The Effect of Hydrophilic Crystalline Admixtures on the Durability of Concrete

One of the causes of the deterioration of concrete buildings are the freeze-thaw cycles to which they are exposed. Namely, the water present in concrete turns into ice when freezing which is of a larger volume than the water from which it was formed and therefore creates pressure on the pore walls. As a result of repeated freeze-thaw cycles, concrete damage occurs in the form of surface scaling or in the form of internal cracking. Crystalline hydrophilic admixtures are powder components added to the concrete mix to reduce the waterproofing of the concrete. The hypothesis of this paper is that crystalline hydrophilic admixtures as substances that reduce the penetration of water into concrete could improve the durability of concrete. Three concrete mixtures were prepared; a reference mixture, a mixture with air entraining agent (additive commonly used to improve concrete's resistance to freeze-thaw cycles), and a mixture with a crystalline hydrophilic admixture. The compressive strength and waterproofing of the hardened concrete samples were tested before and after exposure to freeze-thaw cycles in the chamber. The resistance of concrete to freeze-thaw cycles was expressed through the ratios of these properties after and before the freeze-thaw cycles. The results confirm the possibility of using a crystalline hydrophilic admixture for the purpose of improving the durability of concrete.

Keywords: air entraining agent, crystalline hydrophilic admixtures, freezethaw cycles, compressive strength, waterproofing of concrete

Introduction

The durability of buildings is one of the fundamental requirements placed on building materials. One of the main factors in reducing the durability of materials is the freeze-thaw cycle [1]. Water present in the material during a temperature drop below zero freezes and turns into ice, which has a larger volume than the water from which it was formed, and thus formed ice creates stress on the walls of the material [2], which due to repeated freeze-thaw cycles leads to damage to the material, and thus to a reduction in the durability of the material. Such damage to cement composites occurs either in the form of surface scaling or in the form of internal cracking/damage [3].

According to European legislation, the resistance of concrete to freeze-thaw cycles is tested by procedures prescribed in standards CEN/TS 12390-9 [4] and CEN/TR 15177 [5]. In the procedure described in CEN/TS 12390-9 [4], concrete samples saturated with water or a 3% NaCl solution are exposed to freeze-thaw cycles (56 cycles) and during these cycles, surface scaling of concrete is measured and mass loss is monitored, while a procedure described in CEN/TR 15177 [5] can be used for monitoring damage to the internal structure.

The resistance of concrete to freeze-thaw cycles is usually improved by adding air entraining agents to the concrete mixture [6], and it can also be

2023-5423-CIV - 12 JUN 2023

improved by other additions to concrete such as slag [7], fly ash [8] and silica fume [9].

Crystalline admixtures (CA) are mostly commercially available products from different manufacturers (Xypex, Kryton, Penetron, Harbin) with a dual effect: they reduce the permeability of concrete and heal cracks and are recommended to be added to concrete in amounts of 0.3-2% by weight of cement [10]. The European standard EN 934-2 [11] classifies CA as additives for reducing the water permeability of concrete.

Zhang et al. [12] investigated the effect of CA on mechanical and transport properties and the self-healing ability of cement composite. CA was used in this study at replacement levels of 0, 1.5%, 3% and 4.5% by weight of cement. It was concluded that the addition of CA improves mechanical properties and that a higher content of CA had a more positive impact on mechanical properties. Also, water absorption (transport property) decreased in this study with increasing CA content in mixtures. Gojević et al. [13] in their paper investigate the effectiveness of CA on compressive strength, waterproofing, and self-healing ability of concrete with a water-cement ratio of 0.45 and 0.55. Two mixtures were made in this study for each water-cement ratio, with and without CA addition. The amount of CA in mixtures was 1% by weight of cement. The addition of CA had no effect on compressive strength for both mixtures but reduced water penetration depth. The addition of CA was more effective in reducing water penetration depth for mixtures with a lower water-cement ratio.

The hypothesis of this paper is that crystalline hydrophilic admixtures can improve resistance to freeze-thaw cycles through reduced water permeability. In this paper, we compare the effectiveness of crystalline hydrophilic admixtures and air entraining agents (commonly used concrete additive for improving resistance to freeze-thaw cycles) in terms of resistance to freezethaw cycles.

30

31

1 2

3

4

5 6

7

8

9

10

11

12

13

14 15

16

17

18

19

20

21 22

23

24 25

26 27

28

29

Experimental Part

32 33 34

In the experimental part of the paper, three concrete mixtures were mixed; a reference mixture (M1), a mixture with an air entraining agent (M2) and a mixture with a crystalline hydrophilic admixture (M3).

36 37

35

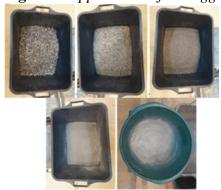
Properties of Aggregates, Binders, and Additives to Concrete

38 39 40

41 42

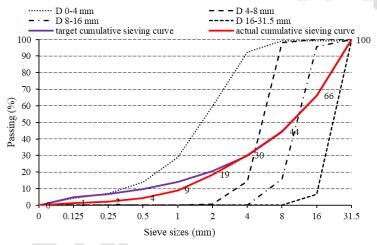
In this research, dolomite was used as an aggregate in fractions 0-4 mm, 4-8 mm, 8-16 mm, and 16-31.5 mm, as well as a dolomite-type filler. Figure 1 shows all aggregate fractions used in this research.

Figure 1. Appearance of the Aggregate and Cement used in the Study



The density of dolomite determined according to EN 1097-6 standard [14] was 2780 kg/m³. The density of filler determined according to EN 1097-6 standard [14] was 2780 kg/m³. Sieve curves for dolomite fractions are shown together with target and actual cumulative aggregate curve in Figure 2, where it should be noted that 5% of the 0-4 mm fraction was replaced with filler.

Figure 2. Fraction Sieve Curves, Target and Cumulative Sieving Curves



The cement used for making concrete mixtures was CEM I 52.5 N with a density of 2960 kg/m³ determined according to EN 196-6 [15].

In all mixtures, the superplasticiser ViscoCrete 5380 shown in Figure 3 was used in the amount of 1% of the mass of cement with a density of 1.08 g/cm³. In mixture M2, the air entraining agent LPS A 94 from Sika was used in the amount of 0.2% of the mass of cement. The density of the air entraining agent is 1.0 g/cm³. In mixture M3, the crystalline hydrophilic admixture Penetron Admix from Penetra shown in Figure 3 was used in the amount of 3% of the mass of cement the density of which was determined according to the standard EN 1097-6 [14] was 2910 kg/m³.

Figure 3. Superplasticiser (left) and crystalline hydrophilic admixture (right)





Composition of Concrete Mixtures

The composition of concrete mixtures is shown in Table 1.

Table 1. Composition of Concrete Mixtures for $1m^3$ of Concrete

Mixture/Components	M1	M2	M3
Cement (kg)	400	400	388
Water (kg)	140	140	140
v/c	0.35	0.35	0.35
Superplasticiser (kg)	4	4	4
Air entraining agent (kg)		0.8	-
Crystalline hydrophilic admixture (kg)	-	-	12
Aggeegate	1957.2	1957.2	1957.2
Dolomite 0-4 mm (kg)	576.6	576.6	576.6
Dolomite 4-8 mm (kg)	195.6	195.6	195.6
Dolomite 8-16 mm (kg)	469.8	469.8	469.8
Dolomite 16-31.5 mm (kg)	685	685	685
Filler (kg)	30.2	30.2	30.2

Properties of Fresh and hardened Concrete

 The consistency of the concrete was determined according to EN 12350-2 [16], the density of fresh concrete according to EN 12350-6 [17], and the pore content according to the standard EN 12350-7 [18]. Figure 5 shows the determination of consistency (a), pore content (b) and density (c). The obtained results are shown in Table 2.

Table 2. Results of fresh concrete testing

Mixture	M1	M2	M3
Consistency (cm)	12	14	11
Density (kg/m ³)	2504	2439	2489
Pore content (%)	1.5	5.0	1.6

Figure 4. Determining Consistency-A), Pore Content-B) And Density Of fresh concrete-c)







Subsequently, cubes (12 cubes of each mixture) measuring 15x15x15 cm were made for testing compressive strength and for determining the depth of water penetration under pressure, both properties before and after exposing the cubes to freeze-thaw cycles. The cubes were cured for 28 days in water and then removed from it, with 6 of them placed in the laboratory and 6 exposed to the action of 56 freeze-thaw cycles according to the procedure described in Item 7 of the CEN/TR 15177 standard [5]. On 3 cubes before and on 3 cubes after the freeze-thaw cycles, the compressive strength (Figure 5) was determined for each mixture according to the EN 12390-3 standard [19].

Figure 5. Testing the Compressive Strength of Concrete



On 3 cubes before and on 3 cubes after freeze-thaw cycles, the depth of water penetration under pressure (Figure 6) was determined for each mixture according to the EN 12390-8 standard [20].

Figure 6. Testing the Depth of Water Penetration under Pressure



Test Results and Discussion

As can be seen in Table 2, the results of testing the properties of concrete in its fresh state for all mixtures achieve a consistency of 12-14 cm according to the slump method, which classifies them in the consistency class S3 according to the EN 206 standard [21]. By looking at the same table, it can be seen that the concretes achieve densities of 2439-2504 kg/m³, and such concrete is classified as normal density concrete according to the EN 206 standard [21]. In the same table, it can be seen that mixture M2 has a higher pore content (5%), which is expected due to the addition of an air entraining agent. The results of compressive strength testing before and after freezing are shown graphically in Figure 7. The labels M1 P, M2 P, and M3 P are mixtures before freezing, while mixtures with labels M1 T, M2 T, and M3 T are treated mixtures, i.e. mixtures that have been subjected to 56 freeze-thaw cycles.

Figure 7. Compressive Strength for Mixtures M1, M2, and M3 before and after freezing

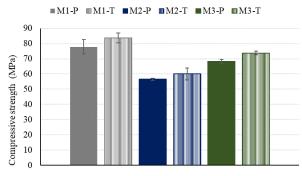


Figure 7 shows that mixture M1 has the best compressive strength before freezing while the worst is mixture M2, which can be expected because of high pore content due to the air entraining agent content in mixture M2.

Figure 8. Compressive Strength Ratio for Mixtures M1, M2, and M3 after and before freezing

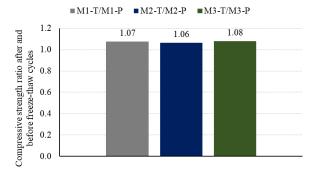


Figure 9. Relative Compressive Strength for all Three unfrozen Mixtures in Relation to M1-P

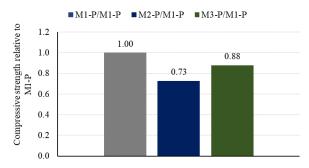
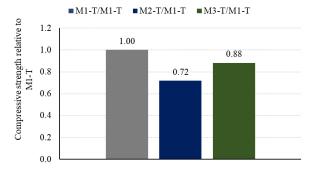


Figure 10. Relative Compressive Strength for all Three Frozen Mixtures in Relation to M1-T



In Figure 8, the ratio of compressive strength after and before freezing for all three mixtures is graphically shown and an increase in compressive strength can be seen in all three mixtures (by 6-8%), and the most in the mixture with the crystalline hydrophilic admixture. This means that the freeze-thaw cycles in the presence of water favoured the development of strength more than they damaged the internal structure of the samples. These results are in line with those shown in [12]. In Figures 9 and 10, the relative compressive strengths for all three mixtures are shown, for non-frozen samples in relation to the reference mixture M1-P (Figure 9), and for frozen samples in relation to the

reference mixture M1-T (Figure 10). From Figures 9 and 10, we can see that the crystalline hydrophilic admixture reduces compressive strength before and after freezing by 12% compared to the compressive strength of the reference mixture while the air entraining agent reduces the compressive strength of concrete by 27% before and 28% after freezing and thawing compared to the compressive strength of the reference mixture.

In Figure 11, the results of the depth of water penetration before and after freezing are graphically shown. In the mentioned figure, it can be seen that the crystalline hydrophilic admixture reduces the depth of penetration of water under pressure, as does as the air entraining agent. The reduction of water penetration by the addition of a crystalline hydrophilic admixture is in line with [13]. Since all three mixtures have a reduction in penetration of water under pressure in treated mixtures, this again means that the freeze-thaw cycles in the presence of water favoured the internal structure more than they damaged it. In Figure 12, from the ratio of the depth of penetration of water under pressure after and before freezing, for all three mixtures, we can see how the test results of the average depth of penetration of water under pressure after 56 freezethaw cycles for mixture M1 are 19% lower compared to the test results of the average depth of penetration of water under pressure for samples without freeze-thaw cycles. In mixture M2 from the shown ratio it can be seen how the water permeability of treated samples is 12% lower compared to untreated samples and in mixture M3 from the shown ratio it can be seen that there is the greatest reduction in water permeability of treated samples by 35% compared to untreated samples.

242526

27

1 2

3

4

5 6

7

8

9

10

11

12

13

14 15

16

17

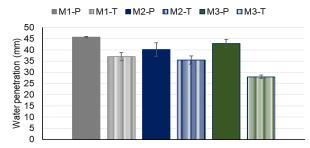
18 19

20

2122

23

Figure 11. Depth of penetration of water under pressure for mixtures M1, M2, and M3 before and after freezing



28 29

30

Figure 12. Ratio of the depth of penetration of water under pressure for mixtures M1, M2, and M3 after and before freezing

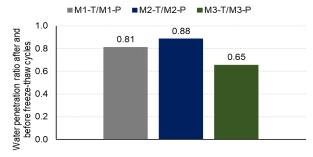


Figure 13. Relative depth of penetration of water under pressure for all three unfrozen mixtures in relation to M1-P

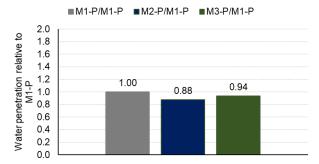
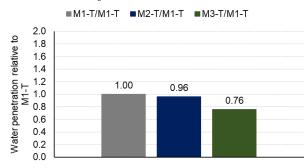


Figure 14. Three Frozen Mixtures Relative Depth of Penetration of Water under Pressure for all in relation to m1-t



 In Figure 13, it can be seen that the crystalline hydrophilic admixture reduces water penetration by 6% and the air entraining agent by 12% compared to the reference untreated mixture. In Figure 14, it can be seen that the crystalline hydrophilic admixture drastically reduces water penetration (by 24%), and the air entraining agent by 4% compared to the reference mixture treated with freeze-thaw cycles. Although in samples before exposure to freeze-thaw cycles, the air entraining agent is more effective than the crystalline hydrophilic admixture, after exposure to freeze-thaw cycles, the crystalline hydrophilic admixture has a more pronounced effect in terms of reducing water penetration than the air entraining agent.

Conclusion

In the experimental part of the paper, three concrete mixtures were mixed; a reference concrete mixture (M1), a concrete mixture with an air entraining agent (M2), and a concrete mixture with a crystalline hydrophilic admixture (M3) in the amount of 3% of the mass of cement. On hardened concrete samples, compressive strength and waterproofing were tested before and after exposing the samples to freeze-thaw cycles in a chamber, and the resistance of concrete to freeze-thaw cycles was expressed through the ratios of these

2023-5423-CIV - 12 JUN 2023

properties after and before the cycles. The results show that the crystalline hydrophilic admixture increases the compressive strength of concrete and drastically reduces penetration of water under pressure in samples that have been exposed to freeze-thaw cycles in a chamber, which leads to the conclusion that it reduces internal damage and increases the resistance of concrete to freeze-thaw cycles as one of the durable properties of concrete.

7 8

References

9 10

27

28

- [1] Surej K. R., (1997), Evaluation and Improvement of Frost Durability of Clay
 Bricks A Thesis in The Centre for Building Studies, Ottawa, Canada
- [2] Pilehvar S., Szczotok M. A., Rodríguez J. F., Valentini L., Lanzón M., Pamies R.,
 Kjøniksen A.-L., (2019), Effect of freeze-thaw cycles on the mechanical behavior
 of geopolymer concrete and Portland cement concrete containing micro-encapsulated phase change materials, Construction and Building Materials, Vol.
 200, p 94-103
- 18 [3] Richardson G. M., (2002), Fundamentals of Durable Reinforced Concrete, First Edition, Spon Press, p 51, 77, 101, 133, 160-179, 194
- [4] CEN/TS 12390-9:2016, (2016), Testing hardened concrete Part 9: Freeze-thaw
 resistance Scaling, CEN: Brussels, Belgium
- [5] CEN/TR 15177:2006, (2006), Testing the freeze-thaw resistance of concrete Internal structural damage, CEN: Brussels, Belgium
- [6] Qiu Y., Peng H., Zhao H., (2020), Study on New Type of Concrete Air-Entraining
 Agent, International Conference on Artificial Intelligence and Electromechanical
 Automation (AIEA), Tianjin, China
 - [7] Nicula L. M., Corbu O., Iliescu M., (2020), *Influence of Blast Furnace Slag on the Durability Characteristic of Road Concrete Such as Freeze-Thaw Resistance*, Procedia Manufacturing, Volume 46, p 194-201
- [8] Islam Md. M., Alam M. T., Islam Md. S., (2018), Effect of fly ash on freeze-thaw
 durability of concrete in marine environment, Australian Journal of Structural
 Engineering, Vol. 19(2), p 1-16
- 33 [9] Zang P., Li Q.-F., (2013), Freezing-thawing durability of fly ash concrete 34 composites containing silica fume and polypropylene fiber, Proceedings of the 35 Institution of Mechanical Engineers, Part L: Journal of Materials: Design and 36 Applications
- [10] García Calvo J. L., Sánchez Moreno M., Carballosa P., Pedrosa F., Tavares F.,
 (2019), Improvement of the Concrete Permeability by Using Hydrophilic Blended
 Additive, Materials, Vol. 12, 2384
- 40 [11]EN 934-2:2012, (2012), Admixtures for concrete, mortar and grout -- Part 2: 41 Concrete admixtures - Definitions, requirements, conformity, marking and 42 labelling, CEN: Brussels, Belgium
- [12] Zhang C., Lu R., Li R., Guan X., (2021), Effect of crystalline admixtures on
 mechanical, self-healing and transport properties of engineered cementitious
 composite, Cement and Concrete Composites, Vol. 124, 104256
- [13] Gojević A., Ducman V., Netinger Grubeša I., Baričević A., Banjad Pečur I.,
 (2021), The Effect of Crystalline Waterproofing Admixtures on the Self-Healing
 and Permeability of Concrete, Materials, Vol. 14, 1860

2023-5423-CIV - 12 JUN 2023

- 1 [14]EN 1097-6:2013, (2013), Tests for mechanical and physical properties of 2 aggregates - Part 6: Determination of particle density and water absorption, 3 CEN: Brussels, Belgium
- 4 [15] EN 196-6:2019, (2019), Methods of testing cement Part 6: Determination of fineness, CEN: Brussels, Belgium
- 6 [16] EN 12350-2:2019, (2019), Testing fresh concrete Part 2: Slump-test, CEN: 7 Brussels, Belgium
- 8 [17]EN 12350-6:2019, (2019), Testing fresh concrete Part 6: Density, CEN: Brussels, Belgium
- 10 [18] EN 12350-7:2019, (2019), Testing fresh concrete -- Part 7: Air content Pressure methods, CEN: Brussels, Belgium
- 12 [19]EN 12390-3:2019, (2019), Testing hardened concrete Part 3: Compressive strength of test specimens, CEN: Brussels, Belgium
- 14 [20] EN 12390-8:2019, (2019), Testing hardened concrete Depth of penetration of water under pressure, CEN: Brussels, Belgium
- 16 [21] EN 206:2021, (2021), Concrete Specification, performance, production and conformity, CEN: Brussels, Belgium