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Moisture evaluation of concrete pavement treated with hydrophobic surface impregnants

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ABSTRACT
Despite excellent service history, concrete pavement faces accelerated deterioration due to water and chemical ingress through micro cracks and surface voids. Surface protection could be an inexpensive way of enhancing the durability of concrete pavement. This research focuses on evaluating the performance of three surface applied hydrophobic materials with different chemistries; fluoropolymer, silicate resin and sodium acetate crystallising material. Tests consisted of a microscopic study to assess the mechanism of treatment, contact angle and pendulum tests to evaluate the hydrophobicity and frictional properties of treatment respectively. Also, surface absorption and water intake tests were conducted to appraise the resistance of treatment to absorb water. It was found that all three materials are capable of developing a hydrophobic effect in concrete, but with different efficacy in reducing water absorption. The rate of water absorption was minimum for sodium acetate compared to fluoropolymer and silicate resin treatments. Scanning Electron Microscope (SEM) analysis revealed different interaction approaches for the three materials with concrete.

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Concrete pavement; rigid pavement; hydrophobic treatment; surface impregnation; water absorption; fluoropolymers; silicate resins; sodium acetate; crystallisation

1. Introduction

Concrete has been employed in the construction of roads and motorways that were designed to serve for more extended periods and reduced maintenance cost than flexible pavement (Delatte 2014). However, concrete pavement is still at the risk of deterioration generated from environmental impacts and climate changes like rainfall, snowfall, and freezing and thawing. Water is one of the main deterioration factors for reinforced concrete since all the mechanical and chemical degradation of concrete is initiated by the presence of water under any circumstances (Willway et al. 2008). In the United Kingdom, Maintenance and repair works of all forms of concrete structures, including highways, are responsible for 45% of the country's activity in the construction industry (Van Breugel 2007). 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 more durable concrete.

Although concrete protection is a well-established technique for enhancing the durability of bridges and coastal structures, the application in concrete pavement is insufficient. The central reservation for using surface applied treatment is the reduction of frictional properties and the possibility of groundwater contamination because of leaching. However, a comprehensive literature search did not find any scientific study to either prove or disprove these concerns.

In latest years, more interest in protecting concrete by hydrophobic impregnation has come to light (Rahman et al. 2013, Al-Kheetan et al. 2018b). Silane and Siloxane impregnants were one of the first effective hydrophobic treatments to be used for enhancing concrete's impermeability of water and resistance to chemical attacks (Basheer et al. 1997, De Vries and Polder 1997, Zhan et al. 2003, Zhan et al. 2005, Dai et al. 2007, Hosoda et al. 2010, Christodoulou et al. 2014). However, some doubts were raised recently regarding the performance and sustainability of these products (Christodoulou et al. 2014). Accordingly, researchers started to look for some alternative and high performance materials that are either extracted from natural resources like natural oils, fatty acids, and animal bloods (Justnes et al. 2004, Albayrak et al. 2005, Wittmann et al. 2011), or industrially manufactured like crystallising materials, moisture blockers, cementitious coatings and silicate materials (Rahman and Chamberlain 2016, Al-Kheetan et al. 2017, Al-Kheetan et al. 2018a, Al-Kheetan et al. 2018b, Al-Kheetan et al. 2018c). If these materials show good performance in concrete pavement protection, there is a considerable potential to apply them in places where the predominantly concrete pavement is used, such as in parking areas, port pavements, runway aprons and taxiways, and a significant proportion of slow and high-speed roads.

In this research, three different protection materials were studied to evaluate their performance against water ingress. The materials were Fluoropolymer, Resin Silicate and Sodium Acetate Crystallising materials. Research on the use of Fluoropolymers in protecting concrete is limited (Zaggia et al. 2009, Krishnan et al. 2013). Fluorine is the primary element forming the Fluoropolymers, which provides them with low friction and improved resistance to aggressive chemicals (Morita et al. 1999,

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Zaggia et al. 2009). Also, studies on these materials showed high water and oil repellency, which drove researchers to apply them as surface hydrophobic impregnants to concrete (Zaggia et al. 2009). Silicate Resin has also been investigated a little in the field of concrete protection. Silicate Resins is a hydrophobic material that forms a coating in the pores of the concrete and works on repelling water (Dai et al. 2010). The Sodium Acetate Crystalline material is also gaining increasing popularity and has shown comparable performance to silane especially when applied on wet surfaces (Rahman et al. 2016).

2. 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 and interaction between the applied materials and concrete. Before running the SEM analysis on the liquid protective materials, they were freeze-dried (lyophilisation) to transfer them into powder to facilitate the testing procedure). In addition, the compatibility between the protective materials and the surface of concrete pavement was assessed regarding skid resistance. Finally, the hydrophobicity of all treated and untreated concrete surfaces was determined by measuring the contact angle between water and the surfaces.

2.1. Materials

All of the three materials were brushed on the surface of concrete with an amount of 200 ml/m^2, following the guidelines of the manufacturer.

Fluoropolymer is water-based colourless compound, and its main components are carbon and Fluorine; it is mainly a Fluorinated carbon chain polymer (Perepelkin 2004). The presence of Fluorine groups in the polymer allows the material to have low surface energy, which results in reducing friction and adhesion and increasing the hydrophobicity of the polymer (Li et al. 2002). The ability of the fluorinated side of the polymer in forming a consistent structure composed of actively arranged –CF_3 groups, gives the material the advantage of being considered for coating purposes, especially in concrete protection (Li et al. 2002).

Silicate Resin is mainly a milky whitish compound, water-based and formed from Silicon and Carbon elements. This compound has a 3-D polymeric structure with Si-O-Si backbone chains and organic R groups linking with silicon atoms (Jia et al. 2009, Zhan et al. 2018), which provides a hydrophobic resistance against water, and high resistance to heat.

Sodium Acetate Crystallising material is mainly formed from sodium acetate with other propriety components that contain carbon and silicon (Abel et al. 1995, Al-Otoom et al. 2007, Pawlenko 2011). This material is characterised by developing high hydrophobicity after its application to concrete, which gives it the advantage to be used as a surface protection agent for concrete. The sodium acetate crystallising material used in this research is a solution that reacts with water to form crystals that line the pores of concrete without blocking them.

2.2. Specimens and testing

C40 concrete was produced for this study with 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 1, was made in agreement with BS 1881–125 (British Standards Institution 2013).

48 cubes with the size of 100 mm × 100 mm × 100 mm were cast 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 Sodium Acetate Crystallising material. 9 cubes were used as a control for comparison. All cubes were treated following the BS EN 1504–2 (British Standards Institution 2004) and the manufacturer instructions by brushing an amount of 200 ml/m^2 of the materials on all the faces of the concrete cubes. Figure 1 outlines a detailed testing programme for the concrete and the number of cubes used in each test.

2.3. Water absorption

Initial Surface Absorption Test (ISAT), as outlined in BS 1881–208 was conducted on 18 concrete cubes to check the resistance of impregnants to water absorption (British Standards Institution 1996). The remaining 30 cubes were also tested for water absorption according to ASTM D 6489 (ASTM 1999). 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 until a constant mass is achieved. For water absorption according to ASTM D 6489-99, cubes were dried in an oven for 24 h at 75°C until a constant mass is reached. Cubes were placed at ambient temperature to cool down, and then one face of each cube was treated with the impregnants. Other faces of the cubes that have contact with water during the test were sealed using a waterproof sealer to prevent water ingress through concrete. Subsequently, Cubes

Table 1. 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

Table 2. 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, 30, 60 min)	Long-term (24, 48 h)
Parameters	Pressure head of 200 mm	Capillary action
Anticipated outcome	Water absorption rate (ml/m^2.s) & Water absorption (%)	Water absorption (%)

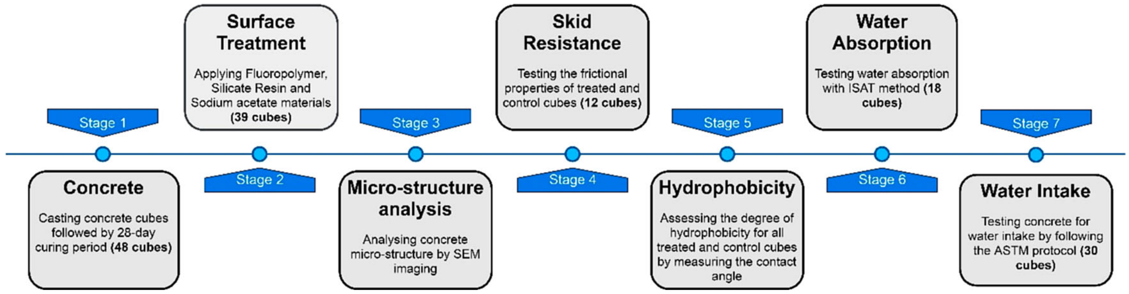

Figure 1. Testing specifications and protocol.

were placed on steel wire mesh inside a container to allow water circulation under them, and then water was filled in the container until the level is about 70 mm from the top of the steel mesh. After 24 and 48 h periods, concrete samples were removed from the container and weighed. Figure 2 shows concrete cubes during testing.

A brief comparison of the two employed tests is outlined in Table 2. Performing the test up to 48 h will indicate material performance for more extended period exposure to water.

2.4. Frictional properties

Frictional properties of 12 cubes of treated and untreated concrete were measured according to the BS EN 13036-4, by using the Pendulum test (British Standards Institution 2003). Those 12 cubes were taken from cubes already used in the water absorption test, and they were left to dry before employing them in this test. Five measurements, on dry and wet surfaces, were taken for each concrete cube, and the Pendulum Test Value (PTV) was then calculated. All the surfaces of tested concrete had the same texture and roughness, to make the comparison between samples more consistent. The same procedure applies to control concrete as well.

2.5. Hydrophobicity measurement

The degree of hydrophobicity of treated surfaces was assessed by measuring the contact angle (θ) between a drop of water and the surface. A goniometer device was used for this purpose; it involves a video recording system, which is attached to a digital image-processing programme (Anderson and Carroll 2011).

Increasing the contact angle results in increasing the hydrophobicity of the surface; surfaces with contact angles higher than 90° are considered hydrophobic. However, if the contact angle has reached or exceeded 150°, then the material is considered super-hydrophobic (Anderson and Carroll 2011).

3. Results and discussion

3.1. Microstructure analysis

All the three surface impregnants, along with concrete treated with these materials were observed under the Scanning Electron Microscope (SEM) with different magnifications, varying from 1000X to 50,000X.

Figure 3 a-c shows the microscopic structure of the three materials and their interaction with concrete.

As noticed in Figure 3a, the freeze-dried structure of Fluoropolymer appears as a mesh (5,000X image on the left), and an in-depth investigation (50,000X image on the left) shows that Fluorine particles are mostly spread on the material surface (Wan et al. 2018). This allows the active content of the attached Fluoropolymer to concrete pores to increase the hydrophobicity and decreases its water absorption. Looking at the interaction between the Fluoropolymer and concrete, it is witnessed that Fluoropolymer is covering a wide area of the concrete cross-section (5,000X image on the right), and a closer look (50,000 image on the right) shows Fluoropolymer as 'pebble' shape particles with smooth surfaces attached to concrete, with sizes less than 200 nm. This size of Fluoropolymer particles allows it to penetrate through most of the pores in concrete and line them without blocking.

The anatomical structure of the Silicate Resin, described in Figure 3b, shows attached clatters of silica resin that works as a unit (5,000X image on the left). When applied to concrete, silica gel will be formed and create strengthening points in the internal concrete structure after its precipitation inside the pores, without completely blocking them (Sandrolini et al. 2012, Franzoni et al. 2013). This refers to the reaction between the attached Silicon Resin with the hydroxyl groups in concrete in the presence of hydrogen bonds that allows silicon resin to adhere to the pores all through the drying time, providing concrete with hydrophobic properties (Pan et al. 2017). On the other hand, and comparing this material with traditional Silane/Siloxane materials, Silane works on penetrating the pores and blocking them, not allowing concrete to breathe, due to the presence of alkoxy group in their molecular structure. Alkoxy group has the ability to react with water inside concrete pores to form Silanol groups that condensate inside the pores and block them (Pan et al. 2017).

When referring to the sodium acetate crystallising material, Figure 3c shows some small crystals attached to each other (1,000X image on the left). An in-depth investigation of their structure (10,000X image on the left) reveals that they have an amorphous structure with smooth surfaces, which boosts their hydrophobic effect (Al-Kheetan et al. 2018b). The reaction of this material with water results in joining sodium acetate crystals with concrete pores, forming a denser concrete structure, and forming another type of crystals containing organosilicon components with a hydrocarbon group that eliminates the hydrophilic properties of silica and converts it into water-repellent agent (2,000X image on the right) (Palomino et al. 2007, Wagh et al. 2010, Wagh et al. 2015). The 10,000X

(a) (b)

Figure 2. Testing concrete for water absorption following (a) a modified ASTM D 6489 testing procedure and (b) ISAT procedure.

image (on the right) in Figure 3c shows a cross-sectional area for treated concrete, where crystals are seen to be attached to concrete texture and packed with each other, covering all the cross-sectional area of concrete.

3.2. Skid resistance

Three individual readings from each swing for the pendulum tester were taken for all treated and untreated concrete samples. Table 3 shows the slip resistance of all the concrete samples after applying the test on dry and wet surfaces. The Pendulum Test Value (PTV) for each case was evaluated by calculating the mean of five swings on every surface.

Comparing the skid resistance of all treated and control concrete based on their PTV values, control samples have shown the highest slipping resistance among all concrete, when concrete is either dry or wet. However, concrete treated with sodium acetate crystallising material and Fluoropolymer materials attained PTV values marginally lower than untreated concrete, and at the same time higher than concrete specimens treated with Silicate Resin. Concrete treated with Silicate Resin achieved the lowest resistance to slipping, with a PTV of 22 when the surface is dry and 18 when it is wet; PTV of concrete treated with Silicate Resin for dry surfaces was even lower than the PTV of all the other materials of wet surfaces.

3.3. Hydrophobicity

The contact angle for treated and control concrete surfaces was measured, and readings from different locations on different samples were taken. Li and Neumann (1992) suggest taking three values for the contact angle with a 30-second interval between each measurement, for an overall period of 90 s. However, in this research, the test was run for a period of two minutes, and the contact angle was measured at 30-second intervals, as suggested by Li and Neumann (1992). Figure 4a-d shows the contact angle between a drop of water and the surfaces of concrete at different time intervals.

Results from this test support outcomes of the skid resistance test obtained in the previous section. It can be noticed from figure 4 and Table 3 that concrete treated with Silicate resin has the highest hydrophobicity and at the same time the lowest skid resistance between all concrete samples. The contact angle of Silicate Resin started at 116° at the beginning of the test and decreased gradually to 107° after 120 s of testing. Concrete treated with Fluoropolymer has exhibited high water-repellence properties with time as well; contact angle was marginally less than concrete treated with Silicate Resin, with a maximum value of 111° at 0 s and a minimum value of 95° at the end of testing. On the other hand, sodium acetate crystallising material has shown the least hydrophobic properties between all treated samples with a contact angle of 82° at 0 s and 29° after 120 s of testing. Despite the low contact angle of sodium acetate crystallising material, its hydrophobicity was two times higher than control concrete, and its slip resistance, as shown in Table 3, was higher than all treated concrete and close to control. This also indicates that sodium acetate crystallising material particles have penetrated through the surface, and have created a lining rather than blocking the pores, allowing concrete to breathe.

Table 3. Skid resistance properties for all treated and untreated concrete in terms of the pendulum test values.

Applied Protective Material	Surface condition	V_1	V_2	V_3	V_4	V_5	PTV
Fluoropolymer	Dry	29	30	28	29	29	29
	Wet	25	23	23	24	23	24
Silicate Resin	Dry	22	22	23	22	23	22
	Wet	19	19	18	17	17	18
Sodium Acetate	Dry	30	29	30	31	29	30
	Wet	24	23	24	24	24	24
Control	Dry	32	32	31	33	31	32
	Wet	26	27	25	26	26	26

3.4. Surface absorption in first 60 min

Concrete absorption of water was investigated by using the ISAT method, for both treated and untreated cubes. Figure 5 illustrates the average water absorption rate for all the concrete mixes at 10 min, 30 min, and 60 min intervals.

A common feature between all treated and untreated concrete specimens, as shown in Figure 5, 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/m^2.s in the case of Silicate Resin, and 0.18 ml/m^2.s in the case of the sodium acetate crystallising material after

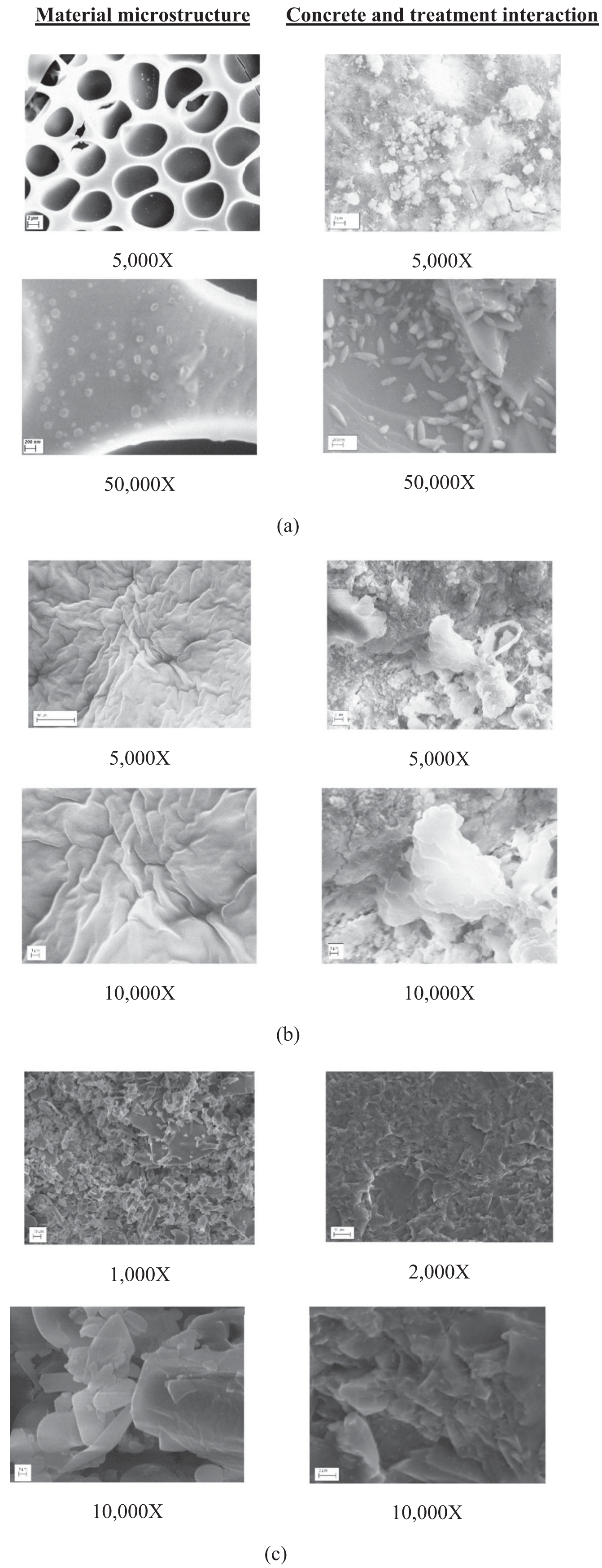

Figure 3. Microstructure of protective materials and the interaction between concrete and the materials: (a) Fluoropolymer, (b) Silicate Resin and (c) Sodium Acetate Crystallising material.

60 min testing. Comparing treated concrete together; concrete treated with sodium acetate showed the least water absorption starting with 0.06 ml/m^2.s at 10 min and finishing with 0.009 ml/m^2.s at 60 min. Both, concrete treated with Fluoropolymers and Silicate Resins, displayed similar performance to each other with a water absorption rate of nearly 0.06 ml/m^2.s at 60 min.

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

Two factors had contributed in the reduction of water absorption in concrete treated with the three materials; their hydrophobic nature, outlined in Figure 4, and their effect on reducing the porosity of concrete (Krishnan et al. 2013, Pan et al. 2017). The three materials exhibit similar mechanism in protecting concrete, and they all depend on their hydrophobic nature and their ability in reducing pores sizes (without blocking them) to reduce water penetration. However, the difference in performance between the three materials might come from their different interaction mechanism with concrete, discussed in section 3.1.

3.5. Water intake during 48 h

In parallel, 30 cubes were tested for water absorption by capillary rise after 24 and 48 h from immersing them in water. Results were obtained as a percentage of the cube's dry weight using the following equation (Equation 1), which is given in ASTM D 6489 (ASTM 1999):

$$\text{Percent Absorption (\%)} = \frac{W_2 - W_1}{W_A} \times 100 \qquad (1)$$

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 performance of each impregnant material after 24 and 48 h of immersing in water is plotted in Figure 6.

Outcomes from this test show similar results to those obtained from the ISAT test. Concrete treated with sodium acetate exhibited the least water absorption rate between all concrete samples, either after 24 h or 48 h 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 sodium acetate. After 24 h 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 and 48 h of immersing with 1.4% after 48 h, whereas concrete treated with Fluoropolymer absorbed 0.87% after 48 h. Control specimens consumed the

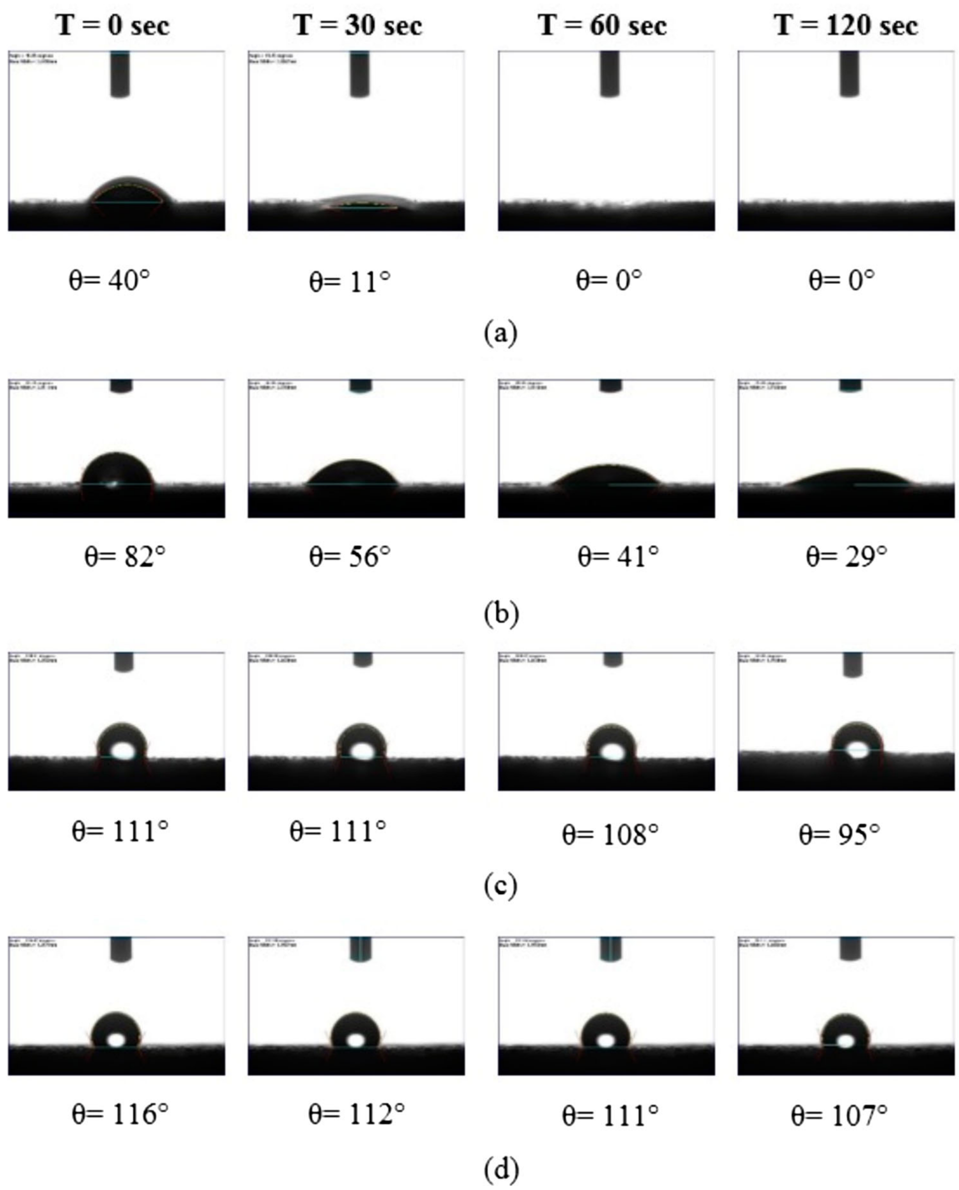

Figure 4. Contact angle for concrete surfaces: (a) untreated, (b) treated with Sodium Acetate Crystallising material, (c) treated with Fluoropolymer, and (d) treated with Silicate Resin.

highest amount of water among all the samples with 1.4% and 1.7% after 24 and 48 h respectively.

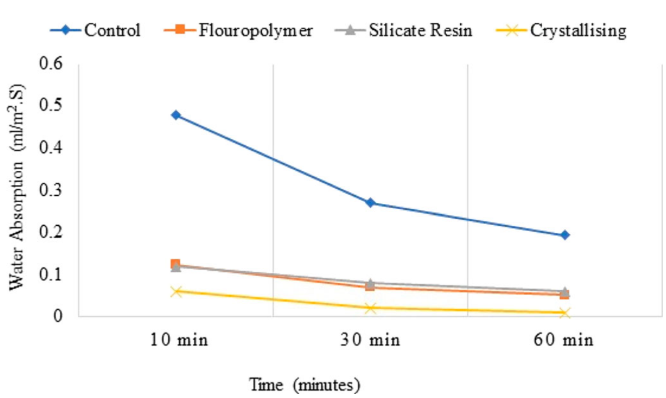

Figure 5. Average surface water absorption rates for control concrete and concrete treated with a Fluoropolymer, Silicate Resin, and Sodium Acetate Crystallising material.

The reduction in water absorption that sodium acetate could achieve in reference to control was around 77% at 24 h of testing and 63% at 48 h of testing. On the other hand, after 48 h of

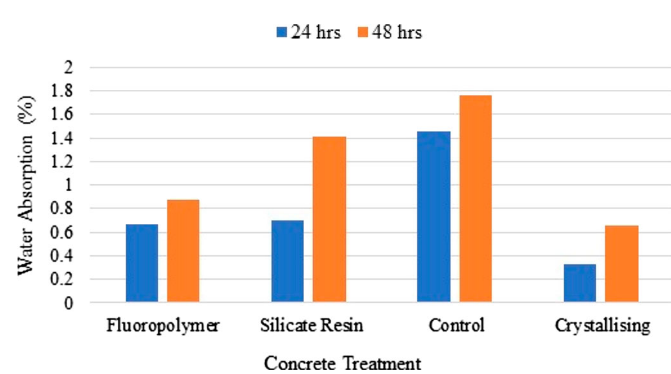

Figure 6. Average percentage of water absorption for treated and untreated concrete after submerging in water for 24 and 48 h respectively.

testing, Fluoropolymer treated concrete achieved a reduction of 51% in water absorption, whereas concrete treated with silicate Resin achieved a 20% reduction in water absorption. After 24 h of testing, both Fluoropolymer and Silicate Resin treated concrete, absorbed 52% less water than untreated concrete.

3.6. 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, derived from the ASTM test, were transferred into a water absorption quantity in millilitres. Table 4 illustrates the water absorption results of both tests starting from 10 min of testing and ending at 48 h. It is worth mentioning that results from the ISAT were transferred into accumulative data so it will have the same trend and measurement as results obtained from the ASTM test.

Even though 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 more protracted periods of time. Also, combining results from both tests will give a close estimation to a real-life situation; water absorption through pavement is either from rainfall or groundwater, and both combined tests are designed to measure water absorption in these situations. The short-term and the long-term water absorption of concrete is shown in Figure 7.

The continuity in water absorption, measured by both tests, could be spotted in Figure 7, as the behaviour of the materials persists on the same pattern in both phases of testing, with concrete treated with sodium acetate showing the least water absorption during the whole period. On the other hand, concrete treated with Fluoropolymer performed similarly to that treated with Silicate Resin during the first 24 h of testing. Nevertheless, Fluoropolymer started to absorb less water and approaches a similar performance to sodium acetate in the second 24 h testing period. However, more confirmations are needed by performing a longer period. To the contrary, concrete treated with Silicate Resin continued to absorb water at higher rates after 24 h of testing, getting closer to the behaviour of the control concrete.

4. Summary and conclusions

Testing the three hydrophobic surface treatments in this research showed promising results in protecting concrete from water absorption. The efficacy of the three materials; Fluoropolymers, Silicate Resins and Sodium Acetate Crystallising material were evaluated by using two methods; ISAT and ASTM water intake method. Also, the hydrophobicity of treated and untreated concrete pavement was assessed to support water absorption results, and to backup results from the skid-resistance test. The compatibility and interaction of treatment with concrete were evaluated by running a microstructure analysis. While sodium acetate showed the least water absorption rate between all other treatments, its hydrophobicity was the lowest (excluding control) which helped in increasing its skid-resistance. Regarding concrete pavement, sodium acetate crystallising material would be the most suitable and compatible treatment, as the material has shown skid-resistance values similar to control and contributed to reducing water permeability of concrete. The presence of sodium acetate in the components of this material helped it to perform better than other treatments, for their reactivity with cement compounds in the presence of water, forming hydrophobic silicate crystals that cover the walls of pores.

Both the BS referenced test, ISAT, and the ASTM based test could be considered as a 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 h of testing. The same materials performed similarly during the first 24 h in the ASTM method as well, reflecting the fact that the ASTM test is a prolonged test that continues the ISAT finding process. Also, sodium acetate crystallising material showed the same pattern and performance in both tests.

Further testing is still needed to assess the long-term performance of these materials and how they affect skid resistance. Also, it is vital to test the skid resistance before and after adding water for long durations to assess how water absorption affects performance.

Table 4. Water absorption of concrete during an extended lifetime of combined testing methods.

		Water Absorption (ml)			
		Control	Fluoropolymer	Silicate Resin	Sodium Acetate
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

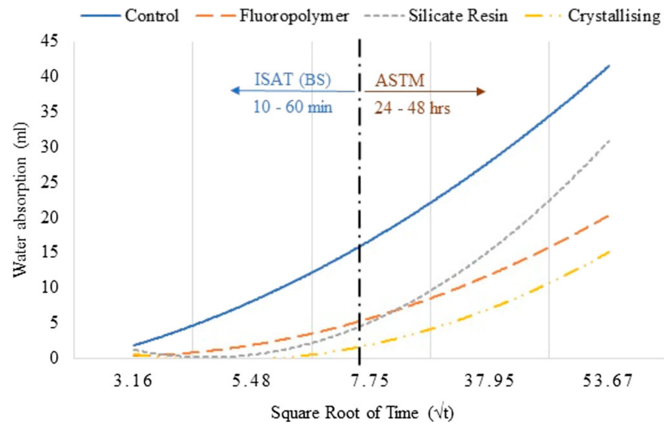

Figure 7. Short-term and long-term water absorption of treated and control concrete over 48 h's period.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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