The Assessment of Prediction Methodology of Concrete Freezing and Thawing Resistance

Mindaugas DAUKŠYS^{1*}, Ernestas IVANAUSKAS², Svajūnas JUOČIŪNAS¹, Darius PUPEIKIS³, Lina ŠEDUIKYTĖ⁴

¹Department of Civil Engineering Technologies, Kaunas University of Technology,

Studentų str. 48, LT-51367 Kaunas, Lithuania

² Building Materials and Structures Research Centre, Kaunas University of Technology, Studentu str. 48, LT-51367 Kaunas, Lithuania

³ Department of Building Materials, Kaunas University of Technology, Studentų str. 48, LT-51367 Kaunas, Lithuania

⁴ Department of Graphics Drawing, Kaunas University of Technology, Studenty str. 48, LT-51367 Kaunas, Lithuania

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Investigation was performed in order to determine whether the predicted frost resistance of concrete in cycles, while using porosity parameters, corresponds to the number of cycles determined experimentally. The porosity parameters of concrete were determined by measuring kinetics of water absorption. The number of concrete freezing and thawing cycles was predicted by a method proposed by Sheikin, according to porosity parameters of concrete. Frost resistance of concrete was determined by using volumetric method of freezing. It was found, that depending on kinetics of water absorption and composition of concrete, closed porosity of concrete varies within the limits from 3.51% to 10.62%, open porosity of concrete varies within the limits from 10.96% to 15.09%, and predicted number of frost resistance of concrete in cycles varies within the limits from 550 to 1000. After volumetric freezing of concrete samples, according to predicted number freezing and thawing cycles, it can be stated, that concrete with fine aggregate did pass predicted number of cycles, but concrete with coarse aggregate did not. In this case, predicted criterion K_F of concrete of freezing and thawing resistance proposed by Sheikin, should be corrected by supplementing it with additional coefficient. The coefficient could be related to the concentration of fine aggregate from the total amount of aggregate.

Keywords: Portland cement, water absorption, porosity parameter of concrete, concrete freezing and thawing resistance prediction.

1. INTRODUCTION

Concrete, as a hardened mixture from binding agents, aggregates, water and additives, is a composite material with complex structure. The structure of concrete has a significant influence on the frost resistance of concrete.

The freezing-thawing resistance of concrete is mostly affected by the capillary porosity and air content in the concrete mixture. These parameters can be controlled in the concrete production process. The factor of concrete freezing-thawing resistance can be estimated from capillary porosity and air content in the concrete. The model of structure of hardened cement paste and concrete is evaluated and used for porosity prediction. The model is based on cement hydration processes, materials proportions and hydration time [1-3].

According to the authors [4], the concrete with bigger course construction has lowest closed porosity and lowest freezing-thawing resistance. With lower coarse aggregate concentration of concrete the average pore size factor λ goes down. Concretes with lower coarse aggregate content have finer pores and better freezing-thawing resistance factor. Pore size uniformity factor α is almost similar in concretes with different coarse aggregate concentration.

Several researches show that frost resistance is a good durability indicator [5-8], mechanisms by which freezing

and thawing are causing damage to concrete are discussed [9-11]. It is important to evaluate damages in concrete caused by freezing, by studding ice formation process in concrete pores. The frost resistance of concrete is affected by its porosity, the presence of water within it, and the environmental conditions [12-19].

The study [20] analyzing freezing and thawing durability of concrete with regard to damages in concrete caused by ice formation in concrete pores showed that frost damage mainly occurs above -10 °C. It means that the freezing rate of concrete pore solution is higher above -10 °C than below -10 °C.

Studies show [21] that ice is a very strong material and its adhesion to hydrophilic materials such as soil or concrete is also very high, it is expected that the infiltration of ice in a moist porous body during freezing will alter its strengths. Ice has both a compressive and a tensile strength under short-term loading, but because ice is viscoelastic, under long-term loading it has none [22]. The presence of dissolved materials is affecting the strength of ice as well as solutes (some of them increase the strength and others decrease it).

The study [23] analyzing characteristics of recycled aggregate concrete (RAC) showed that the main reason why frost resistance of saturated RAC is not satisfying is the high total W/C, which induces higher porosity, lower mechanical characteristics of such concrete as well as the frost resistance of RA themselves.

^{*} Corresponding author. Tel.: +370-37-300479; fax.: +370-37-300480. E-mail address: *mindaugas.dauksys@ktu.lt* (M. Daukšys)

The following methods are used to study changes in concrete characteristics during freezing and thawing cycles: magnetic resonance imaging technique [24], digital laminography as a non-destructive inspection X-ray method [25], electrical resistivity measurements for simultaneous monitoring of temperature and damage in real time [26]. The glue-spall theory is saying [27] that concrete is damaged when ice is cracking on the surface of the material, when the thermal expansion mismatch stress exceeds the strength of the ice.

In a series of experiments [28], the beginning of freezing process, it's continuation and damages caused by it, were studied. Calorimetric, expansion and acoustic methods were applied to monitor heat release, mechanical deformation and damage during a series of frost cycles. The results give an insight into super-cooling, salt segregation, ice front penetration and thawing characteristics. Calorimetric data can be used directly to determine the pore shape parameter λ , and the volume fraction of the pore space saturated with ice [29]. Thermoporometry is very useful for the analysis of pore shape. By combining calorimetric measurements with dilatometry [27], it is possible to calculate the contributions of thermal expansion, pore pressure, and crystallization pressure of ice to the strain observed in a mortar during freezing and thawing cycles. Thermodynamics and the linear theory of elasticity are used to calculate the pressures, stresses, and strains induced into moist concrete during freezing and thawing [30]. Developed experimental program while using ultrasonic waves was used to detect damage in Portland cement concrete [31].

The objective of the study is to determine whether the predicted frost resistance of concrete in cycles, while using porosity parameters, corresponds to the number of cycles determined experimentally, using volumetric method of freezing.

2. MATERIALS AND METHODOLOGY

JSC "Akmenes cementas" (Lithuania) Portland cement CEM I 42.5 R was used for the test (Table 1).

Kvesu quarry sand fraction 0/4 with bulk density 1670 kg/m³, with fineness module 1.67 was used for fine aggregate; 0/2 sand fraction ($\rho = 1550 \text{ kg/m}^3$, fineness module 2.37) was obtained after riddling 0/4 sand fraction through certain sieves, 0/1 sand fraction ($\rho = 1460 \text{ kg/m}^3$, fineness module 2.94) was obtained after riddling 0/4 sand fraction through certain sieves. Gravel fraction 4/16 with bulk density 1327 kg/m³ was used for coarse aggregate. Granuliometric composition of aggregate is presented in Table 2.

Plasticizing admixture based on policarboxile polymers REBAflow 202 (FM) (Remei Blomberg GmbH & Co., Germany) was used with density of solution 1.05 g/ml. The total dosage of admixture was in the range from 1.1 % to 1.7 % of cement.

During the research, dry aggregates were used for concrete mixtures. Cement and aggregates were dosed by weight while water and chemical admixture were dosed by volume. Chemical additives in the form of solutions were mixed with water and used in preparation of concrete mixtures. Concrete mixtures were mixed for 3 minutes in the laboratory in forced type concrete mixers.

Table 1. Physical,	mechanical	properties	and	chemical
compositio	n of Portland o	ement CEM	[42.5 R	

Specific surface area, m ² /kg	360
Particle density, kg /m ³	3110
Dry bulk density, kg /m ³	1200
Normal consistency of cement paste, %	23.8
Volume stability, mm	0.0
Initial setting time, min.	165
Compressive strength after 2 days / after 28 days, MPa	32.7 / 53.3
Loss on ignition, %	1.90
Insoluble materials, %	0.50
SO ₃ , %	2.86
Cl ⁻ , %	0.002
Alkalis, calculated by Na2O equivalent, %	<0.8

Table 2. Granuliometric composition of aggregate

I an ath a fith a	The amount of pour out material, %			
sieve's mesh, mm	Sand fraction 0/1	Sand fraction 0/2	Sand fraction 0/4	Gravel fraction 4/16
32.0	_	_	_	100.0
16.0	-	_	_	98.8
10.0	-	-	-	63.8
8.0	1	-	-	42.0
5.0	1	I	100.0	7.3
4.0	-	Ι	91.2	4.2
2.0	-	100.0	70.3	1.4
1.0	100.0	70.5	49.6	-
0.710	78.5	55.3	38.9	-
0.500	68.1	48.0	33.8	-
0.250	19.4	13.7	9.6	_
0.125	6.7	4.7	3.3	_
0.090	3.1	2.2	1.6	_
0.063	1.5	1.1	0.7	_

The consistency of concrete mixture was determined by LST EN 12350-2, density – by LST EN 12350-6 and air content – by LST EN 12350-7.

Concrete specimens cubes $(100 \times 100 \times 100)$ mm were cured in conditions according to LST EN 12390-2 and tested after 28 days. Density of concrete was determined by LST EN 12390-7, compressive strength – by LST EN 12390-3, freezing and thawing resistance of concrete by volumetric freezing after immersing in water – by LST L 1428.17.

The porosity parameters of concrete were determined by measuring kinetics of water absorption, according to the methodology presented in GOST 12730.4-78. According to this methodology, open porosity (capillary pores), total porosity and closed porosity (air pores) of the concrete are defined.

Relative indicators, characterizing the size of the pores, were determined: λ – the average pore size index and α – the pore size uniformity index. This methodology

is widely used for the prediction of the concrete pores' structure, description of emptiness and frost resistance [32]. A similar methodology (the principles of prediction of durability of concrete) is used in U.S.A. [33, 34].

The prediction of frost resistance of concrete was made in the following sequence: 1. Concrete samples (a cube, $(100 \times 100 \times 100)$ mm) were split into four parts which were dried in an oven at a temperature of 90 °C for 24 hours, 2. Water absorption kinetic of the samples was determined according to GOST 12730.4-78.3. Total, open and closed porosity of the concrete was calculated after determination of water absorption kinetics. The total porosity of the concrete is calculated by equation:

$$P_b = \left(1 - \frac{\rho_b}{\rho_s}\right), \%, \qquad (1)$$

where P_b is the total porosity of concrete, %; ρ_b is the density of concrete, kg/m³; ρ_s is the specific density of concrete, kg/m³.

Open porosity (capillary pores) of concrete is calculated by equation:

$$P_{a} = W_{p} \cdot \frac{\rho_{b}}{1000}, \%, \qquad (2)$$

where P_a is open porosity of concrete (capillary pores), %; W_p is water absorption of concrete, %.

Closed porosity of concrete (air pores) is calculated by equation:

$$P_{u} = P_{b} - P_{a}, \%, \qquad (3)$$

where P_u is closed porosity of concrete (air pores).



Fig. 1. The dependence of frost resistance of concrete on the predicted criterion of concrete of freezing and thawing resistance [1]

Relative indicators, characterizing the size of the pores $(\lambda - \text{the average pore size index and } \alpha - \text{the pore size uniformity index})$, were selected for the tested concrete from the nomogram presented in GOST 12730.4-78 standard.

Concrete freezing and thawing resistance can be predicted using criterion K_F (Sheikin A. E, 1989). According to this criterion concrete is considered to be frost resistant when the volume of closed pores exceeds the increase volume of water in concrete capillary pores during the water freezing [32]:

$$K_F = \frac{P_u}{0.09 \cdot P_a},\tag{4}$$

where K_F is the predicted criterion of concrete of freezing and thawing resistance.

The number of concrete freezing and thawing cycles is predicted according to the empirical dependence, when predicted criterion K_F is known (Fig. 1).

3. RESULTS AND DISCUSSIONS

Technological properties of concrete and physicalmechanical properties of hardened concrete were tested before performing prediction of frost resistance of concrete and durability tests. The composition of concrete mixtures which were randomly selected is presented in Table 3.

Table 3. Composition of concrete mixtures

Materials	The amount of materials for 1 m^3 of concrete mixture, kg			
	B1	B2	B3	B4
Portland cement CEM I 42,5 R	339	339	339	251
Water	208	248	210	171
W/C	0.61	0.73	0.62	0.68
Sand fraction 0/1	1751			-
Sand fraction 0/2		1751		-
Sand fraction 0/4	I	I	1751	855
Gravel fraction 4/16	-	_	_	1105
REBAflow 202 (FM)	3.7	3.4	3.4	0.8

Technological properties (density, slump and air content) of tested concrete mixtures are presented in Table 4. This table shows that, depending on the composition of concrete, density of concrete mixture varies within the limits from 1890 kg/m³ to 2280 kg/m³, slump – from 20 mm to 100 mm and air content of the mixture – from 6.2 % to 9.1 %.

Table 4. Technological properties of concrete mixtures

Technological	Composition of concrete mixtures			
properties	B1	B2	B3	B4
Density, kg/m ³	1890	2000	2140	2280
Slump, mm	20	60	130	100
Air content, %	6.2	7.6	9.1	6.7

Density and compressive strength of samples $(100 \times 100 \times 100 \text{ mm})$ formed of the different concrete mixtures are presented in Table 5. This table shows that, depending on the composition of concrete, density of concrete samples varies within the limits from 2002 kg/m³ to 2241 kg/m³ and compressive strength – from 16.5 MPa to 33.7 MPa.

Prediction of frost resistance of concrete was determined according to the methodology presented in section 2. The concrete porosity parameters determined according to water absorption kinetics are presented in Table 6. This table shows that, depending on the composition of concrete, the total water absorption of tested concrete varies within the limits from 4.97% to 7.93%. The maximum water absorption (7.93%) was obtained with B2 concrete composition and the lowest water absorption (4.97%) – B4 concrete composition.

Table 5. Physical-mechanical properties of concrete

Physical-mechanical	Composition of concrete mixtures			
properties	B1	B2	B3	B4
Density, kg/m ³	2142	2036	2002	2241
Compressive strength, MPa	33.7	22.4	16.5	28.6

Depending on the composition of concrete, the open porosity (capillary pores) of concrete varies within the limits from 15.09% to 10.96% and closed porosity of concrete (air pores) – from 3.51% to 10.62%.

According to the author [31] the frost resistance of concrete depends on concrete's porosity, since water can penetrate only to the open pores. The main influence on the frost resistance of the conglomerate has capillary pores. They are open and easy filled with water [32]. The destructive effect during the freezing period depends on the water content in the material. Air pores, which are appearing in conglomerate because of pulled air, unlike capillary pores, are increasing frost resistance of conglomerate. Air pores are closed and water is not getting into them during immersing process.

Table 6. Total water absorption and porosity of concrete

Parameters	Composition of concrete mixtures			
	B1	B2	B3	B4
Total water absorption of concrete, %	7.72	7.93	6.81	4.97
Total porosity of concrete, %	25.42	24.96	2.44	14.47
Open porosity of concrete, %	14.80	15.09	13.76	10.96
Closed porosity of concrete, %	10.62	9.87	7.68	3.51
λ – average pore size index	0.03	0.69	0.25	0.88
α – pore size uniformity index	0.31	0.46	0.32	0.59

Depending on the composition of concrete, the average pore size index λ of tested concrete varies within the limits from 0.03 to 0.88. The average pore size index λ shows, that pores of B2 concrete composition are very small ($\lambda = 0.03$). Pores of B4 concrete composition are the biggest ones ($\lambda = 0.88$). These kinds of pores, medium size, are common for concrete without air entraining admixture.

Pore size uniformity index α shows, that pore size distribution in concrete, depending on the concrete composition, is similar, and varies within the limits from 0.31 to 0.59; it means that the size of pores in these concretes is similar.

Water absorption kinetics of concrete, depending on the composition of concrete is shown in Fig. 2.

This figure shows, that concretes (B1-B3) without large aggregate absorb more water comparing with concrete (B4) with large aggregate. This is related to the structure of concrete: capillary and closed porosity of concrete (Table 6). Water absorption of B2 concrete composition doesn't differ significantly from B1.



Fig. 2. Water absorption kinetics of concrete

Predicted criterion K_F of concrete of freezing and thawing resistance, calculated from (4) equation, and predicted frost resistance of concrete in cycles, set from dependence presented in Figure 1, are given in Table 7.

This table shows that the highest predicted criterion K_F and predicted frost resistance (according to the number of freezing and thawing cycles) is characteristic for B1 and B2 concrete compositions, when 0/1 (B1) and 0/2 (B2) sand fraction was used for fine aggregate. The lowest predicted criterion K_F and predicted frost resistance (according to the number of freezing and thawing cycles) is characteristic for B4 concrete composition, when sand fraction 0/4 was used for fine aggregate and gravel fraction 4/16 was used for coarse aggregate.

Demonsterne	Composition of concrete mixtures				
Parameters	B1	B2	B3	B4	
Predicted criterion K_F of concrete of freezing and thawing resistance	7.97	7.27	6.20	3.56	
Predicted frost resistance of concrete in cycles	~1000	~950	~850	~550	

 Table 7. Predicted criterion of concrete of freezing and thawing resistance and predicted frost resistance

When porosity parameters are evaluated according to water absorption kinetics, predicted frost resistance of concrete of different compositions varies within the limits from 550 to 1000 freezing and thawing cycles. Taking into consideration the climatic conditions in Lithuania, 9-17 freezing and thawing cycles could occur through the year.

This number of cycles would not be sufficient to destroy the structure of tested concretes, as the effect of 550-1000 freezing and thawing cycles should occur immediately. Over the time, the hydration degree of the cement stone is changing as well as porosity of the stone.

Microporosity of concrete is decreasing and concrete is more resistant to frost when W/C absolute value is lower [31]. Therefore, the durability of such concretes can be measured in a hundred of years and more. Concrete's exploitation conditions have influence as well: under freezing and thawing effect concrete could be constantly in water, ice melting materials and sea water could affect it too.

Frost resistance of concrete was determined using LST L 1428.17. During this experiment, samples were kept in freezer for 2.5 hours and thawed in water of +20 °C degrees for 2.5 hours. Frost resistance experiment in the laboratory took 638 days; this time was required to determine the compressive strength of samples after the 1000 freezing and thawing cycles.

Depending on the composition of the concrete, samples were tested according to the predicted freezing and thawing cycles (Table 7).

If the compressive strength of samples decreased more than 5 % (compared with control one) after the required freezing and thawing cycles, it means that samples did not pass frost resistance test. The results of concrete frost resistance test are presented in Tables 8-9.

Table 8 shows, that compressive strength of B1 concrete composition increased by 9.68 % (compared with control one) after 1000 freezing and thawing cycles, compressive strength of B2 concrete composition increased by 15.96 % after 950 freezing and thawing cycles, compressive strength of B3 concrete composition decreased by 2.03 % after 850 freezing and thawing cycles (but the decrease is not more then 5 %), compressive strength of B4 concrete is characterized by 11.90 % after 550 freezing and thawing cycles (the decrease is more then 5 %). This concrete is characterized by largest pores ($\lambda = 0.88$) and lowest closed porosity (3.51 %).

 Table 8. Change of compressive strength of concrete after the durability test

Composition of concrete	Number of	Change of compressive strength after the durability test		
mixtures	cycles	MPa	%	
D1	500	+3.1	+19.47	
ВІ	1000	+1.6	+9.68	
DJ	450	+5.1	+2.66	
D2	950	+3.7	+15.96	
D2	400	+4.1	+11.84	
БЭ	850	-0.7	-2.03	
D4	300	-2.4	-8.47	
D4	550	-3.4	-11.90	

 Table 9. Change of concrete weight and appearance of cracks after the durability test

Composition of concrete mixtures	Number of cycles	Change of weight after the durability test, %	Visual evaluation
D1	500	-0.08	no cracks appeared
DI	1000	-0.10	no cracks appeared
DJ	450	+0.08	no cracks appeared
D2	950	+0.01	no cracks appeared
D2	400	+0.15	no cracks appeared
Б3	850	-0.49	no cracks appeared
D4	300	+0.00	cracks appeared
Б4	550	+0.11	cracks appeared

Table 9 shows, that weight of B1 concrete composition samples decreased by 0.08 % after 500 freezing and thawing cycles, decreased by 0.10 % after 1000 freezing and thawing cycles (compared with control one); weight of B2 concrete composition samples increased by 0.08 % after 450 freezing and thawing cycles, increased by 0.01 % after 950 freezing and thawing cycles; weight of B3 concrete composition samples increased by 0.15 % after 400 freezing and thawing cycles; weight of B4 concrete composition samples increased by 0.49 % after 400 freezing and thawing cycles; weight of B4 concrete composition samples didn't change after 300 freezing and thawing cycles, but increased by 0.11 % after 550 freezing and thawing cycles. Weight loss of different concrete compositions after certain freezing and thawing cycles is in the range of -0.49 % to +0.15 %.

After durability test, appeared cracks were noticed on samples of B4 concrete composition, formation of defects with other concrete compositions were not noticed (Fig. 3).



Concrete samples after 550 freezing and thawing cycles (B4 concrete composition)

Fig. 3. Concrete samples images after frost resistance test (a cube, (100×100×100) mm)

During the cyclical process of water freezing in concrete capillary pores and thawing process, pores are filled with water and destroying the structure of concrete to complete destruction. This destruction process usually goes from the surface to the inner parts of concrete structure (Fig. 3).

4. DISCUSSIONS

As the objective of the study was to determine whether the predicted frost resistance of concrete in cycles, while using porosity parameters, corresponds to the number of cycles determined experimentally, using the volumetric method of freezing, several randomly selected compositions of concrete mixtures were tested. Only fine aggregate was used for preparation of B1, B2 and B3 concrete mixtures. Gravel was used as a coarse aggregate for preparation of B4 concrete mixture. Gravel is not used in production of frost-resistant concrete, holding more then 200 freezing and thawing cycles. In this case, crushed granite should be used. When porosity parameters are evaluated by water absorption kinetics, the predicted frost resistance of this concrete is 550 freezing and thawing cycles (Table 7). When frost resistance of concrete was determined by LST L 1428.17 requirements, it is clear, that compressive strength decreased by 8.47 % after 300 cycles and 11.90 % after 550 cycles, and it should be not more then 5 % (compared with control one). Samples did not pass the frost resistance test.

B1–B3 are fine-grained concretes. During the process of preparation, together with fine fraction sufficient amount of air is getting into fine-grained concretes. This is the reason why fine-grained concretes have better frost resistance characteristics compared with coarse-grained concretes; have high number of predicted frost resistance of concrete in cycles (Table 7) and do pass this number of cycles during the durability test (Table 8). B4 is coarsegrained concrete. Concentration of fine aggregate from the total amount of aggregate is 0.44 for B4 concrete composition (Table 3). The biggest parts in fine aggregate are not bigger then 4 mm (0/1, 0/2 and 0/4 sand fraction). The biggest parts in coarse aggregate are not smaller then 4 mm (4/16 gravel fraction). The total amount of aggregate consists of fine and coarse aggregates.

Predicted criterion K_F of concrete of freezing and thawing resistance, calculated from (4) equation, is 3.56 for B4 concrete composition, predicted frost resistance is 550 cycles (Table 7). The authors are proposing to correct (4) equation, calculation of predicted criterion K_F , by supplementing it with coefficient K_s , evaluating concentration of fine aggregate from the total amount of aggregate:

$$K_{F} = \left(\frac{P_{u}}{0.09 \cdot P_{a}}\right) \cdot K_{S},$$
(5)

where K_s is the coefficient evaluating concentration of fine aggregate from the total amount of aggregate, parts per unit.

Predicted frost resistance of concrete in cycles for coarse-grained concrete (B4) is higher when criterion K_F presented by A. Sheikin [32] is used comparing with the data obtained from direct volumetric freezing of samples. Table 8 shows, that compressive strength of B4 concrete composition decreased more then 5 % after 300 freezing and thawing cycles. K_s coefficient is presented by authors to correct criterion K_F . K_s coefficient is evaluating concentration of fine parts in coarse-grained concrete, adjusting predicted frost resistance of concrete in cycles so it could be close to the number of cycles obtained from direct volumetric freezing of samples. In this case,

predicted criterion K_F , calculated from (5) equation, is 1.57 for B4 concrete composition and predicted frost resistance of concrete in cycles, set from empirical dependence presented in Fig. 1, is ~200 cycles. While using the volumetric method of freezing, B4 concrete composition would pass 200 freezing and thawing cycles and the decrease of compressive strength would be not more then 5 % (compared with control one).

5. CONCLUSIONS

- 1. The prediction of concrete freezing and thawing resistance according to parameters of porosity proposed by Sheikin is more suitable for evaluation of durability of sand concrete.
- 2. For sand concrete, predicted number of cycles of frost resistance, according to predicted criterion K_F , corresponds to the number of cycles determined experimentally, using volumetric method of freezing.
- 3. The authors propose to correct equation of predicted criterion K_F of coarse-grained concrete of freezing and thawing resistance, by supplementing it with coefficient K_s , evaluating concentration of fine aggregate from the total amount of aggregate.
- 4. For concrete with coarse aggregate, the evaluation of concentration of fine aggregate from the total amount of aggregate, will give more accurate prediction of frost resistance of concrete in cycles, set from empirical dependence.

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