation, and exposure conditions [1]. The air-void structure is the most important factor with regard to the scaling resistance. Numerous field evidences and laboratory testing results e.g., showed that air entrainment reduces frost-salt scaling damage [4].

Deja [5] found that the effectiveness of air entraining admixture into fresh concrete mix was much lower for slag cement concrete than for OPC concrete; for the same air content the dosage of air entraining admixture was even two times higher for the first concrete than for the later. The results published in [6] showed that concrete mixes using slag cement had a lower number of air voids when compared with OPC concrete. The scaling resistance was considerably reduced when used CEM II/B-S 42.5N and CEM III/A 42.5N-HSR/NA instead of CEM I 42.5R [7].

Numerous laboratory testing data have indicated that when exposed to freezing and thawing cycles in the presence of de-icing salts air-entrained concrete incorporating with high fly ash volume showed an unsatisfactory performance [8, 9].

Freezing - thawing of hardened concrete is among the most important problems of the concrete durability. The results for the mixtures investigated in the study [10] showed that a total air

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content of 4% is adequate to provide frost resistance for mixtures with a w/c between 0.36 and 0.50. The purpose of investigation [11] was to examine the influence of the admixtures on porosity and pores size distribution of SCC at constant water/cement ratio, type and volume of aggregate and volume of cement paste. The purpose of study [12] was to propose a method for the prediction of the minimum curing time required for early-age concrete to prevent frost damage. An experimental investigation was conducted using an air-entraining agent and pozzolans additive such as silica fume (SF) and fly ash (FA), to meet the design strengths of 50 and 60 MPa, as well as frost resistance to 300 cycles of freezing and thawing. It is revealed that, as to concrete at a 0.32 water/binder ratio, air entrainment should be a main approach to enhance frost resistance, although the pozzolans could be used to increase long-term strength of concrete [13]. Paper [14] discusses the influence of the high volumes of fly ash and micro poly-vinyl-alcohol (PVA) fibers on the cyclic freeze–thaw resistance and microstructure of the cementitious composites. Samples of concrete with different water/cement ratios and air contents subjected to freeze/thaw cycles cured at temperature about 80 °C were investigated in [15]. Tests for the frost resistance were made on laboratory cements containing different amounts of belite activated by rapid clinker cooling. If freezing begins after the hydration of the belite cements has nearly been completed these cements will offer better frost resistance than cements rich in alite. This is due to the lower portlandite content and to denser microstructure of the cements containing a high amount of belite [16]. Concretes prepared with CEM II/B-M (S-LL) 32.5 R have different behaviors to freeze-thaw depending of slag and limestone percentages [17].

The chemical composition of cement and the fineness of cement have no effect on its frost resistance except for early-age concrete [18].

Entrained air into concrete refers only to the air that is intentionally entrained into a concrete. This kind of air has bubbles whose diameter ranges from $5x10^{-3}$ to 1.2 mm, while accidentally entrained air can take up much more space.

Besides the minimum volume of air bubbles, which provides frost resistance it is important that the bubbles are uniformly distributed in the cement paste. This depends on the spacing factor (average maximum distance of any point in the cement stone to the edge of the nearest bubble), and if it is less than 0.20 mm complete protection and the durability of concrete against frost with de-icing salt is ensured (according to SRPS U.M1.206).

This paper discusses a special property of concrete: - freeze/thaw resistance with and without de-icing salts. This special property of concrete has a great impact on its durability. Tests for freeze/thaw resistance with and without de-icing salts lasted from 1 to 3 months according to the current Serbian standards [19, 20]. Precious time is lost if the test results fail to meet the required properties for concrete. For this reason it is desirable to carry out preliminary investigation promptly and to select a combination of components for concrete with respect to the given recommendations and to compare the results of non-destructive and destructive tests.

Concretes were designed and tested according to Serbian and European norms [19-25].

2. Experimental work

The samples were made from each batch to test the freeze/thaw resistance (15x15x15 cm), resistance to the effects of de-icing agents and water absorption (15x15x7.5 cm) and for the determination of the micropore air spaces factor (15x15x7.5cm). The samples were made in laboratory conditions. As for the samples tested for the resistance to the effects of de-icing agents, the specimens are preconditioned. Seven days before preparing for testing the specimens were cured in the room at temperature of 20 ± 2°C and 65 ± 5 % RH. Plastic frame was set up and sealed, surface was covered with a 3% NaCl solution and specimens were cured at the same conditions for 7 days. After that, they were subjected to 30 freeze/thaw cycles (onecycle consistedoffreezingat -20 ± 2°Cfor 16hours andthawing at+20 ± 2°Cfor 8hours), according to the SRPS U.M1.055 [19]. The material that has scaled off is collected and weighed and the result is expressed in mg/mm^2. The test results are grouped according to the degree of damage given in Table 1 [19].

Table 1

Mass loss after freeze/thaw test, degree of degradation and criteria

Degree of degradation	Mass loss (mg/mm^2)	Criteria
0	0	resistant
1	0.2	resistant
2	0.5	not resistant
3	1	not resistant

The preparation of samples according to SRPS U.M1.055 was carried out as is shown in Figure 1.

The criterion for estimate the frost resistance according to the SRPS U.M1.016 [20] was that the compressive strength of samples exposed to freezing and thawing cycles must be at least 75% of the reference strength (the samples of equivalentage cured in air at 20°C). The

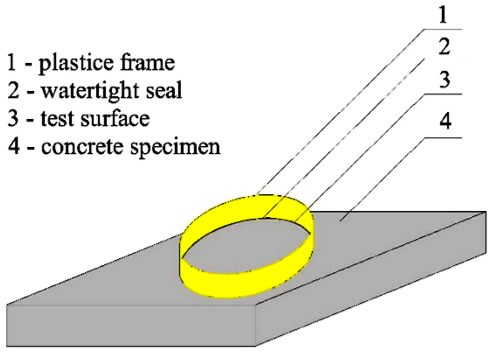

Fig. 1 - The preparation ofsamples according toSRPS.

laboratory conditions in relation to the conditions of the building exploitation are often unrealistic, but they give likely indication for the concrete resistance to frost. One freeze-thaw cycle means that specimens are cured 4h in the water at temperature 20°C and 4h in the freezing chamber at -20°C.

On concrete samples, non-destructive testing has been done according to the recommendation of ASTM C457 standard in order to obtain the results of the total amount of pores, void frequency, average chord length and spacing factor of pores (average maximum distance of any point in the cement stone to the edge of the nearest bubble) in hardened concrete. This US standard [26] describes the complete procedure for this testing and Rapid Air 457 does that automatically.

2.1. Concrete Composition

Component materials that are available in Serbian market were used for this work. Previous trials confirmed their compatibility. Water that was used for mixture preparation was potable water. Five types of cement were used in this work:

- Cement 1: CEM II/ B-M (L-V) 42.5 R, →(Concretes Series **A**)
- Cement 2: CEM I 42.5 R, →(Concretes Series **B**)
- Cement 3: CEM II/A-S 42.5 N →(Concretes Series **C**)
- Cement 4: CEM II/A-M (S-L) 42.5 R →(Concretes Series **D**)
- Cement 5: CEM II/B-M (S-L) 42.5 R, →(Concretes Series **E**)

Certified natural aggregate from river Morava, washed and granulated, with Dmax = 32 mm was used for all concrete series A, B, C, D, E. Admixture “Cementol SPA” (naphtalene sulphonate), which acts as a superplasticizer with strong dispersion action and simultaneously entrains air microbubbles into the concrete, was used (dosage as 1.5% relative to the cement mass).

Mixtures compositions are shown in Table 2.

2.2. Testing results

Fresh concrete was compacted by vibrating needle in container for determination of air content as well as in moulds for making specimens for the testing of specified concrete properties. BAB '87 [21] Art. 32 determines the air content of 3% to 5% for Dmax = 32 mm, for concrete exposed to freeze/thaw during the service life cycle, using an air entraining admixture (being necessary).

SRPS EN 206-1 [22] for the degree of exposure XF3 (environmental conditions that involve high saturation with water without de-icing agents in the case of applying to horizontal concrete surfaces exposed to rain and frost) recommends limit values for composition and properties of concrete which are as follows: the highest W/C = 0.50, minimum strength class C 30/37, minimum quantity of cement 320 kg/m^3, minimum air content in fresh concrete 4% and aggregate in accordance with SRPS EN 12620 with a sufficient freeze-thaw resistance. SRPS EN 206-1 [22] prescribes for the degree of exposure class XF4, max W/C = 0.45, C 30/37, min. cement content C min = 340 kg/m^3, air content ≥ 4%.

Testing was done according to SRPS U.M1.016 [20] at 250 cycles and according to SRPS U.M1.055 [19] at 30 cycles with de-icing salt for preliminary testing of concrete according to BAB '87 [21] in a chamber with automatic tracking of the given regime. All tests presented in this paper were made with the same content of cement of 380 kg/m^3. Five types of cement available on the Serbian market were used. Reference concrete without admixtures, was made with W/C = 0.45. Consistency determined by slump test ranged from 2.5 to 5.0 cm (Class S1-S2). Air content ranged from 1.6 to 2%.

The corresponding concretes with admixtures (mean dosage in the recommended

Table 2

Concrete mixture composition

Components materials	Concrete series											
	Series **A**		Series **B**		Series **C**				Series **D**		Series **E**	
	A1	A2	B1	B2	C1	Z2	C2	X2	D1	D2	E1	E2
Cement, (kg/m^3)	380	380	380	380	380	380	380	380	380	380	380	380
Aggregate, (kg/m^3)	1838	1763	1828	1742	1828	1812	1754	1754	1828	1800	1818	1763
Water,(kg/m^3)	172	151	172	152	172	142	153	151	172	154	172	151
Admixture 1.5%, (kg/m^3)	-	5.7	-	5.7	-	5.7	5.7	5.7	-	5.7	-	5.7
Density, (kg/m^3)	2390	2300	2380	2280	2380	2340	2290	2290	2380	2340	2370	2300
W/C	0.45	0.40	0.45	0.40	0.45	0.37	0.40	0.40	0.45	0.40	0.45	0.40

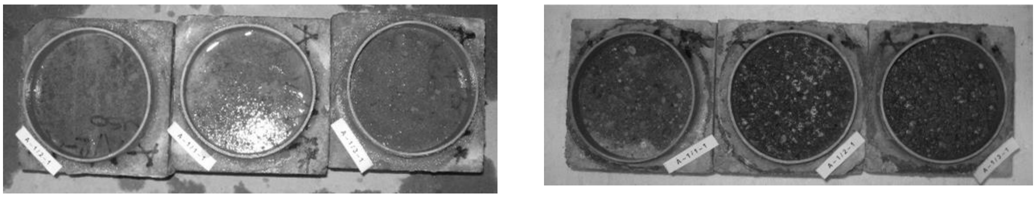

Fig. 2 - The appearance of the samples before and after testing (Concrete Series **A**-A1/1-3).

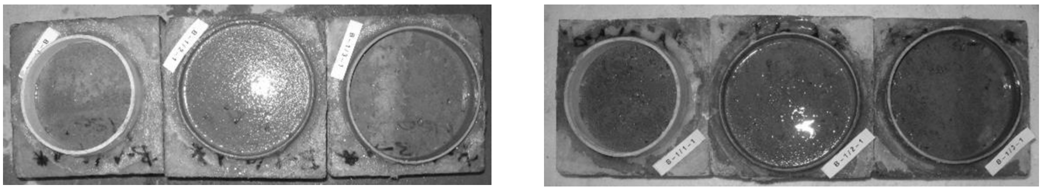

Fig. 3 -The appearance of the samples before and after testing (Concrete Series **B**-B1/1-3)

limits determined by the manufacturer) were designed to maintain consistency as the reference concrete. These were accomplished with a W/C = 0.40, except Z2 series for which it was W/ C = 0.37. Cement CEM II/A-S 42.5 N has smaller specific surface area (3500 cm^2/g, while other cement types have up to 4500 cm^2/g) and lower content of tricalcium aluminate. Air content ranged from 3.3 to 5.1% which is accordingly to the requirements of BAB '87 [21]. Concrete A2, B2 and E2 series complied with the requirements regarding the content of air according to EN 206-1 (≥ 4%). Fresh and hardened concretes testing was performed as is shown in Table 3. The appearance of the samples before test and after 30 cycles with de-icing salt is shown in Figures 2-3.

According to the SRPS EN1338 and 1339 for paving units, weather resistance is determined by testing in accordance with Annex D for freeze/thaw resistance and de-icing salts or in accordance with Annex E for water absorption and compatibility criteria [23, 24].

Only concrete mixtures which were carried out in accordance with the recommendations of BAB87 for concrete exposed to frost an dde-icing salts(air content 3.0 -5.0%) and with EN 206-1 for the degree of exposure XF4 from previous investigation were tested (A2, B2, X2 and E2), except concrete Z2 . For degree of exposure X4 recommends limits values are: w/c≤0.45, minimum strength classC 30/37, cement content ≥ 340kg/m^3 and minimum air content 4.0% [22].

According to SRPS B.B8.010, for water absorption there are no criteria for evaluation [25]. According to SRPSU.M1.055, concrete is resistant if it has the degree of damage 0 or 1, as is given in Table 1 [19]. Testing was done according to SRPS U.M1.055 at 30 cycles for preliminary testing of concrete.

Also, water absorption and freeze/thaw resistance with de-icing salt at 28 cycles was testing according to EN 1338 and EN 1339.

Experimental results are given in Table 3.

According to SRPS U.M1.055 concrete marked A, manufactured with CEM II/ B-M (L-V) 42.5 R showed the worst results and was not resistant to freezing and thawing with de-icing salt. Concrete without admixture has mass loss more than 1 mg/mm^2 (degree damage “3”) and with admixture 0.5 mg/mm^2 (degree damage “2”). These results are in agreement with the research of durability of concrete made with high-volume fly ash blended cement [8, 9].

Concrete marked E, manufactured with CEM II/B-M (S-L) 42.5 R was resistant to freezing and thawing with de-icing salt, but both types, with and without admixture, had mass loss up to 0.2 mg/mm^2 (degree damage “1”). Concrete made without admixture and CEM II/A-S 42.5 N showed the same results. Other concrete types did not have scaling.

All the series of samples tested according to SRPS U.M1.016 achieved the class of frost resistance M-250. This means that after 250 freezing and thawing cycles compressive strength of specimens was greater than 75% of thestrength of the samples of equivalentage cured in air at 20°C.

According to SRPS B.B8.010, water absorption varied between 3.21% and 4.89%.

All tested concretes, which were made according to the recommendations of EN 206-1 had water absorption lesser than 6%, according to EN 1338 and 1339 and satisfied the requirements of those standards. Only the concrete manufactured with CEM II/ B-M (L-V) 42.5 R had mass loss greater than 1 kg/m^2 and not satisfy the conditions for the production of concrete paving units in cold climate.

Concrete resistance to frost action with and without the presence of de-icing salt (agent) is defined by many standards. Non destructive testing is most often done by measuring the quantity, spacing factors and other properties of pores in the hardened concrete. Two representative types of concrete (A1 and B2) with different air content

Table 3

Experimental results

Concrete testing	Concrete series											
	Series **A**		Series **B**		Series **C**				Series **D**		Series **E**	
	A1	A2	B1	B2	C1	Z2	C2	X2	D1	D2	E1	E2
Water reduction, (%)	-	12.2	-	11.6	-	17.4	11.0	12.2	-	10.5	-	12.2
Air content, (%)	1.6	4.2	1.8	5.0	1.8	3.4	5.2	5.0	1.8	3.3	2.0	4.5
Consistency, (cm)	2.5	2.5	3.5	3.5	4.5	4.5	14.5	13.5	2.5	2.5	5.0	5.0
Degree of damage according to [19]	**3**	**2**	**0**	**0**	**1**	**0**	**0**	**0**	**0**	**0**	**1**	**1**
Class of frost resistance according to [20]	**M-250**	**M-250**	**M-250**	**M-250**	**M-250**	**M-250**	**M-250**	**M-250**	**M-250**	**M-250**	**M-250**	**M-250**
Water absorption according to [25]**, (%)**	**4.89**	**4.87**	**4.58**	**4.26**	**4.70**	**4.22**	**-**	**3.21**	**-**	**-**	**4.86**	**4.64**
Mass loss after freeze/thaw test according to [23, 24],**(kg/m^2)**	**-**	**>1.0**	**-**	**≤1.0**	**-**	**≤1.0**	**-**	**≤1.0**	**-**	**-**	**-**	**≤1.0**
Water absorption according to [23, 24]**, (%)**	**-**	**≤6.0**	**-**	**≤6.0**	**-**	**≤6.0**	**-**	**≤6.0**	**-**	**-**	**-**	**≤6.0**

Table 4

Experimental results by device RapidAir 457

Only chords from 30 to 4000 microns are included in results		
	A1	B2
Air Content (%):	1.98	5.21
Specific Surface (mm^{-1}):	13.01	27.48
Spacing Factor (mm):	0.583	0.175
Void Frequency (mm^{-1}):	0.064	0.358
Average Chord Length (mm):	0.307	0.146
Paste to Air Ratio:	15.00	5.39

and degree of damage done by destructive test (3 and 0 respectively) were tested. The aim of this test was to compare testing method with device Rapid Air 457 and classic tests performed on concrete. The results are given in Figures 4 and 5 and Table 4.

Concrete B2, with admixture had finer pores (two times smaller average chord length and two times greater specific surface) than concrete A1 made without admixture.

Spacing factor of pores (average maximum distance of any point in the cement stone to the edge of the nearest bubble) for concrete with admixture is 0.146, while for concrete without admixture it is 0.307. According to SRPS U.M1.206 (national annex of EN 206-1) concrete is resistant to frost action with de-icing salts if spacing factor is less than 0.20 mm [27]. These results confirm with the destructive test according to SRPS U.M1.055, where concrete B2 was resistant to freezing and thawing with de-icing salts (degree of damage “0”), while concrete A1 had the worst results of all tested concrete (degree of damage “3”) as shown in Table 3.

The amount of pores is determined by the size of the pores. Based on the amount of pore diagrams of the distribution of pores in the samples were made (Figures 4-5).

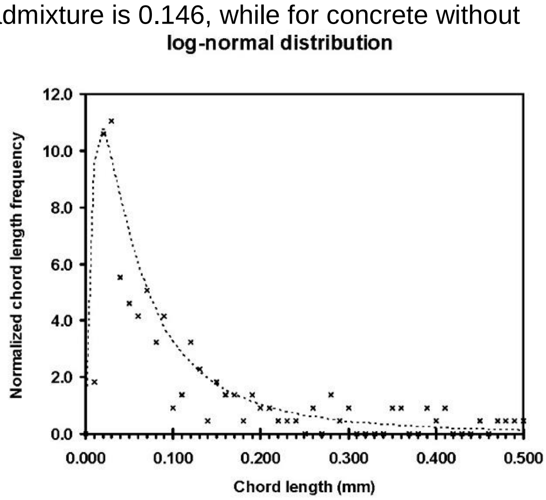

Fig. 4- Chord length distribution for concrete A1.

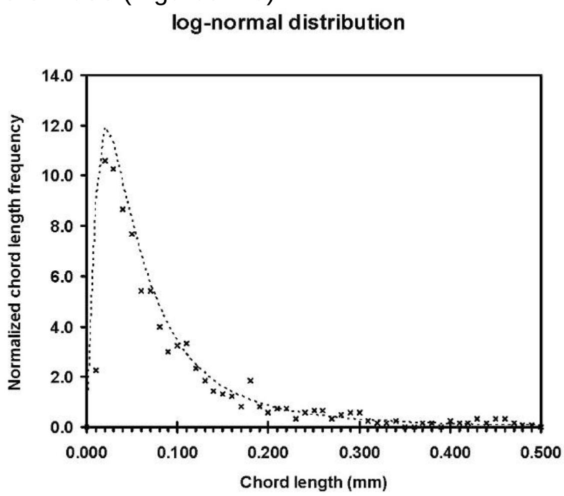

Fig. 5- Chord length distribution for concrete B2.

3. Conclusions

According to SRPSU.M1.055 concrete with CEM II/ B-M (L-V) 42.5 R showed the worst results and was not resistant to freezing and thawing with de-icing salt. Concrete with CEM II/B-M (S-L) 42.5 R was resistant to freezing and thawing with de-icing salt, but both types, with and without admixture had mass loss up to 0.2 mg/mm². Concrete made without admixture and CEM II/A-S 42.5 N showed the same results. Other concrete types did not present scaling and were resistant to freezing and thawing with de-icing salts.

It is certain that the air content and the degree of damage would be different for some other required consistencies (whether they are achieved by changing W/C or the admixture content) with the same amount of cement and the same component materials. Attention should also be paid to the concrete mixing time as well as to the duration of the vibration. Nevertheless, with regard to the situation in many concrete factories, maintaining the W/C according to the dosage is very difficult, which should be especially taken care of.

Besides the respected recommendations, concrete made using cement with 35% fly ash and lime - CEM II/ B-M (L-V) 42.5 R was not freeze/thaw resistant with de-icing salt.

All the series of samples tested according to SRPS U.M1.016 achieved the class of frost resistance M-250 i.e. after 250 freezing and thawing cycles compressive strength of specimens was greater than 75% of the strength of the samples of equivalent age cured in air at 20°C.

All samples were well compacted. In terms of air content, all series with admixture have met the requirements of BAB ′87. The requirements of SRPS EN 206-1 regarding the strength class, cement content and w/c ratio have also been met in all these concrete types, and the air content in the types A2, B2, C2, and E2.

The regimes for freeze/thaw resistance and de-icing salts, as well as the method of sample preparation varied according to SRPS U.M1.055 and EN 1338 and EN 1339. Therefore, without comparativetests it is not possible to classify the concrete according to European standards. In this case, respected recommendations of PBAB 87 and EN 206-1, for concrete exposed to freeze/thaw with de-icing salts, only specimens made with CEM II/B-M (L-V) 42.5 R not satisfied class 3 for weather resistance according to EN 1338 and EN 1339. Also, that specimen was not resistant according to Serbian standard.

Results of concrete resistance at freezing and thawing with de-icing salts, according to SRPSU.M1.055 could be represent the basis for choosing type of cement, but testing concrete according to European standards are necessary for production concrete elements which could satisfied class 3 for weather resistance.

Regarding water absorption (≤6.0% according to EN1338 and EN 1339), it is expected that in the case of established production concrete with cements: CEM II/ B-M (L-V) 42.5 R, CEM I 42.5 R, CEM II/A-S 42.5 N and CEM II/B-M (S-L) 42.5 R would be class 2 for weather resistance according to the same standards.

Measurements taken on the Rapid Air 457 in hardened concrete are close to the total amount of air in fresh concrete.

Assessment after investigation with Rapid Air 457, based on size and spacing factor of pores, is that concrete A1 will be not resistant, while concrete B2 will be resistant to frost in the presence of salt.

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23. xxx, EN 1338:2003, Concrete paving blocks – Requirements and test methods, CEN.
24. xxx, EN 1339:2003, Concrete paving flags – Requirements and test methods, CEN.
25. xxx, SRPS B.B8.010:1980, Testing of natural stone. Absorbtion of water.
26. xxx, ASTM C 457, Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete.
27. xxx, SRPS U.M1.206:2013, Concrete — Specification, performance, production and conformity — Rules for the implementation of SRPS EN 206-1.

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MANIFESTĂRI ŞTIINŢIFICE / SCIENTIFIC EVENTS

CONSILOX 12th –International Hotel-Sinaia, 16-20.09.2016

According to a beautiful tradition, every four years The Conference on Science and Engineering of Oxide Materials – CONSILOX is organized.

Scientific event of high prestige CONSILOX -12 will be hosted by the International Hotel from Sinaia, in the period 16 - 20.09.2016, the place where will meet specialists from university education, centers of research-development-innovation and from production of oxide materials from Romania and from countries with tradition in this range.

General theme of CONSILOX 12 is **“Science of oxide materials in service of durable development”**.

Within this general thematic traditional themes of the Conference will be focused on:

- Fundamentals of science and engineering of oxide materials (physical-chemistry, mechanisms, processes and operations, modelling of properties)
- Inorganic binders
- Glass and vitreous-ceramics
- Traditional and advanced functional and structural ceramics
- Refractory and thermal insulating materials
- Concretes, dry mortars, adhesives for ceramic, and others
- Bio and nano-materials, nano-technologies
- Environment protection in oxide materials industry (including strategies and public politics; assessment of life cycle)

CONSILOX - 12 will have plenary sessions and posters presentation. Also an exhibition will be organized, where sponsors and interested companies may present their own stands with products, research equipment and related information.

Scientific Committee, President:

- **Ecaterina Andronescu** –University POLITEHNICA of Bucharest, Romania

Organization Committee, Executive President:

- **Doru Vladimir Puşcaşu** – CEPROCIM S.A.

Conference Secretary will operate within CEPROCIM S.A. at the following address:

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