Optimum Mix Design for Internally Integrated Concrete with Crystallising Protective Material

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ABSTRACT

In this research, a Silica-based crystallising protective material was integrated into a fresh concrete mix to evaluate its efficacy in reducing water absorption while preserving the compressive strength level of the mixture. An optimum concrete mix design was determined, by producing several concrete mixes with different water to cement ratios (w/c) of 0.32, 0.37, 0.40, and 0.46, and treated with 2% and 4% of the crystallising admixture. Water absorption and the mechanical properties of the treated and control mixes were measured, using the Initial Surface Absorption Test (ISAT) and the compressive strength and the flexural strength tests respectively. Results showed that it is possible to obtain a water-resistant concrete without compromising its compressive strength if the right w/c ratio was used and the proper dosage of the crystallising material was added. In addition, results revealed that treatment is beneficial only in the case of producing concrete with a low w/c ratios of 0.32 and 0.37 and treated with the crystallising material. The compressive strength can increase up to 42% and with a significant drop in water absorption reaches 65%. Treated concrete was analysed thoroughly under the Scanning Electron Microscope (SEM) and X-Ray Diffraction instrument (XRD) to show the development of crystals with time and their interaction with the concrete mix.

Keywords: Fresh concrete, Concrete pavement, Crystallising material, Morphology, Compressive Strength, Flexural Strength, Water absorption, Protection

Introduction

In recent years, concrete is regaining importance in infrastructure engineering for being more energy efficient material as it consumes less fuel, its life is longer than other materials, and it needs fewer maintenance works (Taylor and Patten 2006). Large-scale use of concrete in infrastructure engineering is to build bridges and concrete pavement for roads, airports, ports and in industrial ground floors. In the United Kingdom alone there are more than 61,000 highways and road bridges, most of them are made of reinforced concrete (Rahman and Chamberlain 2016). In the United States, there are more than 158,000 miles of highways and road networks, which are constructed from concrete (Federal Highway Administration 2014). Although these structures were designed and built to withstand deterioration, they still need to be counted for some maintenance procedures, as they are affected by the surrounding environment (Perkins 2002).

The cost of repairing and maintaining concrete bridges, for example, is highly expensive and needs high financial support from highway authorities. As a result, and to reduce the expenses of repairing bridges and any other concrete structure, Purvis et al. (1994) believe that the most cost-effective solution will be through taking some actions at the construction level. In this regard, protecting concrete by adding protective materials at the mixing stage may result in a cost-effective solution for concrete deterioration and distresses.

Protective materials have been under investigation for a long time as a result of the need for adequate concrete protection against probable distresses that would develop in the future due to atmospheric and environmental conditions. A lot of materials with different properties and way of functioning were tested along the previous years, like cementitious coatings, moisture blockers, crystallising materials, and a lot more (Rahman and Chamberlain 2016; Al-Kheetan et al. 2017; Al-Kheetan et al. 2018c). The majority of research conducted in the 1990s and following years concentrated more on silane and siloxane based materials as they have proven their efficacy in protecting concrete and enhancing its durability (Ibrahim et al. 1997; Basheer et al. 1998; Ibrahim et al. 1999; Zhan et al. 2003; Zhan et al. 2005). However, these protective materials have been proven to have harmful effects on the environment as they are made from solvent materials. In addition to that, most research, conducted on this type of materials, focused more on the depth of penetration that silane-based materials could reach (Rahman et al. 2016). This drove many research institutes and companies to look for more environmentally friendly materials, and to study other materials where the penetration depth of these treatments is not a significant problem (Rahman and Chamberlain 2016; Al-Kheetan et al. 2018a). Some of these materials fall under the green treatments, extracted from natural products, like vegetable oils and fatty acids, and animal blood and fats (Justnes et al. 2004; Albayrak et al. 2005; Kevern 2010; Wittmann et al. 2011).

When it comes to highways, treating hardened concrete would involve some inconvenient procedures like closing the roadway to traffic to allow concrete pavement to be impregnated (Sommer 1998). From this point, researchers started to look for new solutions to escape from such inconveniences which are also more cost-effective. Internal impregnation of waterproofing materials into the concrete mix, at the mixing stage, was the most appropriate solution for this issue. Many research were carried out on this discipline, and most of them focused on using silane and siloxane based materials as internal impregnants but with different compositions (Wittmann at al. 2006; Meier and Bauml 2006; Xian et al. 2007; Zhang et al. 2009; Spaeth at al. 2014; Ma at al. 2016). However, most of these treatments negatively affected the compressive strength of the treated concrete regardless of their waterproofing effect. Adding to that, the environmental risks, mentioned previously, that this kind of materials represents due to the existence of solvent agents in their components. From this point, the world started to avoid using such materials and trends toward utilising some environmentally-friendly materials like crystallising, silicate risen, and fluoropolymer admixtures, to drive down environment deterioration (Rahman and Chamberlain 2016; Al-Kheetan et al. 2018a). Pazderka and Hájková (2016) managed to decrease concrete permeability by using a commercially available crystalline material. However, a small reduction in compressive strength was observed when adding the material to the mix. In a recent research, former researchers found that the maximum efficacy of a crystalline material in reducing water absorption will be reached after 12 days from applying the material (Pazderka and Hájková 2017).

Even though most of the research conducted on internal impregnation of fresh concrete reached a high level of waterproofing, compressive strength values were dropped down. Furthermore, all these research were performed only on high water to cement ratio mixes.

This research, which is a continuation to a previous study by authors (Al-Kheetan et al. 2018a; Al-Kheetan et al. 2018b), jumps from the need to test new eco internal impregnants that provide high protection against water ingress without compromising the compressive strength of treated concrete.

Research Objectives

This study emerges from the need to find an optimum mix that combines both; waterproofing and high compressive strength, and to overcome the problem of decreased strength when fresh concrete is internally impregnated to waterproof structures.

The objectives of this research are:

(1) Study the performance of a Silica-based crystallising impregnant added to the concrete mix at early mixing stages, in terms of strength and water permeability.

(2) Evaluate the performance of different percentages of the crystallising material, and their effect on concrete slump when produced with different water to cement ratios.

(3) Produce an optimum concrete mix that contains the optimum w/c ratio and proportion of crystallising material, to reach the maximum possible waterproofing level without negatively affecting the compressive strength.

Experimental Work

Materials

Concrete mixes, with different w/c ratios; 0.32, 0.37, 0.40, 0.46, were produced following the British standards BS 1881-125 (British Standards Institution 2013). During the process of mixing the essential concrete ingredients, the Silica-based crystallising material (A), which conforms to BS EN 1504-2 (British Standards Institution 2004), were added to the mix with two different proportions of 2% and 4%. The mix design proportions for the different mixes are shown in Table 1.

The characteristics and main components of admixture (A) are listed in Table 2.

It is noteworthy that the 2% and 4% proportions of material (A) were added to the total amount of each mix, as stated in the manufacturer instructions, without affecting the proportions of the original mix design.

All the treated mixes were tested to check their resistance to absorb water, and their capability to conserve the compressive strength without dropping down. A control mix, with 0% additive, was produced for each mix for comparisons reasons. The description and coding of each mix are mentioned in Table 3.

Procedure

For the purpose of testing concrete under the proposed objectives, 144 concrete cubes, with 100mm x 100mm x 100mm size, were produced; 48 cubes used as a control mix, 48 cubes treated with 2% of the material (A), and 48 cubes treated with 4% of the material (A). All the produced cubes were conventionally cured in a water tank at a 20 °C temperature for 7, 14 and 28 days before testing them at these periods. In addition, 36 concrete beams with 100mm x 100mm x 500mm size were produced and cured in the same aforementioned conditions; 12 beams used as a control mix, 12 beams treated with 2% of the material (A), and 12 beams treated with 4% of the material (A).

Figure 1 represents an outline of the test specifications, including the number of cubes used for each mix and the tests that were used to assess their performance.

In the beginning, concrete consistency of the treated mixes was evaluated by using the slump test, following the BS EN 12350-2 (British Standards Institution 2009) [31]. Moreover, as shown in the chart, water permeability was tested using the Initial Surface Absorption Test (ISAT) which complies with BS EN 1881-208 (British Standards Institution 1996). This test was carried out after finishing the 7, 14 and 28 days curing periods and removing the cubes from the water bath, and placing them in the lab under a temperature of 20°C to dry until they achieve a constant mass. After finishing the ISAT test, the same samples were used to test the compressive strength of each mix following the BS EN 12390-3 (British Standards Institution 2009a), as the ISAT is a non-destructive test. In addition, flexural strengths of all mixes were determined by testing the beams using the two-point loading method, following the BS EN 12390-5 (British Standards Institution 2009b). Finally, the morphology of admixture (A) and the size and development of its crystals were studied by using the Scanning Electron Microscope (SEM) and X-Ray Diffraction instrument (XRD) respectively.

Results and Discussion

Slump Outcomes

Results from this test are outlined in Table 4 with some observations noted after 28 days of curing.

Although the slump value for the 46/4A mix was very high, this mix did not develop any cracks through the 28 days of curing. Also, like the other mixes, no segregation was observed at all.

In the case of 32/2A and 32/4A, concrete was hard and, as obvious, the slump values for both mixes were zero. However, despite the difficulties in compacting such mixes, a very well compacted concrete was produced with no apparent cracks.

Microstructure Study

Treated concrete specimens were studied under the Scanning Electron Microscope (SEM) at different magnifications ranging between 500X and 12000X, after day one, day three and day seven of casting to evaluate the development and distribution of the crystals, and their interaction with the essential concrete ingredients. Figure 2 illustrates the growth and allocation of crystals with time inside the concrete mix.

Material (A) absorbs some of the water used in the concrete mix to form its crystals. These crystals grow and develop within the first 24 hours of casting concrete, and they integrate within the concrete ingredients at a very early age. This could be noticed from Figures 2 a-f, where the sequence of the micrographs taken from day 1 until day 7, show that the size and distribution of the crystals maintained the same throughout the tested period.

In parallel, treated concrete was tested under the X-Ray Diffractometer (XRD) instrument and analysed by using Scherrer equation to identify the size of crystals, and to check if there is any change in the size during the time (Uvarov and Popov 2007);

$$D= K.\lambda/\beta Cos\theta$$

Where,

D: the crystal size

λ: X-ray wavelength

β: the width of the peak (radians)

θ: Bragg angle

K: Scherrer constant

Testing was progressed for 28 days, and results showed that the growth of the crystals stops after the first 24 hours with a minimum size of 95 nm and maximum size of 200 nm. This range of crystal sizes when compared with the pores of concrete, they were smaller than the 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) (Kumar and Bhattacharjee 2003; Liu et al. 2014). It is witnessed that pores with sizes larger than 10 μm have the greatest effect on compressive strength (Li and Li 2014). This indicates that material (A) can merge easily within the concrete structure, filling most of the existing voids and prevents the formation of more micro-cracks, and preserves concrete’s compressive strength.

Permeability Outcomes

Following the BS EN 1881-208 standardised ISAT test (British Standards Institution 1996), water absorption of the different concrete mixes, treated with 0%, 2% and 4% admixture (A), were tested after 7, 14 and 28 days of curing in a water bath. Figures 3 a-d show the average water absorption rates for 10 minutes, 30 minutes, and 1-hour periods of testing concrete with the ISAT method at 7, 14 and 28 days periods.

Water absorption of all the different mixes, either treated or not, can be noticed to decrease with time but with different efficacies. 32/4A mix has shown the least absorption rate amongst all mixes during the 7, 14 and 28 days periods with zero absorption rates after 30 minutes and 60 minutes of testing on 28 days. This treatment enhanced the performance of the mix by reducing water absorption by 55% of its control mix at the age of 28 days. Also, 37/4A mix showed a proximate performance to the previous mix, with an absorption rate of 0 ml/m^2.s at 60 minutes on 28 days, with a total reduction of 65% in water absorption compared to its corresponding control. On the other hand, concrete with 46/4A revealed the worst performance between all the mixes at all times and periods with absorption rate varies from 0.23 ml/m^2.s at 7 days to 0.10 ml/m^2.s at 28 days (both after 60 minutes of testing). Moreover, in the case of the 0.46 and 0.40 mixes the control mix has performed better than the treated ones with 4% of material (A) at 28 days and after 60 minutes of testing, with a difference in performance of 53% and 40%, respectively, between the treated mixes and the control. The high absorption rates in these treated mixes, in reference to their control, come from the high water quantity used in the mix, compared to the 0.32 and 0.37 mixes, which resulted in high slump values, as shown in Table 4. This high slump indicates the high workability of both mixes resulting from adding the crystallising material. The crystallising material is a dual functioning material that works on absorbing some of the water to form crystals that line the pores of the concrete, and after the formation of these crystals, they work on repelling excess water. Repelling this excess water reduces the amount of water needed to complete the hydration process, which results in the formation of micro-cracks inside the treated concrete. Accordingly, higher absorption rates will be expected for treated concrete like the 46/4A and 40/4A mixes. On the other hand, a minor improvement in water impermeability was observed in the 0.40, and 0.46 w/c ratio mixes when treated with 2% of material (A) and at the age of 28 days.

Compressive Strength Outcomes

Results from the 7, 14 and 28 days compressive strength tests for all concrete mixes, either treated or untreated, are illustrated in Table 5. It also includes the difference between the compressive strengths of treated concrete and its reference control mix, and the variability in individual cubes.

As shown in Table 5, a reduction in compressive strength was observed in all treated mixes that were tested at the age of 7 and 14 days. At the 7 and 14 days periods, more water would be available compared to the 28 days period so that the hydration process will be faster during those periods. With the presence of the crystallising material in the mix, more water will go to activate the crystals which will decrease the total amount of water needed to accelerate the hydration process. This will result in slowing down the hydration process at the

7 and 14 days periods. 46/4A concrete at the 7 and 14 days periods suffered the most significant loss in strength due to the high amount of water in this mix which supports the previous claim.

At the age of 28 days, 32/4A concrete has achieved the highest compressive strength between all treated mixes, with a total enhancement of 31.4% of the related control mix. Also, 37/4A concrete delivered similar performance to 32/4A mix and increased the compressive strength of the mix by 42.2%. On the other hand, the treated mix 46/4A experienced the highest strength loss between all mixes with 32% deficiency of the related control mix. Moreover, all treated mixes with w/c ratio of 0.40 and 0.46 suffered from a strength loss that ranges between 19.8% and 32% related to their control mix. This could be correlated to the high slump values that these mixes delivered (Table 4), which increased their workability, in view of the high w/c ratio of these mixes. Nevertheless, all remaining treating regimes have shown moderate improvement in compressive strength that ranges between 13% and 21%.

Statistical analysis of compressive strength values shows a moderately close cluster of data around the average values.

Flexural Strength Outcomes

Figure 4 shows the results from the two-point loading flexural test for the concrete beams treated with material (A) along with their reference samples and cured for 28 days.

Results from the flexural strength test support the outcomes of both the compressive strength and ISAT tests. It is clear from the figure that treating a 0.46 and 0.40 w/c ratio mixes with any of the proposed concentrations of the crystallising material would result in losing the flexural strength of the mix without any enhancement or even preserving the original flexural strength. 32/4A and 37/4A achieved the highest flexural strength values between all the mixtures with a total improvement of 29% and 18% respectively to their control mixes.

Optimum Mix Design

The aim of the performed tests was to determine the optimum concrete mix that includes the right w/c ratio and the optimum dosage of the protective treatment, in terms of compressive strength and water absorption. ISAT results, for instance, revealed that a mix design with 0.37 w/c ratio and a dosage of 4% of the crystallising material would offer a very high protection level against water ingress with a drop in water absorption of 65% when compared to the corresponding untreated mix. The same treated mix increased the compressive and flexural strengths by 42% and 18% respectively when compared to control. A higher increase in compressive and flexural strengths was observed in the 0.32 w/c ratio mix treated with 4% of material (A), with a rise of 55% and 29% respectively. On the other hand, this mix enhanced water impermeability with an efficacy of 55% compared to its control.

In the case of concrete with high w/c ratios of 0.40 and 0.46 and treated with the crystallising material, a destructive effect was noticed in terms of compressive and flexural strengths. However, water absorption has only increased when treating these mixes with 4% of material (A), and a little reduction in water absorption has occurred when the 2% of material (A) is applied. This means that there is no point in treating concrete mixes with high w/c ratios especially if the treatment works on reducing the desired compressive strength.

The usefulness of this kind of treatment should also be investigated regarding chloride penetration to validate its efficacy.

Summary and Conclusions

Two different dosages, 2% and 4%, of the Silica-based crystallising material (A), were internally impregnated into different fresh concrete mixes with different w/c ratios, to investigate its ability to reduce water absorption and preserve the compressive strength of the original mix. Significant conclusions and observations were drawn from this research are;

(1) Impregnating the crystallising material into fresh concrete reduced the water absorption, tested by ISAT, significantly. A 2% dosage of material (A) relatively reduced water absorption of the 0.40 and 0.46 w/c ratio mixes. Also, a 4% dosage of material (A) in the 0.37 and 0.32 mixes dramatically decreased their water permeability.

(2) The 0.37 w/c ratio mix along with the 0.32 w/c ratio mix, both treated with 4% admixture, showed the best performance, regarding water absorption resistance, among all the mixes. They both prevented water ingress at 30 minutes and 60 minutes testing periods. Additionally, the 0.37 w/c ratio mix treated with 4% admixture showed a significant reduction in water absorption levels close to 65%, and the 0.32 w/c ratio mix treated with 4% admixture reduced water absorption levels by 55%.

(3) Regardless of the positive impact of treating 0.46 w/c ratio mix with 2% of material (A) on waterproofing, a parallel damaging effect has emerged that reduced the 28-days compressive strength of the mix by 23% of the control. Similarly, a reduction of 20% in the 28-days compressive strength was observed in the 0.40 w/c ratio mix treated with 2% admixture.

(4) Results from the 0.46 and 0.40 w/c ratios may suggest the impracticality of treatment, as the compressive and flexural strengths of untreated mixes were less than those treated with 2% admixture, despite the improvement in the impermeability that treatment has achieved. Adding to that, the damaging effect that the 4% dosage has shown on both strength and water absorption.

(5) An optimum mix design could be obtained by treating the 0.32 and 0.37 w/c ratio mixes with 4% admixture. Water absorption has dropped by more than 55% and 65%, respectively, of their untreated mixes, and compressive strength increased by more than 31% and 42%, respectively, above the initially designed strength. Furthermore, an increase of 29% and 18%, respectively, in flexural strength was observed in those mixes.

(6) Based on the previously tested conditions, treatment with the crystallising material (A) is considered useful only in the case of producing concrete with low w/c ratios that range between 0.32 and 0.37.

(7) Analysing treated concrete under the SEM showed that crystals are formed and settled within the detailed texture during the first 24 hours of casting. Also, XRD analysis showed that the size of the shaped crystals is smaller than most of the voids of a normal concrete, making their integration inside the concrete easily.

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List of Tables:

Table 1 - Adopted Mix Design for Different W/C Ratios

Ingredient	**Amount (Kg/m^3)**			
	W/C=0.32	W/C=0.37	W/C=0.40	W/C= 0.46
Cement	513	491	450	457
Water	164	182	180	210
Fine aggregate	658	660	678	660
Coarse aggregate	1068	1070	1092	1073

Table 2 – Characteristics and Constituents of Admixture (A)

Constituent	**Physical and Chemical Properties**	
Silica	Specific gravity	1.6
Proprietary Alkaline Earth Compound	Appearance	Powder
Portland Cement	Boiling point	104 °C
-	Freezing point	-4 °C
-	pH	12 (in water)
-	Solubility	Partially soluble
-	Toxicity	None

Table 3 - Coding of the Different Concrete Mixes and the Accompanying Tests

Code	W/C ratio	Material percentage	Testing
32/0A	0.32	0%	Fresh mixture: Slump test Cured specimens: Initial Surface Absorption Test (ISAT) Compressive strength Flexural strength Scanning Electron Microscope (SEM) X-Ray Diffractometer (XRD)
32/2A		2%	
32/4A		4%	
37/0A	0.37	0%	
37/2A		2%	
37/4A		4%	
40/0A	0.40	0%	
40/2A		2%	
40/4A		4%	
46/0A	0.46	0%	
46/2A		2%	
46/4A		4%	

Table 4 - Concrete Workability for Different Treated Mixes

Concrete mix	Slump (mm)	Comments
32/2A	0	No cracks observed
32/4A	0	No cracks observed
37/2A	5	No cracks observed
37/4A	20	No cracks observed
40/2A	15	No cracks observed
40/4A	70	No cracks observed
46/2A	50	No cracks observed
46/4A	160	No cracks observed

Table 5 - Average Compressive Strength Results for Control and Treated Concrete

	W/C	**Material (A) percentage**	**Compressive strength (MPa)**		**Changes in Strength (%)**
			Average	**Standard Deviation**	
7-days	**0.32**	0%	34.8	1.97	-
		2%	32	1.15	-8.0%
		4%	33.8	1.68	-2.9%
	0.37	0%	30.9	0.94	-
		2%	24.6	1.67	-20.4%
		4%	27	1.90	-12.6%
	0.40	0%	28.6	3.77	-
		2%	24.8	1.79	-13.3%
		4%	26.1	0.70	-8.7%
	0.46	0%	30.1	0.51	-
		2%	20.6	0.56	-31.6%
		4%	19.2	0.64	-36.2%
14-days	**0.32**	0%	39.2	0.63	-
		2%	32.8	1.00	-16.3%
		4%	31.4	4.22	-19.9%
	0.37	0%	35.2	2.25	-
		2%	25.9	1.11	-26.4%
		4%	25.7	0.72	-27.0%
	0.40	0%	38.2	0.95	-
		2%	27.5	2.33	-28.0%
		4%	27	0.78	-29.3%
	0.46	0%	32.8	1.38	-
		2%	26.1	0.64	-20.4%
		4%	20.4	1.24	-37.8%
		0%	42	2.15	-

28-days	**0.32**	2%	47.5	1.68	+13.1%
		4%	55.2	3.00	+31.4%
	0.37	0%	37.4	1.03	-
		2%	45.3	1.89	+21.1%
		4%	53.2	4.12	+42.2%
	0.40	0%	54.6	3.63	-
		2%	43.8	1.49	-19.8%
		4%	40.7	3.93	-25.5%
	0.46	0%	47.8	1.68	-
		2%	36.9	4.66	-22.8%
		4%	32.5	2.48	-32%