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A novel approach of introducing crystalline protection material and curing agent in fresh concrete for enhancing hydrophobicity



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HIGHLIGHTS

- Enhance the performance of concrete under harsh curing conditions.
- Treating fresh concrete with crystallising material and curing agents.
- Tests include water absorption, compressive strength, and microscopic analysis.
- Crystallising material followed by wax-based curing agent improved the strength.
- Applying liquid curing agent with a high w/c ratio increased internal cracks.

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ABSTRACT

A new line of research to enhance the performance of concrete under adverse (harsh) and normal (air cured) curing conditions is presented. A crystallising hydrophobic admixture and curing agents were added to fresh concrete to improve its resistance against severe environmental conditions. A two-stage approach was pursued by adding the crystallising admixture to fresh concrete followed by curing agents, in a wax and liquid forms, in a separate application process, followed by exposing concrete to normal and adverse curing conditions. Results obtained suggests that protecting concrete with the crystallising admixture followed by applying wax based curing agent improves concrete strength and its resistance to water ingress than concrete cured with the liquid curing agent. When following the crystallising-wax treating system under adverse curing conditions, a more conserved strength was noticed compared to that produced by the crystallising-liquid system. Using the liquid curing agent in concrete with high water to cement ratio (w/c) has increased the cracks in the internal structure, while water permeability has decreased, either under normal curing conditions or adverse conditions. Following this protection-curing system in industry would resolve the problem of applying protection on wet surfaces and increase concrete's resistance to deterioration. A microscopic study of the crystallising material was attained with a Scanning Electron Microscope (SEM) to check crystal growth with time.

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1. Introduction

In the UK, there are more than 61,000 highways and bridges constructed in reinforcement concrete [1–3]. Although these structures were designed and built to withstand deterioration, they have proven to demand substantial repair and maintenance, mostly because climatic conditions and winter salting [4,5]. The presence of excess moisture in the concrete leads to winter cycles of freezing and thawing that cause severe damage [5].

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adequate dosage and good penetration in concrete requires unrealistically low moisture content when they are applied to concrete [7]. This refers to pores occupied by water at time of application; when pores are saturated, the penetration of such materials would be difficult due to repellency and lack of available place. Finally, the existence of excess moisture in concrete at the time of applying these materials reduces their performance and efficiency against chloride penetration. Accordingly, alternative hydrophobic materials, that comply with the British Standard BS EN 1504-2 [12], were brought to light to cover the disadvantages of silane and siloxane solvent-based materials. In contrast, water-based materials and others with crystallising mineral components are aimed to function well in the presence of high moisture content in concrete.

Limited researches are available of Crystallising materials. Cementitious crystallising coatings were applied to concrete structures, which are in a direct contact with water like swimming pools and water tanks, in order to protect them from water ingress [13]. This type of coating materials has significantly improved concrete resistance to absorb water, in addition to its innocuous effect on the quality of water. Reiterman and Pazderka [14] tested the efficacy of another type of crystallising coatings, in terms of water absorption and its ability to protect concrete at a thorough depth. They were successful in reducing the absorbed amount of water at depths between 180 and 190 mm from the surface compared to untreated concrete. In a following research, Pazderka and Hajkova [15] studied the effect of adding a crystallising admixture to concrete, at the mixing stage, on reducing its water permeability. Results showed an early interaction between concrete and the added material, where a full waterproofing effect was reached after 12 days of casting. The only dilemma in this kind of admixtures is its negative effect on the compressive strength of concrete which was observed, on a small-scale and negligible level, in the aforementioned research.

In this study, a crystallisation hydrophobic material was applied to fresh concrete, followed by the application of a permanent curing compound. The efficiency of this novel blend is tested in terms of strength and permeability under adverse (harsh) curing regimes. Interest in water activated materials, such as crystallising solutions, acknowledges the improbable occurrence of those ideal low-moisture conditions that favor the established hydrophobic treatments.

The main approach followed by the authors is to protect and extend the service life of concrete by the application of a crystallising hydrophobic solution to the fresh concrete, followed by a curing compound. The hypothesis is that treating concrete with these materials will increase resistance against cracking whilst maintaining strength by controlling excessive hydration during the curing process. Hydrophobic treatments were considered, earlier, by researchers to provide additional protection to reinforcement embedded within concrete from aggressive materials, especially for concrete with high permeability [8,16].

With regard to curing processes and materials, many curing methods and compounds are available and applied in industry to promote hydration and provide protection to concrete [17]. These compounds function as temporary or permanent protection compounds depending on the nature of materials used to produce them. So called permanent curing agents are intended to deliver long duration protection. The influence of these different curing compounds and techniques has been always under study to reduce the negative effect of adverse curing conditions on concrete, as they restrain water movement through concrete pores during its early life [18,19]. Nevertheless, results regarding their effect on concrete properties under adverse conditions are not always beneficial [19].

This paper investigates the early application of a crystallising material followed by a curing compound on fresh concrete and

their influence on its properties under adverse curing conditions. It is an extension of an earlier study conducted by the authors [3] to assess the influence of adverse and normal curing conditions on the performance of the same protective material used here with a wax-based curing agent. The wax-based curing agent is formed from a stable mix of different types of waxes in water.

The objectives of this research are:

- 1) To determine the influence of a successive application of a crystallising protection material followed by a water-based curing compound to fresh concrete, in terms of strength, water absorption, and permeability, when subjected to favorable (normal) and adverse (harsh) curing regimes. The adverse curing consisted of constant high speed air circulation on the specimens to accelerate hydration process.
- 2) To study the effect of applying the protection-curing combination on concrete in respect of potential crack's formation.
- 3) To assess the performance of the protection material and relate its performance to previous research, but with different associated variables; different curing agent and water to cement ratio.

Early application of protection materials to fresh concrete in the presence of a high moisture content has been under investigation by the authors, with promising results [2,3]. Results from a previous study [3] are used to compare outcomes.

2. Experimental work

2.1. Specimen manufacture

C40 concrete was chosen for this study because this type of concrete is generally used for pavement construction and in other structural applications [2,20]. The design mix, as shown in Table 1, was made in agreement with the British Standard BS 1881-125 [21]. A w/c ratio of 0.48 was used which was marginally higher than the previous studies [2,3]. The high-water cement ratio resulted in a slump 65 mm, but no segregation was noted in the compacted specimens.

2.2. Surface applied protection material

The protective material used in this research is a patented material, KLD-1 is an aqueous crystallising waterproofing material applied to concrete with an amount of 2% of cement mass, followed by a wax-based and water-based curing agent. Both materials conform to BS EN 1504-2 [12], and have been tested under the first objective to assess their performance against water absorption. KLD-1 as a dual functioning system works to absorb water to form crystals that reduce moisture movement by closing concrete capillaries, and then form another type of crystals that repel excess water and prevent its penetration through concrete pores.

Table 1
Adopted mixture proportions.

Component	Quantity (kg/m ³)
Cement (CEM II/32.5 N; Sulphates < 3.5%, Chlorides < 0.10%, and initial setting time around 1.25 h)	480
Water	230
Fine aggregate	650
Coarse aggregate	1040
Total	2400
Water/Cement ratio	0.48

Table 2
Mixture coding and description.

Code	Protection material	Curing Agent	Test type
C40KLD-L	KLD-1	Liquid form	ISAT, Permeability, Compressive strength
C40KLD-W	KLD-1	Wax-based	ISAT, Compressive strength (details in ref (3))

The applied curing agent is a commercially available compound that preserves the mixture from outer temperature and offers a water impervious membrane to preserve most of hydration water in the concrete. Thus, this combination is a promising treatment that will give a long-lasting solution for moisture associated problems. The coding and description of the tested mixtures are given in Table 2.

It should be noted that C40KLD-W has been thoroughly investigated in a parallel study [3] and results are compared with C40KLD-L mixtures in this research.

2.3. Material application procedure

Two and half hours after casting concrete in the molds, all cubes were demolded [3] and then KLD-1 was sprayed uniformly in all faces as per the manufacturer instructions. Curing agent was brushed over cubes' surfaces after 1 h of applying the waterproofing. This arrangement allowed KLD-1 to dry on concrete surface before applying the curing agent. Fig. 1a and 1b show, respectively, concrete inside the mold after casting and after removing the mold.

In total 60 cubes with a combination of 100 mm and 150 mm sizes were manufactured. They were cured under two different conditions:

- Condition 1: 30 cubes are placed in laboratory with controlled temperature of 22 °C. This curing condition is referred to, in this research, as normal or favorable curing condition.
- Condition 2: 30 cubes are exposed to forced air generated by electric fans. This curing condition is referred to as adverse or harsh curing condition.

Out of 30 favorably cured cubes, 15 of them were treated by 2% admixture, and 15 were used as a control mix. The same applies to adversely cured cubes; 15 were treated by 2% admixture, and 15 were used as a control mix. Fig. 2 shows the favorable and the adverse curing environment.



(a)



(b)

Fig. 1. C40 concrete in its early age (a) In 150 mm molds (top row) and 100 mm molds (bottom rows), and (b) extracted from the mold.

1.4. Test specifications

All 100 mm cubes were subjected to Initial Surface Absorption Test (ISAT) and compressive strength test at 7, 14, 21 and 28 days of curing, whilst 150 mm cubes were used for permeability test after 28 days of curing. Fig. 3 presents a summary of test specifications. The compressive strength test was operated by following the instructions in BS EN 12390-3:2009 [22], initial Surface Absorption Test was done in according to BS EN 1881-208 [23,24], and the permeability under pressure test was performed in accordance with BS EN 12390-8 [25].

After permeability test, specimens were split into half and depth of penetrated water was measured, as shown in Fig. 4.

It is noteworthy to mention that similar testing procedure were carried out by Reiterman and Pazerka [14] to assess water absorption through concrete treated with a crystallising material. However, they have applied the protection on matured concrete cubes before they slice each cube into prisms with different orien-



Fig. 2. Treated concrete cubes cured under favorable and adverse conditions.

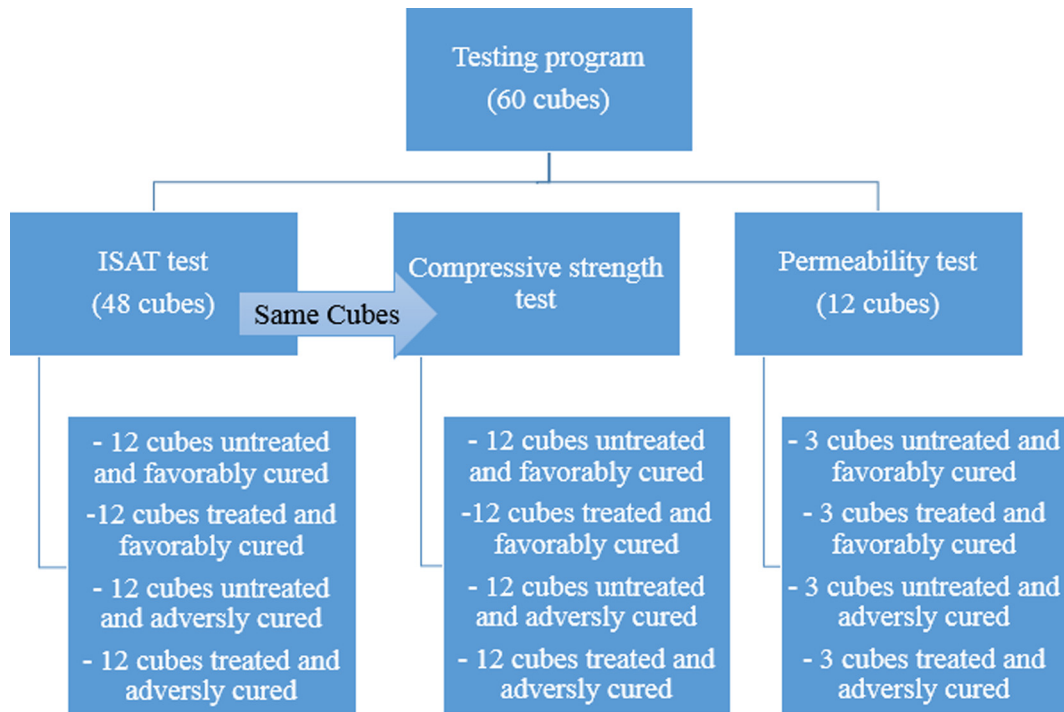


Fig. 3. Test protocol.

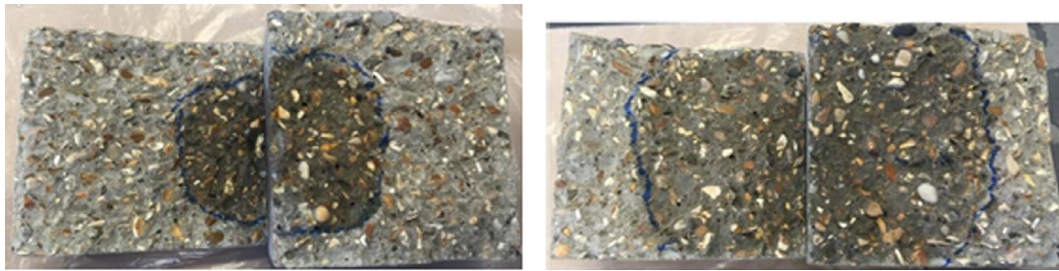


Fig. 4. Water penetration into concrete cubes.

tations, and water absorption was evaluated for each resulted section.

3. Results and discussion

3.1. Microscopic study

The crystallising material KLD-1 was observed under the Scanning Electronic Microscope (SEM) with 500X and 5000X magnifications, to inspect the formation of the crystals and their development with time during a continuous 3 days. Current results obtained from SEM analysis concentrates on investigating the creation of the crystals with time rather than the size and the structure of crystals.

Fig. 5a–d outline the expansion of the crystals under 500X magnification, during the first three days after treatment. It is clear from the figures that crystals are covering wider areas with progressing time, until the whole surface of concrete is fully covered with the crystals. Fig. 5d shows a larger scale capture of the crystals after treating concrete in 3 days and under 5000X magnification. It is also clear from the figures that the gap between the crystals gradually filled up with time.

3.2. Water absorption

Water absorption for C40KLD-L was examined at 7, 14, 21 and 28 days by using the ISAT method, for treated and control mixes, at favorably and adverse curing conditions. Results of this test are outlined in Fig. 6a–d.

All samples showed a declination in water absorption but with different performance. After 28 days of curing, adversely cured concrete singly treated with the curing agent or treated with curing agent followed by KLD-1 has shown the highest absorption rate for water. Both treated and untreated specimens, under this regime, possessed similar performance after one hour of curing, with an absorption rate of $0.60 \text{ ml/m}^2 \text{ s}$. However, favorably cured specimens showed higher performance than those adversely cured. They absorbed less water during the first hour of the test, especially the control mix, which was cured with the water-based curing agent without applying the hydrophobic treatment. Treated cubes absorbed $0.37 \text{ ml/m}^2 \text{ s}$ and untreated ones gave the optimum performance with an absorption rate of $0.09 \text{ ml/m}^2 \text{ s}$. Also, it is important to note that concrete under adverse curing conditions at all curing intervals, starting from day 3 and ending at day 28, had absorption rate values that are close to each other, ranging between 1.38 and $1.75 \text{ ml/m}^2 \text{ s}$. These results support

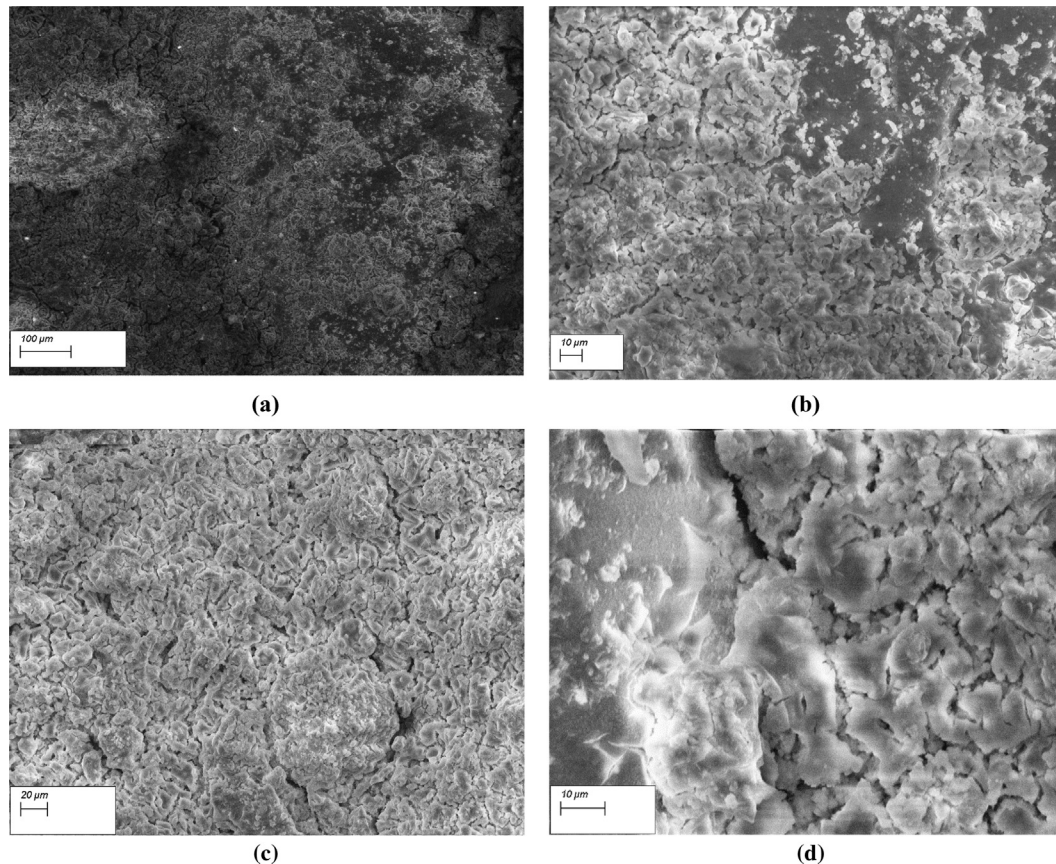


Fig. 5. Crystal formation and development with time as observed under SEM after: (a) 1 day of treatment (500 \times), (b) 2 days of treatment (500 \times), (c) 3 days of treatment (500 \times), and (d) 3 days of treatment (5000 \times).

the conclusion that adverse conditions are highly demanding and increase concrete permeability for water.

An anomaly in results obtained at 7, 14 and 21 days curing periods for untreated concrete under adverse conditions could be spotted in Fig. 6a, b and c respectively. At the period between 7 and 14 days there would be a lot of water available, so hydration will be fast during that period. However, in the presence of harsh environment and lack of protection, the hydration process will be uncontrolled, which will create micro-cracks in the specimens. As a result, these aforementioned variations in ISAT outcomes are resulted from the uncontrolled hydration conditions. In addition, the BS EN 1881–208 mentions that ISAT should be performed on concrete when it has a constant mass (0.1% weight loss in 24 h), which will be unavailable during the 7–14 days period, and this makes the results in that period unreliable to some extent. It is noteworthy to mention that ISAT was performed on the same cubes and on the same sides of the specimens during the 7, 14, 21, and 28 days period, and all the cubes were circulated regularly so all the sides will have the same conditions.

3.3. Compressive strength

Results for compressive strength testing after 7, 14, 21 and 28 days, for treated and control concrete specimens, under favorable and adverse curing conditions are shown in Fig. 7.

Comparing each treated case with its corresponding untreated mix indicates a significant strength loss, especially in the case of favorable curing condition, where treated concrete achieved a 32% drop in strength compared to untreated cubes. In the case of adverse curing conditions, strength loss was, moderately, less sev-

ere than adverse curing conditions. Treated adversely cured specimens exhibited a loss in strength of 17% from their corresponding untreated specimens, which is about half the loss exhibited by concrete under normal curing regimes. The applied protective material uses water to form crystals inside concrete pores; this water is part of the water already used for hydration. Adverse conditions make the situation worse by very fast drying of water in concrete, means less water is available for hydration, which will be reflected on the strength of concrete. This could be noticed in the higher compressive strength of favorable cured concrete than adverse cured concrete. On the other hand, it could be noticed that the protective material has the ability to increase the level of defense when conditions affecting concrete becomes worse than normal, which was shown in Fig. 7; under harsh conditions, the presence of KLD-1 crystals in treated concrete worked on reducing strength loss (compared to its corresponding untreated concrete), which resulted from the uncontrolled hydration. Moreover, early treatment with KLD-1 followed by the water-based curing compound, achieved an increase in concrete strength of 43% for the period from day 7 to day 28, and this increase was about 36% in the case of its corresponding untreated mix.

3.4. Permeability

This test was conducted under 5 bar pressures to examine water penetration through concrete. The average 28-day concrete permeability values under all curing conditions are shown in Table 3.

Untreated adversely cured concrete achieved an average water absorption depth of 59 mm, and the control mix under favorable

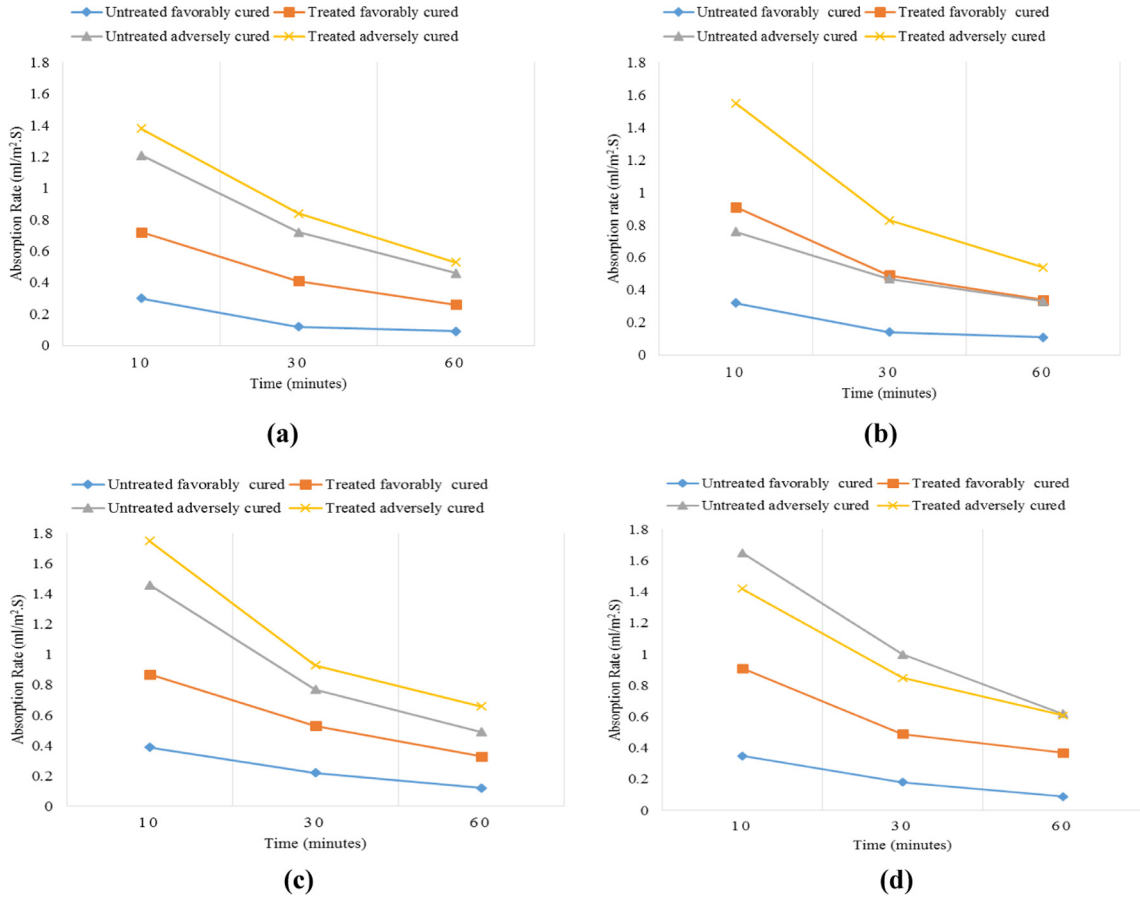


Fig. 6. Concrete sorptivity for different treatment and curing regimes at: (a) 7 days, (b) 14 days, (c) 21 days and (d) 28 days.

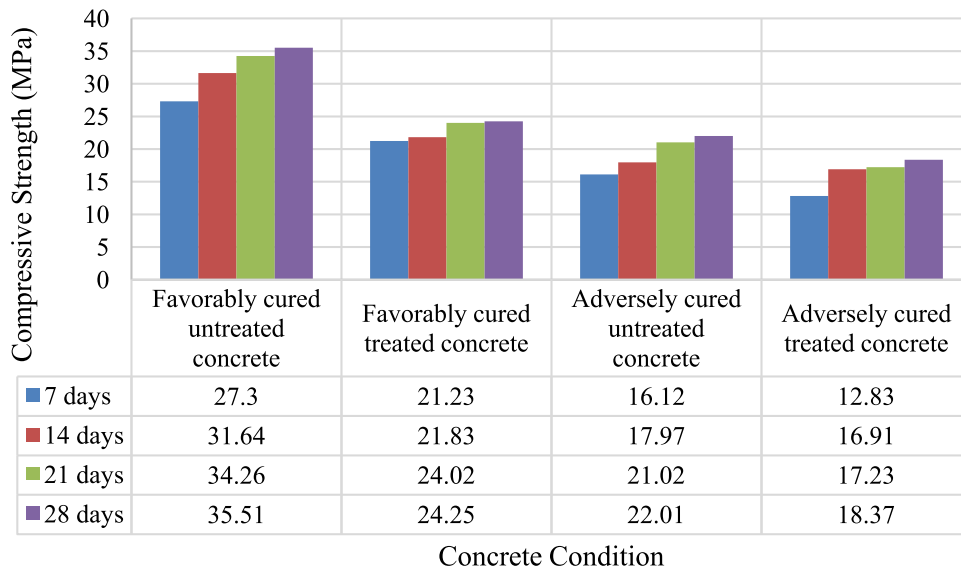


Fig. 7. Compressive strength values for adopted concrete mix under different curing conditions.

curing regime achieved a water penetration depth of 37 mm. In the case of treated favorably and adversely cured concrete no permeability values were found. This is because treated favorably cured concrete starts leaking water after 10 min of initiating the test, and treated adversely cured concrete starts leaking immediately after initiating the test. This was caused by the same process explained in Section 3.3.

3.5. Crack formation

To investigate the presence of cracks and, at the same time, the reason behind the leakage in treated cubes after operating the permeability test, concrete cubes were soaked in water contains blue dye for 48 h. Fig. 8a and 8b show concrete cubes soaked in dyed-water and a concrete cube after removing it from the

Table 3
Permeability outcomes for treated and untreated concrete under favorable and adverse curing conditions.

Case	28-day Permeability (mm)
Untreated favorably cured concrete	37
Treated favorably cured concrete	na
Untreated adversely cured concrete	59
Treated adversely cured concrete	na

dyed-water. After removing specimens from dyed-water, they were placed in the lab for further 48 h to allow excess water to be removed from the surface and evaluate the presence of cracks.

After splitting concrete cubes to two pieces it was noticed that the blue dye has excessively penetrated inside concrete, which proves that concrete contains cracks and due to the existence of these cracks, water applied under 5 bar pressure leaks from concrete.

3.6. Performance of different curing agents

KLD-1 was tested in a previous research [3] associated with a wax-based curing agent and concrete with w/c ratio of 0.46. In the current study, the same protective material was used but with

a water-based curing compound and concrete with w/c ratio of 0.48.

Fig. 9 illustrates water absorption rates for concrete treated with the same protective material, KLD-1, along with wax-based curing agent, and water-based curing agent after 28 days of favorable and adverse curing conditions.

Concrete cured with the wax-based curing agent, either treated with KLD-1 or not, has achieved better performance, in general, than concrete cured with water-based concrete. Under adverse curing conditions, treatment with KLD-1 followed by wax-based curing agent helped in reducing water absorption in concrete significantly when comparing it to its control, contrary to cubes treated with KLD-1 followed by a water-based curing agent, under same conditions, as they exhibited absorption rate like their control mix after 60 min of testing.

Cubes under favorable curing conditions and cured with the water-based compound did not perform much better than those in the adverse regime. Concrete treated with KLD-1 followed by a water-based curing agent under this regime exhibited a similar performance to untreated concrete cured with a wax-based compound under adverse conditions, as they have achieved water absorption rate close to 0.38 ml/m² s after 60 min of testing. Treated concrete followed by a wax-based curing compound under normal curing conditions achieved the best performance among



Fig. 8. C40 concrete: (a) soaked inside dyed-water and (b) absorbed dye after soaking because of cracks presence.

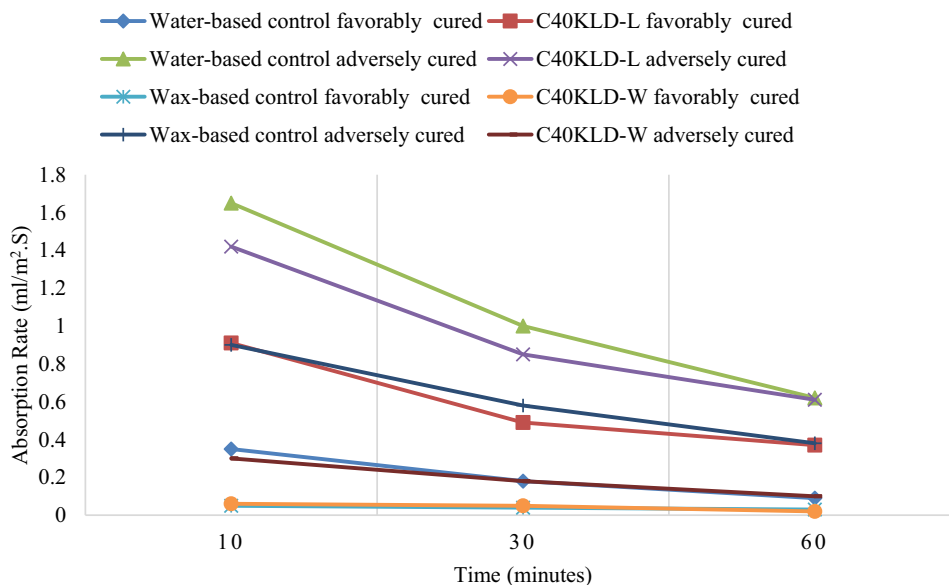


Fig. 9. Concrete sorptivity at 28 days under favorable and adverse curing conditions cured with water-based and wax-based compounds.

Table 4

Strength for treated and untreated concrete cured with water-based or wax-based agent under normal and adverse curing regimes.

Curing agent	Condition	7-day compressive strength (MPa)	28-day compressive strength (MPa)	Preserved strength
Water-based	Favorably cured untreated	27.30	35.51	–32%
	Favorably cured treated	21.23	24.25	
	Adversely cured untreated	16.12	22.01	
	Adversely cured treated	12.83	18.37	
Wax-based	Favorably cured untreated	23.2	40.7	+9%
	Favorably cured treated	22.1	44.2	
	Adversely cured untreated	15.4	17.1	
	Adversely cured treated	22.1	29.7	

all treated and untreated concrete samples with absorption rate around 0.1 ml/m² s. This confirms that using the water-based curing agent had a negative effect on concrete permeability especially when applying it to concrete with high w/c ratio, as it was applied in this study.

Concerning compressive strength, Table 4 summarizes the 7 and 28-day strength values for concrete cured with either curing agents in favorable and adverse environments. Also, the gained strength or lost strength is shown as a percentage for each case.

Curing concrete with a wax-based curing compound has affected concrete strength positively, as strength has increased for concrete cured with this compound, under both adverse and favorable curing conditions, and for cubes either treated or untreated with KLD-1. However, curing concrete with a water-based agent affected strength negatively under all treatment conditions and curing regimes. This gives the KLD-1 and wax-based protection system a virtue over the KLD-1 and water-based system. In the case of adverse curing conditions, wax-based curing agent succeeded in preserving the strength of treated concrete and increasing its value by 74%, in contrast to concrete cured with the water-based agent which suffered from a strength loss of 17%. The same observation can be made for the favorably cured concrete, where treated concrete cured with the wax-based compound had an increase in strength of 9% compared to untreated concrete. This is contrary to the same treated concrete but cured with a water-based compound that lost 17% of its strength compared to untreated concrete.

4. Conclusions

Important conclusions from the study are;

- 1) Applying the crystallising waterproofing material followed by curing agents on concrete has reduced concrete permeability, where water absorption rate values for all cubes have, generally, decreased but with different efficacy between water-based and wax-based curing compounds.
- 2) A significant strength loss was observed in concrete treated with the crystallising material and cured with the water-based compound, either conditioned under normal (favorable) curing conditions or under harsh (adverse) conditions. However, the loss in strength in the case of adverse conditions was less severe than normal curing conditions.
- 3) Concrete mix cured with water-based compound has suffered from a strength loss between 17 and 32% relative to its control mix. On the contrary, wax-based curing compound when accompanied with the waterproofing material enhanced strength levels. Regardless of strength loss as a result of treating concrete with KLD-1 followed by water-based curing agent, the degree of strength improvement was moderately high. For instance, treated concrete under adverse curing conditions and cured with the water-based compound enhanced strength levels from day 7 to day 28

with an increase of 43% in strength. While concrete under the same curing regime but cured with a wax-based compound achieved 34% increase in strength for the same period.

- 4) Treating concrete with KLD-1 followed by water-based curing agent had a destructive effect, as treated cubes have suffered from leakage when tested for permeability under pressure. Significant cracks were found in cubes treated under this regime.
- 5) Increasing the w/c ratio in concrete mix would have a negative effect when using a water-based curing agent. This refers to increasing water content after applying the water-based curing agent. This was not an issue when a wax-based curing agent was used.

4. Future work

Research regarding concrete waterproofing under adverse curing conditions is ongoing, with using different protective admixtures and curing agents. Protecting concrete from chloride attacks, using different admixtures, is also under study.

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