Results in Engineering 6 (2020) 100110

Contents lists available at ScienceDirect

Results in Engineering

journal homepage: www.editorialmanager.com/rineng/Default.aspx

Comprehensive investigation of the long-term performance of internally integrated concrete pavement with sodium acetate

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ARTICLE INFO

Keywords:
Fresh concrete
Concrete pavement
Microstructure
Freezing and thawing
Sodium acetate
Concrete protection

ABSTRACT

The research carried out in this study presents the effectiveness of using sodium acetate as a protective material for concrete pavement. A newly developed freeze-thaw method that depends on the alteration of temperature and humidity is introduced in this research to investigate the efficacy of integrating sodium acetate with concrete with different water to cement ratios (w/c). Results from the introduced freeze-thaw method were compared with the outcomes of a standard freeze-thaw testing method. The distressed concrete was tested for water absorption and compressive strength after finishing six months of freeze-thaw testing. Additionally, the morphology of sodium acetate and its interaction with concrete were investigated by using Scanning Electron Microscope (SEM). Results demonstrated the effectiveness of sodium acetate in protecting concrete.

1. Introduction

Concrete pavement is usually exposed to a combination of mechanical stresses and environmental impacts that accelerate its deterioration rate [1,2]. The presence of de-icing salts in contact with the surface of concrete pavement during winter season can increase the damage of concrete, especially after their penetration in the pores with the absorbed water [3–7]. Moreover, the cyclic freezing and thawing cycles can produce a high pore pressure related to the water phase change inside the pores that lead to initiating cracks within the concrete matrix [8]. Accordingly, it is important to protect such structures from all the weathering actions and chemical attacks to keep it in its original form and serviceability [9,10].

Many protective materials have been used during the years to reduce the rate of deterioration caused by chloride and water ingress through concrete and to inhibit the freeze-thaw damage [11–18]. Surface applied protective materials like silane and siloxane were the most widely used materials in this regard due to their high resistance to different environmental impacts and chemical attacks [16,19–24]. However, few researches considered the protection of concrete pavement, in particular, either by using silane or any other surface applied materials [2]. This refers to their inconvenient application method that requires closing the roads in front of vehicles and their effect on the frictional properties of concrete pavement [2]. Consequently, internally integrated materials were introduced to overcome all the issues associated with the surface applied materials [25–29].

The integration of sodium acetate into concrete for protection purposes is a newly introduced method that aims to improve the drawbacks that are associated with other traditional methods [30]. After the mixing of sodium acetate with concrete, in the presence of water, it forms crystals that line the pores of concrete without blocking them. This material is characterised of being hygroscopic, as it works on absorbing water to form crystals that start to grow after the reaction of sodium acetate with water. After the formation of these crystals, they bond with concrete and work on repelling any excess water from the pores due to the development of hydrophobic properties that results from their reaction with cement during the hydration process [6].

In this research, two freeze-thaw conditions will be applied to concrete, and the performance of the used protective material, under these conditions, will be evaluated. In the first method, concrete was exposed to fast freeze-thaw cycles while it was immersed in water. This method has been widely used in previous studies but with a smaller number of

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https://doi.org/10.1016/j.rineng.2020.100110
Received 20 January 2020; Received in revised form 22 February 2020; Accepted 25 February 2020

cycles than it is used in this research, where the number of cycles in this research is the highest until now [31–35]. In the second freeze-thaw method, a newly developed method is proposed where concrete is only in contact with air and under the effect of temperature change.

2. Materials and test methods

2.1. Materials and specimens

Four concrete mixtures with w/c ratios of 0.32, 0.37, 0.40 and 0.46 were cast following the British Standard BS 1881–125 [36]. A sodium acetate material with some cementitious content was added to all the mixes with two different ratios; 2% and 4% of the cement mass. Additionally, a reference mix with 0% sodium acetate was cast for each mix design for comparison reasons. Table 1 shows the proportions of each concrete mix that was used in this study.

72 concrete cubes with the dimensions of 100 mm × 100 mm x 100 mm were cast and divided into two groups; 36 cubes were tested for freeze-thaw cycles in air and 36 cubes were tested for fast freeze-thaw cycles in water. From each group, 9 cubes were prepared with 0.32 w/c ratio, 9 cubes with 0.37 w/c ratio, 9 cubes with 0.40 w/c ratio and 9 cubes with 0.46 w/c ratio. For each w/c ratio, 3 cubes were integrated with 2% sodium acetate, 3 cubes with 4% sodium acetate and 3 cubes were used as control. Another 36 cubes (treated and control) with the sizes of 100 mm × 100 mm x 100 mm were used to test the compressive strength of concrete before the impact of freeze-thaw test.

For porosity testing, 36 cubes (treated and untreated) with the dimensions of 50 mm × 50 mm x 50 mm were cast. Samples were prepared following the same procedure for preparing the previous cubes.

All concrete samples were cured in a water bath for 28 days. Afterwards, 36 cubes of them were placed in room temperature (21 °C) to dry so they can be tested under the freeze-thaw cycles in air. The other 36 cubes were placed in water for another 7 days until they were fully saturated (to achieve a constant mass).

2.2. Test methods

2.2.1. Exposure to freeze-thaw cycles

In the fast freeze-thaw test, cubes were placed in containers that are filled with water (cubes are immersed in water), and all containers have been placed in Weiss-Voetsch Environmental Testing Chamber C340, following the guidelines of the Chinese standard GB/T 50,082–2009 [37]. Temperature was set to alternate between −10 °C and 6 °C for a duration of 4 h, representing a full rapid freeze-thaw cycle. The internal temperature of concrete cubes ranged between −6 °C and 4 °C during the freezing and thawing periods respectively. In total, 1080 freeze-thaw cycles during 6 months were carried out in this test. After completion of 1080 cycles, the mass of the tested concrete cubes was measured and the change in their masses was calculated.

In the newly developed freeze-thaw test, an environmental chamber that controls humidity and temperature was constructed. The chamber was constructed by utilising an existing industrial freezer as base for the freezing cycles and by introducing heating and humidity units inside the freezer to serve the thawing cycles. Furthermore, the heating and the humidity control units are additionally developed in order to complete the requirements for computer-controlled scheduling of 24 h cycles between temperatures of −20 °C and 20 °C, and constant humidity of 60%. Cubes were placed inside the chamber and it was programmed to run for 6 continuous months with a total of 180 freeze-thaw cycles. The internal temperature of concrete was measured by using embedded thermocouples and it ranged between 17 °C during the thawing process and −16 °C during the freezing process. After completing the freeze-thaw test, the change in the mass of tested concrete in reference to their original mass before the test was recorded. Fig. 1 shows the constructed freeze-thaw chamber with an attached automatic unit for controlling the cycles.

Fig. 2a and b illustrates the temperature alteration during the impact of the standard fast freeze-thaw test (in water) and the newly introduced air freeze-thaw test.

Table 1
Mix designs of the used concrete mixtures.

Ingredient	Amount (kg/m^3)			
	W/C = 0.32	W/C = 0.37	W/C = 0.40	W/C = 0.46
Cement (CEM II/32.5 N; Sulphates < 3.5%, Chlorides < 0.10%, and initial setting time around 1.25 h)	513	491	450	457
Water	164	182	180	210
Fine aggregate (sharp silica sand with uniform grain size distribution between 1 mm and 300 μm)	658	660	678	660
Coarse aggregate (crushed stones with sharp edges and maximum size of 20 mm)	1068	1070	1092	1073

2.2.2. Concrete workability

The consistency of all the concrete mixes was evaluated by using the slump test method following the guidelines of the BS EN 12350–2 [38].

2.2.3. Water absorption

The ability of the sodium acetate compound to preserve concrete from water ingress in harsh environments was assessed by running the ISAT test on the distressed samples (after finishing the freeze-thaw tests). Initial Surface Absorption Test (ISAT) was used to measure the uniaxial water absorption of concrete cubes by following the guidelines of BS 1881–208 [39].

2.2.4. Mechanical properties

Mechanical properties of concrete were determined by running the compressive strength test on all the samples after finishing the freeze-thaw cycles, following the British Standard BS EN 12390–3 [40]. Furthermore, the obtained results were compared with the compressive strength results of the samples before running the freeze-thaw tests.

2.2.5. Porosity

The porosity of all treated and untreated concrete was measured by

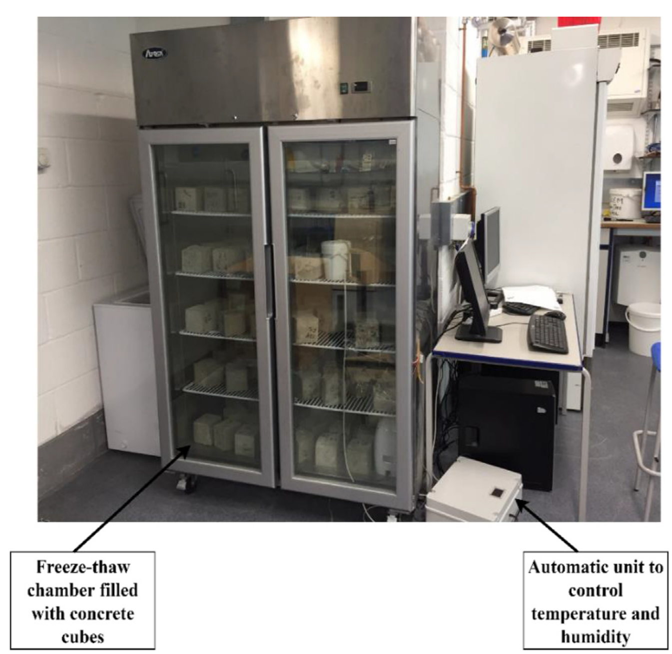


Fig. 1. Concrete samples undergoing the air freeze-thaw cycles.

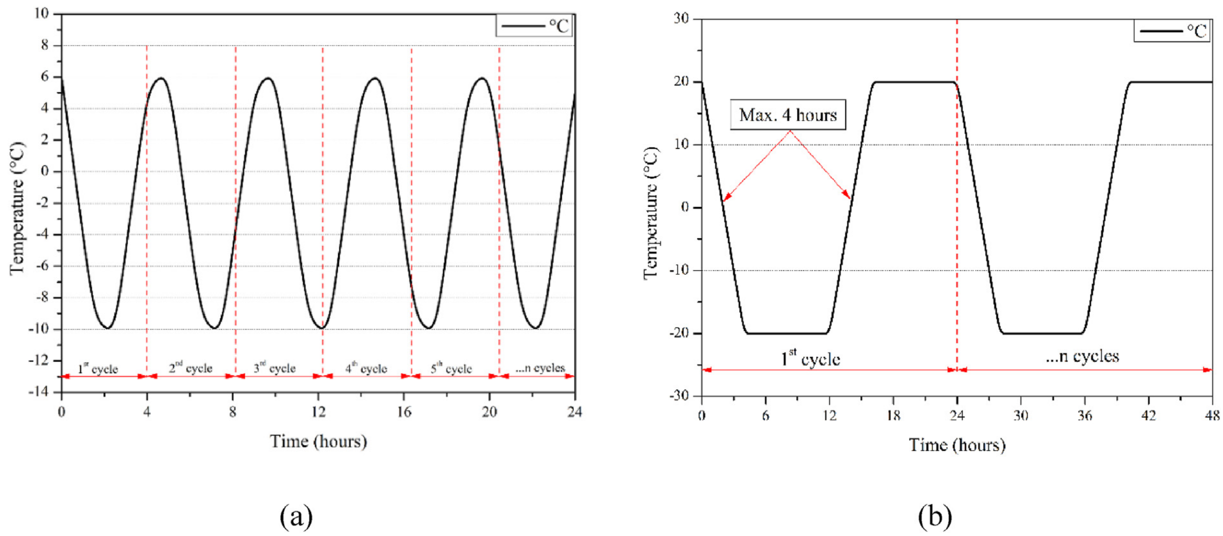


Fig. 2. Temperature alteration during the impact of: (a) fast freeze-thaw test and (b) air freeze-thaw test.

using the vacuum saturation method [41,42]. A vacuum saturation apparatus was used to measure the porosity; it involves a vacuum pump, desiccator and water source.

All samples were dried in oven at a temperature of 105 °C until a constant mass is achieved. Samples were placed in the vacuum saturation apparatus and evacuated for 3 h at a pressure of 90 kPa, and then they were soaked in water for 24 h. At the end of the 24 h period, samples were removed from the vacuum saturation apparatus and their saturated surface dry mass was measured. The porosity of all the samples was calculated by the following equation [42]:

$$Porosity\ (\%) = \frac{M_s - M_d}{M_s - M_b} x100\%$$

where.

M_s: Saturated surface dry mass
M_d: Oven dry mass of the sample in air
M_b: Buoyant mass of the saturated sample in water

2.2.6. Microstructural analysis and interaction mechanism

The morphology of the used sodium acetate compound, its interaction with concrete and the size of its formed crystals were investigated by using the Scanning electron microscope (SEM). Samples were coated with a thin film of gold before analysing them under the SEM since both, the material and concrete, are non-conductive.

3. Results and discussion

3.1. Microstructural analysis

The microstructure of the used sodium acetate compound and its interaction with concrete are shown in Fig. 3 a-d.

The anatomical analysis of the used sodium acetate compound showed that this material is present in a small amorphous shape before its activation (Fig. 3 a-b), which ease their incorporation within the concrete mix. This could be spotted in Fig. 3 c-d where the activated sodium acetate crystals are seen embedded within the pores of concrete. After mixing concrete with sodium acetate, crystals will grow and attach themselves to the interiors of the pores by creating a chemical bond between the CH_3COO^- part of the sodium acetate compound and sodium that already exists in cement in the presence of water (during the hydration process). The formed crystals were found to have sizes that range between 95 and 200 nm when measured on the SEM scale, which ease their attachment to the interiors of the pores. This size of crystals is considered smaller than most of the existing and effective pores in concrete, e.g. macro-pores (>1000 nm), most of the capillary pores (100–1000 nm), most of the meso-pores (10–10000 nm) and some of the transitional pores (10–100 nm) [43,44]. Accordingly, it is believed that the formed crystals will work on lining the pores instead of blocking them.

According to previous research by authors [6,45], it is believed that a replacement process will take place after the activation of the sodium acetate crystals. The –OH groups will be replaced by the $–CH_3$ groups, which in turn bond with Silicon atoms in cement and result in forming a hydrophobic component integrated within the mix (organosilicon bonds) [46–49]. This could result in improving the hydrophobicity of concrete treated with sodium acetate.

3.2. Concrete workability

Results from the slump test are outlined in Table 2.

It is witnessed that increasing the added amount of the sodium acetate compound to concrete increases its consistency and flow properties. For instance, concrete with w/c ratio of 0.37 and 0% sodium acetate has shown 0 mm slump value, however adding 2% and 4% sodium acetate to the mix increased the slump to 5 mm and 20 mm respectively. Even in concrete with high w/c ratio, the slump value increased significantly with adding the sodium acetate. Furthermore, the slump value of concrete with 0.46 w/c ratio and treated with 4% sodium acetate has reached 160 mm, which is considered a high slump though acceptable [38]. Despite the high slump values, especially for treated concrete mixes that has a high w/c ratio, concrete did not show any sign of segregation in matured concrete after 28 days of curing.

3.3. Porosity

The average results of the porosity test are illustrated in Table 3.

It is witnessed from the results that adding sodium acetate to the mix does not have a significant effect on the porosity of concrete. Adding sodium acetate to concrete with w/c ratios of 0.32 and 0.37 has slightly reduced the porosity of the mix, and adding sodium acetate to concrete with high w/c ratios of 0.40 and 0.46 has contributed in a slight increase in the concrete's porosity. This insignificant increase/reduction in the porosity of treated concrete may refer to the small size of the formed crystals that work on lining the pores of concrete instead of entirely blocking them, which supports the outcomes of the morphological analysis in section 3.1. On the other hand, the small increase in the porosity of concrete with high w/c ratios might indicate the formation of

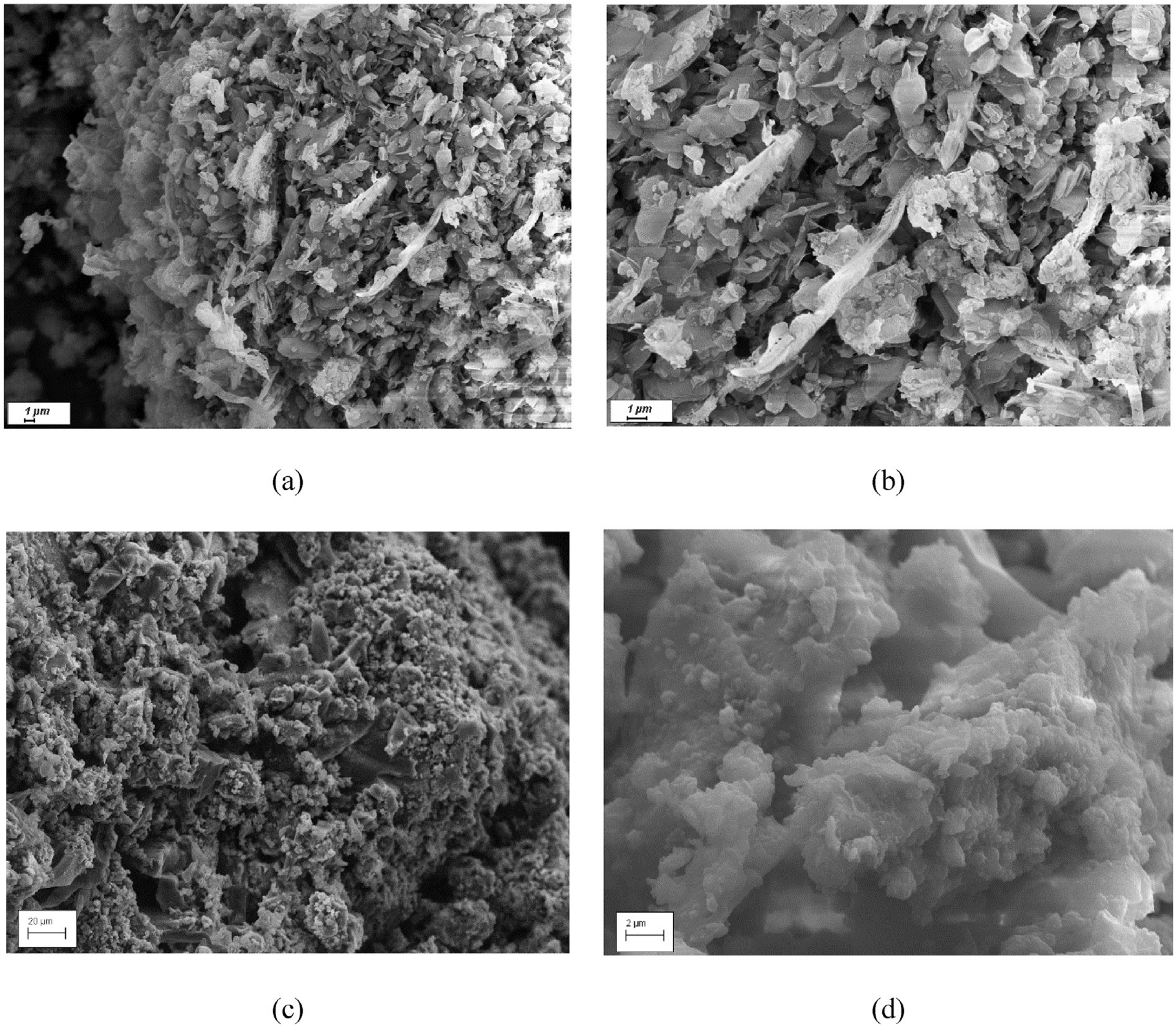


Fig. 3. SEM micrographs for sodium acetate compound before mixing with concrete: (a) at 5000X, (b) at 10000X, and the interaction between sodium acetate and concrete: (c) at 5000X, (d) at 10000X

Table 2
Slump values of all concrete samples.

w/c ratio	Sodium acetate %	Slump (mm)
0.32	0	0
0.32	2	0
0.32	4	0
0.37	0	0
0.37	2	5
0.37	4	20
0.40	0	5
0.40	2	15
0.40	4	70
0.46	0	25
0.46	2	50
0.46	4	160

Table 3
Porosity of control and internally integrated concrete.

w/c ratio	Sodium acetate %	Average porosity %
0.32	0%	7.57%
	2%	7.32%
	4%	7.28%
0.37	0%	8.16%
	2%	8.02%
	4%	7.96%
0.40	0%	8.84%
	2%	9.17%
	4%	9.36%
0.46	0%	9.23%
	2%	9.83%
	4%	10.17%

microcracks in the concrete mix due to treatment, contrary to concrete with low w/c ratios.

3.4. Mass change (after freeze-thaw)

3.4.1. Fast freeze-thaw cycles (water impact)

The change in the concrete samples' mass is directly related to the degree of deterioration that developed from the freeze-thaw cycles. In the freeze-thaw test in water, it is witnessed that concrete has suffered from mass loss during the applied 1080 cycles. Fig. 4 illustrates the damage in all concrete samples in terms of mass loss percentage during the freeze-thaw test in water.

In general, all concrete samples have shown a reduction in their masses during the test, and this reduction increases with increasing the number of applied cycles. The main reason for this trend could be attributed to the damage of the internal pores of concrete during the cyclic and fast freeze-thaw process, especially that pores were saturated with water before starting the test. This damage to the pore structure can cause surrounding water to move freely in and out concrete, which leads to the scaling of concrete surface (separation of paste from the surface)

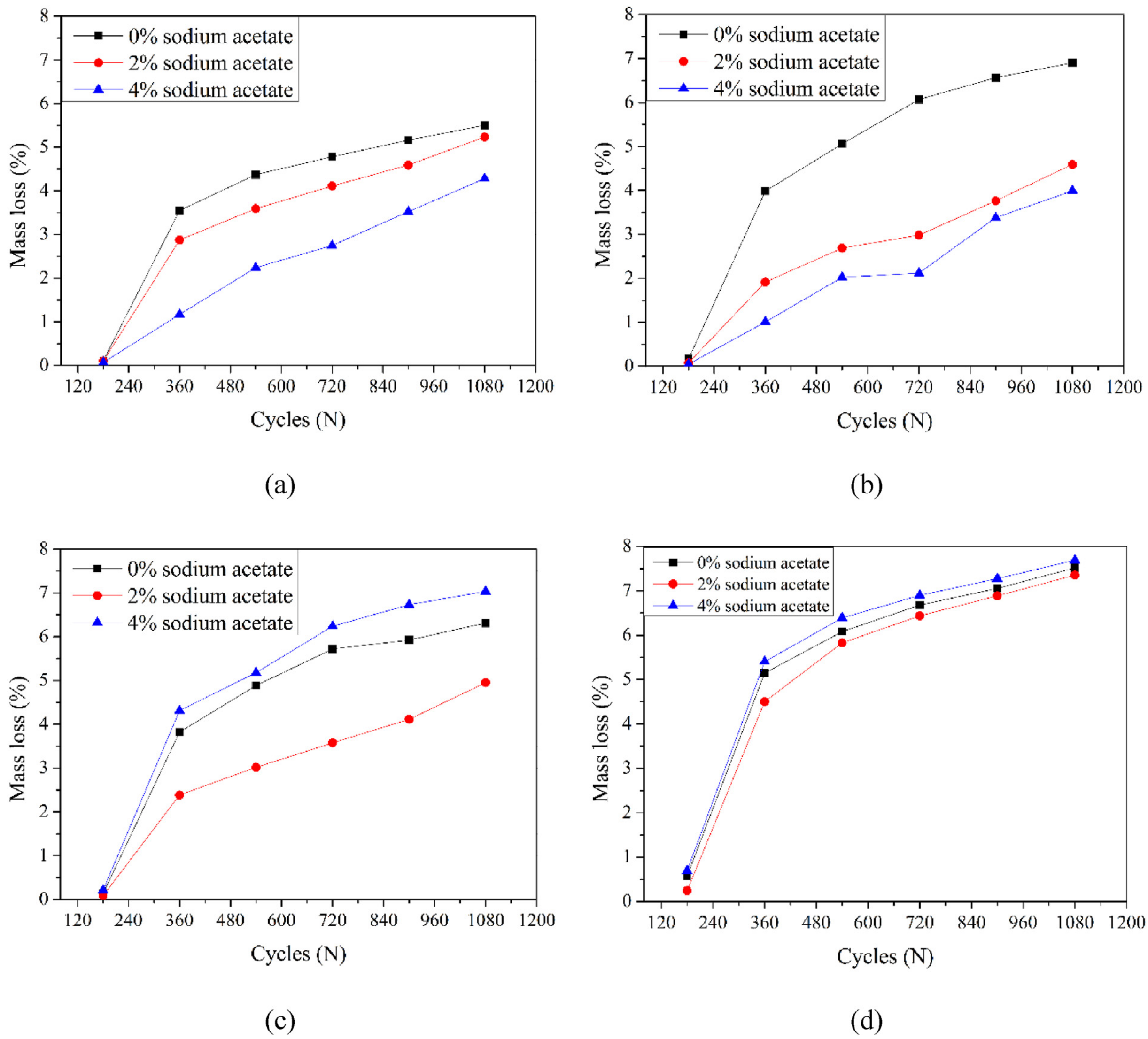


Fig. 4. Mass loss percentage during the water freeze-thaw test in treated and untreated concrete with w/c ratio of: (a) 0.32, (b) 0.37, (c) 0.40 and (d) 0.46.

[32]. The scaling of concrete was visibly noticed in all concrete samples, either treated or control, as shown in Fig. 5, where most of concrete samples showed similar appearance. The detected scaling in this test is believed to be severer than what is observed in real life (naturally-occurring scaling) because of the fast alteration in temperature and because of the high number of applied cycles in this test.

It can be clearly observed, in Fig. 4, that the development in the loss of mass in concrete with low w/c ratios was less than that in high w/c ratios' concrete. This could be spotted in the gradient of the produced graphs, where concrete with w/c ratios of 0.32 and 0.37 (Fig. 4a and b) have shown a slow progress in the mass loss than concrete with w/c ratios of 0.40 and 0.46. More specifically, treatment of 0.32 and 0.37

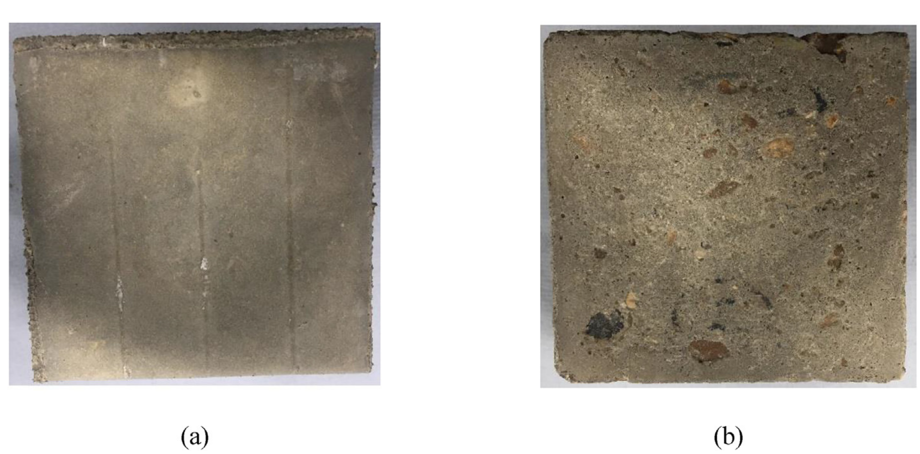


Fig. 5. The surface of concrete sample: (a) before the fast freeze-thaw impact and (b) after the fast freeze-thaw impact.

concrete with 4% of sodium acetate has exhibited the least mass loss amongst all concrete mixtures, where the mass loss in both mixes was around 4%. On the other hand, adding 4% sodium acetate to concrete with 0.40 and 0.46 w/c ratio increased the mass loss, where it reached 7% for 0.40 concrete and 7.7% for 0.46 concrete, which is even higher than untreated concrete.

Concrete with low w/c ratio would have lower porosity than concrete with high w/c ratio (Table 3), which is reflected on the amount of capillary water exposed to frost action; less exposure in the case of low w/c ratio concrete as it has less capillary water. In addition, lowering the porosity would participate in reducing the movement of water to areas, like air bubbles, where water could be entrapped and initiates cracks due to its freezing and thawing inside these areas [50–52]. Adding the 4% sodium acetate to the low w/c ratio concrete participated in increasing its resistance to freeze-thaw, where mass loss decreased significantly compared to control. The presence of sodium acetate inside the pore structure of concrete would contribute in reducing the movement of water through the pores; either water that already exists inside the pores or the surrounding water. Adding to that, treating low w/c ratio concrete with 4% sodium acetate has proven its ability to reduce water absorption of concrete, which could be reflected on the saturation process that was performed on concrete prior to starting the freeze-thaw test. In other words, low w/c ratio concrete and treated with 4% sodium acetate was less saturated than other mixes, which reduces the amount of capillary water that is subjected to frost action.

On the other hand, adding 4% sodium acetate to high w/c ratio concrete, like the 0.40 and 0.46 mixes, has increased the damage to concrete. This could be attributed to the presence of high-water content and high sodium acetate content at the same time. Their presence in relatively large contents contributes in increasing the consistency of the mix, which in turn takes part in increasing the air voids content and microcracks (Table 2).

3.4.2. *Air freeze-thaw cycles*

Contrary to the freeze-thaw test in water, concrete cubes were observed to increase their masses during the freeze-thaw test in air. Fig. 6 illustrates the change in concrete's mass during the freeze-thaw test in air.

All concrete cubes have shown a general trend of increasing their masses with increasing the applied number of cycles, with different performance for each mix. This increase in mass is attributed to the absorption of concrete to moisture from the surrounding environment due to the applied humidity of 60% during the test. Water is believed to be absorbed through the pore structure of concrete during the thawing process. When the freezing phase starts, the absorbed water will freeze inside the pores and expands, which will create some microcracks inside the pore structure. These cracks will increase the absorption of concrete to moisture and participate in increasing the mass.

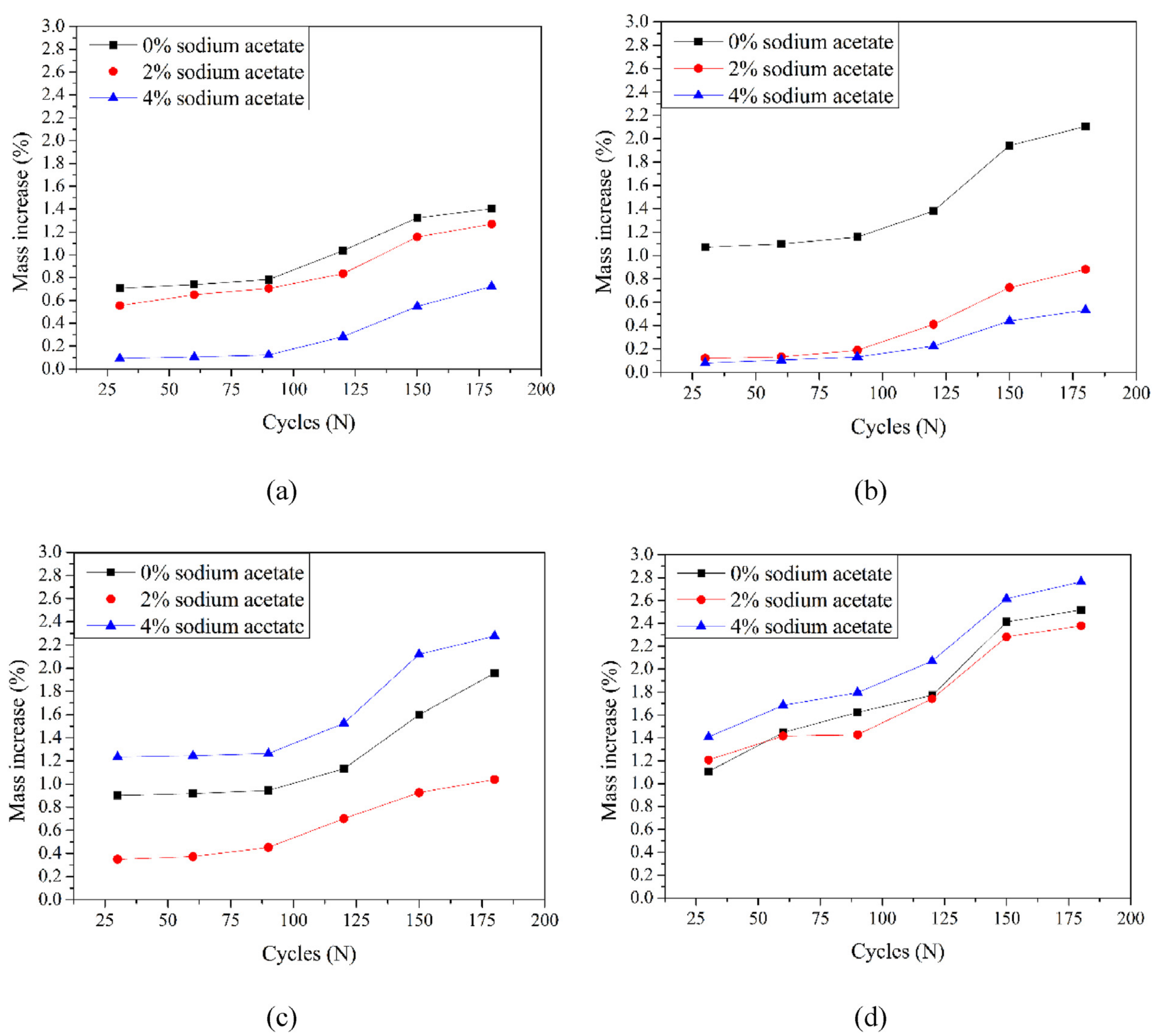


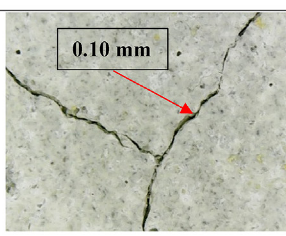
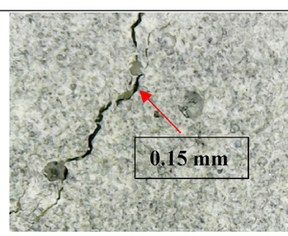
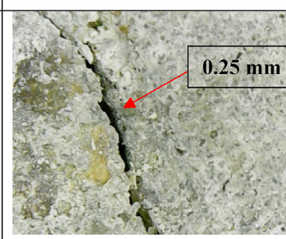
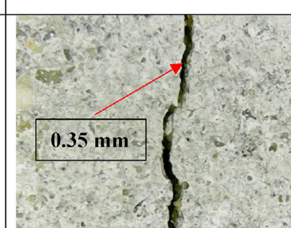
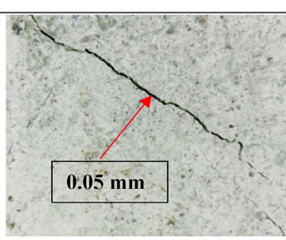
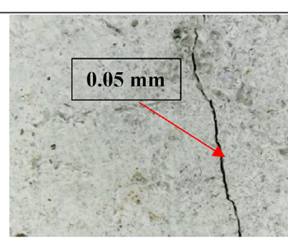
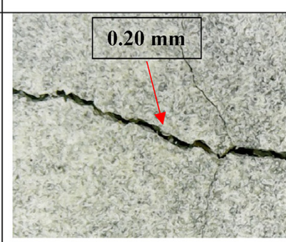
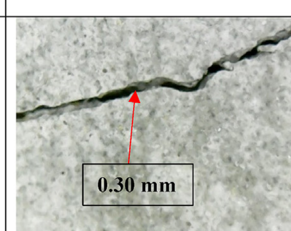
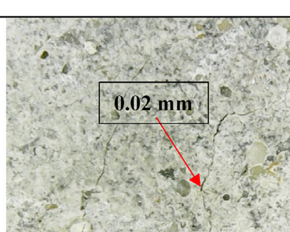
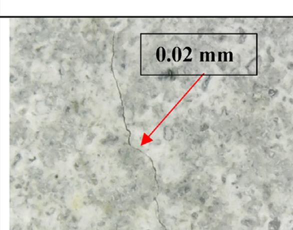
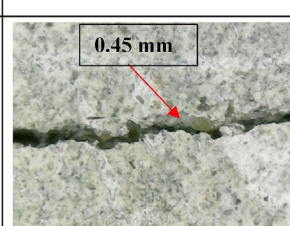
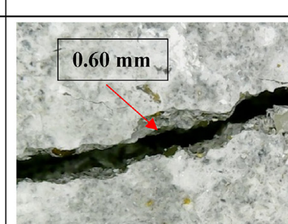
Fig. 6. Mass increase percentage during the air freeze-thaw test in treated and untreated concrete with w/c ratio of: (a) 0.32, (b) 0.37, (c) 0.40 and (d) 0.46.

During all the cycles, concrete with low w/c ratio (0.32 and 0.37) has gained less weight than concrete with high w/c ratio (0.40 and 0.46). This could be attributed to the low porosity of concrete with the lower w/c ratio (Table 3), which decreases their absorption to water during the thawing process. Accordingly, the amount of water that is exposed to frost action in the freezing process will be less, which will reduce the initiated microcracks inside concrete. Treating 0.32 and 0.37 concrete with 4% sodium acetate has shown the least weight gain between all mixes, which is compatible with the results obtained from the freeze-thaw in water test. The 4% sodium acetate when added to concrete with 0.32 and 0.37 w/c ratio may participate in forming enough organosilicon bonds inside the pores of concrete, which are capable of reducing the absorbed water during the thawing process (section 3.1). This will reduce the risk of frost action during the freezing phase. On the other hand, treating concrete with w/c ratio of 0.32 with 2% sodium acetate did not significantly enhance the performance of the mix when compared to control. This might refer to two reasons: the low amount of water needed to activate the sodium acetate crystals and the low amount of the used sodium acetate compound itself. The presence of the sodium acetate in such low quantity restricts the distribution of its active content all over the mix. Adding to that, the used water during mixing will not be enough to continue the hydration process and at the same time activates the crystals of the material. This was not observed in the case of concrete with w/c ratio of 0.37 and treated with 2% material due to the presence of relatively enough water for hydration process and activating the crystals.

Concrete with w/c ratio of 0.40 and 0.46 and treated with 4% sodium acetate have shown the highest mass increase between all mixes, even higher than control. This may be a result of the high workability of these mixtures (Table 2), which contributed in forming some microcracks in concrete prior to the freeze-thaw test. Adding to that, the high porosity of these mixes will work with the previously mentioned microcracks on facilitating the absorption of moisture during the thawing process. After absorbing the moisture and with the start of the freezing process, water will expand inside the pores and creates more microcracks. These factors would ensure to increase the mass of such mixtures. On the other hand, adding 2% sodium acetate to concrete with w/c ratio of 0.40 has, somehow, resisted the increase in the mixture mass when compared to its own control mix. However, its performance was less efficient than 0.32 and 0.37 mixes treated with 4% sodium acetate.

One thing to be observed in the freeze-thaw in air testing method is the absence of scaling from concrete surfaces, unlike the freeze-thaw in water method where scaling forms an obvious characteristic. Regardless of the absence of scaling in the air method, some cracks have developed on the surface of concrete, which confirms with the mass increase during all the cycles. Table 4 shows some microscopic images (500X) for the developed cracks and their sizes on the surface of treated and untreated concrete after 180 cycles. It is clear from the images that the severest

Table 4
Development of cracks after 180 freeze-thaw cycles in air.

	w/c ratio			
Sodium acetate (%)	**0.32**	**0.37**	**0.40**	**0.46**
0%				
2%				
	w/c ratio			
Sodium acetate (%)	**0.32**	**0.37**	**0.40**	**0.46**
4%				

cracks have developed in concrete mixes with w/c ratios of 0.40 and 0.46 and treated with 4% sodium acetate. These micro-images support the previous claim about the formation of microcracks in these mixtures that caused them to absorb more moisture from the surrounding environment during the test. On the other hand, concrete mixes with w/c ratios of 0.32 and 0.37 and treated with 4% sodium acetate have shown the least development for cracks. The developed cracks in these mixes were much less severe than those developed in concrete with w/c ratios of 0.40 and 0.46.

3.4.3. Comparative analysis of air and water freeze-thaw investigations

Both proposed freeze-thaw methods have shown different deterioration effect on concrete. As discussed earlier, the water freeze-thaw method was distinguished by the scaling impact on the surface of concrete accompanied with mass loss. Furthermore, the air freeze-thaw has worked on developing cracks on the surface of concrete with an increase in the total mass of concrete. This might refer to the more aggressive effect of water on concrete than air and humidity. Adding to that, the fast freeze-thaw cycles used in the water method would impose more destruction to concrete than the slow cycles used in the air method (1080 cycles compared to 180 cycles during 6 months). Fig. 7 shows a comparison between the effects of both freeze-thaw methods on the internally integrated concrete at the end of the 1080 cycle for the water freeze-thaw and at the end of the 180 cycle for the air freeze-thaw.

It is witnessed from the graphs that both freeze-thaw methods have shown a relatively similar performance for all the developed concrete mixes. Regardless of their different mechanisms of deterioration, the increase in the mass of concrete caused by the water freeze-thaw was compatible with the reduction in mass caused by the air freeze-thaw. For instance, concrete with w/c ratio of 0.46 and integrated with 4% sodium acetate has shown the same deterioration level in both methods, with a maximum reduction in mass of 7.7% when exposed to water freeze-thaw and maximum increase in mass of 2.8% when exposed to air freeze-thaw. In accordance with that, all the other developed mixes have followed the

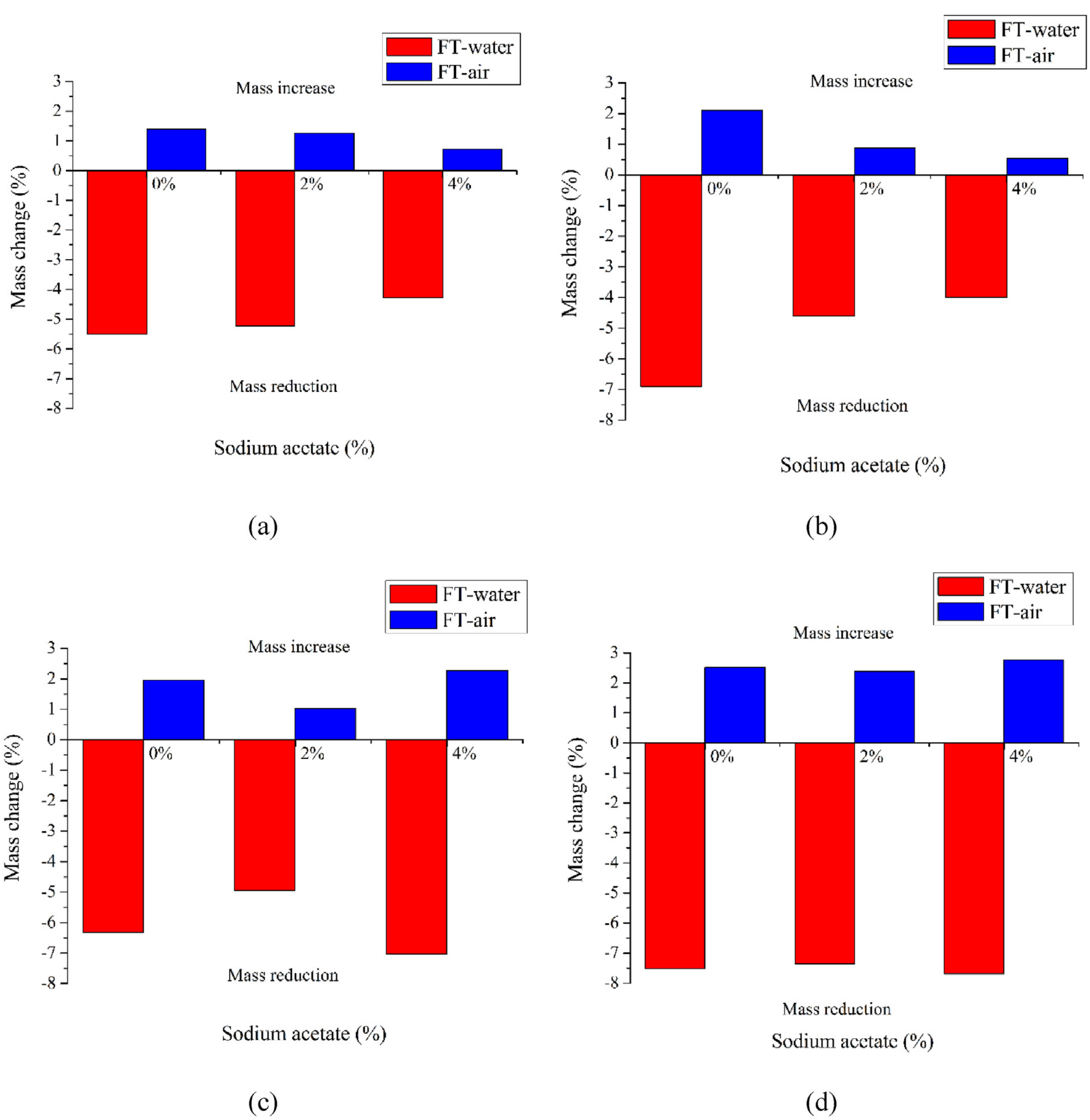


Fig. 7. Deterioration rate of concrete at the 1080 water freeze-thaw cycle and 180 air freeze-thaw cycle for internally integrated concrete with w/c ratio of: (a) 0.32, (b) 0.37, (c) 0.40 and (d) 0.46.

same pattern of deterioration in both tests.

The increase in the mass of all the internally integrated concrete, either with 2% or 4% sodium acetate and regardless of the mixture's w/c ratio, under the impact of the air freeze-thaw supports the outcomes of the morphological analysis in section 3.1. The formed crystals work on lining the pores of concrete without blocking them, which allows concrete to breathe. This was observed from the increase in the concrete's mass after the exposure to 60% humidity (without soaking in water), where moisture worked on penetrating concrete through its pores at the thawing process.

3.5. Water absorption after freeze-thaw

The ability of the sodium acetate compound to preserve concrete from water ingress in harsh environments was assessed by running the ISAT test on the distressed samples. Concrete samples have shown a general increase in water absorption after the impact of freeze-thaw cycles in water. Fig. 8 demonstrates the water absorption rate of concrete after the impact of 1080 cycles under the immersion effect.

ISAT results were in line with the mass change outcomes of concrete after the impact of freeze-thaw tests; both the water and air tests. Concrete samples with w/c ratios of 0.32 and 0.37 and treated with 4% sodium acetate have shown the least water absorption rates among all mixtures. This amount of treatment has helped water absorption rate to drop by 78% and 85% for mixtures with 0.32 and 0.37 w/c ratios respectively compared to their control mixtures (Fig. 8a and b). Even when both mixtures were treated with 2% sodium acetate they have shown a high ability to reduce water absorption. This might be a result of using a compatible amount of water and sodium acetate, where no excess water is present in the mix, and the amount of water was suitable to initiate the reaction between sodium acetate and cement, and at the same time water was enough to continue the hydration process. Despite the scaling effect that the freeze-thaw cycles have imposed on concrete (Fig. 5), sodium acetate has proven its efficacy against water absorption. This could give an indication that the protection and the presence of this material is not only near the surface of concrete but all over the structure.

In the case of concrete with w/c ratios of 0.40 and 0.46 and treated with 4% sodium acetate, an obvious destructive effect could be seen from Fig. 8c and d. Water absorption of these two mixes has significantly increased compared to their control and to other mixtures after the impact of freeze-thaw. This might refer to their high porosity when they were first cast (Table 3) and the formation of some microcracks in concrete after the impact of freeze-thaw cycles. Moreover, the high w/c ratio, especially in concrete with w/c ratio of 0.46, and the highly added dosage of the sodium acetate compound will contribute in increasing the formation of the hydrophobic organosilicon content. The presence of this

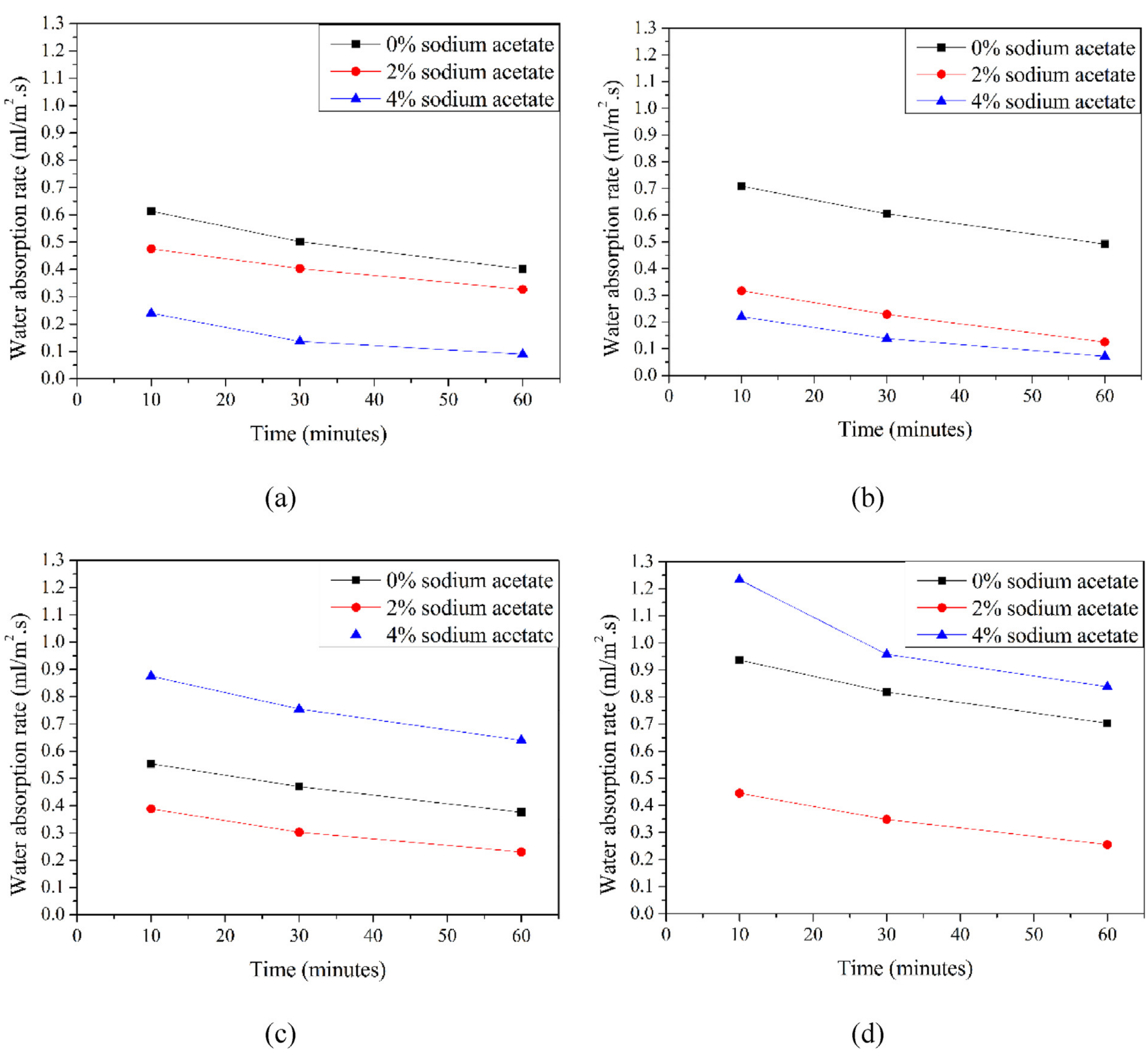


Fig. 8. Water absorption of concrete after the impact of 1080 freeze-thaw cycles in water for concrete with w/c ratio of: (a) 0.32, (b) 0.37, (c) 0.40 and (d) 0.46.

hydrophobic content in large amounts in concrete at early ages will probably work on reducing the needed amount for the hydration process by repelling water out from concrete. This will lead to the formation of some microcracks in concrete during its curing and before initiating the freeze-thaw test. With the impact of the freeze-thaw test, these microcracks will develop into larger cracks, and concrete will absorb more quantities of water.

Referring to the results of water absorption of concrete under the impact of 180 freeze-thaw cycles in air, a clear increase in permeability can be noticed. Fig. 9 illustrates the effect of freeze-thaw cycles in air on concrete's absorption of water.

Water absorption rates under the effect of air have followed a similar trend to those in water but with less absorbing rates. This is expected since the severity of the test in water is higher than that in air.

Similar to the freeze-thaw test in water, concrete with w/c ratios of 0.32 and 0.37 and treated with 4% sodium acetate have shown the least absorption rate between all mixes, which refers to the compatibility between the used amount of water and the added percentage of the admixture. This could be linked with the micro-images in Table 4, where these mixtures have developed less cracks with small sizes.

On the other hand, concrete with w/c ratios of 0.40 and 0.46 and treated with 4% sodium acetate suffered from increasing the absorption rate of water. This issue could be explained by following the same reasons for their high absorption rates after the impact of freeze-thaw cycles in water. Also, their high tendency to absorb moisture during freeze-thaw in air, as noticed in section 3.4.2, confirms with current outcomes, because of the formation of large microcracks (Table 4) and the original high porosity of concrete (Table 3).

3.6. Compressive strength (After freeze-thaw)

After finishing both freeze-thaw tests, in water and air, all samples have been tested for compressive strength to measure the ability of treatment to preserve the strength of concrete in harsh conditions. Table 5 shows the compressive strength and standard deviation values of concrete after their exposure to cyclic freeze-thaw in water and air.

It is witnessed that concrete under the impact of cyclic freeze-thaw in water has suffered from a drop in its compressive strength values when compared to concrete under freeze-thaw cycles in air. Once more, this refers to the severity of the applied conditions in the case of the freeze-thaw test in water. The frost action of the freeze-thaw test in water may damage the internal pores of concrete due to the presence of water inside the pores. The expansion of water inside the pores will damage the pore system and initiates cracks inside concrete, which in turn will weaken it and reduce its strength.

Comparing these results with those obtained before the impact of freeze-thaw in water (Fig. 10), a clear reduction in compressive strength could be noticed. Concrete with w/c ratio of 0.46 and treated with 4%

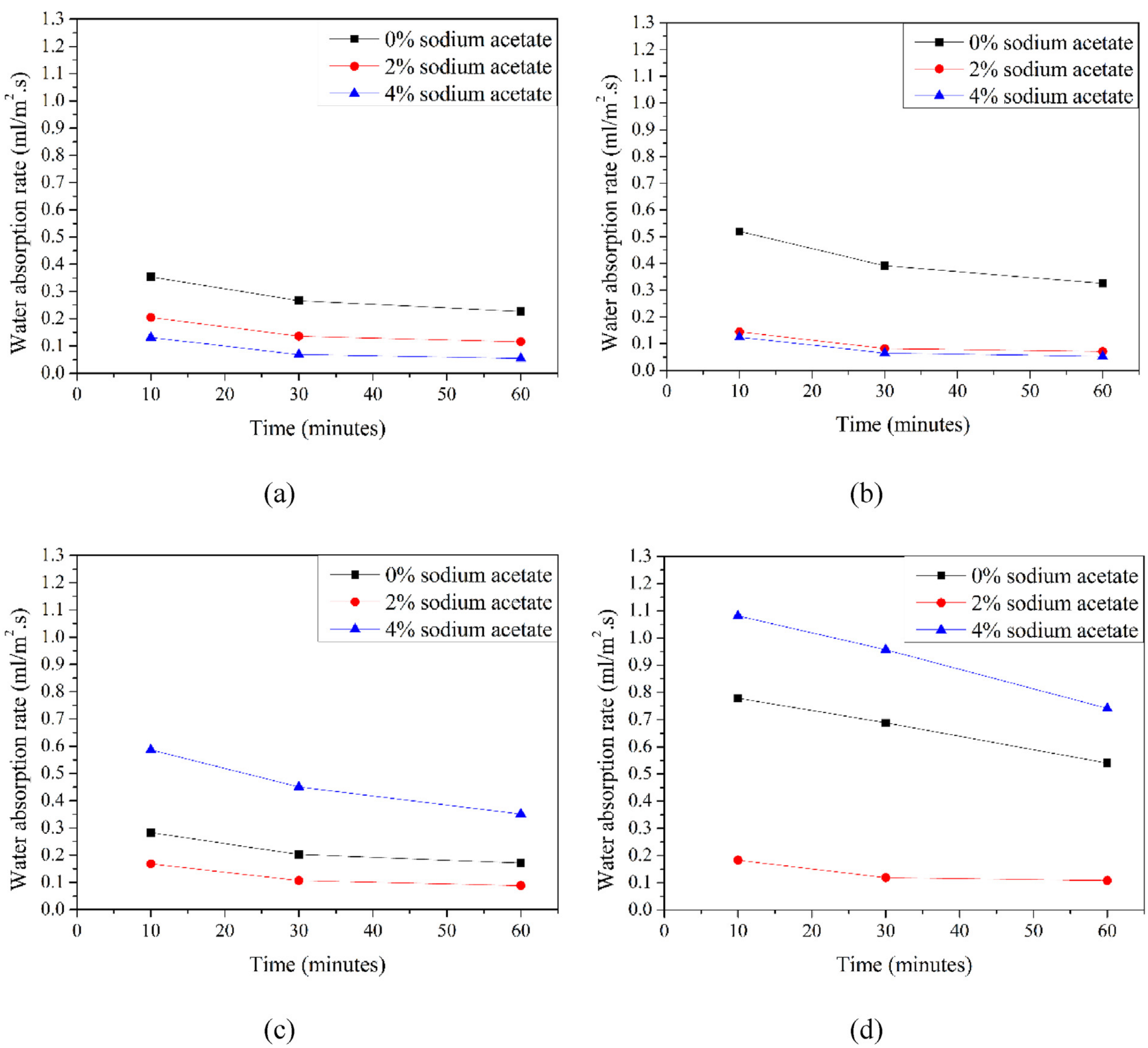


Fig. 9. Water absorption of concrete after the impact of 180 freeze-thaw cycles in air for concrete with w/c ratio of: (a) 0.32, (b) 0.37, (c) 0.40 and (d) 0.46.

Table 5
Compressive strength of concrete after the impact of freeze-thaw cycles.

		Compressive strength (MPa)											
		Freeze-thaw in water						Freeze-thaw in air					
Sodium acetate %		0%	SD	2%	SD	4%	SD	0%	SD	2%	SD	4%	SD
w/c ratio	**0.32**	38.7	1.1	42.1	1.5	50.5	0.7	38.8	0.9	47.7	1.6	53.5	0.8
	0.37	31.2	0.9	39.2	1.3	46	2	34.8	2.8	45.2	2.3	50.5	1.3
	0.40	45.5	1.2	36.4	1.2	32.9	0.2	54.4	1.8	42.1	1.9	43.3	0.3
	0.46	36.1	0.8	29.1	1	21.2	0.4	45.1	2.3	37	1.6	30.3	1.6

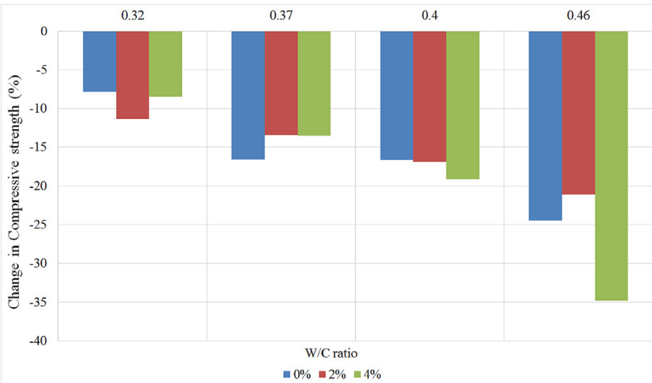


Fig. 10. The impact of freeze-thaw cycles in water on concrete's compressive strength compared to original strength.

sodium acetate has suffered from 35% reduction in its strength, which shows that treatment with high w/c ratios will have a negative effect on the durability of concrete.

The reduction in compressive strength was less severe in the case of low w/c ratio concrete, especially concrete with w/c ratio of 0.32 and treated with 2% and 4% sodium acetate. This reflect the high influence of treatment on low w/c ratio concrete, as it helps in making its structure denser (Table 3) and with minimum cracks even under harsh environments.

Compressive strength of concrete that was under the impact of freeze-thaw cycles in air did not show any reduction in compressive strength, which refers to the low severity of this freeze-thaw test. Also, the major impact of the freeze-thaw cycles in air might only affect the near-surface zone of the concrete, without affecting the internals of concrete, or they may have a minor effect on the interiors of concrete.

4. Conclusions

The long-term performance of concrete treated with 2% and 4% sodium acetate has been tested through two freeze-thaw tests that operate differently; freeze-thaw under the effect of water and freeze-thaw under the effect of air. Both tests have run for 6 continuous months but with different number of cycles and different speeds. In the freeze-thaw test under the effect of water, saturated concrete was immersed in water and placed under the impact of fast 1080 freeze-thaw cycles during the 6 months. However, in the freeze-thaw test under the effect of air, concrete has been under the impact of slow temperature alteration with 180 cycles during the 6 months. Both tests have been destructive to concrete but with severer effect in the case of freeze-thaw cycles in water.

Concrete with low w/c ratios of 0.32 and 0.37 have managed to resist deterioration more than concrete with high w/c ratios of 0.40 and 0.46. Treating the low w/c ratio concrete with the sodium acetate compound has increased its resistance to deterioration unlike the high w/c ratio concrete. Treating high w/c ratio with sodium acetate has increased its deterioration rate more than untreated concrete, and deterioration was noticed to increase with increasing the added dosage of treatment. The opposite was noticed in the low w/c ratio concrete, where increasing the dosage has improved concrete's resistance to deterioration. After both freeze-thaw tests have finished, water absorption and compressive strength of the deteriorated concrete were evaluated. All concrete samples have shown an increase in water absorption. However, this increase was minimum in the case of concrete with low w/c ratio, especially when treated with 4% sodium acetate. The maximum absorption rate was noticed in concrete with 0.46 w/c ratio and treated with 4% sodium acetate. Similar results were noticed after testing the deteriorated concrete for compressive strength. However, in the case of concrete that suffered from the air cyclic freeze-thaw, its compressive strength values did not change and remained similar to original values of non-deteriorated concrete.

Acknowledgement

Authors would like to acknowledge the help and support provided by the Experimental Technique Centre (ETC) at Brunel University London and its researchers, namely: Dr. Nico Nelson, Dr. Lorna Anguilano, Dr. Safiyeh Haghani, and Dr. Ashley Howkins. Also, the help of Dr. Vejen Hlebarov is highly acknowledged.

References

[1] P. Zhang, F.H. Wittmann, M. Vogel, H.S. Müller, T. Zhao, Influence of freeze-thaw cycles on capillary absorption and chloride penetration into concrete, Cement Concr. Res. 100 (2017) 60–67.
[2] M.J. Al-Kheetan, M.M. Rahman, D.A. Chamberlain, Moisture evaluation of concrete pavement treated with hydrophobic surface impregnants, Int. J. Pavement Eng. (2019), https://doi.org/10.1080/10298436.2019.1567917.
[3] Y. Farnam, H.S. Esmaeeli, P.D. Zavattieri, J. Haddock, J. Weiss, Incorporating phase change materials in concrete pavement to melt snow and ice, Cement Concr. Compos. 84 (2017) 134–145.
[4] Y. Farnam, S. Dick, A. Wiese, J. Davis, D. Bentz, J. Weiss, The influence of calcium chloride deicing salt on phase changes and damage development in cementitious materials, Cement Concr. Compos. 64 (2015) 1–15.
[5] Y. Farnam, A. Wiese, D. Bentz, J. Davis, J. Weiss, Damage development in cementitious materials exposed to magnesium chloride deicing salt, Construct. Build. Mater. 93 (2015) 384–392.
[6] M.J. Al-Kheetan, M.M. Rahman, D.A. Chamberlain, Fundamental interaction of hydrophobic materials in concrete with different moisture contents in saline environment, Construct. Build. Mater. 207 (2019) 122–135.
[7] W. Sun, R. Mu, X. Luo, C. Miao, Effect of chloride salt, freeze–thaw cycling and externally applied load on the performance of the concrete, Cement Concr. Res. 32 (12) (2002) 1859–1864.
[8] K. Li, Durability Design of Concrete Structures: Phenomena, Modeling, and Practice, John Wiley & Sons, 2017.
[9] S.W. Tang, Y. Yao, C. Andrade, Z.J. Li, Recent durability studies on concrete structure, Cement Concr. Res. 78 (2015) 143–154.
[10] P.K. Mehta, P.J.M. Monteiro, Concrete: Microstructure, Properties, and Materials, third ed., McGraw-Hill, New York; London, 2006.
[11] L. Bertolini, B. Elsener, P. Pedeferri, E. Redaelli, R.B. Polder, Corrosion of Steel in Concrete: Prevention, Diagnosis, Repair, John Wiley & Sons, 2013.
[12] C.M. Dry, Three designs for the internal release of sealants, adhesives, and waterproofing chemicals into concrete to reduce permeability, Cement Concr. Res. 30 (12) (2000) 1969–1977.
[13] Z. Lu, X. Zhou, The waterproofing characteristics of polymer sodium carboxymethyl-cellulose, Cement Concr. Res. 30 (2) (2000) 227–231.
[14] M.J. Al-Kheetan, S.H. Ghaffar, O.A. Madyan, M.M. Rahman, Development of low absorption and high-resistant sodium acetate concrete for severe environmental conditions, Construct. Build. Mater. 230 (2020) 117057.
[15] M.J. Al-Kheetan, M.M. Rahman, D.A. Chamberlain, Development of hydrophobic concrete by adding dual-crystalline admixture at mixing stage, Struct. Concr. 19 (5) (2018) 1504–1511.
[16] M.J. Al-Kheetan, M. Rahman, B.N. Muniswamappa, D. Chamberlain, Performance enhancement of self-compacting concrete in saline environment by hydrophobic

surface protection, Can. J. Civ. Eng. 46 (8) (2019) 677–686, https://doi.org/10.1139/cjce-2018-0546.
[17] S.H. Ghaffar, M. Al-Kheetan, P. Ewens, T. Wang, J. Zhuang, Investigation of the interfacial bonding between flax/wool twine and various cementitious matrices in mortar composites, Construct. Build. Mater. 239 (2020) 117833.
[18] M.J. Al-Kheetan, M.M. Rahman, D.A. Chamberlain, Remediation and protection of masonry structures with crystallising moisture blocking treatment, Int. J. Building Pathol. Adaptation 36 (1) (2018) 77–92.
[19] H. Herb, A. Gerdes, G. Brenner-Weiß, Characterization of silane-based hydrophobic admixtures in concrete using TOF-MS, Cement Concr. Res. 70 (2015) 77–82.
[20] J. De Vries, R.B. Polder, Hydrophobic treatment of concrete, Construct. Build. Mater. 11 (4) (1997) 259–265.
[21] F. Tittarelli, G. Moriconi, The effect of silane-based hydrophobic admixture on corrosion of reinforcing steel in concrete, Cement Concr. Res. 38 (11) (2008) 1354–1357.
[22] A. Brenna, F. Bolzoni, S. Beretta, M. Ormellese, Long-term chloride-induced corrosion monitoring of reinforced concrete coated with commercial polymer-modified mortar and polymeric coatings, Construct. Build. Mater. 48 (2013) 734–744.
[23] L. Falchi, E. Zendri, U. Müller, P. Fontana, The influence of water-repellent admixtures on the behaviour and the effectiveness of Portland limestone cement mortars, Cement Concr. Compos. 59 (2015) 107–118.
[24] J.G. Dai, Y. Akira, F.H. Wittmann, H. Yokota, P. Zhang, Water repellent surface impregnation for extension of service life of reinforced concrete structures in marine environments: the role of cracks, Cement Concr. Compos. 32 (2) (2010) 101–109.
[25] V.G. Cappellesso, N. dos Santos Petry, D.C.C. Dal Molin, A.B. Masuero, Use of crystalline waterproofing to reduce capillary porosity in concrete, Journal of Building Pathology and Rehabilitation 1 (1) (2016) 9.
[26] L.W. Teng, R. Huang, J. Chen, A. Cheng, H.M. Hsu, A study of crystalline mechanism of penetration sealer materials, Materials 7 (1) (2014) 399–412.
[27] H. Justnes, T.A. Østnor, N. Barnils Vila, October. Vegetable oils as water repellents for mortars, in: Proceedings of the 1st International Conference of Asian Concrete Federation, 2004, pp. 28–29. Chiang Mai.
[28] J.T. Kevern, Using soybean oil to improve the durability of concrete pavements, International Journal of Pavement Research and Technology 3 (5) (2010) 280–285.
[29] Z. Ma, F.H. Wittmann, J. Xiao, T. Zhao, Influence of freeze-thaw cycles on properties of integral water repellent concrete, J. Wuhan Univ. Technol.-Materials Sci. Ed. 31 (4) (2016) 851–856.
[30] A. Al-Otoom, A. Al-Khlaifa, A. Shawaqfeh, Crystallization technology for reducing water permeability into concrete, Ind. Eng. Chem. Res. 46 (16) (2007) 5463–5467.
[31] P. Zhang, F.H. Wittmann, M. Vogel, H.S. Müller, T. Zhao, Influence of freeze-thaw cycles on capillary absorption and chloride penetration into concrete, Cement Concr. Res. 100 (2017) 60–67.
[32] H.S. Shang, T.H. Yi, Freeze-thaw durability of air-entrained concrete, Sci. World J. 2013 (2013), https://doi.org/10.1155/2013/650791, 2013.
[33] C. Jianxun, Z. Xizhong, L. Yanbin, D. Xianghui, L. Qin, Investigating freeze-proof durability of C25 shotcrete, Construct. Build. Mater. 61 (2014) 33–40.
[34] H.S. Shang, T.H. Yi, Y.P. Song, Behavior of plain concrete of a high water-cement ratio after freeze-thaw cycles, Materials 5 (9) (2012) 1698–1707.
[35] Y. Wu, B. Wu, Residual compressive strength and freeze–thaw resistance of ordinary concrete after high temperature, Construct. Build. Mater. 54 (2014) 596–604.
[36] British Standards Institution BS 1881-125, 2013. *Testing C*oncrete. Methods for Mixing and Sampling Fresh Concrete in the Laboratory. British Standards Institution, London
[37] China Academy of Building Research, GB/T 50082-2009 Standard for Test Methods of Long-Term Performance and Durability of Ordinary Concrete, Ministry of Housing and Urban-rural Development of China, Beijing, 2009, pp. 10–14.
[38] British Standards Institution BS EN 12350-2, Testing Fresh Concrete. Slump Test, British Standards Institution, London, 2009.
[39] British Standards Institution BS 1881-208, Testing Concrete. Recommendations For the Determination of the Initial Surface Absorption of Concrete, British Standards Institution, London, 1996.
[40] British Standards Institution BS EN 12390-3, Testing Hardened Concrete. Compressive Strength of Test Specimens, British Standards Institution, London, 2009.
[41] American Society for Testing and Materials, ASTM C1202: Standard Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration, American Society for Testing and Materials, Philadelphia, Pa, 1997.
[42] M. Safiuddin, N. Hearn, Comparison of ASTM saturation techniques for measuring the permeable porosity of concrete, Cement Concr. Res. 35 (5) (2005) 1008–1013.
[43] R. Kumar, B. Bhattacharjee, Porosity, pore size distribution and in situ strength of concrete, Cement Concr. Res. 33 (1) (2003) 155–164.
[44] J. Liu, K. Tang, Q. Qiu, D. Pan, Z. Lei, F. Xing, Experimental investigation on pore structure characterization of concrete exposed to water and chlorides, Materials 7 (9) (2014) 6646–6659.
[45] M.J. Al-Kheetan, M.M. Rahman, D.A. Chamberlain, Optimum mix design for internally integrated concrete with crystallizing protective material, J. Mater. Civ. Eng. 31 (7) (2019), 04019101.
[46] L.E.M. Palomino, Z. Pászti, I.V. Aoki, H.G.D. Melo, Comparative investigation of the adhesion of Ce conversion layers and silane layers to a AA 2024-T3 substrate through mechanical and electrochemical tests, Mater. Res. 10 (4) (2007) 399–406.
[47] P.B. Wagh, S.V. Ingale, S.C. Gupta, Comparison of hydrophobicity studies of silica aerogels using contact angle measurements with water drop method and adsorbed water content measurements made by Karl Fischer's titration method, J. Sol. Gel Sci. Technol. 55 (1) (2010) 73–78.
[48] P.B. Wagh, R. Kumar, R.P. Patel, I.K. Singh, S.V. Ingale, S.C. Gupta, D.B. Mahadik, A.V. Rao, Hydrophobicity measurement studies of silica Aerogels using FTIR spectroscopy, weight difference method, contact angle method and KF Titration method, J. Chem. Biol. Phys. Sci. 5 (3) (2015) 2350.
[49] M.J. Al-Kheetan, M.M. Rahman, Integration of anhydrous sodium acetate (ASAc) into concrete pavement for protection against harmful impact of deicing salt, J. Occup. Med. 71 (12) (2019) 4899–4909.
[50] C. Foy, M. Pigeon, N. Banthia, Freeze-thaw durability and deicer salt scaling resistance of a 0, 25 water-cement ratio concrete, Cement Concr. Res. 18 (4) (1988) 604–614.
[51] M. Chougan, E. Marotta, F.R. Lamastra, F. Vivio, G. Montesperelli, U. Ianniruberto, A. Bianco, A systematic study on EN-998-2 premixed mortars modified with graphene-based materials, Construct. Build. Mater. 227 (2019) 116701.
[52] S.H. Ghaffar, M. Al-Kheetan, P. Ewens, T. Wang, J. Zhuang, Investigation of the interfacial bonding between flax/wool twine and various cementitious matrices in mortar composites, Construct. Build. Mater. 239 (2020) 117833.