

A-2-4 Comparative study of different hydrophobic materials in concrete pavement protection

Mazen J. Al-Kheetan

*Division of Civil Engineering, Department of Mechanical, Aerospace and Civil Engineering, College of Engineering, Brunel University London, Kingston Ln, Uxbridge, United Kingdom, UB8 3PH.
Mazen.al-kheetan@brunel.ac.uk*

Mujib M. Rahman

Division of Civil Engineering, Department of Mechanical, Aerospace and Civil Engineering, College of Engineering, Brunel University London, Kingston Ln, Uxbridge, United Kingdom, UB8 3PH.

Denis A. Chamberlain

Division of Civil Engineering, Department of Mechanical, Aerospace and Civil Engineering, College of Engineering, Brunel University London, Kingston Ln, Uxbridge, United Kingdom, UB8 3PH.

ABSTRACT: With the world moving towards the use of alternative environmentally friendly hydrophobic materials, in the intention of protecting concrete from water ingress and accelerated deterioration, this research focuses on testing three different hydrophobic; Fluoropolymer, Silicate Resin and Crystallizing materials. A number of C40 concrete cubes were treated with these materials in the purpose of evaluating their performance in terms of water absorption, and water intake. Both ASTM and British Standards were used to evaluate the effectiveness of the treatment and to make a comparison. Microscopic study was carried out to observe their development with time, and how their polymers and crystals are formed. Results from the tests were compared with a control mix. Test results showed that all of these three materials had a hydrophobic effect on water and can be utilized in concrete protection purpose. Water absorption was noticed to be reduced in all specimens treated with the three materials but with different rates.

KEY-WORDS: Concrete, concrete pavement, hydrophobic treatment, surface impregnation, water absorption, fluoropolymers, silicate resins, crystallisation.

INTRODUCTION

Concrete has been employed in the construction of roads and motorways that were designed to serve for longer periods and reduced maintenance cost than flexible pavement [1]. However, concrete pavement is still under the risk of deterioration generated from environmental impacts and climate changes like rainfall, snowfall, and freezing and thawing. Water is one of the main factors for reinforced concrete, since all the mechanical and chemical degradation of concrete is initiated by the presence of water under any circumstances [2]. The bill of maintaining and repairing all forms of concrete structures, including highways, in the UK alone exceeded £1x10¹⁰ in 1982 [3]. As a result, an urgent need to protect concrete from water and aggressive ions that water carries, has emerged recently to reduce the expenses of concrete maintenance and to produce a more durable concrete.

Until recently, the most widely used concrete protection material was silane, siloxane and their derivatives [4-7]. However, the main issue that has emerged concerning the use of these materials is their harmful impact on the environment, and toxic effect on operatives [8]. Accordingly, researchers focused more on nature-friendly materials that are either extracted from natural resources like natural oils, fatty acids, and animal bloods [9-11], or industrial materials that have a low risk on environment like crystallising and silicate materials [12]. During past years, various alternative materials have emerged - for example- cementitious coatings [13], moisture blockers [14], and hydrophobic impregnation [15-18]. These materials have shown variable performance in the laboratory as well as in the field application. It is therefore necessary, further scrutiny before adopted to wider application.

In this research, three chemically different protection materials were studied to evaluate their performance against water ingress. The materials were Fluoropolymer, Resin Silicate and Crystallising materials. Research on the use of Fluoropolymers in protecting concrete is limited [19, 20]. Fluorine is the main element forming the

Fluoropolymers which provides them with a low friction and an improved resistance to aggressive chemicals [21, 19]. In addition, studies on these materials showed high water and oil repellence which drove researchers to apply them as surface hydrophobic impregnants to concrete [19]. Silicate Resin has also been investigated a little in the field of concrete protection. Silicate Resins is hydrophobic material that forms a coating in the pores of the concrete and works on repelling water [22]. Crystalline material is also gaining increasing popularity and have shown comparable performance against silane material especially when applied in wet concrete [23].

EXPERIMENTAL PROGRAMME

The experimental procedures of this research involve determining the water absorption of protected concrete by capillary action, and the water absorption rate under constant head pressure. Two standardised water absorption tests on concrete cubes were used followed by Scanning Electron Microscopy (SEM) testing to evaluate the structural formation with time.

Materials

A brief overview of three protection materials and their application rate are outlined in Table 1.

Table1. Specifications and details of the tested hydrophobic impregnants

Material	Base	Colour	Application	General information
Fluoropolymer	Water based	Colourless	200 ml/m ²	Resistance to temperature, chemical and weather.
Silicate Resin	Water based	Colourless	200 ml/m ²	Provides hydrophobic resistance against water, has a high resistance to heat which makes it very suitable to be used in hot areas
Crystallising	Water based	Colourless	200 ml/m ²	Non-toxic and non-hazardous material, forms crystals that protect concrete from freeze thaw deterioration, and decrease water penetration and salts ingress. It allows the surface of concrete to breathe.

Specimens and testing

AC40 concrete was produced for this study with a water to cement ratio of 0.46. Slump value for this mix was found to be 70 mm. The mix design of the concrete, shown in Table 2, was made in agreement with BS 1881-125 [24].

Table 2. Concrete mix proportions following BS 1881-125

Component	Quantity (Kg/m ³)
Cement	457
Water	210
Fine aggregate	660
Coarse aggregate	1073
Total	2400
Water/Cement ratio	0.46

48 cubes with the size of 100 mm x 100 mm x 100 mm were casted and cured for 28 days in a curing room, with 60% humidity and a temperature of 20 °C. 39 cubes were treated with the three materials; 13 cubes with Fluoropolymers, 13 with Resin Silicates, and 13 with the Crystallising material. 9 cubes were used as a control for comparison. All cubes were treated following the BS EN 1504-2 [25] and the manufacturer instructions by brushing an amount of 200 ml/m² of the materials on all the faces of the concrete cubes. Fig. 1 outlines a detailed testing program for the concrete and the number of cubes used in each test.

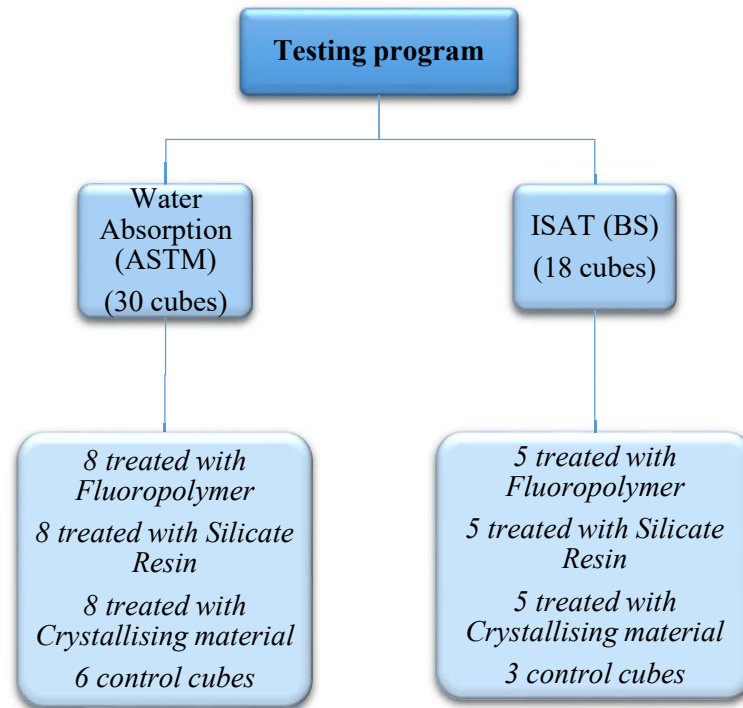


Fig.1. Testing specifications and protocol

Initial Surface Absorption Test (ISAT), as outlined in BS 1881-208 was conducted on 18 concrete cubes in order to check the resistance of impregnants to water absorption [26]. The remaining 30 cubes were also tested for water absorption according to ASTM D 6489-99 [27]. To ensure consistency, the test procedures to the ASTM D 6489-99 were followed by using concrete cubes instead of cylindrical cores as specified in the standard.

ISAT test was operated on the cubes after 28 days of curing, and after drying them till a constant mass is achieved. For water absorption according to ASTM D 6489-99, cubes were dried in an oven for 24 hours at 75 °C until a constant mass is achieved. Cubes were placed in ambient temperature to cool down and then one face of each cube was treated with the impregnants. Other faces of the cubes, that have a contact with water during the test, were sealed using a waterproof sealer to prevent water ingress through concrete. Fig. 2 shows concrete cubes during testing. Cubes were placed on steel wire mesh inside a container to allow water circulation under them. Water was filled in the container until the level is about 70 mm from the top of the steel mesh. After 24 hours and 48 hours periods, concrete samples were removed from the container and weighed.



Fig.2. Testing concrete for water absorption following a modified ASTM D 6489-99 testing procedure

A brief comparison on the two employed tests is outlined in Table 3. Performing test up to 48 hours will give an indication of material performance for longer period exposure to water.

Table 3. Comparison between the BS water absorption method and the ASTM water intake method

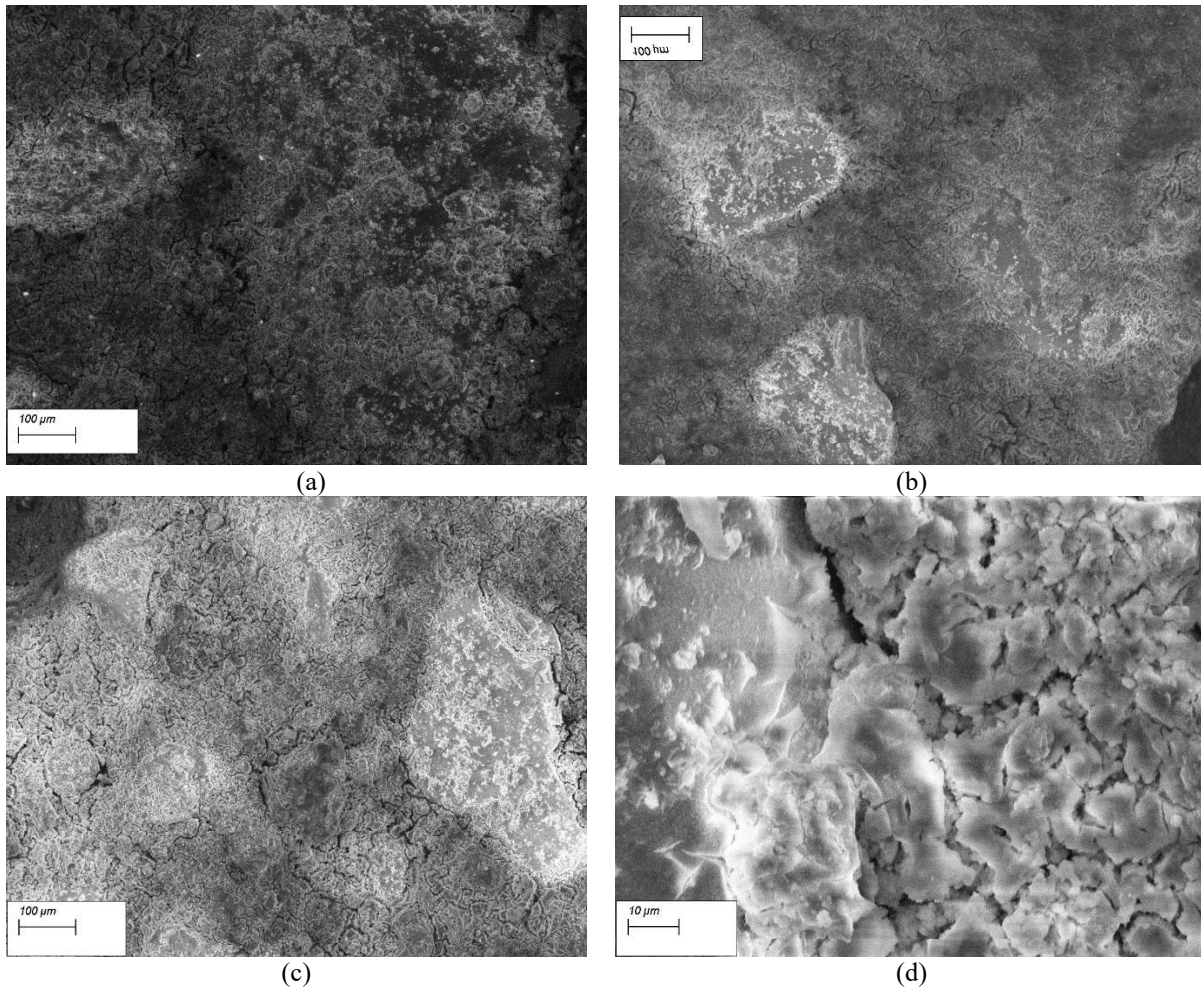
	ISAT (BS 1881-208)	Water intake (ASTM D 6489)
Testing duration	Short-term (10 minutes, 30 minutes, 60 minutes)	Long-term (24 hours, 48 hours)
Parameters	Pressure head of 200 mm	Capillary action
Anticipated outcome	Water absorption rate ($\text{ml/m}^2\cdot\text{s}$) & Water absorption (%)	Water absorption (%)

RESULTS AND DISCUSSION

Formation of crystals

All the three surface impregnants were observed under the Scanning Electron Microscope (SEM) with a 500X and 5000X magnifications. The Crystallising material was observed for 3 continuous days to monitor the development of the crystals with time. Current research focuses more on crystals formation development with time rather than studying the shape and size of the crystal itself. These two parameters will be studied in the subsequent research.

Fig. 3a-3c show the development and growth of crystals, under 500X magnification, after day one, day two and day three respectively. It is clear from Fig.s how crystals develop and grow with time and spread all over the surface. Fig. 3d shows the crystals growth after 3 days under 5000X magnification. Moreover, Fig.s 3e and 3f show the distribution of the Fluoropolymer coating and Silicate Resin correspondingly after day one. It can be seen that the distribution of material is uniform throughout the surface.



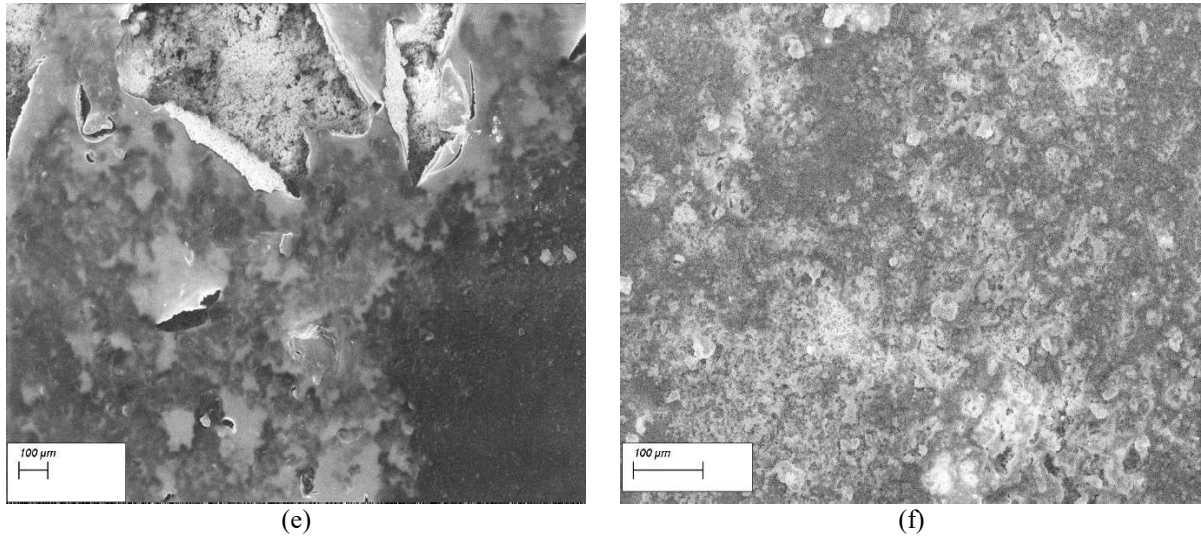


Fig.3. A microscopic view for concrete treated with: (a) Crystallising material after 1 day of application (500X), (b) Crystallising material after 2 days of application (500X), (c) Crystallising material after 3 days of application (500X), (d) Crystallising material after 3 days of application (5000X), (e) Fluoropolymer after 1 day of application (500X), and (f) Silicate Resin after 1 day of application (500X)

Surface absorption in first 60 minutes

Concrete absorption for water was investigated by using the ISAT method, for both treated and untreated cubes. Table 4 illustrates the water absorption rate for all the cubes, their average and standard deviation under each case and timing period.

Table 4. Statistical analysis for all cubes tested with ISAT

	Water absorption rate (ml/m ² .s)			Average water absorption rate (ml/m ² .s)			Standard Deviation		
	10 min	30 min	60 min	10 min	30 min	60 min	10 min	30 min	60 min
Control	0.37	0.26	0.19	0.48	0.27	0.19	0.086	0.022	0.021
	0.58	0.3	0.22						
	0.48	0.25	0.17						
Fluoropolymer	0.045	0	0	0.12	0.07	0.05	0.076	0.058	0.043
	0.018	0	0						
	0.195	0.13	0.095						
	0.19	0.12	0.09						
	0.17	0.1	0.075						
Silicate Resin	0.01	0	0	0.12	0.08	0.06	0.089	0.066	0.051
	0.015	0	0						
	0.22	0.15	0.1						
	0.18	0.13	0.12						
	0.17	0.12	0.08						
Crystallising	0.035	0	0	0.06	0.02	0.009	0.029	0.026	0.011
	0.03	0	0						
	0.09	0.055	0.021						
	0.1	0.05	0.025						
	0.045	0	0						

Sorptivity average values are plotted in Fig. 4 for all the concrete mixes at 10 minutes, 30 minutes, and 60 minutes intervals.

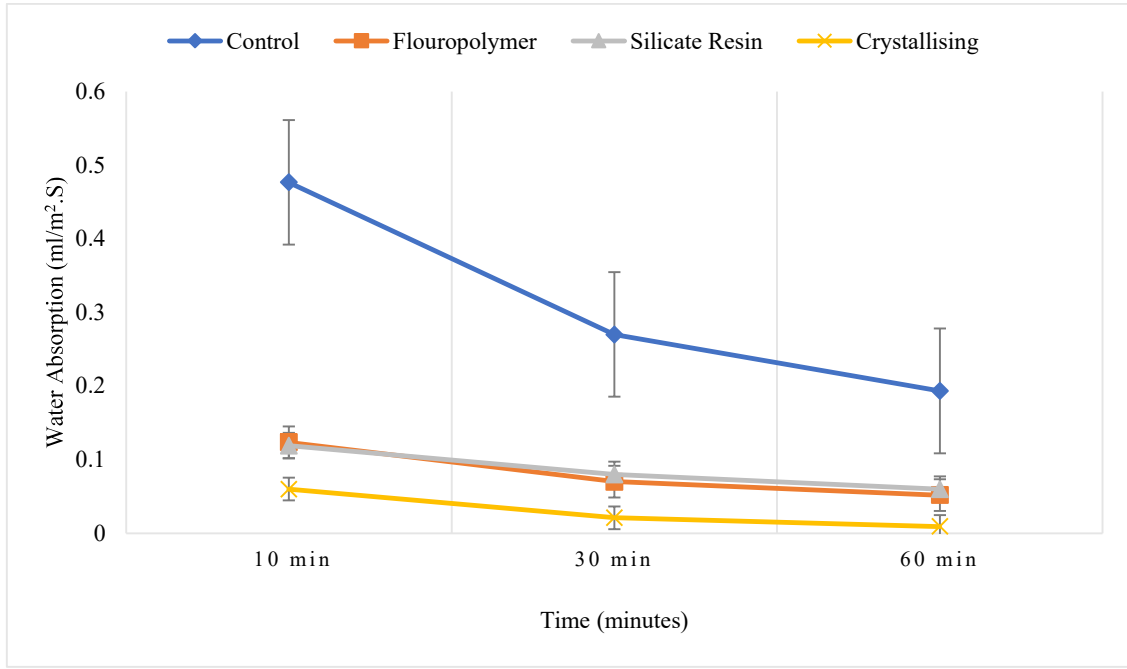


Fig.4. Surface water absorption rates for control concrete and concrete treated with a Fluoropolymer, Silicate Resin, and Crystallising material

A mutual feature between all treated and untreated concrete specimens, as shown in Fig. 4, is the reduction of water absorption rates with time. However, treated concrete showed better performance than control concrete with a difference of 0.13 ml/m².s in the case of Silicate Resin, and 0.18 ml/m².s in the case of the Crystallising material after 60 minutes testing. Comparing treated concrete together; concrete treated with a Crystallising material showed the least water absorption starting with 0.06 ml/m².s at 10 minutes and finishing with 0.009 ml/m².s at 60 minutes. Both, concrete treated with Fluoropolymers and Silicate Resins, displayed similar performance to each other with a water absorption rate of nearly 0.06 ml/m².s at 60 minute.

When comparing the three different treatments with each other, in reference to control concrete, concrete treated with Crystallising material showed a 95% efficacy in performance, after 60 minutes, compared to 69% to concert treated either with Fluoropolymers or Silicate Resins. This, undoubtedly, proves the efficacy of the three impregnants, regardless the difference in performance between them, and the high impact they provide in protecting concrete from water penetration.

Water intake during 48 hours

In parallel, 30 cubes were tested for water absorption by capillary rise after 24 hours and 48 hours from immersing them in water. Results were obtained as a percentage of the cube's dry weight using the following equation (Eq. 1), which is given in ASTM D 6489-99 [27]:

$$\text{Percent Absorption (\%)} = \frac{W_2 - W_1}{W_A} \times 100$$

where;

W_A : dry weight of concrete samples before applying the material (g).

W_1 : Weight of the concrete samples after applying impregnant and sealer (g).

W_2 : Weight of concrete samples after immersing in water (g).

The water absorption of individual cubes, their average values and the statistical analysis for the results obtained from this test after 24 hours and 48 hours periods are outlined in Table 5. Calculations of Standard deviation for all samples shows similar values during both 24 hours and 48 hours periods. These values were less than 1 and very close to 0 which mean that water absorption values for all concrete samples are uniform and very close to the average water absorption. Same observations apply to standard deviation values obtained from the ISAT test (Table 4).

Table 5. Statistical analysis for treated and control cubes tested for water absorption during 24 hours and 48 hours

	Water absorption (%)		Average water absorption (%)		Standard Deviation	
	24 hours	48 hours	24 hours	48 hours	24 hours	48 hours
Control	1.7	1.97	1.45	1.77	0.034	0.034
	0.68	0.79				
	1.82	2.23				
	1.65	2.05				
	1.39	1.77				
	1.45	1.79				
Fluoropolymer	0.71	0.89	0.66	0.87	0.046	0.046
	0.72	0.85				
	0.84	1.06				
	0.82	1.03				
	0.17	0.49				
	0.64	0.85				
	0.66	0.83				
	0.75	0.96				
Silicate Resin	1.29	1.89	0.70	1.41	0.038	0.038
	1.39	2.04				
	0.8	1.48				
	0.65	1.49				
	0.45	1.12				
	0.24	0.84				
	0.35	1.12				
	0.45	1.31				
Crystallising	0.47	0.96	0.33	0.66	0.059	0.06
	0.54	1.26				
	0.32	0.90				
	0.59	0.99				
	0.21	0.32				
	0.14	0.23				
	0.13	0.24				
	0.24	0.38				

The performance of each impregnant material after 24 hours and 48 hours of immersing in water are plotted in Fig. 5. Outcomes from this test show similar results to those obtained from the ISAT test. Concrete treated with the Crystallising material exhibited the least water absorption rate between all concrete samples, either after 24 hours or 48 hours of immersing. On the other hand, the performance of concrete treated with the Fluoropolymer and the Silicate Resin materials was less efficient than the concrete treated with the Crystallising material. After 24 hours of immersion, both Fluoropolymer and Silicate Resin, showed similar performance with water absorption of 0.7%.

However, concrete treated with Silicate Resin started to absorb more water in the period between 24 to 48 hours of immersing with 1.4% after 48 hours, whereas concrete treated with Fluoropolymer absorbed 0.87% after 48 hours. Control specimens absorbed the highest amount of water among all the specimens with 1.4% and 1.7% after 24 hours and 48 hours respectively.

The reduction in water absorption that Crystallising material could achieve in reference to control was around 77% at 24 hours of testing and 63% at 48 hours of testing. On the other hand, after 48 hours of testing, Fluoropolymer treated concrete achieved a reduction of 51% in water absorption, whereas concrete treated with silicate Resin achieved 20% reduction in water absorption. After 24 hours of testing, both Fluoropolymer and Silicate Resin treated concrete, absorbed 52% less water than untreated concrete.

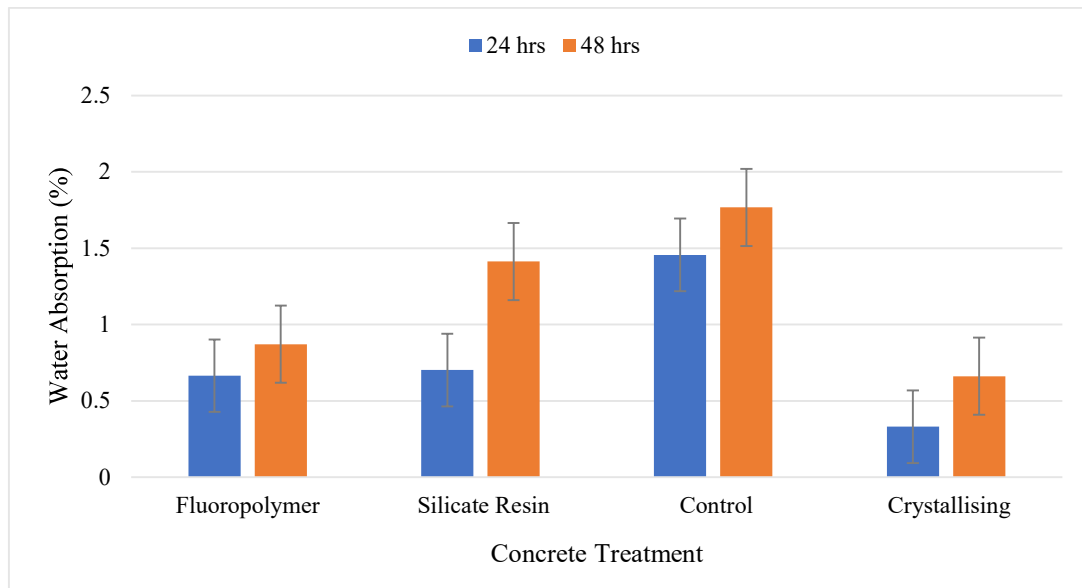


Fig.5. Percentage of water absorption for treated and untreated concrete after immersing in water for 24 hours and 48 hours respectively

Comparative analysis

In order to combine the outcomes from both tests, ISAT and water intake, the rate of water absorption, obtained from the ISAT test, and the percentage of water intake, obtained from the ASTM test, were transferred into a water absorption quantity in millilitres. Table 6 illustrates the water absorption results of both tests starting from 10 minutes of testing and ending at 48 hours. It is worth mentioning that results from the ISAT were transferred into accumulative data so it will have the same trend and measurement as the results obtained from the ASTM test.

Table 6. Water absorption of concrete during an extended life time of combined testing methods

		Water Absorption (ml)			
		Control	Fluoropolymer	Silicate Resin	Crystallising
Time (minutes)	10	2.72	0.70	0.68	0.34
	30	7.33	1.90	2.05	0.7
	60	13.94	3.68	4.10	1.02
	1440	32.58	14.66	15.35	7.50
	2880	39.55	19.20	31.01	15.01

Despite the fact that both tests operate in different ways, and they represent two different concepts for water absorption; water absorption by capillary suction and water absorption under pressure head, their outcomes could be linked together to have a full scale measurement that covers longer periods of time. The short-term and the long-term water absorption of concrete are shown in Fig. 6.

The continuity in water absorption, measured by both tests, could be spotted in Fig. 6, as the behaviour of the materials persists on the same pattern in both phases of testing. Concrete treated with a Crystallising material shows the least water absorption during the whole period. However, it is noticeable that it has the tendency to absorb more water if testing persisted beyond the 48 hours period. On the other hand, concrete treated with Fluoropolymer performed similarly to that treated with Silicate Resin during the first 24 hours of testing. Nevertheless, Fluoropolymer started to absorb less water and approaches a similar performance to Crystallising material in the second 24 hours testing period. However, more confirmations are needed by performing a longer period. To the contrary, concrete treated with Silicate Resin continued to absorb water in higher rates after 24 hours of testing, getting closer to the behaviour of the control concrete.

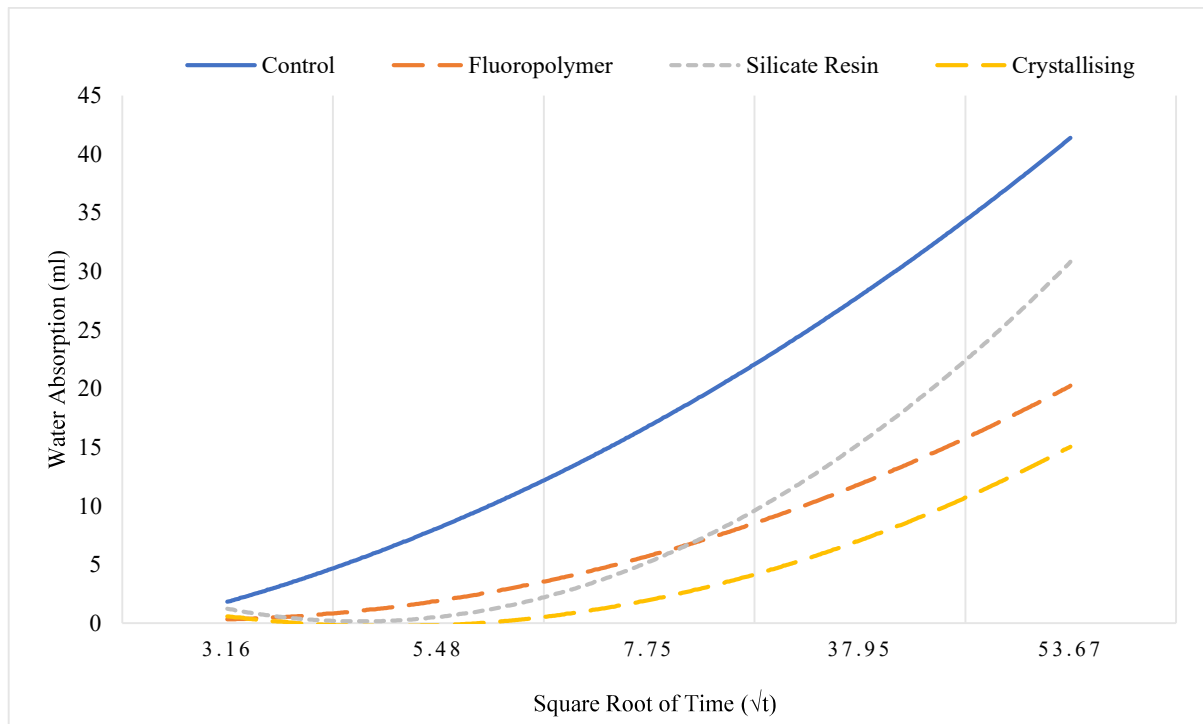


Fig.6. Short-term and long-term water absorption of treated and control concrete over 48 hour's period

SUMMARY AND CONCLUSIONS

Three different hydrophobic surface impregnants; Fluoropolymers, Silicate Resins and Crystallising material were tested in this research to evaluate their efficacy against water absorption using two methods; ISAT and ASTM water intake method. Important conclusions and observations were captured from this study and are listed below:

1. Crystallising material exhibited the least absorption rate for water in both tests, the ASTM and the ISAT tests. From ISAT results, an efficacy of 95% was observed after 1 hour from exposing treated concrete to a head of 200 mm. on the other hand, outcomes from the ASTM test showed an efficacy of 77% after 24 hours, and 63% after 48 hours from exposing treated concrete to a water head of 70 mm.
2. Fluoropolymer and Silicate Resin treated concrete showed a similar performance in the ISAT test, and during the first 24 hours of the ASTM test. After the 24 hours of testing, Silicate Resin started to be less effective than the Fluoropolymer with a modest efficacy of 20% at 48 hours of testing, while Fluoropolymer treated concrete achieved 52% efficacy at the same interval.
3. Despite the fact that concrete treated with Crystallising impregnant performed better than concrete treated with the Fluoropolymer, either during the 60 minutes testing or the 48 hours testing, Fluoropolymers managed to keep the rate of increase in water intake during the ASTM testing less than the Crystallising materials. In other words, an increase of 24% in water absorption, for 24 hours, was observed in specimens treated with Fluoropolymers, and an increase rate of 50% was observed in concrete treated with Crystallising material at the same period.
4. Both the BS referenced test, ISAT, and the ASTM based test could be considered as continuation and complementary to each other. This could be observed from the similar results that both tests imparted. For example, in the ISAT test, Silicate Resin and Fluoropolymer treated concrete exhibited the same performance during 1 hour of testing. The same materials performed similarly during the first 24 hours in the ASTM method as well, reflecting the fact that ASTM test is a prolonged test that continues the ISAT finding process. Also, Crystallising material showed the same pattern and performance in both tests.

ACKNOWLEDGMENT

The authors appreciate and acknowledge the assistance and support of the Experimental Technique Centre ETC at Brunel University London, for providing their facilities to be used by authors. Also, authors acknowledge the contribution of Dr. Nico Nelson, technician at ETC, and Mrs. Nita Verma, senior analyst at ETC, for their help and support.

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