

**TECHNICAL PAPER**

Development of hydrophobic concrete by adding dual-crystalline admixture at mixing stage

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Funding information

Mu'tah University, Jordan

A novel approach has been presented to add a dual-crystalline hydrophobic admixture in fresh concrete for improving hydrophobicity against chloride and harmful chemicals. Dual-crystalline material can utilize water of the fresh concrete to form crystals, but the challenge is to maintain adequate hydration and strength while improving hydrophobicity. This study presents the results from a comprehensive laboratory investigation on the application of 1, 2 and 8% of crystallizing aqueous and cementitious hydrophobic mineral in fresh concrete. Despite the high slump in the fresh mixture, no segregation was observed in the matured concrete. There was a marginal reduction of strength when a high percentage of admixture was used. Despite this, significant reduction of water absorption was observed indicating greater hydrophobicity. The optimum performance was found in mixtures with 2% admixture. Mixture with 2% aqueous hydrophobic admixture revealed marginal strength gain compared to 2% crystalline cementitious hydrophobic admixture, although water protection appears to be better in cementitious mixture.

KEYWORDS

admixture, compressive strength, crystal structure, fresh concrete, hydrophobic treatment, permeability, water absorption

1 | INTRODUCTION

Although concrete is known for its long serviceability, its performance could be greatly compromised when exposed to a harsh environment like bridges, concrete pavements, and coastal defenses. The deterioration is mainly caused by excessive water, chloride penetration and attack by other harmful substance like chemicals, fuel spillage, etc. In the United Kingdom, for example, there are about 61,000 highways and bridges, most of them are reinforced concrete pavements with reinforced steel, their maintenance expenses reached more than £4 billion in the period 2012–2013.^{1,2} They are subjected to rainfall of 1,420 mm as recorded in the year 2012, and about 1,120 mm in 2014.^{3,4} The easy

entrance of water into concrete can produce many disadvantages affecting its desired performance and durability and could bring many problems into being; including corrosion of the embedded steel, and leakage. Also, some water-associated issues like freezing and thawing could drag concrete to a series of deterioration stages.⁵ Reflecting the need to protect concrete from moisture damages and to decrease its permeability without compromising its strength, the research presented in this study jumps from the improper performance and benefits of in situ protection. Also, it is worth mentioning that there is a need to get over the fact of insufficient penetration of protection materials in concrete and try to develop a way of application on concrete so that it will cover the whole mass of concrete.³ The purpose of this study is to investigate the performance of a crystallizing material mixed at mixing stage, in different percentages, within the concrete mixture.

Discussion on this paper must be submitted within two months of the print publication. The discussion will then be published in print, along with the authors' closure, if any, approximately nine months after the print publication.

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Chemical admixtures are mainly used in concrete either when it is in the fresh state or when it is matured with the intention to improve strength, durability, and impermeability.⁶ Durability could be regarded as a feature that depends on concrete permeability since it manages the grade of penetration that aggressive chemicals can reach, and it also controls water movement at some point in freezing and heating.⁷ From this point, limiting water penetration through concrete surface will improve its resistance to spalling, safeguard the embedded steel from corrosion, and suppress deterioration.⁸ One of the methods commonly used to enhance concrete defense and expand its service life is surface protection materials. This treatment reduces the risk of chloride ingress and water penetration by diffusion and capillary absorption.⁹

Researchers have focused intensely on coating materials and pore blockers, in their studies, in the last decade.^{10–13} Although they form an impervious barrier on the surface of concrete, they lock moisture inside the concrete, if they were used in wet conditions, leading to continuous degradation of their protective layer, by the vapor formed inside concrete and internal vapor pressure.⁹ Since the mid-1980s, in Great Britain, crystallizing materials were used to manage moisture movement and access of chloride ions that instigate steel corrosion.¹⁴ These materials are well-known polymers which are distinguished from other surface treatments, as they do not react, chemically, with silicates found in concrete, and they make concrete surface repellent to water and permeable to vapor at the same time.^{9,15} Pazderka¹⁶ tested two different crystalline materials in concrete to evaluate their performance against water permeability. One material was applied on the surface of matured concrete and the other material was integrated into the concrete mix at mixing stage. The crystalline coating showed higher efficacy than the crystalline admixture in reducing water absorption.

Impregnants, which contain silane and siloxane materials, were particularly the most widely used protective materials to treat reinforced concrete in the United Kingdom for many years.¹⁷ Nevertheless, due to the existence of solvent materials in these treatments, it is recommended to avoid using such materials. As a result, there is increasing demand of using water-based silane, silane creams, and crystallization mixtures, especially after their compliance with the British standards for protecting concrete, BS EN 1504-2.^{2,18} Also, the consents conferred by the Highway Authorities in the United Kingdom to use these materials, which springs from the need to drive down environmental deterioration, has encouraged studies to expand in this field.¹⁹

Previous studies have claimed that silane-based protective materials could reach, when applied to concrete surface, a modest penetration, around 10 mm.²⁰ However, manufacturers of other materials have claimed to achieve penetration depths more than 10 mm, as a high part of its silane

material is an active content. One of the previous studies, conducted by one of the authors, has indicated that one of the materials with high active content achieved a penetration depth more than 20 mm.¹⁷

On the other hand, it is necessary to accept the fact that there is a noticeable divergence between the results obtained in laboratory trials, and the onsite conditions that reduce their aimed performance.³ This is, most likely, because of weather conditions and presence of internal moisture in the host structures. These circumstances could be overcome by controlling the applied amount or dosage of the protection materials in the fresh concrete if they do not negatively affect concrete properties. In this study, different proportions of protection material were tested, and their influence on concrete properties was monitored. Additionally, this research is an extension of a previous study conducted by the authors, where one proportion of an aqueous protection material was used and tested under different curing regimes; conventional and adverse conditions.² Results and observations from this study are to support outcomes of that research.

2 | RESEARCH SCOPE AND OBJECTIVES

Reflecting the need of applying protective materials on concrete, this research project investigates the influence of early application of some of the promising protective materials on fresh concrete, mainly mixing them with concrete components at the mixing stage. If successful, this research will eliminate the concern over the depth of penetration of the surface applied impregnation.

The objectives of this study are:

1. To assess the performance of a cementitious crystallizing material when implanted within the concrete mixture at mixing stage.
2. To compare the performance of the protective material, when applied in different proportions at mixing stage and in different forms (powder and liquid), and their effect on strength and water absorption.

An aim regarding the performance of the protective treatment under different curing conditions has been under study by the authors, and earlier research in this regard showed encouraging results.² After all, crystallizing minerals, have a virtue over other types of admixtures, especially silane- and siloxane-based materials, as they are environmentally friendly, and have a better affinity with water. This advantage gave confidence in this study to mix the material with concrete components at mixing stage, instead of applying on the surface of the matured concrete. This material has the feature of repelling water too.

TABLE 1 Approved mix proportions

Component	Quantity (kg m ⁻³)
Cement	479
Water	230
Fine aggregate	625
Coarse aggregate	1,066
Total	2,400
Water/cement ratio	0.48

3 | EXPERIMENTAL WORK

3.1 | Materials

At the time of on-going research with other materials being investigated, first results of this research, regarding strength and water absorption, are related to the protection material LYN-1, a cementitious crystallizing material which is extracted from animals and conforming to the terms of related British Standards.²¹ C40 concrete was used in this research, as the UK standards for concrete testing requires employing the C40 concrete in such tests. Concrete mixes, in this study, are made in compliance with BS 1881-125.²² Some trial mixes were conducted, with the approved mix design shown in Table 1. The water to cement ratio chosen in this study is 0.48. This refers to the nature of the cementitious material used in the current research as a protection material, in contrast to the material used previously which was a water-based aqueous material.² The latter would increase the mix workability.

It is important to mention that the different percentages of the material LYN-1 would be added to the overall

mixture amount, shown in Table 1, without affecting its ingredients proportions.

3.2 | Test specimens and testing

Forty, 100 mm, concrete cubes were produced, 16 for the control mix, 4 cubes treated with 1% admixture, 16 cubes treated with 2% admixture, and 4 treated with 8% admixture. Cubes were cured in a water bath for different periods before tests were carried out.

Figure 1 represents a diagram for the quantity and purpose of the C40 cubes used to study and achieve the formally mentioned objectives. The protective material was applied according to the corresponding manufacturer instructions, in different proportions: 1, 2, and 8%. Cubes have been divided into four groups; each group contains cubes that represent the control mix, and the 1, 2, and 8% treatments, cured in a water bath for 3, 7, 14, and 28 days.

All specimens were subjected to Initial Surface Absorption Test (ISAT) first. The ISAT method was adopted because it is a non-destructive test. Therefore, the same specimens could be tested again for compressive strength. Also, the ISAT test is adaptable, to an extent has the potential for in situ adaptation.²³

Instructions on the test procedures and specimens adjustments are followed as provided in the BS 1881-208.²⁴

3.3 | Procedure

Since the admixture is in the form of solids, it is recommended, by the manufacturer, to stir it properly with water till all the particles of the admixture are fully distributed and

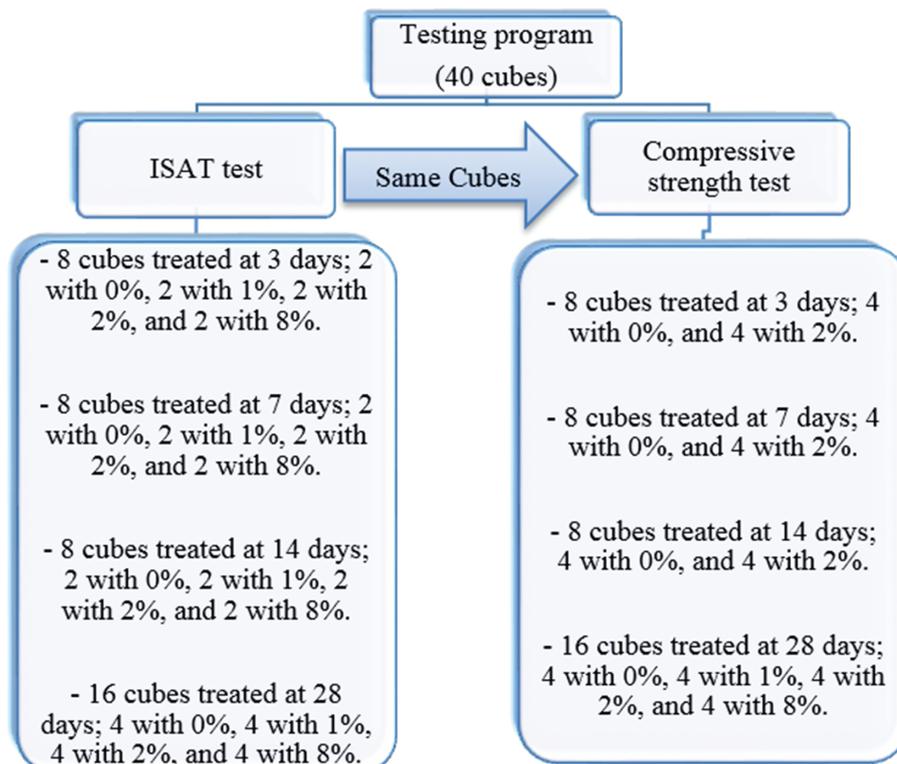


FIGURE 1 Specimens used for testing absorption and compressive strength

TABLE 2 Slump values for treated and control concrete

Mixture type	Slump (mm)	Comments
C40: 0% admixture	40	No cracks appeared
C40: 1% admixture	60	No cracks appeared
C40: 2% admixture	190	No cracks appeared
C40: 8% admixture	210	No cracks appeared

dissolved in water. The admixture is then added with concrete ingredients and concrete cubes are normally produced. The consistency of all mixtures was checked by their slump value as shown in Table 2.

It should be noted that despite the very large slump, mixture with 8% admixture did not show any sign of segregation and/or internal cracks in the matured concrete after 28 days of curing (Figure 2).

All the cubes are left for a duration of 24 hr to ensure that concrete has set and become stiff and ready to be demolded, except for the cubes with 8% admixture content, as they need more time to set, they were demolded after 72 hr. This decision was taken after a previous trial to demold the 8% admixture content cubes after 24 and 48 hr, where concrete found green at these times. This refers to the high workability of concrete treated with 8% admixture where it increases with increasing the dosage of material LYN-1.

All cubes were cured in a water bath at a temperature of 20°C, and ISAT testing was operated at 3, 7, 14, and 28 days, respectively. Before conducting the ISAT test and after removing the cubes from water, at the formally mentioned intervals, they should be placed in the lab at a temperature of 20°C, to let them dry until a constant mass is achieved, then cubes must be placed in a cooling cabinet till there temperature drops 2°C from the room temperature.

After testing the cubes with the ISAT method, same cubes are tested for compressive strength in accordance with BS EN 12390-3 (British Standards²⁵). However, control cubes and cubes with 2% admixture content are the only cubes tested for 3, 7, 14, and 28 days, when subjected to compressive strength test. Only, 1% and 8% admixture content cubes are tested at 28 days. This refers to the fact that, the 1% content is considered a small amount and the 8% is considered a very high amount; representing the two extremes. Admixture content of 2% is considered the most appropriate amount.

4 | RESULTS

4.1 | Concrete absorption of water

The influence of applying different proportions of the material LYN-1 on water absorption for the C40 concrete is outlined in Figure 3a–d, which show the absorption rate for concrete at 10, 30, and 60 min periods and at the end of 3, 7, 14, and 28 days interval.

Concrete absorption for water declines for all specimens, with similar performance for concrete with 1 and 2% material contents. This similarity in performance is obvious at a 60-minute timing at all curing stages, as they reach similar absorption rate. Both have touched absorption rates that range between 0 and 0.01 mL m⁻² s⁻¹ in 14 days duration at 60 min timing, and they achieved their optimum performance before reaching the 28 days period. Specimens with 8% material content reached 0.05 mL m⁻² s⁻¹ absorption rate at 60 min timing in the 14 days duration and its influence has persisted inadequately until it reached 0.03 mL m⁻² s⁻¹ absorption rate, which makes it less effective than the formally mentioned proportions. To make it more clear, the higher absorption rates for specimens treated with 8% admixture compared to those treated with 1 and 2% admixtures, refer to the higher dosage of 8% admixture that was reflected in higher workability values. This resulted in retaining some air voids in concrete and forming greater void spaces than those formed in concrete treated with lower admixtures dosages.

On the other hand, the control mix specimens showed a considerable absorption rate and the highest among all samples. In their best condition, they achieved 0.17 mL m⁻² s⁻¹ absorption rate at 60 min timing during 28 days interval. This, clearly, demonstrates the effectiveness of the treatment, and its positive influence on protecting concrete against water ingress.

4.2 | Concrete compressive strength

Results for 3, 7, 14, and 28 days compressive strength (MPa) test for control and treated concrete cubes are shown in Table 3. However, 1 and 8% admixture content cubes are tested only on 28 days, and strength values at 3, 7, and 14 days for these contents are not included in the table. Considering only the 2% treatment regime, for all the periods, refers to the fact that this proportion represents an average value between two extreme values. Although, both the 1 and 8% treatments were tested under the 28 days compressive strength. Treatment with 2% admixture achieved

**FIGURE 2** Concrete cube treated with 8% LYN-1 admixture without segregation

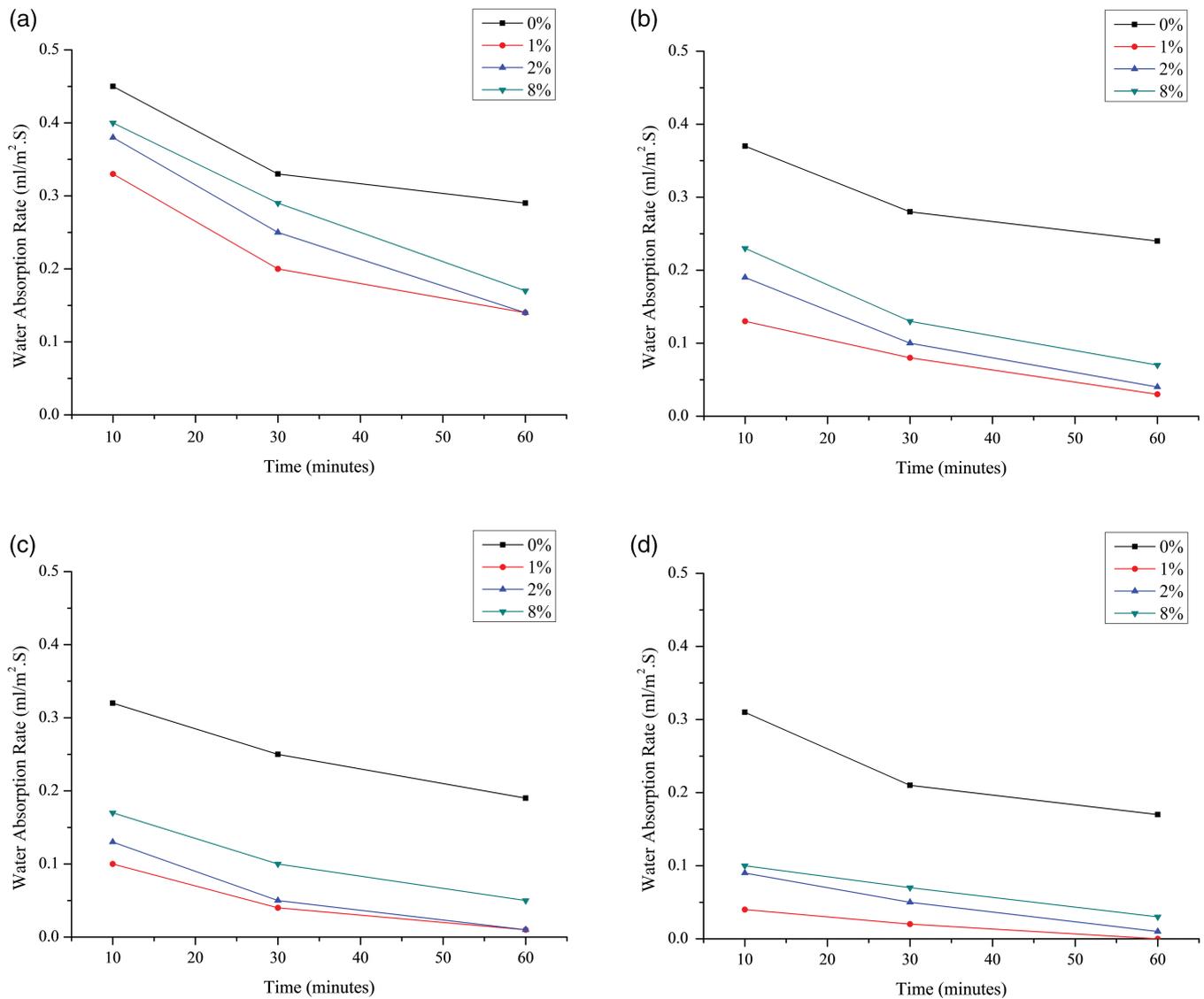


FIGURE 3 Concrete absorption rates during the first hour of applying different ratios of material LYN-1 at: (a) 3 days, (b) 7 days, (c) 14 days, and (d) 28 days

the most advantageous performance between treated cubes on 28 days, regarding strength. However, the 28-day strength values of all treated cubes have values, slightly, higher than 30 MPa. Furthermore, all treated cubes experienced some strength loss at 28-day. This reduction in strength was observed, possibly due to excess w/c ratio. The addition of the admixture leads to excessive workability indicated by the complete slump. Also, the change in treated concrete strengths relative to the control is shown in the table.

Concrete treated with a concentration of 1% of material LYN-1 achieved compressive strength of 75% of the corresponding control strength. This percent was achieved in the control mix in the period that falls between 3 and 7 days. Moreover, concrete treated with 8% material content achieved 79% of the control strength. Finally, 2% content of material LYN-1 gave the concrete 81% of control strength on 28 day, which makes the 2% treatment regime the most suitable one in terms of strength, regardless the loss in strength. In other words, treating concrete with 2%

TABLE 3 The effect of applying material LYN-1 on concrete strength

Crystalline admixture content (%)	Compressive strength (MPa)				Changes in strength compared to control at 28 day
	3 days	7 days	14 days	28 days	
0	28.2	37.2	39.6	41.6	0%
1	na	na	na	31.4	-25%
2	16.2	24.5	29.8	33.6	-19%
8	na	na	na	32.8	-21%

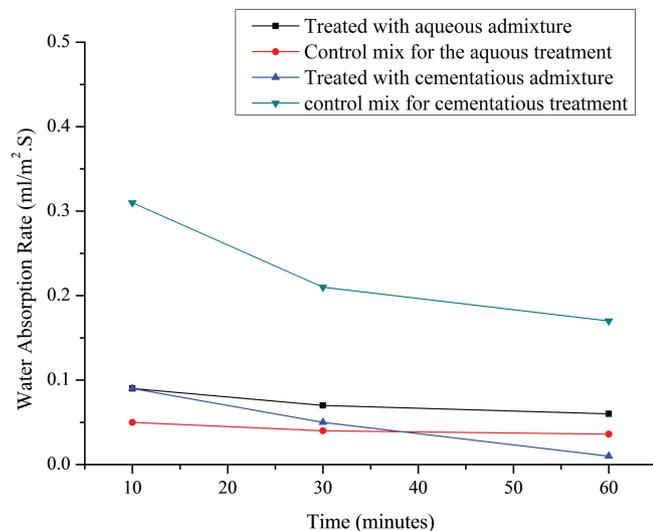


FIGURE 4 Water absorption rates for C40 concrete under conventional curing conditions

admixture exhibited the least change in strength compared to control, which was about 19% drop from the achieved control strength value. On the other hand, adding 2% concentrate to concrete achieved an increase in the strength of 37% during an interval of 21 days (from day 7 to day 28), and this increase was about 11% in the case of the control mix.

5 | PERFORMANCE OF DIFFERENT ADMIXTURES

Figure 4 outlines the ISAT sorptivity for concrete with 2% aqueous admixture, and cementitious admixture along with control mix corresponding to each case, at 28 days of normal curing regimes. It should be noted that the w/c ratio for this mixture was slightly reduced to 0.46, to accommodate the extra water in the aqueous hydrophobic mix.

Treated concrete with 2% aqueous admixture, under normal curing conditions, exhibited water absorption levels higher than its corresponding control mix, contrary to treated specimens with cementitious material, where cubes absorbed less water at 28 days for the same applied material content. This would give the cementitious material advantage over aqueous material especially knowing that sorptivity for the water-based material reached about $0.06 \text{ mL m}^{-2} \text{ s}^{-1}$ after the first hour at 28 days, which is about six times the sorptivity of the cementitious material for the same period. When referring to compressive

strength, Table 4 outlines the 7 and 28 days compressive strengths for both, treated cubes with the 2% aqueous material content and untreated cubes, under normal curing regime.² Also, the preserved strength value is shown in the table, as a ratio between the change in strength values for treated and untreated control cubes to that control strength value.

Favorable curing conditions affected concrete positively, where it has gained more strength instead of losing part of it as what happened in the current case of adding the cementitious material. However, preserved strength was kind of modest as it safeguarded only 5% of strength relative to its control mix.

It is noteworthy to mention that in a study conducted by Pazderka and Hájková,²⁶ a crystallizing material was integrated into the concrete mix, at mixing stage, with an amount of 2% of the cement and cured under favorable conditions. Results showed that a reduction in water absorption, for treated concrete, was observed after 12 days from casting the concrete, with efficiency exceeded 20% compared to control. On the other hand, a small drop of 1.6% in the compressive strength has resulted in treated samples when compared to control.

6 | MICROSTRUCTURE INVESTIGATION

The formation and the distribution of the crystals of the added admixture were observed under the scanning electron microscope (SEM) with $\times 500$ and $\times 5,000$ magnifications, for 1 day, 3 and 7 days period. Crystals can be easily recognized from Figure 5a–f, which show the increased growth and distribution of crystals during the time.

Treated concrete samples were also tested and analyzed under the X-ray diffractometer (XRD) instrument to know the size of crystals formed. Samples that were 28-day-old were investigated under the XRD to ensure full growth and distribution of crystals. Results showed that the minimum size of the formed crystals was 95 nm and the maximum size was 200 nm. Comparing crystal sizes with the pore size of concrete, LYN-1 particles were smaller than macropores, the entrained air voids, the entrapped air voids, and the pre-existing microcracks of a normal concrete, which means that LYN-1 could integrate easily within any concrete mix filling the most of the voids and not allowing new voids to be formed.

TABLE 4 Strength values for treated and untreated concrete with different types of materials under favorable curing conditions

Protection material	Material content	7-day compressive strength (MPa)	28-day compressive strength (MPa)	Preserved strength
Liquid admixture	Control	22.7	39.9	+5%
	2%	24.1	42.0	
Cementitious admixture	Control	37.2	41.6	-24%
	2%	24.5	33.6	

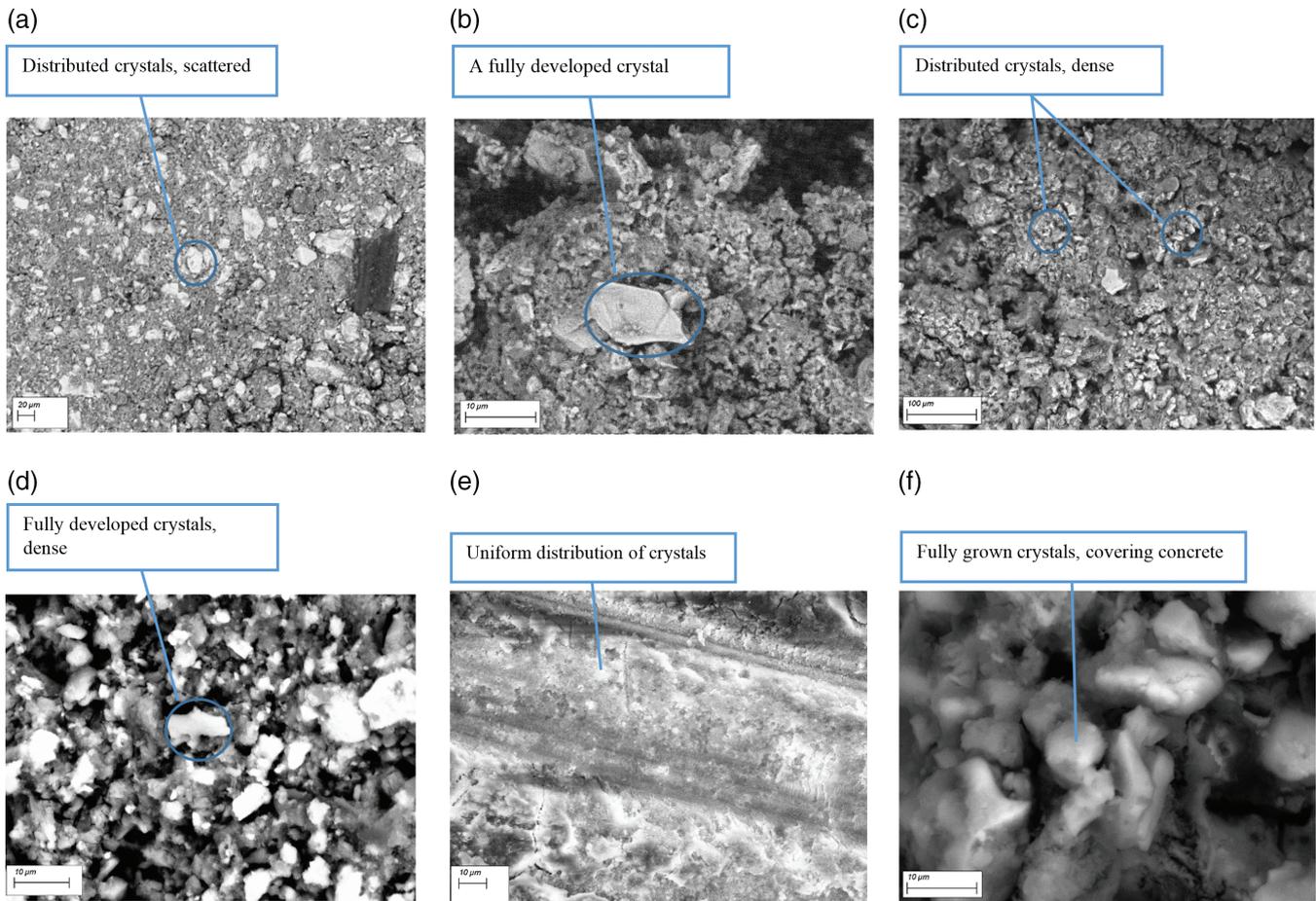


FIGURE 5 Scanning electron microscope (SEM) analysis for treated concrete after (a) 1 day under $\times 500$ magnification, (b) 1 day under $\times 5,000$ magnification, (c) 3 days under $\times 500$ magnification, (d) 3 days under $\times 5,000$ magnification, (e) 7 days under $\times 500$ magnification, and (f) 7 days under $\times 5,000$ magnification

7 | CONCLUSIONS

Significant conclusions and observations from the research are:

1. The application of the crystallizing protection materials with concrete mixture has a reducing influence on concrete permeability, where the absorption of water has decreased dramatically. All admixtures contents reduced concrete permeability but with different effectiveness.
2. Two percent addition of the aqueous material marginally enhanced compressive strength, indicating better dispersion of aqueous material in the mixture. Despite the moderate reduction in strength due to the addition of cementitious admixture, the rate of strength gain was significantly higher. For example, 2% cementitious admixture to concrete gave a boost to strength values from day 7 to day 28, where an increase of 37% in strength was achieved in that period. Whereas untreated concrete attained an increase of 11% during the same period.
3. Overall, treating concrete with a concentration of 2% admixture gave optimum performance in terms of permeability, and it reduced water absorption rate to a

nearly of 0. Eighty-one percent of the 28-day control strength was achieved by adding 2% of material LYN-1 to the concrete mix, which makes this proportion of admixture the most effective one in the matter of strength. This material has also demonstrated better protection than aqueous hydrophobic admixture.

4. Regardless of increasing the workability of concrete when adding a high percentage (8%) of hydrophobic admixture, neither segregation nor thermal cracking has taken place.

ACKNOWLEDGMENT

Authors highly appreciate the great help and support provided by Mr Neil Macfadyen, Technician at the Civil Engineering Research Centre, Brunel University London. Also, the support of the Experimental Technique Centre (ETC) at Brunel University is highly appreciated. Finally, M.J.A. would like to pay special thanks to Mu'tah University, Jordan, for providing financial support.

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How to cite this article: Al-Kheetan MJ, Rahman MM, Chamberlain DA. Development of hydrophobic concrete by adding dual-crystalline admixture at mixing stage. *Structural Concrete*. 2018;19:1504–1511. <https://doi.org/10.1002/suco.201700254>