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Araştırma Makalesi / Research Article

Efficiency of Self-Healing Chemical Additives on the Freeze/Thaw Resistance of Cement Composites

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Abstract

Self-healing of concrete is the process in which the material regenerates itself healing present cracks. It can be either autogenous/natural self-healing or autonomous/artificial self-healing. Autonomous self-healing techniques include the use of crystalline hydrophilic additives, the bacterial method, and the microencapsulation method. Since internal cracking is one of the damage forms that are occurring by freeze/thaw cycles, the idea in this paper was to estimate the effectiveness of artificial self-healing techniques such as crystalline hydrophilic additive and microcapsules on the freeze/thaw resistance of cement composites. The reference mortar mixture, the mortar mixture with crystalline hydrophilic additive as well as the mortar mixture with toluene diisocyanate (TDI) microcapsules (both additives added in 2% by cement weight) were prepared. Hardened mortar samples are subjected to freezing and thawing cycles according to CEN/TS 12390-9:2006 and the amount of scaling material due to freeze/thaw cycles have been measured. The amount of scaling material was lower in case of both mortar mixtures with self-healing additives than in case of reference mortar mixture which indicate that self-

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healing chemical additives used here could improve resistance of the cement composites to freeze/thaw cycles.

Anahtar kelimeler: *Crystalline hydrophilic additive, TDI microcapsules, Cement composites, Freeze/thaw resistance*

Çimento Kompozitlerinin Donma/Çözülme Direnci Üzerinde Kendi Kendini İyileştiren Kimyasal Katkı Maddelerinin Etkinliği

Öz

Betonun kendi kendini iyileştirmesi, malzemenin mevcut çatlakları iyileştirerek kendini yenileme sürecidir Bu ya otojen/doğal kendi kendini iyileştirme ile ya da otonom/yapay kendi kendini iyileştirme ile olabilir. Otonom kendi kendini iyileştirme teknikleri arasında kristalli hidrofilik katkı maddelerinin kullanımı, bakteriyel yöntem ve mikroenkapsülasyon yöntemi yer alır. İçsel çatlamalar, donma/çözülme döngüleri tarafından meydana gelen hasar biçimlerinden biri olduğundan, bu makalenin amacı, kristal hidrofilik katkı maddesi ve mikrokapsüller gibi yapay kendi kendini iyileştirme tekniklerinin çimento kompozitlerinin donma/çözülme direnci üzerindeki etkinliğini değerlendirmektir. Bu amaçla, şahit harç karışımı, kristal hidrofilik katkılı harç karışımı ve toluen diizosiyanat (TDI) mikrokapsüllü harç karışımı (her iki katkı da çimento ağırlığına göre 2% oranında eklenmiştir) hazırlanmıştır. Sertleşmiş harç numuneleri CEN/TS 12390-9:2006 'ya göre donma/çözülme döngülerine tabi tutulmuş ve donma/çözülme döngülerinden kaynaklanan tufal malzeme miktarı ölçülmüştür. Kendi kendini iyileştiren katkı maddelerine sahip her iki harç karışımında tufal malzeme miktarının, burada kullanılan kendi kendini iyileştiren kimyasal katkı maddelerinin çimento kompozitlerinin donma/çözülme döngülerine karşı direncini artırabileceğini gösteren referans harç karışımına göre, daha düşük olduğu görülmüştür.

Anahtar kelimeler: *Kristal hidrofilik katkı maddesi, TDI mikrokapsüller, Çimento kompozitleri, Donma/çözülme direnci*

1. Introduction

The durability is one of the main requirements placed on building structures and materials. Freezing and thawing cycles are considered to be one of the main factors reducing the durability of these materials (Koroth, 1997). When the temperature drops below zero, water present in the material freezes and turns into ice having a larger volume than the water from which it was formed, and thus formed ice creates stresses on the walls of the material (Pilehvar et al, 2019). Repeated freeze/thaw cycles lead to material damage. Such damage in cement composites occurs either in the form of surface scaling or in the form of internal cracking (Richardson, 2002). According to the European legislation, the resistance of concrete to surface scaling has to be tested according to the Technical Specification CEN/TS 12390-9:2006 while the Technical Report CEN/TR 15177:2006 can be used to monitor the internal cracking occurred by freeze/thaw cycles (CEN/TS 12390-9:2006, 2006; CEN/TR 15177:2006, 2006). The resistance of a concrete to freeze/thaw cycles is usually improved by adding an air entraining agent to the mixture but it can also be improved by other admixtures such as slag, fly ash, and silica fume (Qiu et al, 2020; Nicula et al, 2020; Islam et al, 2018; Zang & Li 2013).

As previously mentioned, internal cracking (i.e. the formation of cracks in the interior of the cement composite) is one of the damage forms that are occurring by freeze/thaw cycles. The self-healing of cracks is an already known phenomenon, especially when it comes to autogenous or natural self-healing (Byoung Sun & Young, 2019). Namely, in cracked cement composite, the non-hydrated cement particles react with water that is present and this reaction restarts the hydration process creating hydration products that will fill the cracks. Autogenous healing can heal cracks up to 0.1 mm (Žáková et al, 2020; Park & Choi, 2020). However, nowadays, artificial methods of self-healing, the so-called autonomous self-healing, are employed which can make also the wider cracks healed. Autonomous self-healing techniques include the use of crystalline hydrophilic additives, the bacterial method, and the microencapsulation method (Danish et al, 2020). Crystalline hydrophilic additive can induce non-hydrated cement to form crystals that bridge the cracks in concrete, but for their effectiveness, a constant presence of water in concrete is necessary (Danish et al, 2020). Today, such crystalline hydrophilic admixtures are commercially available (Xypex, Kryton, Penetron) and it is recommended to put them in a concrete in the amounts of 0.3-2 % by weight of cement (Calvo et al, 2019; Cappellesso et al., 2016; García-Vera et al, 2019). Their primary purpose is to reduce the water permeability of concrete. Bacterial spores, on the other hand, produce calcite by their own respiration to repair cracks, and the healing process is very time-consuming (Danish et al, 2020). In the process of self-healing by the method of microencapsulation, microcapsules previously installed in concrete and settled in the path of crack formed, brake and their "healing" content is poured into the crack, sealing it (Danish et al, 2020). According to the content of the core material, microcapsules can be one-component and two-component (Du et al, 2019). When it comes to two-component, Kanellopoulos et al used gelatin and gum acacia as a membrane, and sodium silicate as a "healing" substance/core (Kanellopoulos et al, 2017). Li, Zhu, Zhao and Jiang (2016) used melamine urea-formaldehyde resin as a membrane and epoxy resin as a "healing" substance/core, with n-butyl glycidyl ether as the substance to synthesize the previously mentioned two components. Tong and Li (2013) used melamine urea formaldehyde as a membrane, epoxy resin as a core, and sebacate hydrazide as a substance for their synthesis. Du et al (2019, 2020, 2021) made capsules from paraffin as a membrane, toluene diisocyanate (TDI) as a core and perfluorotributylamine for their preparation. However, successful encapsulation is only possible if the mechanical resistance of the microcapsules used can overcome the internal forces in the concrete. Microcapsules should have a sufficient proportion of the substance that seals the crack, and a sufficiently strong membrane to be mixed into concrete, and therefore researchers, in addition to components for microcapsule preparation, vary the temperature as well as mixing speed and time in the process of microcapsule preparation (Du et al, 2019; Kanellopoulos et al, 2017; Li et al, 2016; Tong & Li, 2013; Du et al, 2020; Du et al, 2021; Mao et al, 2020). Some of these authors use a constant percentage of microcapsules, and some vary the percentage of microcapsules in a cement composite (Du et al, 2019; Kanellopoulos et al, 2017; Li et al, 2016; Du et al, 2020; Du et al, 2021). In the latter case, the percentage of these microcapsules in the cement composite ranges from 1.5-6 % by cement weight (Du et al, 2019; Kanellopoulos et al, 2017; Li et al, 2016; Tong & Li, 2013; Du et al, 2020; Du et al, 2021).

Danish et al. (2020) systematizes self-healing methods into chemical (crystalline hydrophilic additives and microcapsules) and biological (bacteria). The use of chemical self-healing additives to improve the freeze/thaw resistance of concrete is a poorly researched topic in the scientific literature. So far, one paper has been found investigating the use of a crystalline hydrophilic additive to improve the freeze/thaw resistance of concrete and one paper investigating the use of microcapsules for the same purpose (Wang et al, 2019; Du et al, 2021). The effect of a crystalline hydrophilic additive in the amount of 0.5 % by the cement weight is observed in and the effect of microcapsules in the amount of 3% by the cement weight on the freeze/thaw resistance of concrete is observed in and the results are promising (Wang et al, 2019; Du et al, 2021). No papers comparing the effectiveness of these two methods in improving the freeze/thaw resistance of concrete have been found so far.

It is expected that the addition of any additive will increase the total cost of concrete. For example, concrete containing 400 kg of cement/1 m^3 of concrete with 0.05-0.2% by cement weight of air entraining agent (dosage according to manufacturer's recommendation) and at a price of about 1,33 € per kg of air entraining agent, would be more expensive than concrete without additives for 0,27-1,06 €. For the same amount of cement, a crystalline hydrophobic additive at a price of 10,37 € per kg and in the amount of 0.3-2% by cement weight would increase the price of concrete for 12,5-82,96 €. The cost of microcapsules preparation is unknown at the moment and, therefore, it is not possible to specify the total cost of concrete containing them but, no doubt, an increased price of such concrete is expected. However, it should be emphasized that the air entraining agent reduces the strength of concrete while the crystalline hydrophilic additives do not affect the compressive strength of cement composite and microcapsules can even improve it (Abas Abdela Salem et al, 2017; Cappellesso et al, 2016; García-Vera et al, 2019; Gojević et al, 2021; Du et al, 2019; Du et al, 2021).

According to Danish et al the maintenance costs of buildings are more than two times higher than the costs of concrete production, while Du et al claims that about 50% of the construction costs belong to the costs of its maintenance (Danish et al, 2020; Du et al, 2019). In addition, it should be considered that there are buildings or their parts that are difficult to access to repair, such as: bridges, water tanks, structures subjected to chemical reactions, prefabricated tunnel parts, nuclear installations, dams, concrete pavements, pylons, plane runways (Danish et al, 2020; Gardner et al, 2018). In order to reduce the maintenance costs and to extend the life of hard-to-reach buildings, it could be beneficial to additionally invest in the composition of the concrete mix and make it self-healing and more resistant to freeze/thaw cycles.

This paper compares the efficiency of a crystalline hydrophilic additive and the TDI microcapsules on the freeze/thaw resistance of concrete. The microcapsules used in this paper were prepared in the laboratory using the procedure previously described in the available literature (Du et al, 2019).

2. Materials and methods

Mortar mixtures

Three different mortar mixtures were prepared: reference mortar mixture - R, mortar mixture with crystalline hydrophilic additive (PenetronAdmix) - M1 and mortar mixture with TDI microcapsules - M2. CEM II / A-M (S-V) 42.5 N cement with a density of 3.0 kg/dm^3 and sand with a density of 2.65 kg/dm^3 were used to prepare the mixtures. An enlarged view of the microcapsules is shown in Figure 1a while a comparative view of the crystalline hydrophilic additive and the microcapsules used in the experimental part is shown in Figure 1b.

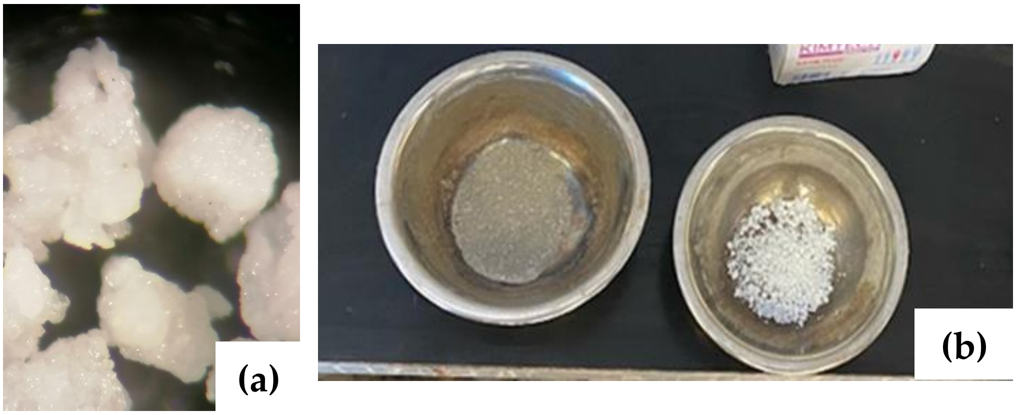


Figure 1. Appearance of: (a) TDI microcapsules (enlarged) and (b) crystalline hydrophilic additive (left) and TDI microcapsules (right)

The composition of TDI microcapsules is shown in Table 1. To prepare the microcapsules, 10 g of paraffin beads were weighed and then heated to 75 ° C until complete melting. Further, 20 g of toluene diisocyanate (TDI) were added and the mixture was stirred with a mechanical stirrer for 3 h at a constant temperature of 75 °C and rotation speed of 600 rpm. Upon cessation of heating, 100 cm3 of perfluorotributylamine (PFTBA) was added to the mixture, resulting in the formation of microcapsules which were isolated by vacuum filtration and dried at 40 °C for 24 h.

Both chemical self-healing additives were added into the mortar mixtures in the amount of 2% by cement weight. The crystalline hydrophilic additive is a powdered material partly composed of cement. Therefore, to keep constant the amount of powdered material, this additive was used as a partial replacement of cement in mortar mixture M1. Analogously, the cement was replaced partially with microcapsules in the mortar mixture M2. Table 2 shows the composition of mortar mixtures.

Table 1. Composition of TDI microcapsules

Paraffin beads (g)	**TDI (g)**	**PFTBA (cm^3)**
10	20	100

The consistence of the mortar mixtures was tested in accordance with EN 1015-3:2000/A1:2005. Figure 2 shows the mortar workability test while the measurement results are presented in Table 3.

Table 2. Composition of mortar mixtures

Mortar mixtures	Component mass (g)				
	Cement	Sand	Water	Crystalline hydrophilic additive	TDI microcapsules
R	450	1550	225	-	-
M1	441	1550	225	9	-
M2	441	1550	225	-	9

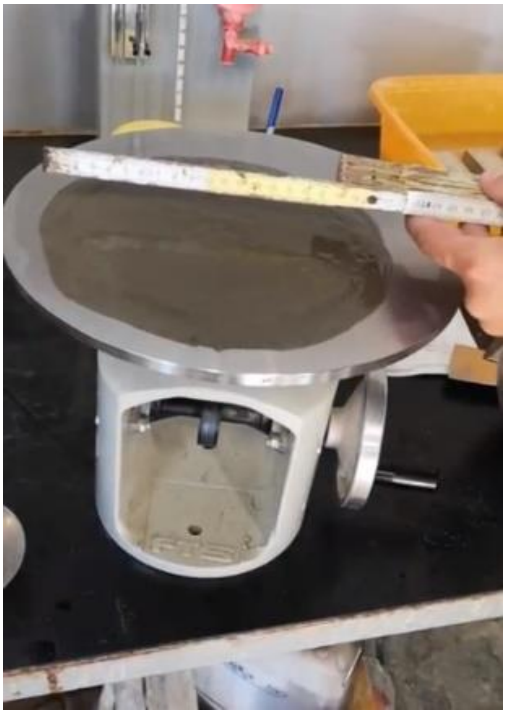


Figure 2. Testing the mortar consistence by flow table

Table 3. Results of mortar consistence testing

Mortar mixture	R	M1	M2
Consistence (cm)	21	20	23

Testing the resistance of mortar to freezing and thawing cycles

Fresh mortar was poured into the prism molds measuring 4x4x16 cm. Samples of hardened mortar were removed from the molds after 24 hours (Figure 3) and immersed in water until 28 days of age.

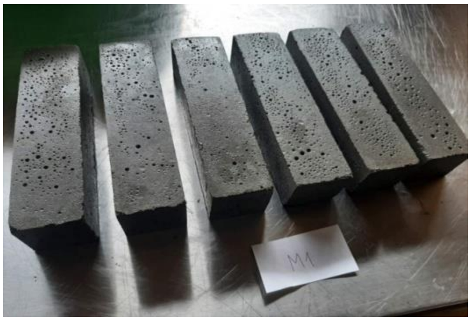
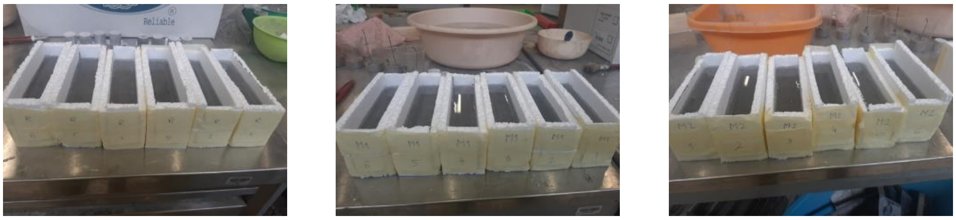


Figure 3. Samples of hardened mortar

After 28 days of curing in water, the prisms were placed in molds made of styrofoam and the edges around the upper surface of the mortar prisms and molds sealed with the sanitary silicone. A 3% solution of NaCl in distilled water was poured onto the saturated samples (Figure 4). The samples thus prepared were placed in a chamber (Figure 5) and treated as prescribed by CEN/TS 12390-9:2006.



(a) (b) (c)

Figure 4. Mortar samples prepared for exposure to freeze/thaw cycles: (a) reference mortar mixture (R), (b) mortar mixture with crystalline hydrophilic additive (M1) and (c) mortar mixture with TDI microcapsules (M2)

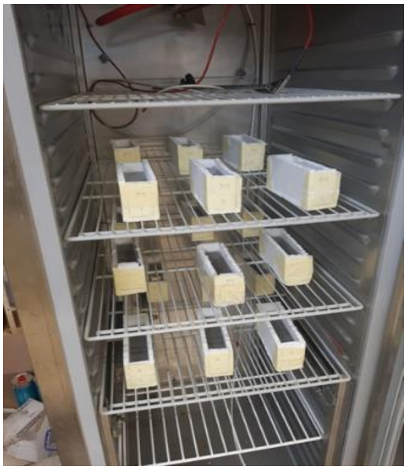
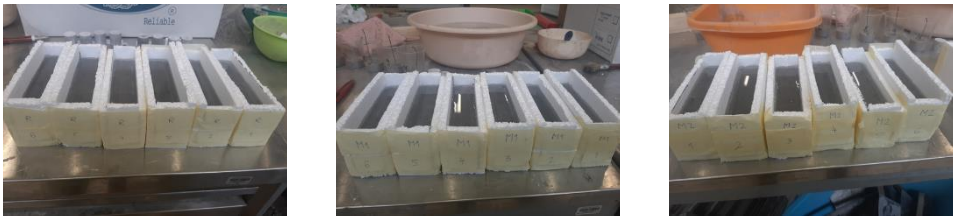


Figure 5. Mortar samples in the chamber

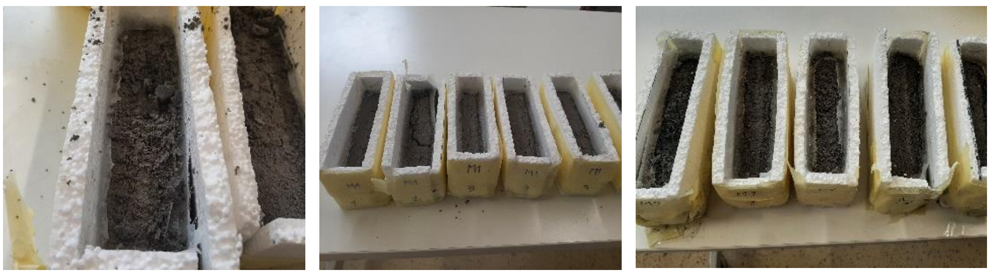
3. Results and Discussion

After the 28th and 56th cycles of freezing and thawing, the samples were taken out of the chamber and a solution was poured out along with the scaling concrete. The solution was poured over the filter paper to collect all the scaling material/concrete. Figures 6 and 7 show the appearance of the prism series R, M1 and M2 after 28th and 56th cycles. Figure 8 shows a comparison of the collected material formed by scaling of all 6 prisms within the same series of samples (series R, M1 and M2) after 28 cycles while, as shown in Figure 7, all samples were completely decomposed after 56 cycles and it was not possible to measure the weight loss.



(a) (b) (c)

Figure 6. Appearance of mortar prisms after 28 cycles of freezing and thawing: (a) reference mortar mixture (R), (b) mortar mixture with crystalline hydrophilic additive (M1) and (c) mortar mixture with TDI microcapsules (M2)



(a) (b) (c)

Figure 7. Appearance of mortar prisms after 56 cycles of freezing and thawing: (a) reference mortar mixture (R), (b) mixture with crystalline hydrophilic additive (M1) and (c) mortar mixture with TDI microcapsules (M2)

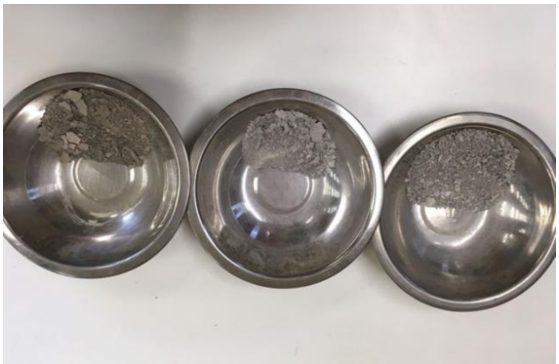


Figure 8. Total residues of scaling material of mortar mixture M2 (left), mortar mixture M1 (in the middle) and reference mortar mixture R (right)

Table 4. Masses and amount of scaling material after 28 cycles of freezing and thawing Composition of mortar mixtures

Mortar	**R**		**M1**		**M2**	
mixture/prism	**m (g)**	**a (kg/m²)**	**m (g)**	**a (kg/m²)**	**m (g)**	**a (kg/m²)**
1	4,97	0,78	3,57	0,56	8,01	1,25
2	5,34	0,83	5,27	0,82	6,21	0,97
3	6,86	1,07	3,33	0,52	6,61	1,03
4	8,54	1,33	6,32	0,99	4,08	0,64
5	8,60	1,34	2,95	0,46	5,01	0,78
6	9,76	1,53	2,40	0,38	3,90	0,61
Σ/average value; standard deviation	44,07	1,15; 0,3	23,84	0,62; 0,23	33,82	0,88; 0,25

The masses (m) and the amount of scaling material per unit area (a) are shown in Table 4

The total mass of scaling material of all six prisms for the reference mortar mixture (R) is 44.07 g, mortar mixture M1 - 23.84 g and mortar mixture M2 - 33.82 g. The average value of the amount of scaling material in mortar mixture R is 1.15 kg /m2, mortar mixture M1 0.62 kg /m2 and mortar mixture M2 0.88 kg /m2. From the above it can be concluded that the weight loss due to 28 cycles of freezing and thawing in both mortar samples with self-healing chemical admixtures (M1 and M2) is lower than for reference mortar R. This means that using the crystalline hydrophilic additive (PenetronAdmix) in mortar mixture M1 and the TDI microcapsules in mortar mixture M2 can improve the resistance of cement composites to freeze/thaw cycles. However, no mortar mixture has survived 56 freeze/thaw cycles.

4. Conclusion

The paper compares the efficiency of two self-healing chemical methods (crystalline hydrophilic additive and TDI microcapsules) on the freeze/thaw resistance of the cement composites. Three mortar mixtures were prepared: a reference mixture, a mixture with crystalline hydrophilic additive, and a mixture with the addition of the TDI microcapsules, with 2% of each additive by cement weight. Hardened mortar samples are subjected to freeze-thaw cycles according to CEN/TS 12390-9:2006. and the amount of scaling material due to freeze/thaw cycles have been measured. The testing results indicate that the self-healing chemical additives used here could improve the freeze/thaw resistance of cement composites.

Here presented results are a part of a preliminary research on the possibility of using self-healing additives to improve the freeze/thaw resistance of concrete. Given the affirmative research results, the authors will further expand their research to concrete mixtures level and with variation in the quantity of self-healing additives.

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