Construction and Building Materials 230 (2020) 117057

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Development of low absorption and high-resistant sodium acetate concrete for severe environmental conditions

Mazen J. Al-Kheetan [a,b], Seyed Hamidreza Ghaffar PhD [a,*], Omar Abo Madyan [a], Mujib M. Rahman [a]

[a] Department of Civil and Environmental Engineering, Brunel University London, Kingston Ln, Uxbridge, Middlesex UB8 3PH, United Kingdom
[b] Department of Civil and Environmental Engineering, College of Engineering, Mutah University, P.O. Box 7, Mutah, Karak 61710, Jordan

HIGHLIGHTS

- Influence of harsh curing conditions on concrete integrated with sodium acetate.
- The interaction between sodium acetate and concrete under extreme curing conditions.
- Strength development and concrete's resistance to extreme environments.
- Enhancing the impermeability of concrete treated with sodium acetate.

ARTICLE INFO

Article history:
Received 28 July 2019
Received in revised form 17 September 2019
Accepted 20 September 2019
Available online 26 September 2019

Keywords:
Sodium acetate
High resistant concrete
Extreme curing
Water absorption
Compressive strength

ABSTRACT

This research presents new insight on the performance of concrete when integrated with sodium acetate and cured under extremely harsh environmental conditions: freezing temperature of −25 °C and hot temperature of 60 °C. Mechanical properties, water absorption, microstructural analysis and interaction mechanism of concrete and sodium acetate were evaluated by conducting the compressive strength test, Initial Surface Absorption Test (ISAT), Scanning Electron Microscope (SEM) analysis and Fourier-transform Infrared Spectroscopy (FTIR) analysis. Despite the harsh curing conditions, results showed an enhancement of 64% in compressive strength when 4% (based on the weight of cement) sodium acetate is incorporated within concrete with w/c ratio of 0.32 and cured under temperature of 60 °C. Also, water absorption was observed to decrease by more than 79% when 2% sodium acetate is added to concrete with w/c ratio of 0.32. SEM and FTIR analyses revealed the formation, high distribution and strong bonding of sodium acetate crystals within the concrete's micropores.

1. Introduction

The influence of extreme weather conditions on concrete, either when it is fresh or matured, have been reported to cause serious problems to the body of the concrete structure. Extremely high temperatures, for instance, increase the evaporation rate of water that is necessary for hydration process, which leads to crack propagation and strength reduction [1–4]. Likewise, placing concrete under cold weather conditions will initiate cracks in conjunction with spalling and strength reduction due to the early frost of concrete [5]. Therefore, protecting concrete structures that are exposed to cold or hot environments has become a necessity to reduce their deterioration rate and extend their service life [6–9].

In latest years, more interest in protecting concrete by hydrophobic impregnation has come to light [10,11]. Silane and Siloxane impregnates were one of the first effective hydrophobic treatments to be used for enhancing concrete's water impermeability and resistance to extreme weather conditions [12–14]. The efficacy of this kind of materials depends mainly on the 'penetration depth; higher penetration depths result in more effective protection [15]. However, recently some doubts were raised regarding the performance and effect of such products on the environment [14]. The deficiency of Silane and Siloxane materials to protect damped concrete surfaces and the presence of some organic solvents in their chemical composition, which negatively affects the ecosystem, encouraged researchers to seek for some new water-tolerant and environmentally friendly materials [8,16–18]. Accordingly, researchers developed some new protective materials and techniques, natural and synthetic, that are ecologically friendly and aim to work well in the presence of water. Incorporating sodium acetate in the concrete mix is a new

* Corresponding author.
E-mail addresses: mazen.al-kheetan@mutah.edu.jo (M.J. Al-Kheetan), seyed.ghaffar@brunel.ac.uk (S.H. Ghaffar), omar.abomadyan@brunel.ac.uk (O.A. Madyan), mujib.rahman@brunel.ac.uk (M.M. Rahman).

https://doi.org/10.1016/j.conbuildmat.2019.117057
0950-0618/Crown Copyright © 2019 Published by Elsevier Ltd.

technique developed recently to overcome the problems associated with surface protection of concrete. Integrating sodium acetate within the concrete mixture has shown its ability to enhance concrete's durability and extend its service life without affecting its strength [16].

In previous work by authors, concrete integrated with sodium acetate has been tested and analysed under normal conditions, and the interaction mechanism between concrete and sodium acetate has been studied [16]. Current work will discuss the ability of sodium acetate to preserve concrete from deterioration when it is exposed to harsh curing conditions.

Therefore, the main objectives of this research are: (1) studying the influence of extreme curing conditions on the performance of concrete when integrated with sodium acetate, (2) establishing an aggregated understanding for the interaction between sodium acetate and concrete under extreme curing conditions and (3) investigating the strength development and concrete's resistance to extreme environments.

2. Materials and test methods

2.1. Materials and sample preparations

A protective material formed mainly from sodium acetate as an active content with other cementitious material was used in this research. The core of the proposed technology, shown in Fig. 1, depends on the dual functionality of the material. Sodium acetate absorbs water to form crystals that settle inside the pores to line them [16,19]. The formed crystals were found to develop hydrophobic properties that enables them to fend off excess water from the pores and increase its impermeability (Fig. 1).

A total of 72 cubes with the dimensions of 100 mm × 100 mm × 100 mm were produced. Two different concrete mixtures with w/c ratios of 0.32 and 0.46 were used to study the influence of water content on the performance of the protective material, following the guidelines of BS 1881-125 [20]. A sodium acetate compound was added to the fresh mix in two ratios of 2% and 4% of the cement mass. A control mix with 0% protective material was also produced for each mix design for comparison purposes. Table 1 illustrates the mix designs for the two types of concrete mixes.

To study the effect of extreme weather conditions on the produced concrete, all samples were cured in an environmental confined chamber. A set of the concrete samples were cured under freezing conditions with a temperature of −25 °C, and the other set were cured under hot conditions with a temperature of 60 °C.

Table 1
Proportions of the used concrete mixtures.

Ingredient	Amount (kg/m^3)	
	W/C = 0.32	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	457
Water	164	210
Fine aggregate (sharp silica sand with uniform grain size distribution between 1 mm and 300 μm)	658	660
Coarse aggregate (crushed stones with sharp edges and maximum size of 20 mm)	1068	1073

2.2. Test methods

Experimental procedure was divided into three main parts; 1) water absorption, 2) compressive strength and 3) microstructure analysis. Initial Surface Absorption Test (ISAT) was used to assess the ability of the protective material (sodium acetate) to enhance the impermeabilty of concrete following the guidelines of the BS 1881-208 [21]. Testing was carried out on all samples for durations of 10, 30 and 60 min for each sample and after 7 and 28 days of curing (hot and freezing curing conditions and benchmarked against control samples with no addition of sodium acetate). The compressive strength of concrete was assessed at 7 and 28 days of same curing conditions BS EN 12390-3:2009 [22]. Finally, the interaction between sodium acetate and concrete constituent materials, were investigated using the Scanning Electron Microscope (SEM), this included the distribution of sodium acetate within concrete and their functionality in the pores. All samples were coated with a thin gold film before placing them under the SEM to make them conductive.

3. Results and discussion

3.1. Microstructural analysis

The formation of the sodium acetate inside the pores of concrete and the effect of harsh environment on its functionality within concrete were investigated through SEM. Fig. 2 shows untreated concrete (0% of sodium acetate) after its exposure to both extreme environments, this was carried out to better

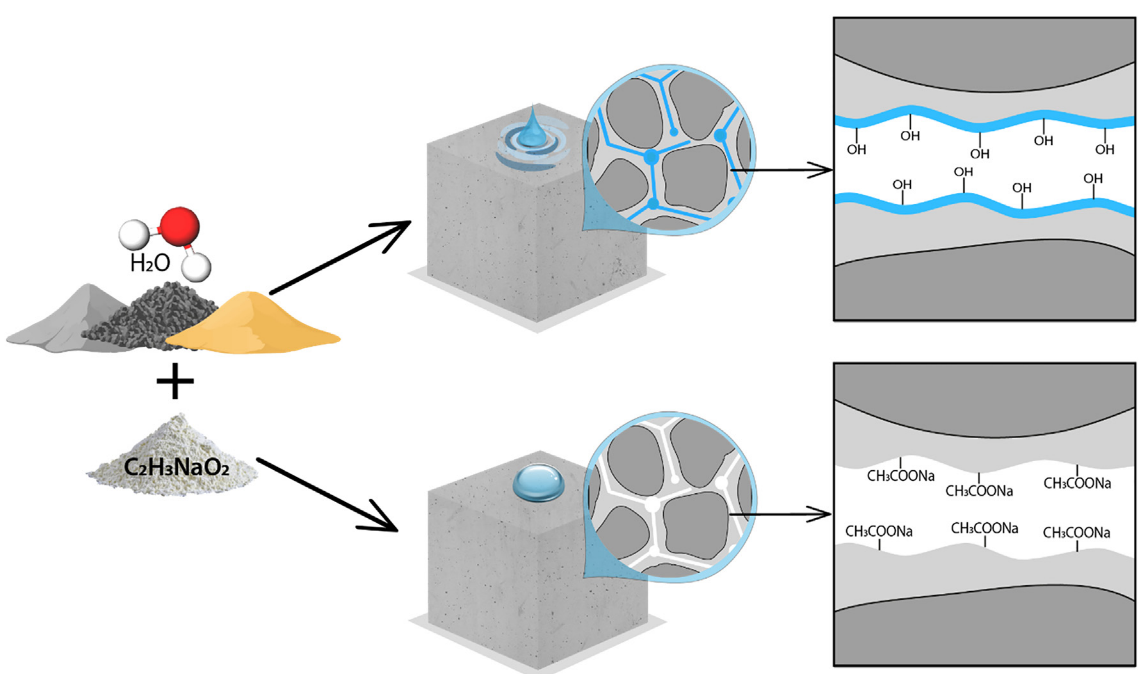

Fig. 1. Schematic illustration of the interaction and bonding between sodium acetate and concrete.

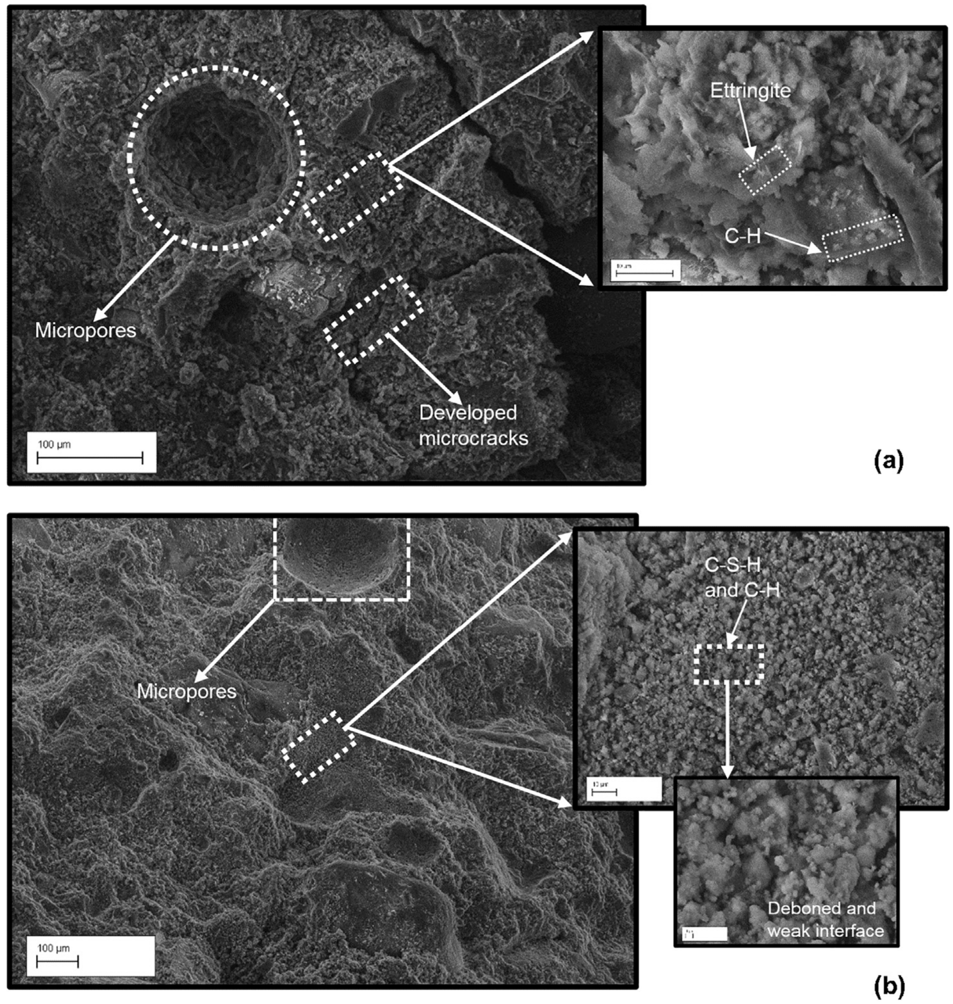

Fig. 2. Morphology of untreated concrete after its curing under extreme conditions of: (a) −25 °C (100×, 5000× and 10,000×) and (b) 60 °C (500×, 5000× and 10,000×).

interpret the effects of such curing conditions on the microstructure of concrete.

As illustrated in Fig. 2a and b, extreme curing conditions have negatively affected the control samples, where freezing condition (−25 °C) (Fig. 2a) has led to the introduction of some microcracks through the internal parts of the concrete samples. Curing concrete under freezing conditions, especially at the beginning of the hydration process, would result in freezing the available water that is necessary for hydration process inside the pores. This will cause frozen water to expand inside the pores and will lead to: (1) increasing the crack propagation rate within the pores due to water expansion, (2) delaying the hydration process as long as water is still frozen and not promoting the hydration process, and (3) creating weak cement-aggregate bonds [16].

Interestingly, curing control samples under high temperature of 60 °C did not induce any evident microcracks in concrete (Fig. 2b). However, an obvious wearing effect for temperature rise was observed on the tested cross-sectional areas, where a weak bond has developed between hydrated cement particles and aggregates. Additionally, a reduction in the presence of ettringite and other hydration products within the microstructure of concrete is clearly observed (Fig. 2b). This can be due to the rapid moisture loss from concrete at an early age which will influence the development of hydration during time. Moreover, high temperature can result in increased shrinkage development and self-produced stress in the microstructure of concrete [23].

On the other hand, incorporating sodium acetate as an admixture within concrete has led to an improved morphology of concrete when cured under extreme environmental conditions. Fig. 3a and b show the ‘sodium acetate-concrete’ matrix interfaces, where a full illustration of the bond between the components of the used protective material and concrete is revealed.

As shown in Fig. 3a, incorporating the sodium acetate compound within the concrete mixture has greatly reduced crack propagation when concrete is under freezing conditions. Additionally, the presence of sodium acetate has led to development of a denser microstructure compared to control in Fig. 2a. This can mean a greater resistance to the weathering effect of the freezing environment. Also, the presence of sodium acetate crystals inside the pores in dense formations and distribution will increase the rigidity of the pores.

The integration of sodium acetate in concrete while curing it at 60 °C has helped in reducing the weathering effect of high temperatures (Fig. 3b). The high distribution of sodium acetate crystals within concrete and its high resistance to heat (>300 °C) [24] has resulted in preserving the internal structure of concrete from debonding and the reservation of the hydration products from weathering.

In a recent research by authors, FTIR analysis of sodium acetate and concrete was conducted to give a thorough explanation for their interaction mechanism [16]. Fig. 4 shows the FTIR spectra of sodium acetate and its interaction with concrete.

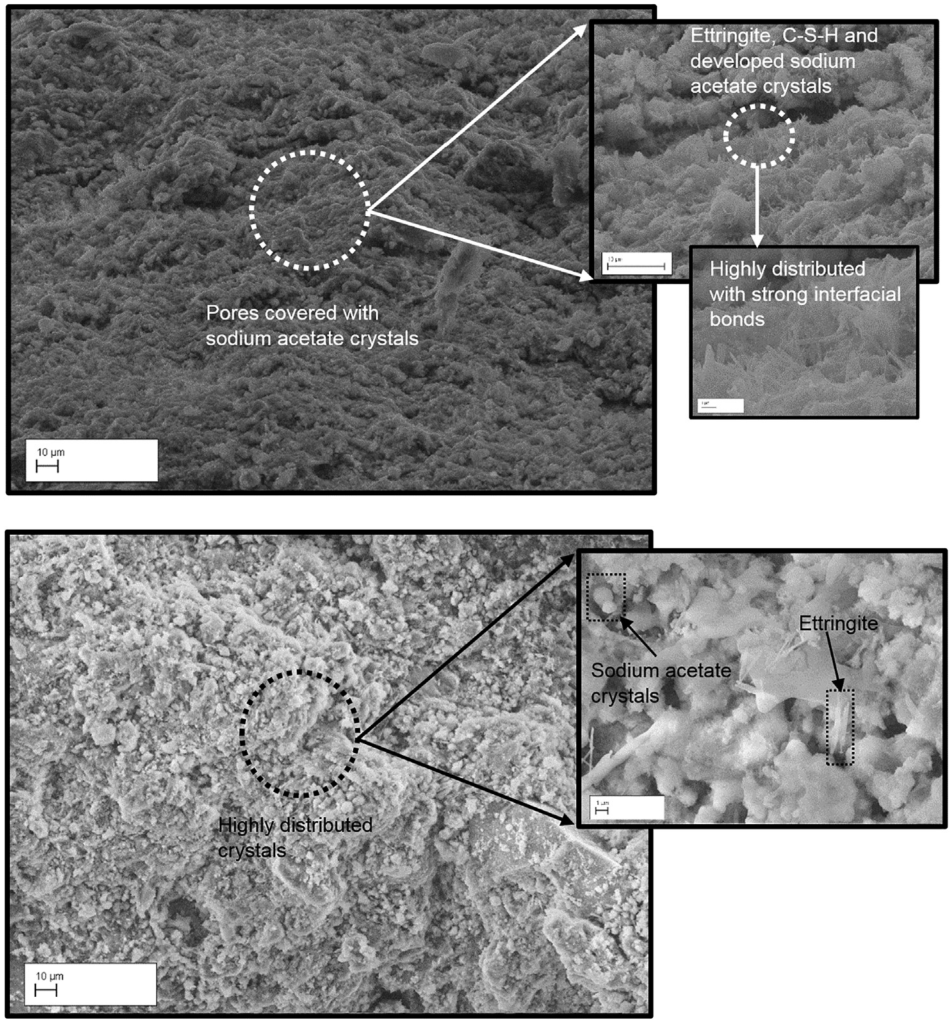

Fig. 3. Morphology of concrete incorporated with sodium acetate and cured under extreme conditions of: (a) −25 °C (1000×, 5000× and 20,000×) and (b) 60 °C (1000×, 5000× and 20,000×).

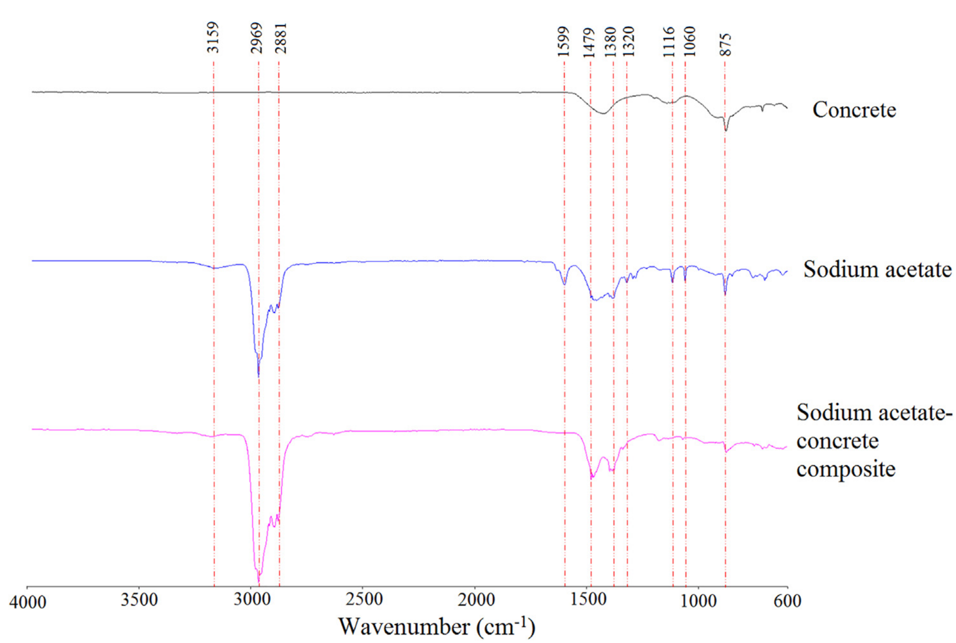

Fig. 4. FTIR analysis of sodium acetate and concrete [16].

As seen in the analysis (Fig. 4), a significant increase in the transmittance of peaks at 2969 cm^{-1} and 2881 cm^{-1}, which correspond to —CH and —OH stretching vibrational bonds, can be spotted. This increase in the hydrogen bond could refer to the activation of sodium acetate in the presence of water, where it dissociates in water as described in the following reaction [25,26]:

$$CH_3COONa \rightarrow CH_3COO^{-} + Na^{+}$$

CH_3COO^{-} ions are understood to connect strongly with cement through their reaction with sodium to form dual functioning crystals that line the pores and work on repelling water [16]. This reaction is believed to increase the intensity of the —CH bond (as seen in Fig. 4). On the other hand, some sodium hydroxide (NaOH) is believed to form after the reaction between the dissociated Na^{+} ions and water that already exists in concrete, which results in an increase in the intensity of —OH bonds [16]. Also, dissociated CH_3COO^{-} might contribute in forming small quantities of acetic acid (CH_3COOH) after its reaction with water, which subsequently leads in increasing the —OH bonds intensity [16].

The increase in the —CH bond can also be attributed to the replacement of —OH groups by —CH_3 groups that bond with silicon that already exists in cement to form hydrophobic organosilicon bonds [16].

3.2. Water absorption properties

The effect of sodium acetate as a protective material on the permeability of concrete, when exposed to extreme environmental conditions, was determined through the ISAT method. As depicted in Fig. 5, adding 4% of sodium acetate either to concrete with w/c ratio of 0.32 or concrete with w/c ratio of 0.46 has increased its water absorption rate after extreme curing for 7 days. Moreover, curing both concrete samples at 60 °C (Fig. 5b and d) has increased their water absorption rate more than 77% for the 0.32 mix and 60% for the 0.46 mix (when compared with concrete). Moreover, freezing conditions have slightly contributed in increasing the water absorption of the aforementioned mixtures. The higher increase in water absorption for concrete treated with 4% sodium acetate and cured under high temperature can be attributed to: (1) the formation of large amounts of NaOH (as explained in the microstructural analysis section) that increase the mixtures' consistency (as shown by Al-Kheetan et al. 2019 [16]) which therefore increases the air voids and microcracks in concrete; and (2) exposing concrete to high temperature of 60 °C at an early age can contribute towards drying water which is necessary for the continuation of the hydration process and the formation of the needed crystals (less water means less formed hydrophobic crystals).

On the other hand, exposing concrete to freezing conditions for 7 days (Fig. 5a and c) showed a less severe influence on its water absorption for both w/c ratio mixtures. Moreover, adding 4% sodium acetate resulted in an increased water absorption rate to more than 4% and 20% for concrete with w/c ratios of 0.32 and 0.46 respectively. This increase in water absorption may refer to the influence of low temperature on the hydration process, where cold weather slows the setting time of concrete [27]. Low

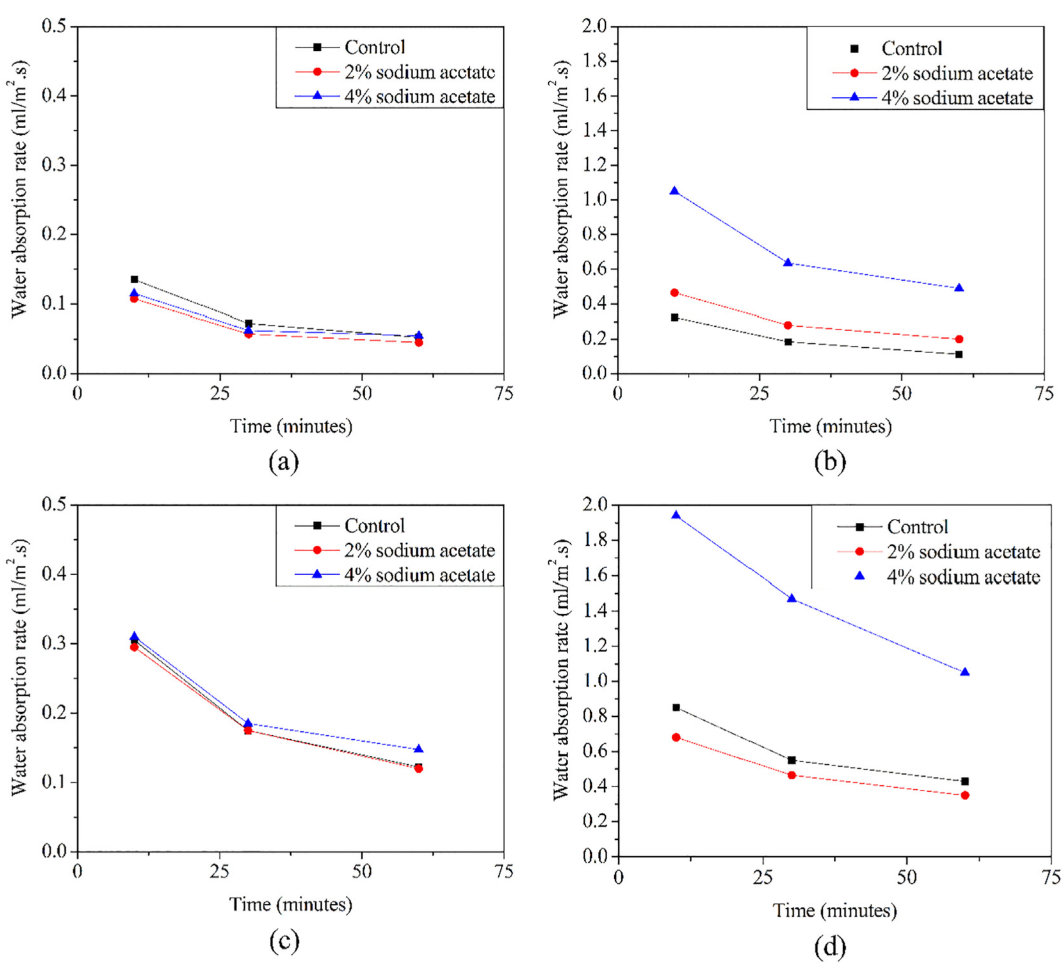

Fig. 5. Water absorption rate at 7 days for concrete with (a) w/c = 0.32 cured under −25 °C, (b) w/c = 0.32 cured under 60 °C, (c) w/c = 0.46 cured under −25 °C and (d) w/c = 0.46 cured under 60 °C.

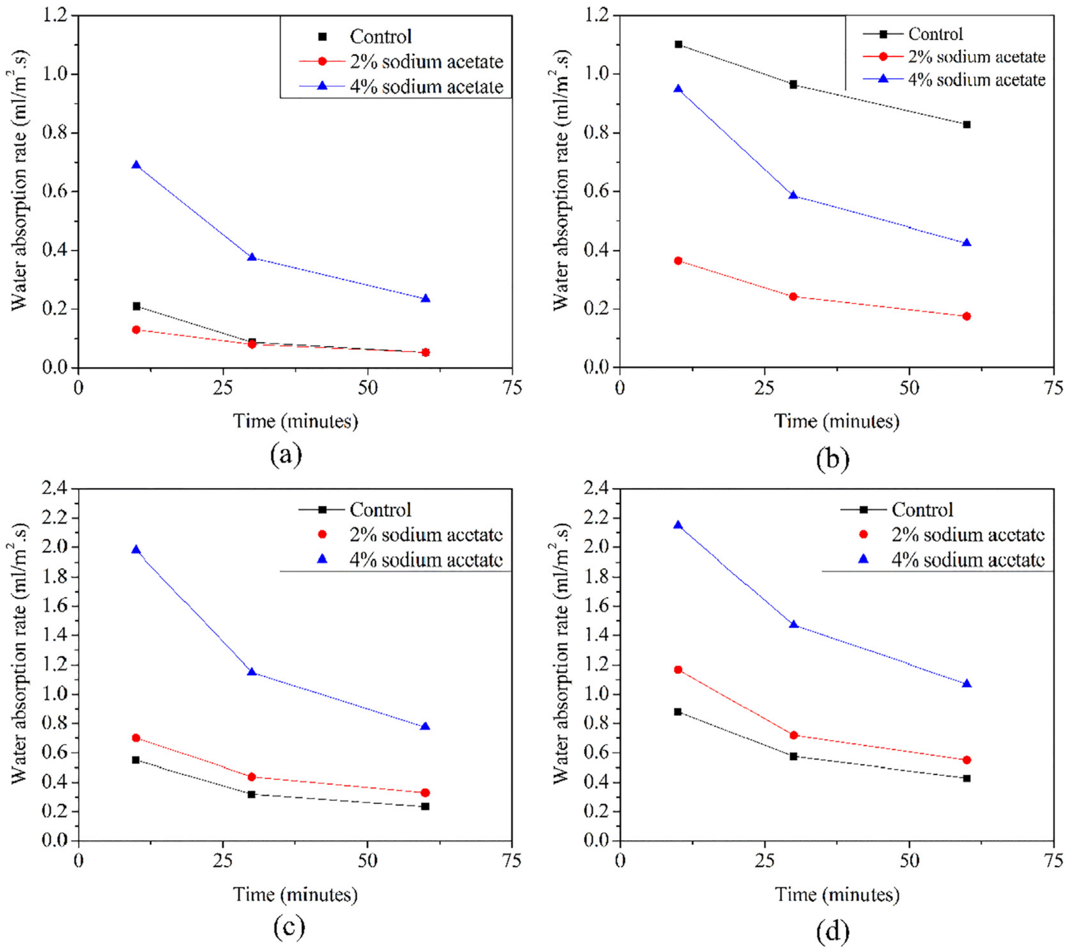

Fig. 6. Water absorption rate at 28 days for concrete with (a) w/c = 0.32 cured under −25 °C, (b) w/c = 0.32 cured under 60 °C, (c) w/c = 0.46 cured under −25 °C and (d) w/c = 0.46 cured under 60 °C.

temperature can also lead to a sharp thermal gradient through the whole depth of the concrete microstructure, which in turn results in the formation of internal stresses and the propagation of micro-cracks [28].

Water absorption properties were also assessed after 28 days of curing under extreme temperatures of −25 °C and 60 °C. As shown in Fig. 6, adding 2% of sodium acetate to concrete with w/c ratio of 0.32 has managed to reduce the water absorption rate by 79% when cured at 60 °C (compared with control). Moreover, adding the same dose of sodium acetate led to the preservation of the water absorption rate of concrete with w/c ratio of 0.32 when cured at −25 °C. The high resistance of concrete to both extreme curing conditions when treated with the 2% sodium acetate (Fig. 6a and b) can be explained by: (1) the demand of 2% sodium acetate to water to form its crystals is less than the demand of the 4% dose, which makes the available water during high temperature curing enough to continue the hydration process and, at the same time, activating the crystals of sodium acetate, (2) the presence of sodium acetate crystals in the pores, during low temperature curing, will help in reducing the thermal gradient through concrete, which will decrease the internal pressure inside the pores, and (3) the formation of enough organosilicon bonds inside the pores of concrete that are capable of reducing the absorbed water either after high temperature curing or low temperature curing (as shown in the microstructural analysis section).

It is worth pointing out the high reduction in water absorption was mainly observed in treated concrete with w/c ratio of 0.32 and cured under 60 °C. This might refer to the influence of high temperature on accelerating the hydration process. Also, the presence of sodium acetate in its full crystal form, as seen in Fig. 3b, under high temperature contributed in reducing water intake to very low levels.

3.3. Compressive strength development

The influence of curing different concrete mixtures at extreme temperatures on the compressive strength properties was assessed at 7 and 28 days. As highlighted in Fig. 7, the addition of sodium acetate has greatly enhanced the protection level of concrete under extreme conditions, as it worked on preserving the strength of control (no drop in compressive strength) when added to concrete with high w/c ratio. Moreover, sodium acetate even worked on increasing the strength of concrete when added to concrete with low w/c ratio. The addition of sodium acetate, either by 2% or 4%, with w/c ratio of 0.32 led to increase in concrete's strength at 7 and 28 days despite the extreme curing conditions. Adding 4% sodium acetate increased the strength of 0.32 concrete by more than 64% (compared to control) even after it's curing in hot conditions. Moreover, adding 2% sodium acetate to concrete with w/c ratio of 0.46 worked on preserving its strength when exposed to cold temperature and increased it by 8% when exposed to high temperature (both after 28 days of curing and compared to control). These results reflect the high influence of treatment on concrete and its ability to combat extreme harsh conditions, as

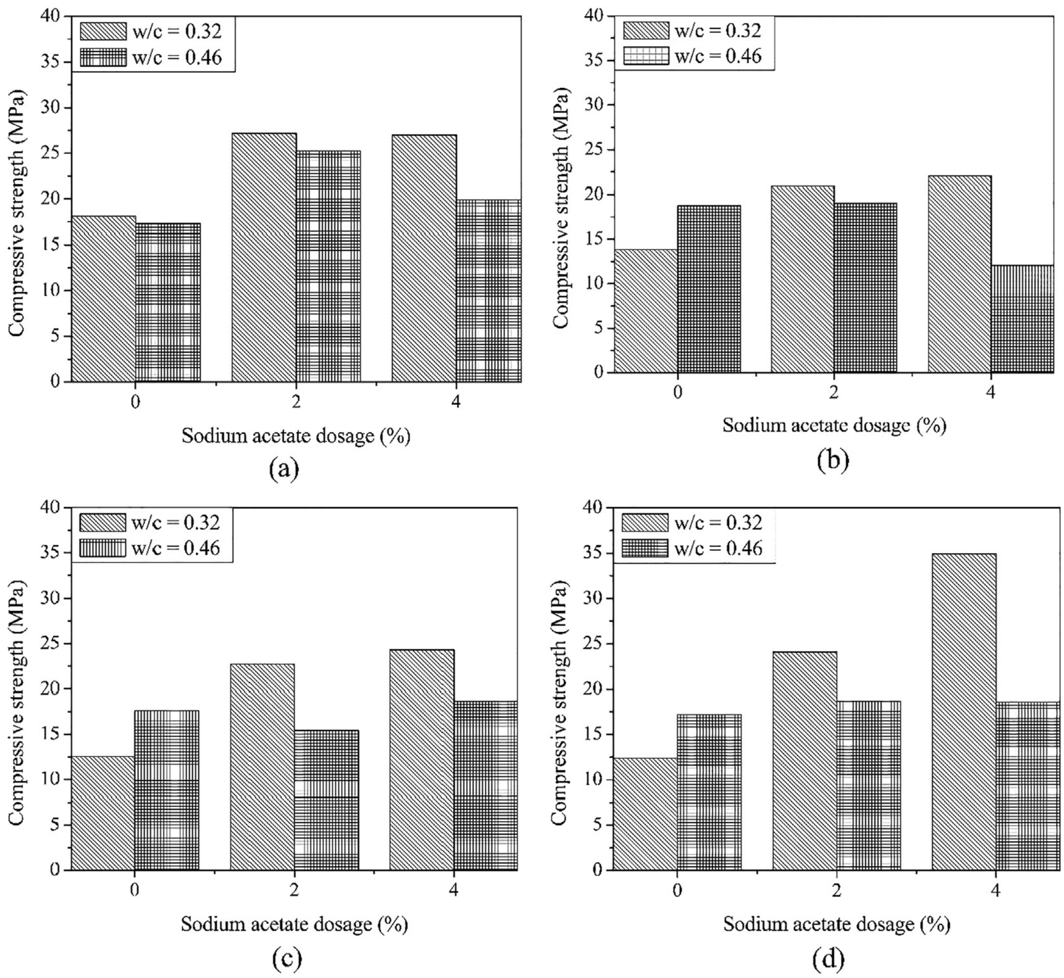

Fig. 7. Compressive strength of concrete after: (a) curing under −25 °C for 7 days, (b) curing under −25 °C for 28 days, (c) curing under 60 °C for 7 days and (d) curing under 60 °C for 28 days.

treatment participates in making the concrete's structure denser and with minimum microcracks (refer to microstructural analysis section).

4. Conclusions

In this research, the performance of concrete integrated with sodium acetate has been investigated when it is exposed to two extreme curing conditions; freezing temperature of −25 °C and high temperature of 60 °C. The effectiveness of treatment to protect concrete was evaluated by investigating the mechanical, physical and microstructural properties. The following conclusions can be drawn from this research:

1. Adding sodium acetate compound to concrete increased the interfacial bonds between hydration products and aggregates. It was shown that sodium acetate covers the macropores (>1000 nm), most of the capillary pores (100–1000 nm), most of the mesopores (10–10,000 nm), and some of the transitional pores (10–100 nm) of concrete which leads to enhanced rigidity.
2. The incorporation of sodium acetate compound established strong —CH bonds with concrete. Also, a hydrophobic organosilicon bond was formed inside the pores upon the addition of sodium acetate.
3. Adding 2% sodium acetate to concrete with low w/c ratio of 0.32 enhanced its impermeability by 79% when concrete is cured at high temperature (60 °C). Moreover, the 2% sodium acetate preserved concrete with w/c ratio of 0.32 from absorbing water when exposed to freezing curing conditions (−25 °C).
4. A negative effect was noticed when adding sodium acetate to concrete with high w/c ratio of 0.46, where an increase in water absorption was observed.
5. The compressive strength of concrete, even when cured under freezing and hot conditions was increased as a result of the addition of sodium acetate.

The durability and long-term performance of concrete integrated with sodium acetate is currently under assessment.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.conbuildmat.2019.117057.

References

[1] J. Ortiz, A. Aguado, L. Agullo, T. Garcia, Influence of environmental temperatures on the concrete compressive strength: Simulation of hot and cold weather conditions, Cem. Concr. Res. 35 (2005) 1970–1979.
[2] S. Alsayed, M. Amjad, Effect of curing conditions on strength, porosity, absorptivity, and shrinkage of concrete in hot and dry climate, Cem. Concr. Res. 24 (1994) 1390–1398.
[3] M.J. Al-Kheetan, M.M. Rahman, D.A. Chamberlain, Fundamental interaction of hydrophobic materials in concrete with different moisture contents in saline environment, Constr. Build. Mater. 207 (2019) 122–135.
[4] 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 (2019) 04019101.
[5] J. Ryou, Y. Lee, Use of tabletting & coating accelerator for the prevention of early-frost of concrete in cold weather, Cold Reg. Sci. Technol. 87 (2013) 1–5.
[6] 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 (2018) 1504–1511.
[7] M.J. Al-Kheetan, M.M. Rahman, D.A. Chamberlain, Influence of early water exposure on modified cementitious coating, Constr. Build. Mater. 141 (2017) 64–71.
[8] M.J. Al-Kheetan, M.M. Rahman, D.A. Chamberlain, Influence of crystalline admixture on fresh concrete to develop hydrophobicity, Transportation Research Board 96th annual meeting (No. 17-02487), 2017.
[9] 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 (2019) 677–686.
[10] M.J. Al-Kheetan, M.M. Rahman, D.A. Chamberlain, Remediation and protection of masonry structures with crystallising moisture blocking treatment, Int. J. Build. Pathol. Adapt. 36 (2018) 77–92.
[11] 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) 1–9.
[12] J. De Vries, R. Polder, Hydrophobic treatment of concrete, Constr. Build. Mater. 11 (1997) 259–265.
[13] P. Basheer, L. Basheer, D. Cleland, A. Long, Surface treatments for concrete: assessment methods and reported performance, Constr. Build. Mater. 11 (1997) 413–429.
[14] C. Christodoulou, H. Tiplady, C. Goodier, S. Austin, Performance of silane impregnants for the protection of reinforced concrete, Concr. Sol. (2014) 385–392.
[15] J. Dai, Y. Akira, F. 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, Cem. Concr. Compos. 32 (2010) 101–109.
[16] M.J. Al-Kheetan, M.M. Rahman, Integration of anhydrous sodium acetate (ASAc) into concrete pavement for protection against harmful impact of deicing salt, JOM (2019) 1–11.
[17] M. Rahman, N. Alkordi, A. Ragrag, S. Kamal, D. Chamberlain, Moisture efficacy of impregnant in concrete protection, Transportation Research Board 95th Annual Meeting, 2016.
[18] M.C. Bubalo, K. Radošević, I.R. Redovniković, J. Halambek, V.G. Srček, A brief overview of the potential environmental hazards of ionic liquids, Ecotoxicol. Environ. Saf. 99 (2014) 1–12.
[19] A. Al-Otoom, A. Al-Khlaifa, A. Shawaqfeh, Crystallization technology for reducing water permeability into concrete, Ind. Eng. Chem. Res. 46 (2007) 5463–5467.
[20] British Standards Institution. BS 1881-125:2013: Testing concrete. Methods for mixing and sampling fresh concrete in the laboratory. 2013.
[21] British Standards Institution. BS 1881-208:1996: Testing concrete. Recommendations for the determination of the initial surface absorption of concrete. 1996.
[22] British Standards Institution. BS EN 12390-3:2009: Testing hardened concrete. Compressive strength of test specimens. 2009.
[23] P. Lura, K. van Breugel, I. Maruyama, Effect of curing temperature and type of cement on early-age shrinkage of high-performance concrete, Cem. Concr. Res. 31 (2001) 1867–1872.
[24] United States Environmental Protection Agency. Sodium Acetate 127-09-3 | DTXSID2027044. https://comptox.epa.gov/dashboard/dsstoxdb/results?search=DTXSID2027044 (Accessed 05 July 2019)
[25] B. Sharma, Objective Question Bank in Chemistry, Krishna Prakashan Media, 1997.
[26] R. Chang, Physical chemistry for the biosciences, University Science Books, Sausalito, USA, 2005.
[27] C.K. Nmai, Cold weather concreting admixtures, Cem. Concr. Compos. 20 (1998) 121–128.
[28] A.Y. Nassif, M.F. Petrou, Influence of cold weather during casting and curing on the stiffness and strength of concrete, Constr. Build. Mater. 44 (2013) 161–167.