

Frost Resistance of Concretes with Low-Clinker Cements Depending on Curing Time

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ABSTRACT

The article discusses the test results concerning the compressive strength, water absorption, and frost resistance of concretes produced with three types of cement: CEM I, CEM II/B-M (S-V), and CEM III/A, evaluated after different curing periods (28, 56, and 90 days). Additionally, to assess the effects of the minimum 4% air entrainment recommended by the EN 206 standard, concrete mixes with the same composition but containing an air-entraining admixture used consistently at 0.15% of the cement mass, were prepared. To achieve a more detailed characterization of the concretes, tests on the concrete mixes were also performed, and the physical properties of the concretes, including water absorption, density, and total porosity, were measured. The paper also presents significant results on the pore distribution in the air-entrained concretes, confirming the achievement of very good basic air-entrainment parameters in all concretes. Based on the test results, it was found that both the type of cement and the introduction of an air-entraining admixture significantly influence not only the frost resistance of the concretes but also their compressive strength depending on the curing time of the specimens. This is particularly evident in the low-clinker cements CEM II and CEM III. It was observed that in the case of concretes with these cements, it is possible to attain nearly no reduction in compressive strength after undergoing 150 cycles of freezing and thawing following a 90-day curing period.

Keywords: low-clinker cement, type of cement, frost resistance of concrete, pore distribution in concrete, compressive strength, concrete curing time.

INTRODUCTION

Durability of building materials pertains to a material capacity to retain its functional properties under the influence of aggressive environmental conditions and mechanical loads throughout its entire service life, without the need for significant maintenance actions [1, 2]. Currently, the concrete durability is one of the key criteria for choosing construction materials. It is determined by the appropriate selection of components during the design phase, correct execution technology, proper curing, exposure to corrosive environments, mechanical loads, and maintenance [3]. One of the most crucial features responsible

for the concrete durability is its resistance to frost. Theories on the deterioration of water-saturated concrete under the influence of sub-zero temperatures have been developed by Powers [4], Powers and Helmuth [5], Litvan [6], Fagerlund [7, 8], and Setzer [9]. A review of existing theories regarding the physical characteristics of processes occurring during the freezing of concrete can also be found in works [10, 11, 12]. The mechanism of concrete degradation due to cyclic exposure to sub-zero temperatures is associated with thermal deformations of concrete and physical phenomena. As water freezes, it increases in volume, causing internal stresses within the concrete, initially leading to micro-cracks and spalling, and in the

long term, potentially resulting in the destruction of the concrete element [10, 11, 12].

Frost damage can manifest as a deterioration of the internal structure and as a surface scaling of the concrete [13]. Internal damage may be evident via a reduction in strength and a loss of dynamic modulus of elasticity, and microcracks caused by increased stresses during the freezing of water [14, 15]. Surface scaling primarily results in a reduction of mass at the concrete surface [16, 17]. While surface spalling of the cement paste does not directly impact the concrete’s strength, it can lead to increased susceptibility to moisture absorption, which is significant given the capillary-porous structure of concrete [13].

Material-structural protection of concrete exposed to low temperatures can be ensured by maintaining an appropriate balance between pores that collect water capable of freezing and pores that act as a protective buffer, which due to their size, cannot fill with water [18]. In the technology of frost-resistant concrete, two methods are known for achieving such a water balance [19]:

- sealing the concrete structure by lowering the water-cement ratio while simultaneously increasing/maintaining an appropriate content of cement and reducing capillary pores;
- air-entraining the concrete mix.

The air-entraining technology involves introducing small, evenly distributed air bubbles into the concrete mix. The primary function of these bubbles is to absorb excess water from the freezing capillary pores [20].

The EN 206 standard [21] defines four exposure classes for concrete exposed to the harsh effects of cyclic freezing and thawing:

- XF1 – moderately water-saturated environment without the use of de-icing agents;
- XF2 – moderately water-saturated environment with the use of de-icing agents;
- XF3 – highly water-saturated environment without the use of de-icing agents;
- XF4 – highly water-saturated environment with the use of de-icing agents.

The EN 206 standard [21] recommends an air content in the concrete mix of at least 4%, noting that the maximum value is the “specialized minimum value plus 4%”. The national supplement to the EN 206 standard, PN-B 06265 [22], recommends a minimum air content in concretes exposed to XF2-XF4 environments, depending on the maximum aggregate size (Table 1).

The procedure for testing air content in concrete mixes is described in the EN 12350-7 standard [23]. It distinguishes two testing methods: the pressure gauge method and the water column method. The frost durability of concrete becomes even more critical when freezing occurs when de-icing salts are present. Concrete surfaces such as roads, bridges, and pavement slabs are most exposed to these conditions. Commonly used de-icing substances applied in winter can accelerate the degradation process of concrete under low-temperature conditions, particularly causing surface scaling. According to some estimates, the destruction process can accelerate up to four or five times in such conditions [24]. The most used de-icing agent in concrete pavement technology is NaCl. According to Rusin [12], the salt solution in the pores of concrete increases the intensity of the destructive mechanism associated with the rise in hydraulic pressure. This leads to an increase in water viscosity because of the higher concentration of the salt solution, an increase in concrete absorbability, and a reduction in the crystallization temperature, which increases the amount of unfrozen water capable of moving through the capillary networks.

For concrete pavements, the EN 206 standard [21] specifies exposure classes XF3-XF4 due to frost effects. The recommended limit values for these classes are presented in Table 2.

Undoubtedly, all structural changes in concrete due to cyclic freezing are connected to the presence of water within the concrete. It is widely recognized that there is a correlation between concrete durability in environments subjected to cyclic freezing and thawing and its water

Table 1. Recommended air content for XF2-XF4 classes depending on the largest aggregate size [22]

The largest aggregate size (mm)		8	16	32	64
Air content (%)	XF2	≥ 5.5	≥ 4.5	≥ 4.0	≥ 3.5*
	XF3				
	XF4				

Note: * Concrete with a consistency of V0 and w/c ≤ 0.4 can be produced without additional air-entraining.

Table 2. Recommended limit values for classes XF1-XF4 [21]

Specification	XF1	XF2	XF3	XF4
Maximum w/c ratio	0.55	0.55	0.50	0.45
Minimum strength class	C30/37	C25/30	C30/37	C30/37
Minimum cement content (kg/m ³)	300	300	320	340
Aggregate frost resistance class according to PN-EN 12620	–	F2	F1	F1

absorption. Concrete, being a porous material composed of a network of capillary pores, open pores, and closed pores, naturally absorbs water under atmospheric pressure [25]. When characterizing concrete, we often refer to its mass water absorption. This is understood as the proportion of the maximum mass of water absorbed by the material to the dry mass of the sample. The European standard EN 206 [21] does not mention concrete water absorption. The testing process is detailed in the outdated standard PN-B-06250 [26]. According to the standard, the concrete's water absorption should not exceed 5% for those exposed directly to atmospheric conditions and 9% for concrete protected from direct exposure to atmospheric conditions.

Water absorption is an important material property; however, the suitability of concrete should not be determined based on this single property without a thorough analysis of the operational conditions of the structure [27].

The frost resistance of concrete relies on the selection of appropriate technological and material factors [12, 28]. One of the most crucial material factors influencing the frost resistance of concrete is the type of cement, including its specific surface area, alkali content, gypsum dehydration, and hydration rate [19]. It is generally advised to increase the dose of air-entraining admixture when the cement has a higher specific surface area or low alkali content. [26]. Finely ground cement requires more water to properly form stable air bubbles in the mix. Utilizing cements with mineral additives (CEM II/B-M, CEM III) reduces the overall air content within the concrete mix compared to Portland cement CEM I. It is widely believed that the greatest problems with proper air entrainment occur in concrete mixes containing fly ash. Even small amounts of fly ash can reduce the mix's air content by 1% [29, 30]. This is attributed to the presence of fine coke in fly ash, which has a large specific surface area, which can absorb the surfactant on its surface [30]. Furthermore, if the fine coke is unevenly distributed within the concrete

mass, it can create areas with varying air content [31, 32, 33]. Concretes made with slag cement are particularly susceptible to frost damage. According to research conducted by Giergiczny [34], using cement with a higher slag content can reduce the total air content in hardened concrete by more than 3% and an increase in the air pore spacing factor L by over 0.1 mm. Additionally, the presence of slag influences the distribution of air particles by reducing the micropore content by over 1.6%. Although pozzolanic mineral additives reduce capillary pores, their effect on the frost resistance of concrete is variable and requires the use of different amounts of an air-entraining admixture [35, 36, 37]. Achieving proper air entrainment in concrete made with cement containing a high level of mineral additives is challenging and requires selecting the amount of air-entraining admixture (AEA) based on the type and amount of mineral additive in the cement. Moreover, the effect of additives on the demand, efficiency, and stability of AEA is variable [38]. Despite existing problems with achieving the desired air-entrainment parameters, the use of cements with additives is becoming increasingly popular. In addition to the ecological aspect, i.e., reducing the consumption of cement clinker and lowering CO₂ emissions into the atmosphere, pozzolanic additives can improve pore structure, increase hydration, and enhance the durability of concrete [39, 40].

Cements with additives, such as pozzolanic additives, may also require a longer time to achieve full compressive strength. Therefore, it is often recommended that testing of concretes with these cements be conducted after a longer period, such as 56 or 90 days, to obtain more comprehensive information about their quality and durability. Testing after a shorter period may not provide a complete picture of the material's properties, potentially leading to incorrect conclusions about its durability. The PN-B-06265 [22] standard introduces the concept of equivalent time for frost resistance testing, which is different from the compressive strength curing time and refers to the

time after which concrete should be tested. The testing times based on the type of cement used are presented in Table 3.

This paper details the results of tests on the frost resistance, water absorption, and compressive strength of concrete produced with three types of cement (CEM I, II, and III) evaluated after different curing periods (28, 56, and 90 days). Additionally, to evaluate the effectiveness of air entrainment in concretes made with different types of cement, both air-entrained and non-air-entrained concretes, with a constant admixture amounting to 0.15% of the cement mass, were tested. To provide a broader characterization of the tested concretes, the concrete mixes were tested and the concrete physical properties, such as water absorption, density, and total porosity, were determined.

MATERIALS AND METHODS

Materials

Cements

Three types of cement were used in the concretes: CEM I 42.5 N SR3/NA (designated as CEM I 0 or CEM I 0.15), CEM II/B-M (S-V) 42.5 R (designated as CEM II 0 or CEM II 0.15) and

CEM III/A 42.5 N-LH/HSR/NA (designated as CEM III 0 or CEM III 0.15).

Explanations of the full cement designations:

- CEM I 42.5 N SR3/NA – Portland cement with low alkali content and high sulphate resistance, with low early strength classes;
- CEM II/B-M (S-V) 42.5 R – slag-ash cement with high early strength classes;
- CEM III/A 42.5 N-LH/HSR/NA – blast furnace cement with low heat of hydration, low alkali content and high sulphate resistance, with low early strength classes.

Concrete specimens with a designation of 0 (CEM I 0, CEM II 0, CEM III 0) indicate un-aerated concretes. Specimens with a designation of 0.15 (CEM I 0.15, CEM II 0.15, CEM III 0.15) indicate concretes with an aeration admixture of 0.15% of the cement mass (m.c.). The chemical and physical properties of the cements are presented in Table 4.

Table 3. Testing time based on the type of cement used

Type of cement	Curing time (days)
CEM I R, CEM II/A R	28
CEM I N, CEM II/A N, CEM II/B NR, CEM IV/A	56
CEM III, CEM IV/B, CEM V	90

Table 4. Chemical and physical properties of the cements

Property		CEM I 42.5 N SR3/NA	CEM II/B-M (S-V)42.5 R	CEM III/A 42.5 N LH/HSR/NA
Setting time (min)	initial	184	173	220
	end	227	217	270
Water demand (%)		26.9	32.5	31.4
Volume demand (mm)		1.1	0.6	1.0
Specific surface area (cm ² /g)		3638	4727	4610
Compressive strength (MPa)	after 2 days	25.6	26.4	13.5
	after 28 days	56.4	56.7	58.4
Chemical analysis (%)	SiO ₂	20.01	27.27	31.41
	Al ₂ O ₃	4.49	8.27	6.98
	Fe ₂ O ₃	3.73	3.70	2.18
	CaO	63.59	53.40	50.51
	MgO	1.00	1.84	4.71
	SO ₃	3.01	2.73	1.97
	Cl	0.062	0.038	0.070
	Na ₂ O	0.23	0.32	0.32
	K ₂ O	0.52	0.68	0.51
Na ₂ O _{eq}	0.57	0.77	0.65	
Insoluble residue (%)		1.09	11.97	2.23
Loss on ignition (%)		3.08	1.82	0.41

Aggregates

All concretes were made with granite crushed stone having a grain size of 2–16 mm and natural sand having a grain size of 0–2 mm. Despite the widespread availability of natural gravel, granite crushed stone was deliberately used to eliminate the possibility of alkali-aggregate reactions, which often occur in the presence of reactive grains from the carbonate rock group in gravels. The tested aggregates bulk densities were: sand 2.65 kg/dm³, and granite crushed stone 2.64 kg/dm³. The particle size gradation of the fine and coarse aggregates has been shown in Table 5.

Admixtures

To achieve the minimum air-entrainment level recommended in EN 206 [21], an air-entraining

admixture derived from polycarboxylic acids was used. The properties of the air-entraining admixture, based on data supplied by the manufacturer, are shown in Table 6.

Concretes

To investigate the effect of cement type and concrete age on the frost resistance of concrete, a total of 6 concretes were examined after being cured in water for 28, 56, and 90 days. In the compared concretes, the same volume of cement and a similar volume of aggregate with a sand point of 36% were used, as well as a fixed w/c ratio of 0.44. Thus, all concretes meet the recommendations of both the EN 206 standard [21] and the PN-B-06265 standard [22] relating to the XF4 exposure class, which require $w/c \leq$

Table 5. Sieve analysis of the fine and coarse aggregates (%)

Sieve size (mm)	Sand	Coarse aggregate (granite)
16	100	100
8	100	51
4	100	23
2	100	2
1	91	–
0.5	60	–
0.25	8	–
0.125	1	–

Table 6. Chemical and physical properties of the air-entraining admixture

Property	Method	Admixture dosage	Requirements according to EN 934-1 and EN 934-2	Measurement result	Compliance
Homogeneity	Visually	–	Homogeneous during use	Homogeneous	Compliant
Color	Visually	–	Uniform, according to manufacturer's description	Transparent	Compliant
Density	ISO 758	–	1000 ±20 kg/m ³	1000 kg/m ³	Compliant
Dry substance content	EN 480-8	–	1.6 ±0.16%	1.50%	Compliant
pH value	ISO 4316	–	5.0 – 9.0	7.12	Compliant
Water-soluble chlorides	EN 480-10	–	≤ 0.1%	0.02%	Compliant
Alkali content Na ₂ O _{eq}	EN 480-12	–	≤ 2.0%	1.02%	Compliant
Air content in concrete mix (entrained air)	EN 12350-7 concrete mix according to EN 480-1	0.08% binder mass	Tested mix ≥ 2.5% volume above content in control mix. Total air content from 4% to 6%	6.0 ≥ 2.5 + 2.0% 4.0 ≤ 6.0 ≤ 6.0%	Compliant
Compressive strength after 28 days (at constant consistency)	EN 12390-3 concrete mix according to EN 480-1	0.08% binder mass	After 28 days ≥ 75%	89%	Compliant
Pore distribution characteristics in hardened concrete	EN 480-11	0.08% binder mass	≤ 0.200 mm	0.155 mm	Compliant

0.45, $C_{min} = 340$ kg (Table 6), and the use of aggregate with frost resistance corresponding to the (F) category according to EN 12620 [41]. For each concrete, an air-entraining admixture based on sulfonated sodium alpha-olefin was used at a constant 0.15% of the cement mass (c.m.). The compositions of all concretes are shown in Table 7.

Methods

The air content within the concrete mix was determined using the pressure gauge method as per EN 12350-7 [23]. The consistency of the mix, depending on its fluidity, was measured using the slump test method [42] or the Vebe test method [43].

The physical parameters of the concrete were determined after 28, 56, and a 90-day curing period in water at a temperature of (20 ± 2) °C. The specific density was measured using the pycnometric method on powdered material following the procedure outlined in EN 196-6 [44]. The material was dried to a constant mass at a temperature of $(105 \div 110)$ °C and sieved through a 0.08 mm sieve.

The apparent density was determined following the procedure outlined in EN 12390-7 [45] on 6 specimens with dimensions of $150 \times 150 \times 150$ mm. The sample volume was determined using the hydrostatic method by water displacement.

The total porosity was determined as the ratio of apparent density to specific density according to the BS EN 1936:2006 [46]:

The air content was determined using the relationship between specific density and bulk density according to the formula:

$$P = \left(\frac{\rho_r - \rho_b}{\rho_r} \right) \cdot 100\% \quad (1)$$

where: ρ_b – apparent density (g/cm^3); ρ_r – specific density (g/cm^3).

Weight-based water absorption was determined in accordance with the withdrawn Polish standard PN-B-06250 [26] as the ratio of absorbed water to the sample’s dry mass. The test was performed on 4 specimens measuring $150 \times 150 \times 150$ mm.

Compressive strength was measured after 28, 56, and a 90-day curing period in water at a temperature of 20 ± 2 °C. The test was conducted according to EN 12390-3 [47] on 6 specimens with dimensions of $150 \times 150 \times 150$ mm. An Advantest 9 press from Controls having a maximum force range of 3000 kN, meeting the requirements of BS EN 12390-4 [48], was used for strength tests.

Frost resistance was determined in accordance with the withdrawn Polish standard PN-B-06250 [26] on 12 specimens measuring $150 \times 150 \times 150$ mm. The specimens were examined after 28, 56, and 90 days of curing. Frost resistance was determined as the difference in compressive strength and mass of the specimens after 150 cycles of alternating freezing at -18 ± 2 °C and thawing at 18 ± 2 °C. Specimens were considered frost-resistant if the mass difference ratio before and after freezing did not exceed 5%, and the variation in compressive strength between the frozen and non-frozen specimens, which were kept in water at 20 ± 2 °C throughout the test period, did not exceed 20%.

The characterization of air pores in the hardened concrete sample was determined according to EN 480-11 [49]. The test was performed on specimens measuring $100 \times 150 \times 40$ mm cut from the interior of 150 mm cubic blocks after 28 days of curing. Preparation of the surface for testing included wet grinding and polishing to achieve a matte finish, followed by applying zinc paste for contrast. For each prepared sample, the micropore content A_{300} and the spacing factor \bar{L} were measured. Microscopic analysis was performed using an optical microscope at $100 \times$

Table 7. Compositions of the tested concretes (kg/m^3)

Component	Concrete designation		
	CEM I 0/CEM I 0.15	CEM II 0/CEM II 0.15	CEM III 0/CEM III 0.15
Cement CEM I 42.5 N SR3/NA	365	–	–
Cement CEM II/B-M (S-V)42.5 R	–	345	–
Cement CEM III/A 42.5 N-LH/HSR/NA	–	–	347
Sand 0–2 mm	693	700	700
Crushed granite 2–8 mm	486	492	491
Crushed granite 8–16 mm	726	737	736
Water	162	154	154
Admixture, % c.m.	0/0.15	0/0.15	0/0.15

magnification along a measurement line with a total length of 1200 mm per sample to record the chord lengths of the pores and the lengths of lines passing through the solid phase (Fig. 1).

RESULTS AND DISCUSSION

Table 8 presents the properties of the concrete mixes, including air content and consistency. The analysis of the concrete mix consistency using the Vebe method allows for the assessment of the plasticity and workability of concrete. A shorter Vebe time indicates higher plasticity, which is beneficial in many construction applications. A longer Vebe time indicates less workable mixes, which may require additional adjustments by dosing a plasticizing admixture [50].

The consistency classes designated K3-K4 represent the degree of liquidity of the concrete mixture in accordance with the old nomenclature of PN-B-06250 [26], where K3 means plastic consistency and K4 means semi-fluid consistency. Concrete mixes CEM I 0, CEM II 0, and CEM II 0.15 achieved a plastic consistency class K3, while mixes CEM I 0.15, CEM III, and CEM III 0.15 achieved a semi-fluid consistency class K4. The consistency testing of concrete mixes showed an increase in fluidity in all mixes due to the use of a constant amount of air-entraining admixture equal to 0.15% of the cement mass. The mixes

from the CEM I series had the highest fluidity and the highest air content (6.1%). The worst workability was observed in mixes containing CEM II/B-M (S-V) cement, in both air-entrained and non-air-entrained series. According to Gopalan et al. [51], this is because the addition of fly ash to cement results in a reduction in the amount of water in the concrete mix, which influences the degree of air entrainment.

Concrete mixes containing blast furnace slag CEM III/A are characterized by lower viscosity, which facilitates their placement on construction sites. The addition of slag to cement makes the mix more fluid, which is advantageous for elements with complex shapes or reinforced components [52]. Furthermore, Rudnicki et al. [53] note that mixes with CEM III cement have a longer setting time, which is crucial when there is a need to maintain workability for an extended period.

Among non-air-entrained concretes, the lowest air content was observed in mixes containing fly ash cement CEM II 0, while the highest air content was observed in mixes containing pure Portland cement CEM I 0. An air-entraining admixture at 0.15% of the cement mass resulted in air entrainment levels of 4.3–6.1%, based on the type of cement used. The air-entraining admixture was most effective in the mix containing pure Portland cement (6.1%), while the lowest was in the mix with fly ash cement (4.3%). According to the PN-B 06265 [22] standard, for the aggregate

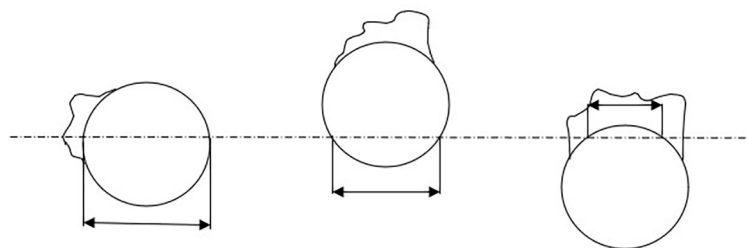


Figure 1. Example of chord length observations along the measurement line

Table 8. Concrete mixtures properties

Series	Air content	Slump test	Vebe test	Consistency class according to EN 206	Consistency class according to PN-B-06250
	(%)	(mm)	(s)	(-)	(-)
CEM I 0	2.7		7	V3	K3
CEM I 0.15	6.1	50	3	S2/V4	K4
CEM II 0	2.0		9	V3	K3
CEM II 0.15	4.3		8	V3	K3
CEM III 0	2.5	70	5	S2/V4	K4
CEM III 0.15	5.1		3	V4	K4

used (0–16 mm), it is recommended that the concrete mix be air-entrained to at least 4.5%. Pressure method tests showed that the air-entrained concrete mixes CEM I 0.15 and CEM III 0.15 met this requirement.

One of the most important parameters of a concrete mix is its air content. The total air content in a concrete mix, measured using the pressure gauge method, includes air from air-entraining admixtures, plasticizing admixtures, air accidentally trapped during mixing, and surface pores and voids resulting from insufficient vibration. The effectiveness of air entrainment depends on various factors, such as the type of cement used.

Blast furnace cement CEM III/A, a blend of Portland clinker and a significant amount of blast furnace slag, is often used in construction due to its unique properties, such as improved sulfate resistance and resistance to alkali-silica reaction, low shrinkage, low heat of hydration, and lower CO₂ emissions during production [54]. Regrettably, the presence of blast furnace slag in the cement can diminish the effectiveness of air-entraining admixtures. Studies by Giergiczny et al. [55] indicate that concrete mixes containing slag cement require a higher amount of air-entraining admixture compared to concrete containing pure Portland cement. Deja [56] discovered that the effectiveness of the air-entraining admixture in fresh mixtures was notably lower for slag cement concrete compared to concrete with CEM I cement. For the same air content, the dose of air-entraining admixture was up to twice as high as for concrete with pure Portland cement. Cement

CEM II/B-M (S-V) is a type of Portland cement with fly ash added, which constitutes 6% to 20% of the cement mass. Fly ash is a residue from coal combustion in power plants and is widely used in concrete due to its positive impact on the properties of the concrete mix. However, fly ash in cement can reduce the effectiveness of air-entraining agents. Studies conducted by Justnes et al. [1, 57] indicate that fly ash can adsorb particles of the air-entraining agent, leading to a decrease in the amount of entrained air.

The physical properties of concretes, such as specific density, bulk density, mass water absorption, and total porosity, tested at different intervals, are presented in Table 9–10 and Figures 2–3.

No significant differences were noted in the specific densities of the concretes, both within the same cement series and across different time periods. This is expected due to the lack of substantial changes in the mix design of the tested concretes. However, a decrease in apparent density was observed as a result of the addition of extra air into the mix using the air-entraining admixture.

Analysing the results of total porosity, an increase in porosity was observed in all air-entrained concretes. The highest increase in porosity was recorded in the CEM I 0.15 concretes, with an increase of approximately 55% in comparison to concrete without air entrainment. The smallest increase in total porosity was noted in the CEM II 0.15 concretes, with an increase of about 17% compared to the non-air-entrained CEM II 0 concrete. Over time, a decrease in total porosity was observed in almost all tested series as the

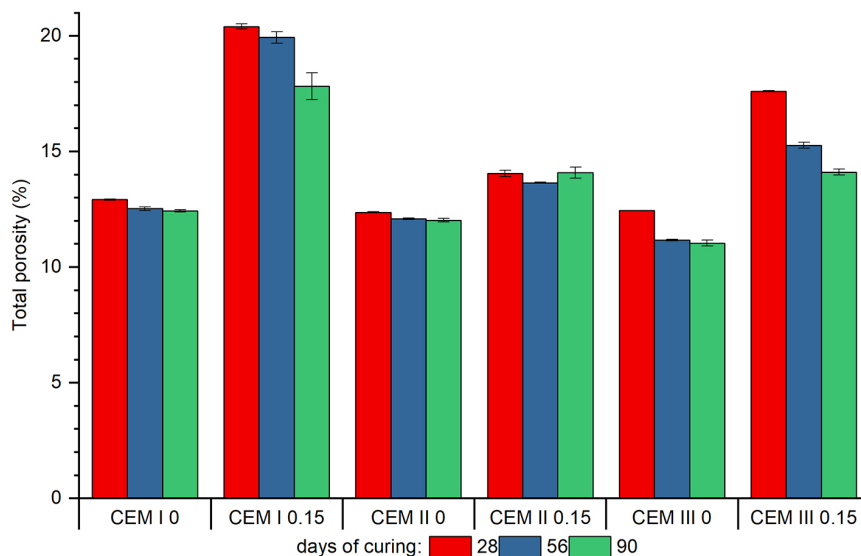


Figure 2. Total porosity of tested concretes

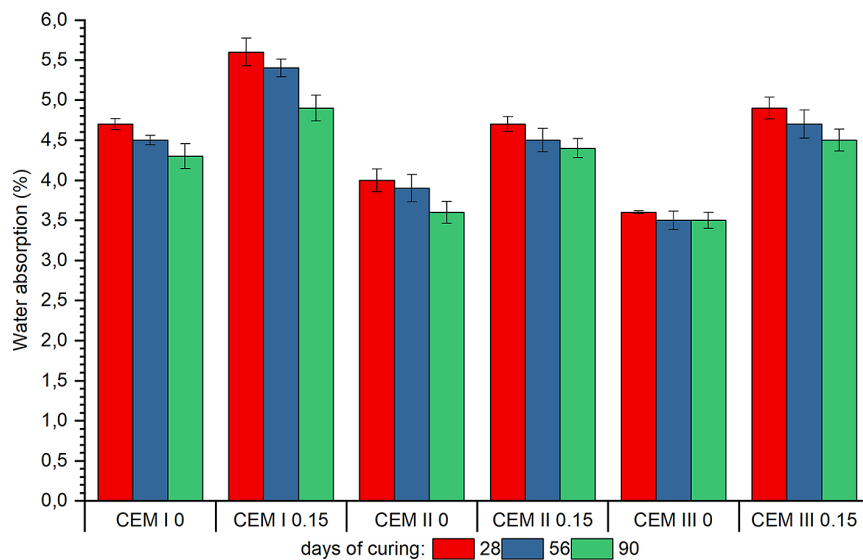


Figure 3. Water absorption of tested concretes

Table 9. Physical properties of tested concretes

Days of curing (days)	28		56		90	
	Specific density (kg/dm ³)	Bulk density (kg/dm ³)	Specific density (kg/dm ³)	Bulk density (kg/dm ³)	Specific density (kg/dm ³)	Bulk density (kg/dm ³)
CEM I 0	2.64	2.30	2.64	2.31	2.64	2.31
	0.00*	0.00*	0.01*	0.01*	0.01*	0.01*
	0.02**	0.07**	0.03**	0.35**	0.31**	0.23**
CEM I 0.15	2.65	2.11	2.65	2.12	2.65	2.18
	0.00*	0.01*	0.00*	0.06*	0.00*	0.02*
	0.19**	0.51**	0.05**	1.13**	0.02**	2.66**
CEM II 0	2.63	2.31	2.63	2.32	2.63	2.33
	0.01*	0.00*	0.01*	0.01*	0.01*	0.00*
	0.23**	0.14**	0.21**	0.16**	0.18**	0.33**
CEM II 0.15	2.62	2.25	2.62	2.26	2.61	2.24
	0.01*	0.01*	0.01*	0.02*	0.01*	0.02*
	0.23**	0.63**	0.35**	0.14**	0.21**	1.06**
CEM III 0	2.63	2.30	2.63	2.34	2.60	2.31
	0.01*	0.00*	0.00*	0.01*	0.01*	0.01*
	0.47**	0.02**	0.07**	0.11**	0.20**	0.57**
CEM III 0.15	2.57	2.12	2.57	2.18	2.57	2.21
	0.00*	0.00*	0.00*	0.01*	0.01*	0.01*
	0.09**	0.13**	0.03**	0.59**	0.31**	0.58**

Note: *standard deviations, **coefficients of variation.

Table 10. Air content of tested concretes

Series	Air content after 28 days	Air content after 56 days	Air content after 90 days
	(%)	(%)	(%)
CEM I 0	12.88	12.50	12.49
CEM I 0.15	20.38	20.00	17.74
CEM II 0	12.17	11.79	11.41
CEM II 0.15	14.12	13.74	14.18
CEM III 0	12.55	11.03	11.15
CEM III 0.15	17.51	15.18	14.01

specimens matured. The exception was the CEM II 0.15 concrete, where a slight increase in porosity was noted after 90 days of curing compared to the 56-day results. The highest tightness after 28 days of curing was noted in the CEM II 0 concretes, at approximately 88%, while after 56 and 90 days, the highest tightness, approximately 89%, was observed in the CEM III 0 series specimens.

Mass water absorption tests are compatible with porosity tests. The largest water absorption was observed in the CEM I 0.15 concretes, which decreases as the curing period progresses. It is worth noting that CEM I 0.15 series also had the highest air content within the concrete mix (6.1%). Among the concretes tested after 28 days of curing, the lowest water absorption value of 3.6% was obtained in concretes with slag cement CEM III. This series also exhibited the lowest water absorption after 56 and 90 days of curing, at 3.5%.

The obtained results confirm the studies by Giergiczny et al. [34], who examined concretes made with CEM I, CEM II/B-M (S-V), and CEM III/A cements after 28 and 90 days of curing. The authors observed higher tightness and lower water absorption in concretes made with slag cements compared to pure Portland cement, despite using an increased amount of air-entraining admixture. Similar conclusions were noted in studies on fly ash-slag concretes by Sikora et al. [58]. The compressive strength results of the concretes after 28, 56, and 90 days of curing are shown in Figure 4 and Table 11. Classifying the non-air-entrained concretes based on their 28-day compressive strength revealed that all achieved the same class, C50/60, which is somewhat unexpected for the CEM III concrete. It was anticipated that this

concrete would have both a significantly lower average strength and a lower class compared to the other concretes due to the 42.5N cement class. Differences appear in longer curing periods. Both the CEM II 0 and CEM III 0 concretes experienced an increase in strength, resulting in a class upgrade to C55/67, meaning that according to EN 206, they are classified as high-strength concretes [21]. The introduction of an air-entraining admixture caused a significant decrease in strength in all concretes, the largest being in the CEM I 0.15 concretes, reaching up to 34%, and in the CEM III 0.15 concretes, up to 28%, with the smallest decrease in the CEM II 0.15 concretes at 15%. Consequently, the concrete classes were reduced to C30/37 for CEM I 0.15, C45/55 for CEM II 0.15, and C30/37 for CEM III 0.15. This aligns with the air content within the concrete mixes (highest in CEM I 0.15 and lowest in CEM II 0.15) as well as the quantity and pozzolanic activity of the additives introduced into the cements.

Expected compressive strength results over time calculated in accordance with ACI 209 [59] are shown in Table 12. The results show a comparison of the achieved compressive strengths of the concretes after 28, 56 and 90 days with the expected strengths and calculated according to ACI 209 [59]. The study showed that the CEM I 0 and CEM II 0 concretes had not achieved the expected compressive strengths after 56 and 90 days of curing, obtaining about 3.7–5.1 MPa lower results than expected. In contrast, CEM III concretes where the achieved compressive strengths are higher than expected. In the case of the aerated concretes, in all cases results exceeding the expected results by about 1.4-6.9 MPa were achieved.

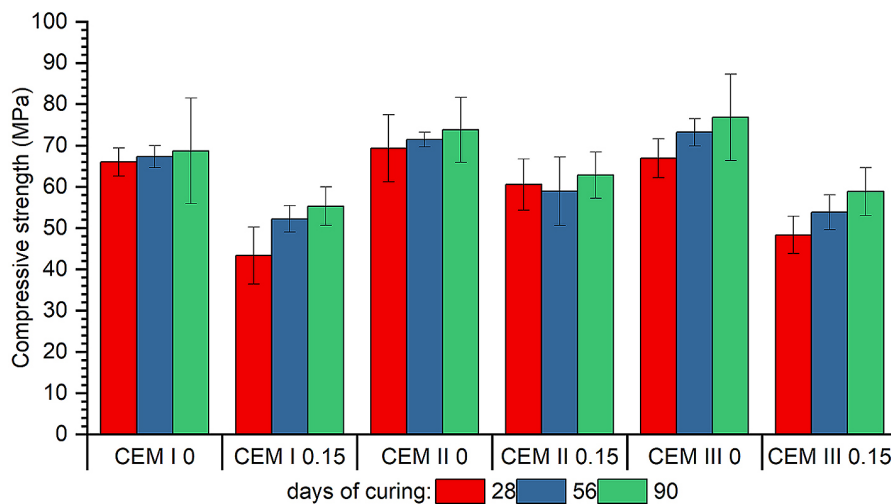


Figure 4. Compressive strength of tested concretes

Table 11. Compressive strength of tested concretes after 3 different curing times (28, 56, 90 days)

Series	Compressive strength after 28 days	Compressive strength after 56 days	Compressive strength after 90 days
	(MPa)	(MPa)	(MPa)
CEM I 0	66.0	67.3	68.7
	3.38*	2.68*	6.21*
	4.06**	3.66**	12.74**
CEM I 0.15	43.3	52.2	55.3
	6.95*	3.16*	4.72*
	14.14**	8.33**	11.86**
CEM II 0	69.3	71.4	73.8
	8.08*	1.80*	7.94*
	10.44**	2.03**	9.55**
CEM II 0.15	60.5	58.9	62.8
	6.23*	8.31*	5.58*
	8.47**	12.03**	8.55**
CEM III 0	66.9	73.2	76.8
	4.68*	3.28*	10.49*
	5.51**	3.45**	11.90**
CEM III 0.15	48.3	53.8	58.8
	4.48*	4.20*	5.80*
	7.86**	8.59**	12.32**

Note: *standard deviations, **coefficients of variation.

Table 12. Expected compressive strength over time calculated in accordance with ACI 209 [59]

Series	Achieved compressive strength after 28 days	Expected compressive strength after 56 days calculated in accordance with ACI 209	Achieved compressive strength after 56 days	Expected compressive strength after 90 days calculated in accordance with ACI 209	Achieved compressive strength after 90 days
	(MPa)	(MPa)	(MPa)	(MPa)	(MPa)
CEM I 0	66.0	71.6	67.3	73.8	68.7
CEM I 0.15	43.3	47.0	52.2	48.4	55.3
CEM II 0	69.3	75.2	71.4	77.5	73.8
CEM II 0.15	60.5	65.7	58.9	67.6	62.8
CEM III 0	63.9	72.6	73.2	74.8	76.8
CEM III 0.15	48.3	52.4	53.8	54.0	58.8

The analysis results of parameters describing the air pore characteristics are shown in Table 13. Despite the lack of information in Polish standards regarding the threshold values of key parameters of pore distribution in concrete, older international literature provides information on these values and their impact on the characteristics of air pores [11].

The most used parameters for assessing the frost resistance of air-entrained concretes are the micropore content A_{300} and the spacing factor. The spacing factor determines the maximum distance of any point within the cement paste in millimeters from the edge of an air pore. The

micropore content A_{300} is a parameter that defines the air content in air pores with a diameter up to 300 μm [49].

The technical requirements for the pore structure in hardened concrete can be found in the document issued by GDDKiA in 2019 regarding Cement Concrete Pavements D-05.03.04 [60]. According to this document, the micropore content A_{300} should not be less than 1.5%, and the pore spacing factor should not exceed 0.200 mm [61, 62]. In accordance with these guidelines, all air-entrained concretes achieved the intended parameters, with the pore spacing factor ranging from 0.090 to 0.127 mm and the micropore

Table 13. Properties of air pores according to EN 480-11 [49]

Series	28-days specimens		56-days specimens		90-days specimens	
	Micropore content A_{300}	Spacing factor \bar{L}	Micropore content A_{300}	Spacing factor \bar{L}	Micropore content A_{300}	Spacing factor \bar{L}
	(%)	(mm)	(%)	(mm)	(%)	(mm)
CEM I 0.15	3.93	0.104	4.11	0.111	4.03	0.117
CEM II 0.15	4.85	0.090	4.55	0.105	4.61	0.099
CEM III 0.15	3.96	0.112	3.79	0.127	3.88	0.115

content ranging from 3.79 to 4.85%. The highest A_{300} values at all tested curing periods were found in the CEM II/B-M (S-V) series concretes, while the lowest values were observed in the CEM III/A series concretes. Furthermore, there appears to be a correlation between the decrease in the spacing factor and the increase in A_{300} content.

According to Neville [10], fly ash in Portland cement can improve the characteristics of air-entrained concrete by creating more stable and finer air bubbles, which can lead to enhanced frost

resistance. Findings from the tests assessing frost resistance are showcased in Table 14 and Figure 5.

The frost resistance results can be considered quite surprising due to the compressive strength reductions, especially after 28 days of concrete curing. The mass losses of the concretes are negligible, as confirmed by the macroscopic evaluation of the specimens. Theoretically, the best compressive strength results were expected in the CEM I concretes, considering their highest Portland clinker content and thus the fastest development of

Table 14. Results of frost resistance of tested concretes after different curing periods following 150 F-T cycles

Specimens	28-days specimens		56-days specimens		90-days specimens	
	Mass difference	Compressive strength difference	Mass difference	Compressive strength difference	Mass difference	Compressive strength difference
	(%)	(%)	(%)	(%)	(%)	(%)
CEM I 0	-0.6	18.4	-0.7	15.5	-0.5	14.7
CEM I 0.15	0.1	8.2	0.2	6.7	0.2	6.2
CEM II 0	-0.1	10.3	0.0	7.6	0.0	2.6
CEM II 0.15	0.2	4.6	0.1	5.6	0.1	0.4
CEM III 0	0.0	12.1	0.0	11.7	0.0	9.5
CEM III 0.15	0.2	9.6	0.2	7.4	0.2	0.7

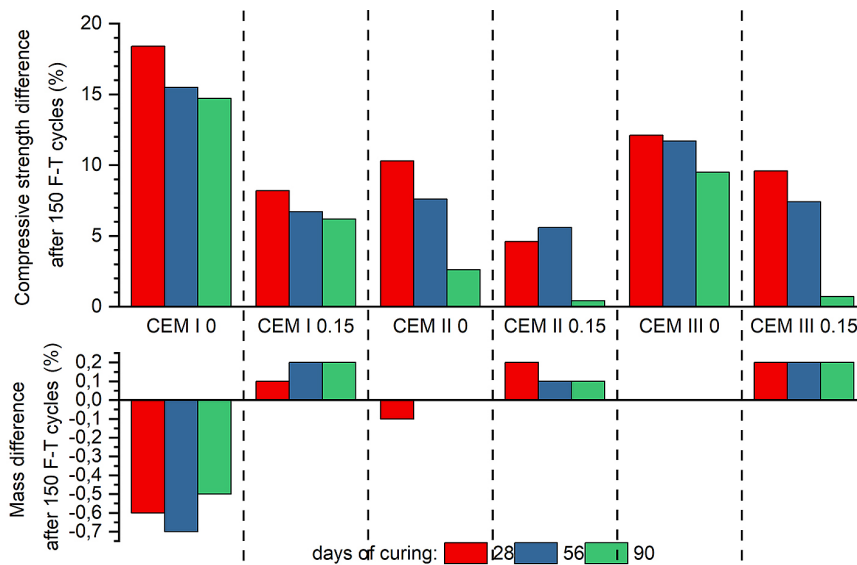


Figure 5. Frost resistance of tested concretes

a dense structure in the hardened cement paste. Nevertheless, the outcomes indicated all CEM I 0 and CEM I 0.15 concretes exhibited the worst strength parameters. This was particularly noted in the CEM I 0 concrete, where the compressive strength decrease was on the verge of the allowable 20% drop according to PN-B-06250 [26], being 18.4% after 28 days of curing and around 15% in later curing periods. In the other concretes, CEM II and CEM III, the strength reductions were lower, by 8%, with slightly better values obtained in the CEM II and CEM II 0.15 concretes. It's important to highlight that the inclusion of the air-entraining admixture at 0.15% of the cement mass significantly improved the strength characteristics. In comparison, air-entrained concretes experienced half the compressive strength reduction observed in non-air-entrained concretes. The impact of curing time on the strength reductions of the frozen concretes was observed, with larger differences occurring in the air-entrained concretes, particularly in the CEM II 0.15 and CEM III 0.15 concretes. In the CEM II 0.15 concrete, the strength reduction after 28 days of curing was 4.6%, and after 90 days, it was 2.6%. In the CEM III 0.15 concrete, the corresponding reductions were 9.6% and only 0.7%. Similar results were also obtained from Rudnicki et al. [53], who studied the frost resistance of air-entrained pavement concretes with CEM I 42.5 R-NA and CEM III/A 42.5 HSR-NA cements following 150 cycles of freezing and thawing. In these studies, both types of concrete met the requirements of PN-B-06265 [22], but the smallest strength reductions, around 4%, were obtained in the concrete with CEM III cement. However, some researchers indicate possible problems with the frost resistance of concretes when using low-clinker two- or three-component cements [63], particularly suggesting that concretes with three-component cements have lower resistance compared to those with two-component cements. The authors suggest a possible increase in capillary porosity, leading to more places where destructive freezing can occur. Consequently, this results in reduced resistance to freezing and thawing [63]. However, the obtained test results do not confirm these observations; on the contrary, the worst results were obtained in the CEM I concretes, both with and without the inclusion of the air-entraining admixture. Similar findings were reported by other researchers [64], who observed higher resistance in concrete with CEM/B 32.5 N-LH compared to concrete

with CEM I 42.5R when conducting frost resistance tests in the presence of de-icing salt after 28 cycles of freezing/thawing. Conversely, Giergiczny et al. [55] reached different conclusions, also studying the frost resistance of concretes when exposed to de-icing salts. They noted disturbances in the air void distribution in the air-entrained hardened concrete and consequently increased surface scaling of the tested low-clinker cements concretes (greater mass loss of specimens after frost resistance testing over 56 cycles of freezing/thawing, particularly in the CEM III/A concrete) despite good strength parameters after frost resistance testing. This suggests the need for further research on concrete elements exposed to more complex environmental conditions, such as horizontally embedded concretes (e.g., paving blocks, concrete pavements, sidewalk slabs, etc.), as the recommendations of PN-B-06250 [26], despite being in use in Poland for many years, may be insufficient.

CONCLUSIONS

The article discusses the physical, mechanical, and durability properties of concretes produced using various types of cement (CEM I, CEM II, and CEM III) tested at three-time intervals (28, 56, and 90 days of curing). From the results obtained, the following conclusions can be drawn:

1. Analyzing the compressive strength results, the extended curing time benefited only the low-clinker cements, CEM II and CEM III, by classifying these concretes into a higher strength class. The CEM I cement achieved the same compressive strength class after 28, 56, and 90 days of curing, likely due to the relatively stable concrete structure formed by the 28th day of curing.
2. Similarly, analysing the frost resistance of concretes based on strength reductions, it was found that concretes with CEM II and CEM III cements achieved significantly better results compared to CEM I. Using an air-entraining admixture, the strength reductions after 90 days of curing were almost negligible. This confirms the validity of the PN-B-06265 standard recommendations and the introduction of the so-called equivalent time in frost resistance testing. However, the test results indicate that for CEM II/B cements, this time should be extended to 90 days, similar to CEM III.

3. Cements with pozzolanic additives, such as CEM II and CEM III, reduce the effectiveness of air-entraining in concrete mixes. An air-entraining admixture at 0.15% of the cement mass resulted in air content levels of 4.3-6.1% in the concrete mixes, varying with the type of cement used. The highest effectiveness of the air-entraining admixture was observed in the mix with pure Portland cement (6.1%), while the lowest was in the mix with fly ash cement (4.3%).
4. All air-entrained concretes exhibited increased porosity. The largest increase in porosity was observed in CEM I 0.15 concretes, which was approximately 55% greater compared to concrete without air entrainment. The smallest increase in porosity was noted in CEM II 0.15 concretes, which was about 17% higher than in CEM II 0 concretes. Over time, a decrease in total porosity was observed in almost all tested series, except for the CEM II 0.15 series, where a slight increase in porosity was noted after 90 days of curing compared to the 56-day curing results;
5. The lowest mass water absorption was observed in the CEM III series concretes, with 3.5% after 28 days and 3.6% after 56 and 90 days of curing.
6. All air-entrained concretes met the recommendations specified by GDDKiA [60], achieving a pore spacing factor within the range of 0.090–0.127 mm and micropore content within the range of 3.79–4.85%. The highest micropore content A_{300} at all tested curing periods was found in the CEM II/B-M (S-V) series concretes, while the lowest values were observed in the CEM III/A series concretes. Analysing the results revealed a correlation between the increase in A_{300} micropore content and the decrease in pore spacing factor.

The findings of this study indicate that it is not only possible to use low-clinker cements for frost-resistant concretes, but it is also possible to achieve higher resistance to cyclic freezing and thawing with these cements in comparison to concretes with CEM I cements.

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