WARSAW UNIVERSITY OF TECHNOLOGY Index 351733
FACULTY OF CIVIL ENGINEERING
COMMITTEE FOR CIVIL AND WATER ENGINEERING
POLISH ACADEMY OF SCIENCES ISSN 1230-2945

DOI: 10.24425/ace.2024.149868
ARCHIVES OF CIVIL ENGINEERING
Vol. LXX ISSUE 2 2024

pp. 357 –369

Research paper

Assessment of the properties of mortars made of sorel cement

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Abstract: The purpose of laboratory tests was to determine the effect of sodium silicate and selected hydrophobic agents on the basic physical parameters and freeze-thaw durability of mortars made with Sorel cement in variable proportions. In order to determine the mortars' parameters, samples of the dimensions of $4 \times 4 \times 16$ cm and boards of the diameters of $1 \times 4 \times 16$ cm were prepared. Parameters such as water absorption, capillary absorption, compressive and flexural strength and frost resistance were tested. Mortar supplemented with sodium silicate in the quantity of 2.6% of all components demonstrated the best properties. None of the other hydrophobic agents that were used to mitigate the negative effects of water on Sorel cement mortars demonstrated such positive properties. Flexural strength tests of all mortar batches, performed on cuboid samples and boards of the thickness of 1 cm, demonstrated a similar improvement in strength. The lowest value of compressive strength was recorded for the reference batch at 46.6 MPa, whereas the highest value was recorded for the second batch containing sodium silicate, at 49.8 MPa. During the testing of frost resistance, the lowest reduction of compressive and flexural strength was recorded for the reference mortar and for mortar with sodium silicate. All mortars were varied in the MgO/$MgCl_2$ ratio and the total amount of water, the observed effects may be caused by other variables. However it is possible to notice the positive effect of selected hydrophobic agents.

Keywords: magnesium oxychloride cement, sodium silicate, hydrophobic agents, freeze-thaw durability

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1. Introduction

The reduction of the amount of carbon dioxide and the associated carbon footprint is the main direction of construction development. This can be achieved in many known ways including modifications in the concrete mix (i.e. applying mineral additives or industrial waste), but also using cement substitutes [1, 2]. Products that utilize magnesium oxychloride cement as a binder can demonstrate low compressive and flexural strength. This is because of their low resistance to atmospheric conditions, among other factors [3]. To protect elements against the detrimental effects of water, their surfaces were covered in various coatings e.g. wax and turpentine [4]. Today Sorel cement can be used for the production of industrial floors, indoor plasters, imitation marble and other stone products, or for the repair or renovation of historic buildings, [5, 6]. Researchers renewed their interest in materials based on magnesium binder following the Olympic Games in Beijing, where MgO boards were used to finish Olympic venues [7]. Apart from a number of advantages (resistance to abrasion, fire, salt, quick setting and curing, high early strength, and others), elements made of Sorel cement also have many disadvantages that need to be considered to avoid misusing them in the construction process [8]. The most common disadvantages of elements made of Sorel cement are their brittleness and poor resistance to variable weather conditions, including freeze-thaw durability [8–14]. Despite the many publications from around the world on Sorel cements [15], it is still a topic of great interest to many researchers [16, 17]. The mechanisms of chemical reactions in the formation of magnesia cements were explained already in the 1950s [18, 19]. A summary of the tests on Sorel cement was compiled and presented by W. Kurdowski and F. Sorrentino in 1983 [20]. The authors paid particular attention to the possibility of improving its resistance to atmospheric conditions by using compounds such as sulphates, borax, or mixtures of calcium sulphate and silicates. Researchers also successfully used melamine and formaldehyde urea during testing. The addition of these agents delayed the growth of compressive strength, but improved water resistance of elements made of Sorel cement. Many researchers [21, 22] have carried out wide-ranging laboratory tests to determine the resistance of MgO elements to water. The analysis of the results of tests performed on elements made of Sorel cement has led to the idea that the introduction of additives to the cement slurry might be a simple, but effective method of improving their resistance to water. The problem with this method is that the quantity of soluble phosphate ions in a solution that could effectively improve the resistance of elements to water has to be very small (below 1% of the weight of slurry) [23]. The authors have proven that the addition of orthophosphoric acid H_3PO_4, or preferably soluble phosphates, can greatly improve the water resistance of Sorel cement slurry by improving the endurance of such elements, they has also examined how soluble phosphates can improve the water resistance of elements made of magnesia cement [24]. Soluble phosphates can significantly improve the resistance of elements to water. So the strength retention factor of hardened slurry increases with the increase of the mass fraction of phosphates in the slurry. If the mass fraction of phosphates is between 0.5–1.0% of the slurry mass, it means that the mortar is suitable for most engineering applications. There is a number of specific applications in the building sector where Sorel cement can and should be used, but there are also environments where this cement should not be used for the production of certain elements [25, 26]. Every engineer must remember to perform preliminary laboratory tests before any new application of this binder in the building industry [27]. The subject of Sorel cement tests and application has been only vaguely discussed in existing literature [2, 3]. The issue that needs to be resolved is the determination of the effect of sodium silicate, 12 mm long glass fibers and selected hydrophobic agents on the degree of reduction of the influence of atmospheric conditions on the parameters of elements consisting of Sorel cement. The reduction of the influence of atmospheric conditions on the durability parameters of elements consisting of Sorel cement still poses a major challenge.

2. Testing programme

The purpose of the planned laboratory tests was to determine the effect of sodium silicate and selected hydrophobic agents on the basic physical parameters and freeze-thaw durability of mortars made with Sorel cement in variable proportions. To conduct research four batches of mortars with different proportions of the specific components have been prepared. The composition of all mortars was prepared in accordance with Table 1.

Glass bubbles used in the mortars was made of glass cullet that was milled to the form of fine powder. It demonstrates properties such as non-flammability, low density and high compressive strength. It also has good noise and thermal insulating properties. It is also resistant to alkali and other chemical compounds. Glass bubbles are very light, weighing in the range of 140–530 kg/m^3, Adding tis material to concrete mixtures or mortars may contribute to the reduction of the weight of the end product. Additionally, the fact that glass bubbles are almost spherical in shape means that the mortar is more workable, which for example facilitates the spreading of mortar over the surfaces of walls or ceilings [29]. Glass fibers added to the mortars were 12 mm long. They contribute to the reduction of shrinkage cracks and microfractures, reduce absorption and water permeability, increase frost resistance and contribute to higher resistance to abrasion, compression or tension. Mortars with fibers demonstrate better workability and resistance to the segregation of components. Fibers were added to mortar during the mixing of dry components. The parameters of the fibers were as follows: diameter 30.5 microns, density 0.91 g/cm^3, Young modulus 3500 N//mm^2, tensile strength 0.32–0.42 kN/mm^2, melting point 168°C, zero absorbency, full chemical resistance, transparent/white color. Magnesium oxide is used for the production of building materials. Elements made of magnesium oxide are fire resistant, resistant to moisture and mold and demonstrate high endurance. Magnesia is highly resistant to fire, even in high temperatures, but has low electrical conductivity, which means that it can transfer heat but doesn't transfer electric power. Glass powder used for the production of the mortars was made of high quality milled glass. Its main advantage is its low absorption. It is a non-flammable material that does not dissolve in water, odorless, with a melting point of approx. 1000°C. The granularity of powder used for testing was between 0 μm and 125 μm. The chemical composition of the glass powder is as follows: $Fe_2O_3 < 0.2\%$; MgO

Table 1. Composition of mortars used for laboratory tests

Batch name	Components	Quantity, [kg/m^3]	Content of component, [%]
1	MgO	998	39.0
	$MgCl_2$	592.8	23.1
	Glass powder	553.3	21.6
	Glass fibre, length 12 mm	10.7	0.4
	Glass bubbles 1–2 mm	170.7	6.7
	Water	224	8.7
	Polycarboxylate plasticiser	13.3	0.5
2	MgO	952.7	42.1
	MgCl2	691.3	30.6
	Glass powder	129.1	5.7
	Glass fibre, length 12 mm	12.5	0.6
	Glass bubbles 1–2 mm	199.2	8.8
	Water	196.8	8.7
	Polycarboxylate plasticiser	15.5	0.7
	Sodium silicate	64.5	2.9
3	MgO	940.2	36.7
	$MgCl_2$	563.6	22.0
	Glass powder	527.7	20.6
	Glass fibre, length 12 mm	12.8	0.5
	Glass bubbles 1–2 mm	161.4	6.3
	Water	286.9	11.2
	Polycarboxylate plasticiser	20.5	0.8
	Surface-applied water-repelling agent	48.7	1.9
4	MgO	940.2	36.7
	$MgCl_2$	563.6	22.0
	Glass powder	527.7	20.6
	Glass fibre, length 12 mm	12.8	0.5
	Glass bubbles 1–2 mm	161.4	6.3
	Water	286.9	11.2
	Polycarboxylate plasticiser	56.4	2.2
	Internal water-repelling agent	12.8	0.5

$< 4.0\%$; $Na_2O > 14\%$; $SiO_2 > 65\%$; $CaO > 8.0\%$; Al_2O_3 0.5–2.0%, [30]. Magnesium chloride is a highly hygroscopic material that easily dissolves in water. When mixed with water, it transforms into hexahydrate $MgCl_2 \cdot 6H_2O$ of the density of 1.57 g/cm^3, which decomposes in temperatures above 117°C. Magnesium chloride was dissolved in a part of the mixing water and added to dry components before the addition of mixing water. The aim to addition of sodium silicate to mortar was to increase its resistance to moisture and to protect the mortar against water penetration. A measured amount of sodium silicate was mixed with some of the mixing water and poured into the wet mortar components at the end of mixing. The polycarboxylate plasticizer was added to the mortars to reduce the amount of mixing water without affecting the consistency of the mortars. It enables the production of stronger, high-density mortars. The plasticizer was added directly to wet mortar components. The water-repelling agent is an additive that protects mortar against the effects of rainfall, surface water, air moisture and ground water. Mortar with this additive demonstrates lower capillary absorption and higher frost resistance. The surface-applied water-repelling agent is a single-component, reactive silane-based paste – commercial product available on the market. This product reduces the absorption of water and aggressive substances present in the air and in the water and also improves frost resistance. Additionally it doesn't form a layer and does not affect vapor permeability. Water-repelling impregnation was applied in accordance with standard PN-EN 1504-2. After 7 days of curing, the samples were coated twice using a surface-applied water repelling agent. The interval between the application of successive layers was 24 hours. The depth of penetration was $\geq$ 10 mm. The capillary absorption test was performed in accordance with PN-EN 13057:2004, [31] on $4 \times 4 \times 16$ cm samples. After the removal of the formwork, samples were cured for 7 days in dry air conditions, at +18°C. Afterwards, the samples were placed in an air conditioned chamber at +40°C for a period of 72 h. Before starting the test, the samples were cooled down, weighed and placed in a water-filled container, submerging them to the level of 3–5 mm. The water was regularly refilled. Changes in the weight of the samples were recorded. The samples were weighed after 12 min, 30 min, 1 h, 2 h, 4 h, 24 h, until the recording of two identical weight measurements of the samples. The standard water absorption test was performed in accordance with PN-88/B-06250 [32]. Cuboid samples of the dimensions $4 \times 4 \times 16$ cm were prepared for this test. The preparation of these samples was identical to their preparation for the capillary rise test. Dry samples were weighed and submerged in a vessel to the level of ½of their height. The samples were completely submerged in water after 24 hours. Changes in weight were measured every 24 hours, until the recording of two identical measurements. Flexural strength was tested in accordance with standard PN-EN 12390-5:2001 [33]. Before testing, samples were cured in constant temperature/humidity conditions at ±18°C for a period of 14 days. The test consisted of the measurement of the flexural strength of cuboid beams of the dimensions $4 \times 4 \times 16$ cm and previously cut out and prepared slabs of the dimensions $1 \times 4 \times 16$ cm. Frost resistance was tested in accordance with standard PN-88/B-06250. This method is based on the estimation of the loss of weight and compression strength of tested samples that were subject to periodic frosting and de-frosting. Before being placed in a chamber, the samples were saturated with water. The samples were then weighed and placed in an air-conditioned chamber. The samples were frozen for 4 h at – 18°C ±2°C, and were then de-frosted when fully submerged in water for 4 hours at +18°C ±2°C. The samples were frozen for 4 h at – 18°C ±2°C, and were then de-frosted when fully submerged in water for 4 hours at +18°C ±2°C.

3. Research results

According to standard PN-EN 13057, the results of capillary absorption tests should be given as per the square root of time in hours as it has been presented on Fig. 1.

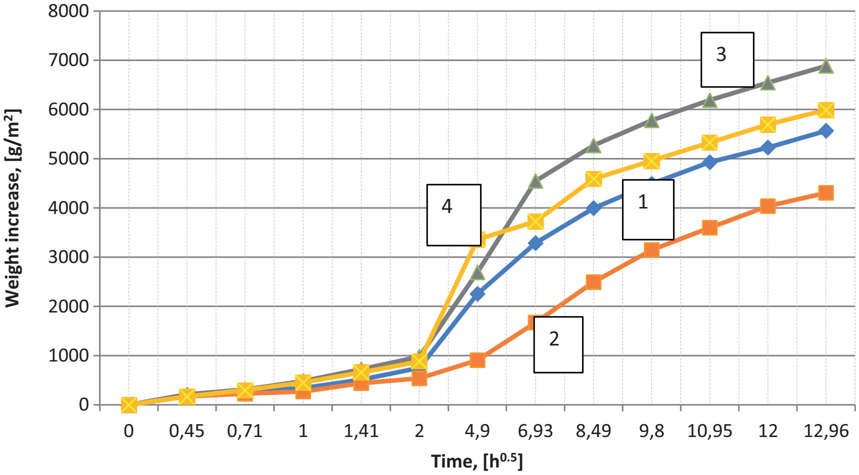

Fig. 1. Increase in weight of tested mortars due to capillary absorption, [internal research]

The obtained results demonstrated that the lowest weight increase due to capillary rise was recorded for the second batch mortars, at 4.3 kg/m^2. These mortars, apart from the standard components, contained sodium silicate, which significantly sealed the capillary pores of the mortars. This mortars also differs significantly in the MgO/MgCl$_2$ ratio and the amount of water, the observed effect may also be due to other variables. The highest weight increase was recorded for the third batch of mortars. The first and fourth batch achieved similar weight increases below 6 kg/m^2. The results of water capillary absorption test in percent of total weight increase has been presented on Fig. 2.

The performed laboratory tests have revealed that the lowest weight increase during absorption tests was recorded for 1st batch mortars, at 4.6%. The highest weight increase was recorded for 4th batch mortars which contained a water-repelling agent, at 6.1%. Flexural strength tests were performed on beam-shaped samples of the dimensions 4 × 4 × 16 cm as it has been shown in Table 2 and Fig. 3.

The highest flexural strength was recorded for the 2nd batch with the addition of sodium silicate, which stood at 9.97 MPa after 14 days of curing. The lowest value of flexural strength was recorded for the 4th batch, at 8.27 MPa. A list of flexural strength results recorded for slabs of the dimensions 1 × 4 × 16 cm is presented below in Table 3.

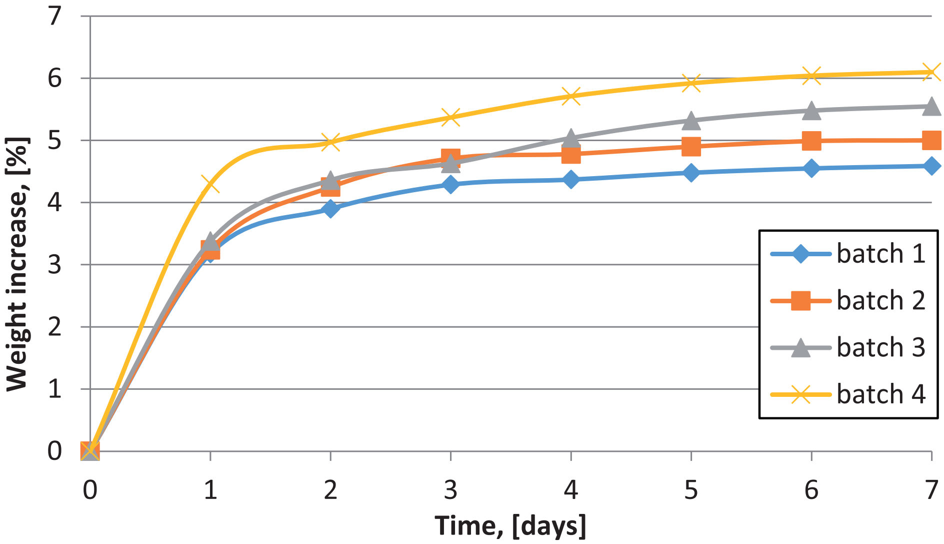

Fig. 2. Increase in weight of mortars during water absorption testing, [internal research]

Table 2. Increase in flexural strength of tested mortars on the basis of samples of the dimensions $4 \times 4 \times 16$ cm, [internal research]

Batch name	R_f, [MPa]	Standard deviation S	Coefficient of variation μ, [%]
1	9.3	0.657	7.11
2	9.9	0.508	5.09
3	9.8	0.59	6.02
4	8.3	0.58	6.97

Table 3. Increase in the flexural strength of slabs of the dimensions $1 \times 4 \times 16$ cm, [internal research]

Batch name	R_f, [MPa]	Standard deviation S	Coefficient of variation μ, [%]
1	9.79	0.345	17.34
2	9.7	0.698	14.85
3	9.97	0.568	5.7
4	9.93	0.467	4.69

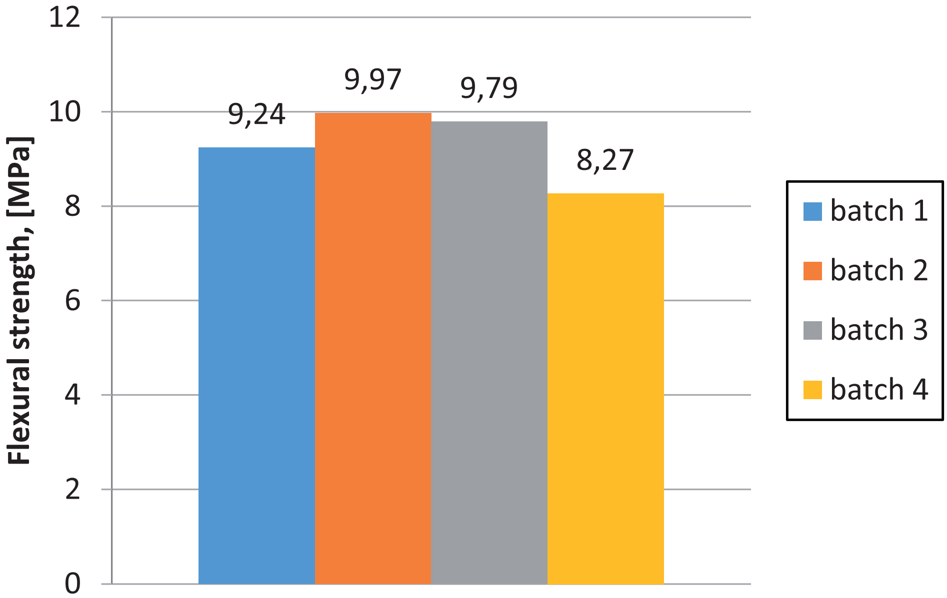

Fig. 3. Increase in flexural strength of tested mortars after 14 days of curing, [internal research]

The results of flexural strength tests obtained for 1 cm thick mortar slabs are slightly different from the flexural strength of tested beams of the dimensions 4 × 4 × 16 cm. Similar values of flexural strength were achieved for all batches. The testing of compressive strength was carried out on batches of the dimensions 4 × 4 × 16 cm, cured over a period of 14 days in dry air conditions at +18°C, results has been shown on Table 4 and Fig. 4.

Table 4. Increase in compressive strength of tested mortars, [internal research]

Batch name	R_f, [MPa]	Standard deviation S	Coefficient of variation μ, [%]
1	46.6	6.41	13.76
2	49.8	2.83	5.68
3	48.3	1.21	2.79
4	47.9	2.27	4.73

The highest value of compressive strength was recorded for the 2nd batch, which apart from the basic components included sodium silicate in the quantity of 2.6% of all components. The results for all other batches were similar. Relevant literature sources state that the final increase in compressive strength of Sorel cement mortars should amount to approximately 50 MPa [3]. During our tests, all batches demonstrated the correct value

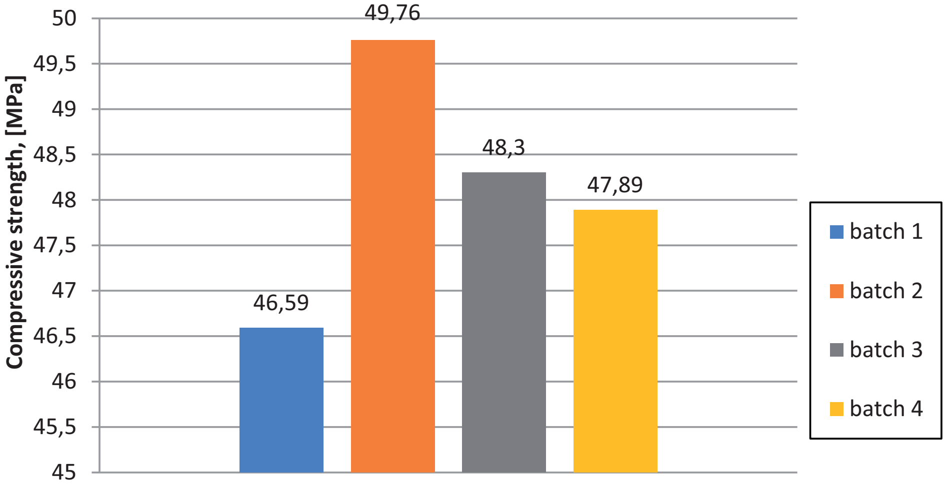

Fig. 4. Increase in compressive strength of tested mortars after 14 days of curing, [internal research]

of increase in compressive strength. In order to determine the percentage reduction in the flexural strength of mortars exposed to freeze-thaw cycles, the authors proceeded in accordance with guidelines described in PN-88/B-06250. During the test, samples were subjected to 150 freeze-thaw cycles (requirements for frost resistance level F150). Additionally the samples were inspected visually and periodically weighed in order to determine the variations in their weight, the results has been shown on Fig. 5 and Table 5.

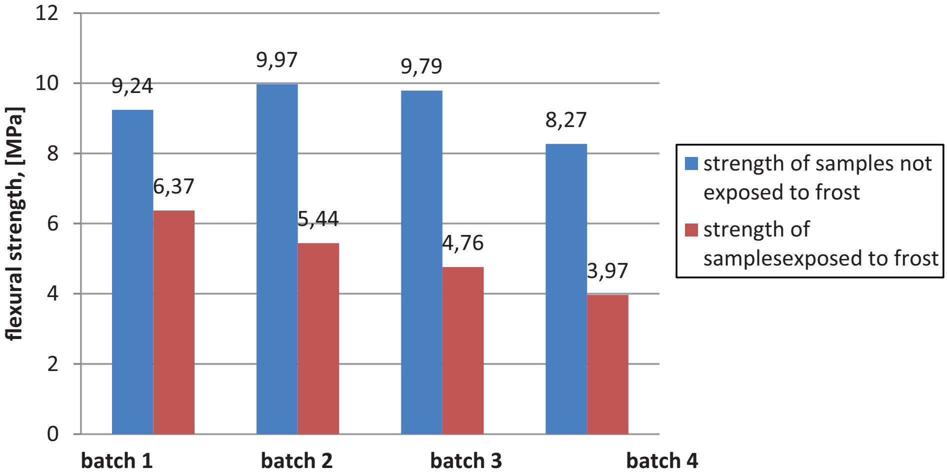

Fig. 5. Comparison of the flexural strength of mortars stored in laboratory conditions and exposed to frost, [internal research]

Table 5. The reduction of flexural strength of mortars exposed to frost, [internal research]

Batch name	R_f, [MPa]	Standard deviation S	Coefficient of variation μ, [%]
1	9.24	6.37	31.06
2	9.97	5.44	45.44
3	9.79	4.76	51.38
4	8.27	3.97	52.19

Analysis of the flexural strength tests performed on cuboid samples of the dimensions 4×4×16 cm that were exposed to frost has revealed that the 1st batch maintained the highest value of flexural strength. The loss of flexural strength for this batch amounted to 31.06%. This was a reference series that was not supplemented with any water-repelling agents. The highest loss of flexural strength was recorded for the 4th batch, which included the internal water-repelling agent, and amounted to 52.19%. The loss of compressive strength of samples exposed to frost was also determined, the results has been shown on Fig. 6 and Table 6.

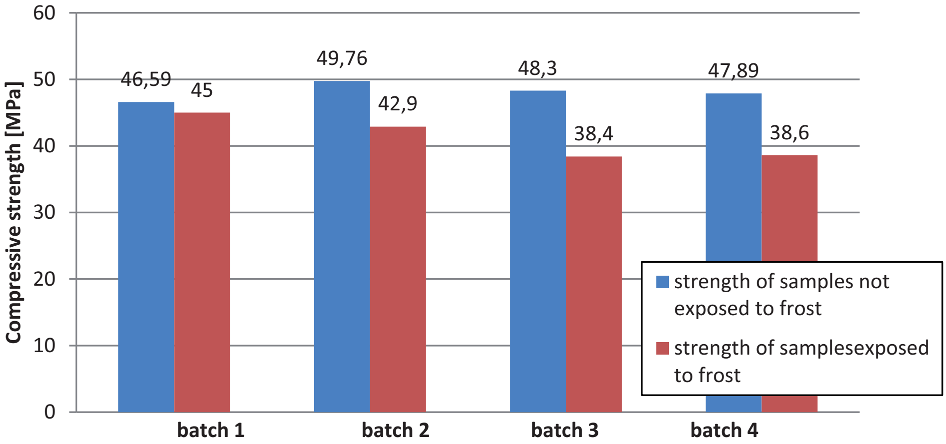

Fig. 6. Comparison of the compressive strength of mortar batches stored in laboratory conditions and exposed to freeze-thaw cycles, [internal research]

An analysis of the results of compressive strength tests of samples exposed to freeze-thaw cycles has revealed that the lowest loss of 3.46% was recorded for the 1st batch in relation to the reference batch stored in laboratory conditions. The highest loss of compressive strength was recorded for the 4th batch that was exposed to frost, of the value of 9.3 MPa, i.e. 19.34%.

Table 6. The reduction of compressive strength of mortars exposed to frost, [internal research]

Batch name	R_f, [MPa]	Standard deviation S	Coefficient of variation µ, [%]
1	46.6	45.0	3.46
2	49.8	42.9	13.85
3	48.3	38.4	18.34
4	47.9	38.6	19.34

4. Conclusions

The testing programme comprised the preparation of four batches of mortars, which differed in terms of their composition and utilised different water-repelling agents. Following an analysis of the results of capillary absorption and standard water absorption, it was observed that the additive of sodium silicate can significantly improve the watertightness of the tested mortars. However, these mortars also differs significantly in the MgO/$MgCl_2$ ratio and the amount of water, the observed effect may also be due to other variables. The other agents did not demonstrate the sufficient weight-reducing properties of the tested mortars. Flexural strength tests performed on cuboid beams demonstrated that the addition of sodium silicate improved the properties of the tested mortars and increased their strength. But the results of flexural strength tests did not vary greatly for all batches. Flexural strength tests performed on mortar slabs of the thickness of 1 cm, on the other hand, revealed that samples of all batches achieved similar results to those achieved by beams of the dimensions $4 \times 4 \times 16$ cm. The results of compressive strength tests of the mortars have revealed that the batch with the addition of sodium silicate achieved the highest value of fcm at 49.8 MPa. The results of the strength tests for all mortars were very similar to one another, with the highest difference being 3.2 MPa between the reference batch and the batch with the sodium silicate additive. The results of laboratory tests of the frost resistance of mortars have revealed that mortars in the reference batch demonstrated the lowest loss in compressive and flexural strength in relation to mortars not exposed to freeze-thaw cycles.

Following the completion of the planned laboratory tests, we can conclude that the best properties were recorded for mortars of the 2nd batch with the sodium silicate additive. They demonstrated better properties during capillary rise tests, as well as flexural and compressive strength tests. All mortars were varied in the MgO/$MgCl_2$ ratio and the amount of water, the observed effect may also be due to many other variables, which can be continued in further research.

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Ocena właściwości zapraw wykonanych z cementu Sorela

Słowa kluczowe: cement Sorela, szkło wodne (krzemian sodu), środki hydrofobowe, mrozoodporność

Streszczenie:

Celem badań było określenie wpływu szkła wodnego oraz wybranych środków hydrofobowych na parametry zapraw wykonanych z cementu Sorela w zmiennych proporcjach. Do określenia parametrów zapraw wykonano próbki o wymiarach 4 × 4 × 16 cm oraz płyty o wymiarach 1 × 4 × 16 cm. Badano takie parametry jak: nasiąkliwość, podciąganie kapilarne, wytrzymałość na ściskanie i zginanie oraz mrozoodporność. Zaprawa z dodatkiem szkła wodnego w swoim składzie w ilości 2,6% wszystkich składników wykazała najlepsze właściwości. Pozostałe środki hydrofobowe zastosowane do ograniczenia negatywnego wpływu wody na zaprawy wykonane z cementu Sorela nie wykazują aż tak pozytywnych cech. Badania wytrzymałości na zginanie wykonane dla każdej serii zapraw na próbkach prostopadłościennych i płytach o grubości 1 cm wykazują podobne przyrosty wytrzymałości. Najmniejszą wytrzymałość na ściskanie zanotowano dla serii referencyjnej wynoszącą 46,6 MPa, zaś najwyższą dla serii drugiej (ze szkłem wodnym) – osiągnięto 49,8 MPa. W badaniu mrozoodporności po 150 cyklach zamrażania – rozmrażania najmniejszy spadek wytrzymałości na ściskanie i zginanie zanotowano dla zaprawy referencyjnej oraz zaprawy ze szkłem wodnym.

Received: 2023-08-11, Revised: 2023-09-12