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# Experimental Investigation on Hydrophobic Treatment of Cement Concrete with Organosilane Based Compound

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## ABSTRACT

Concrete is the most widely used construction material owing to its good strength, mouldability, and robustness. However, its durability has always been a cause of concern arising, mainly due to the fact that concrete is highly porous, microcracked, and hydrophilic in nature, making water ingress into it unavoidable. Water ingress is the primary cause of all major durability-related issues in concrete, such as freezing and thawing, reinforcement corrosion, carbonation, efflorescence, etc. It is thus evident that to prevent deterioration and thereby increase durability and service life of concrete structures, water ingress into it must be minimized. This can be accomplished by a number of methods, out of which hydrophobic treatment of concrete is nowadays becoming popular. Surface Hydrophobic Treatment and Integral/Bulk Hydrophobic Treatment are two main ways to induce water repellency in concrete. In this work, the efficacy of integrally incorporating a silane product into concrete and providing a surface treatment using the same product on the mechanical, durability, and physical properties have been studied. The integral modification did not yield satisfactory results in the case of waterproofing the composites with the Water Contact Angle values lying below 90˚ in the hydrophilic range. Whereas, the surface treatment reported a successful hydrophobic modification reporting a Water Contact Angle value as high as 157.1˚. The Water Contact Angle values for all surface-modified samples were over 150˚, which lies in the superhydrophobic category, along with the composites exhibiting a self-cleaning behavior with very little effect on the compressive and tensile strength.

**Keywords:** Hydrophobic, Porous, Silane, Durability, Concrete.



## 1. INTRODUCTION

Concrete faces multiple issues that threaten the serviceability of the structure, durability being one such major issue. High porosity, hydrophilic nature, and the presence of microcracks and fractures facilitate water penetration into the concrete. Water penetration is the reason behind all major durability-related issues in concrete [1, 2]. It might weaken or even negate the protection offered by concrete to the reinforcing bars. Corrosion, freeze-thaw cycles and penetration of harmful ions and substances not only corrode the steel bars but also may react with cement paste and give rise to unwanted deleterious products; all result from water penetration into the concrete [3-5]. All these issues can be negated if water penetration into the concrete can be prevented or reduced. There are various methods that can be used to reduce water penetration into the concrete, out of which the hydrophobic modification methods are continually becoming popular [6-8]. Hydrophobic treatments alter the concrete characteristics from hydrophilic to hydrophobic, lowering the water absorption and resulting in an increase in concrete durability [9]. Surface Hydrophobic Treatment and Integral/Bulk Hydrophobic Treatment are two main ways to induce water repellency in concrete, and both methods have been explored in the current investigation. This paper aims to develop a hydrophobic concrete using an organosilane-based waterproofing agent with the commercial name Zycosil+. In this investigation, integral and surface hydrophobic modification is carried out to see the development of hydrophobicity on the substrate. The research program was subdivided into three parts. In the first part, the raw materials are obtained and analyzed to determine their chemical and physical properties. In the second stage, the organosilane compound is admixed into the concrete in different percentages in an effort to form an integrally hydrophobic concrete. In addition, the surface treatment with the organosilane-based compound was provided on the hardened concrete samples without any admixture to produce a hydrophobic surface concrete. The third stage involves conducting various mechanical, physical, and durability tests on the untreated, integrally treated and surface-treated specimens to determine the effect of the hydrophobic compound on the properties of concrete.

## 2. MATERIALS AND METHODS

### *2.1. CLASSIFICATION OF SURFACES BASED ON HYDROPHOBICITY AND DIFFERENT TREATMENT METHODS*

A surface is said to be hydrophobic if it repels water and does not allow its easy penetration. A water droplet, when placed on a hydrophobic surface, does not wet it, whereas, in the case of a hydrophilic surface, the water droplet wets the surface and easily penetrates into the surface, as shown in Figure 1.

**Figure 1.** View showing a hydrophilic surface with the help of a water droplet

**Figure 2.** View showing hydrophobic surface with the help of a water droplet

The surfaces are classified as being hydrophilic/ hydrophobic based on their Water Contact Angle (WCA) values, as shown in Table 1. Water Contact Angle values quantify the wettability of the surface and help in their classification.

**Table 1.** Classification of surfaces based on WCA values [1][10]

| S.No. | Type of surface | Water Contact Angle |
|---|---|---|
| 1 | Hydrophilic Surface | θ < 90˚ |
| 2 | Hydrophobic Surface | 90˚<θ < 120˚ |
| 3 | Over hydrophobic | 120˚θ < 150˚ |
| 4 | Superhydrophobic | θ > 150˚ |

Hydrophobicity can be induced either by

- Surface treatment or
- Integral / Bulk modification [11]

Surface treatment is provided on the hardened concrete specimens by either brushing the hydrophobic agent onto the surface or by impregnation by immersing the specimens in hydrophobic solutions. This treatment poses one main problem the treatment is effective so long as the surface is not damaged/worn out. In case the surface gets damaged, the internal hydrophilic surface is exposed, making the concrete prone to water penetration, thereby defeating the purpose of the treatment.

In the case of integral/bulk modification, the hydrophobic additive is added to concrete during the mixing phase itself, eliminating the extra step of coating/immersion involved in the surface treatment method. The bulk treatment does not pose any such challenge of being prone to surface damage, and the entire concrete volume exhibits hydrophobicity [12, 13]. If the surface breaks or a fracture/crack forms when using integral hydrophobic concrete, the newly exposed surface still remains hydrophobic, because of which its capability to repel water is not compromised [14]. This reason provides an advantage of using integral treatment over surface treatment. The most commonly used compounds for providing hydrophobic treatment to concrete and mortars are silanes and siloxanes, and a combination of these two [15-18].

## *2.2 MATERIALS USED*

The materials used in the experimental work are water, fine aggregates, coarse aggregates, cement, and an organosilane-based compound with the commercial name Zycosil+, which is used in conjunction with the bonding agent Zycoprime. Zycosil+ is a waterproofing organosilane-based agent. It seals the surface up to nanopore level and can penetrate up to 2 mm depth inside the surface, thereby becoming a part of the surface. It converts the nature of the substrate from hydrophilic to hydrophobic. Zycoprime is an acrylic co-polymer emulsion used as the bonding agent. Zycosil+ is mixed with the Zycoprime and water in the recommended ratio specified by the manufacturers, which is a ratio of 2 parts of ZycoPrime, 1 part of ZycoSil+ & 20 parts of water to prepare the solution which is to be used in concrete. In the present work, OPC of grade 53 has been used, which was available in the institute laboratory. River sand and crushed stones are used as fine and coarse aggregates, respectively, available in the institute laboratory.

## *2.3 METHODOLOGY*

The mix is designed for M35 structural grade concrete with a water-cement ratio of 0.45 as per Indian Standards for mix design. The percentage of silane added in an effort to form an integrally hydrophobic concrete was 1%, 2%, 3%, 4%, 5%, and 6% by weight of the binder. A reference mix termed the control mix was prepared without any silane addition. The number of materials obtained as per the mix design is presented in Table 2.

**Table 2.** Quantity of material worked out as per IS code

| S.No. | Material | Quantity (kg/m$^3$) |
|---|---|---|
| 1. | Cement | 380 |
| 2. | FA | 670 |
| 3. | CA | 1320 |

A drum mixer is used for mixing concrete mixes. The required quantities of cement, fine aggregates, coarse aggregate, and water were weighed and dry-mixed. The order of dry mixing is coarse aggregates and fine aggregates, followed by cement. Zycosil+ is then weighted as a percentage weight of the binder, after which its aqueous solution is prepared by adding in Zycoprime and water. The ratio of Zycosil+, Zycoprime, and water is kept at 1:2:20, as suggested in the product usage by the manufacturers. Trials for the same were performed in order to determine the optimum ratio of the products used, which coincided with the one provided by the manufacturer. Water is added to the dry mixed components, and the mixing is carried on for a few more minutes in order to create a homogenous mixture. After thorough mixing of the contents in the drum, the aqueous solution of Zycosil+ was added to the mix, and mixing was continued till a homogeneous mass was obtained. The concrete mix is then poured into molds, compacted with the tamping rod, and then vibrated in order for any air bubbles to be released. All samples are de-molded after 24 hours of the casting and then stored in the curing tank until the testing age of 7 days, 28 days, and 56 days. For each test, at a specified age, 3 samples were cast for a single variation of the silane.

## *2.4 TESTS PERFORMED ON THE CONCRETE SPECIMENS*

The following tests are carried out on hardened concrete at three different curing ages i.e. 7-days, 28-days and 56-days after their casting.

### 2.4.1 COMPRESSIVE STRENGTH TEST

Several properties of concrete are directly related to its compressive strength making this test one of the most important tests conducted on hardened concrete. For this test, cube samples of size 150mm x 150mmx 150mm are cast and tested at specified curing days. The average value of the 3 samples tested is used as a representative value for strength for each mix, with the average rounded off to two digits after the decimal. As shown in Figure 4, the samples are tested using an automated Compression Testing Machine (CTM) with a load capacity of 3000 kN. According to IS: 516-1959 [19], the load is applied progressively at a rate of 14 N/mm$^2$ /min. The machine is stress-controlled, and the maximum load at which the specimen fails is recorded.

**Figure 4.** Testing of cube in CTM showing the failure of the sample

### 2.4.2 SPLIT TENSILE STRENGTH TEST

Split tensile strength is performed to get an idea about the tensile strength of concrete. The test is performed on cylindrical samples at each curing age, and the average value of the three samples tested is used as a strong value for that specific age for each mix. At each age, three cylindrical specimens with a diameter of 100 mm and a height of 200 mm were tested in order to determine the split tensile strength of concrete specimens, and the average of the results so obtained is reported. As illustrated in Figure 5, the samples are examined using an automated Compression Testing Machine (CTM) with a load capacity of 3000 kN and a load rate of 1.8 N/mm$^2$ /min, as per IS: 5816- 1999 [20].

**Figure 5.** Testing of cylinder by CTM for split tensile strength

This test is performed to check the rate of water absorption of concrete by measuring the increase in mass of a sample after the absorption of water with the time when one surface of the sample is exposed to water. This test was done as per ASTM Standard C 1585–04 [21]. The testing sample is a 100 ± 6 mm diameter disc, and the height is 50 ± 3 mm. These samples are made by cutting cylinders of 100 mm diameter and 200 mm height with the help of a stone cutting machine.The obtained cylindrical discs are first put in an oven at a temperature of 100 ± 5 °C for 24 hours, following which, the samples are put in a desiccator by applying silica gel to prevent moisture absorption after allowing samples to cool for 24 hours to ambient temperature. The circumferential surface is then wax-coated, and the top face is covered with a plastic sheet to enable uniaxial water flow via the sample's uncoated surface, i.e, from the base. The initial mass of the disc is then noted. The pan is then filled with tap water such that the water level is 1-3 mm above the supportive device. The samples are put on the supportive device, and the stopwatch is started at the same time. The weight of the sample is recorded after 30 min, and a similar procedure is followed for all the mixes. The absorption value calculated by using relation

$$I = M/(A*D)$$

where, I = Absorption (in mm)
M = change in the mass of the sample (in g)A = Area exposed (in $mm^2$)
D = Density of water (in $g/mm^3$)

Sorptivity gives us an idea about the pore structure of the concrete. The lower sorptivity value means the concrete has a higher resistance to the water, and the higher value means that the concrete has less resistance toward the water.

### *2.4.3 SESSILE DROP TEST*

This test helps determine if the sample is hydrophobic or not with the help of Water Contact Angle (WCA) measurements. In this experimental work, the sessile drop test is performed for the integral hydrophobic concrete as well as for surface-modified hydrophobic concrete. For the integral hydrophobic method, first, the samples are cut from the various concrete mixes, as shown in Figure 6, and the WCA measurements are taken. For the surface hydrophobic method, the silane solution is made then the samples are coated with the solution with the help of a brush and kept for 24 hours for drying. For double coating of the silane solution, the same sample is coated again by dipping and kept for drying. A similar procedure is followed for triple coating, following which the WCA measurements are taken. Before performing the test, the power to Acam water contact angle measuring equipment is turned on and set up prepared for the test (Figure 7). Now, the syringe is filled with the water, and initial adjustments are made. The sample is placed on the platform and adjusted in such a way that the high-resolution camera captures the image of the sample. The water droplet is then placed on the sample through the prefilled syringe attached to the equipment by lifting the platform. Once the water droplet is placed, measurements of the water contact angle are made with the help of the software. The test is repeated for various faces, and the average of three values is considered the representative value for the specimen.

**Figure 6.** Cutting of the sample for WCA measurement

**Figure 7.** Setup for Water C ontact Angle-ACAM WCA measuring equipment

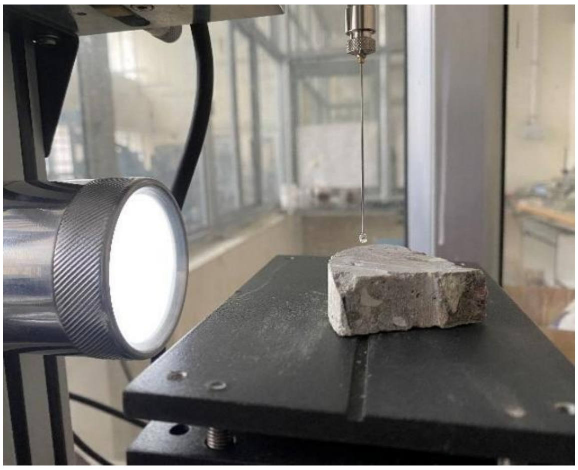

**Figure 8.** Dispensing the water droplet from the Syringe onto the concrete specimen

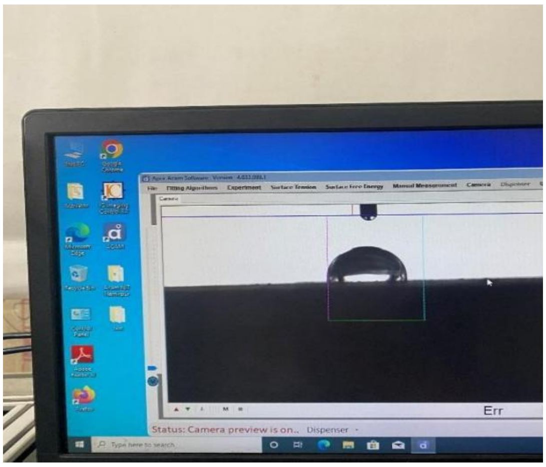


**Figure 9.** Measuring the WCA of the water droplet

## 3. RESULTS AND DISCUSSION

### *3.1. COMPRESSIVE STRENGTH*

The compressive strength of concrete mixtures at various curing ages is shown in Table 3. The mix with no silane or 0% silane is considered the control mix. It is observed that there is no significant change in the 7 day compressive strength, but there is a decrease in 28 days and 56 days compressive strength of various mixes having silane when compared with the control sample. It is also observed that the decrease in strength is more when the percentage of silane replacement is more. This might be attributed to a delay in cement hydration caused by the silane product. Average compressive strength values of single, double, and triple-coated samples are reported in Table 3. Not much change in the compressive strength of coated samples is observed when compared with the control samples. This is attributed to the fact that the product merely coats the surface without interfering with cement hydration during the setting and hardening processes.

**Table 3.** 7-day, 28-day,56-day compressive strength test results

| Percentage of Silane | 7-day Strength ($N/mm^2$) | 28-day Strength ($N/mm^2$) | 56-day Strength ($N/mm^2$) |
|---|---|---|---|
| **0** | 18.67 | 40.00 | 41.34 |
| **1** | 19.56 | 35.12 | 35.56 |
| **2** | 17.78 | 33.34 | 34.22 |
| **3** | 19.78 | 32.23 | 32.67 |
| **4** | 18.45 | 31.12 | 32.44 |
| **5** | 19.12 | 30.67 | 31.55 |
| **6** | 18.14 | 28.44 | 30.88 |
| **Surface coated samples** | 18.33 | 40.14 | 41.23 |

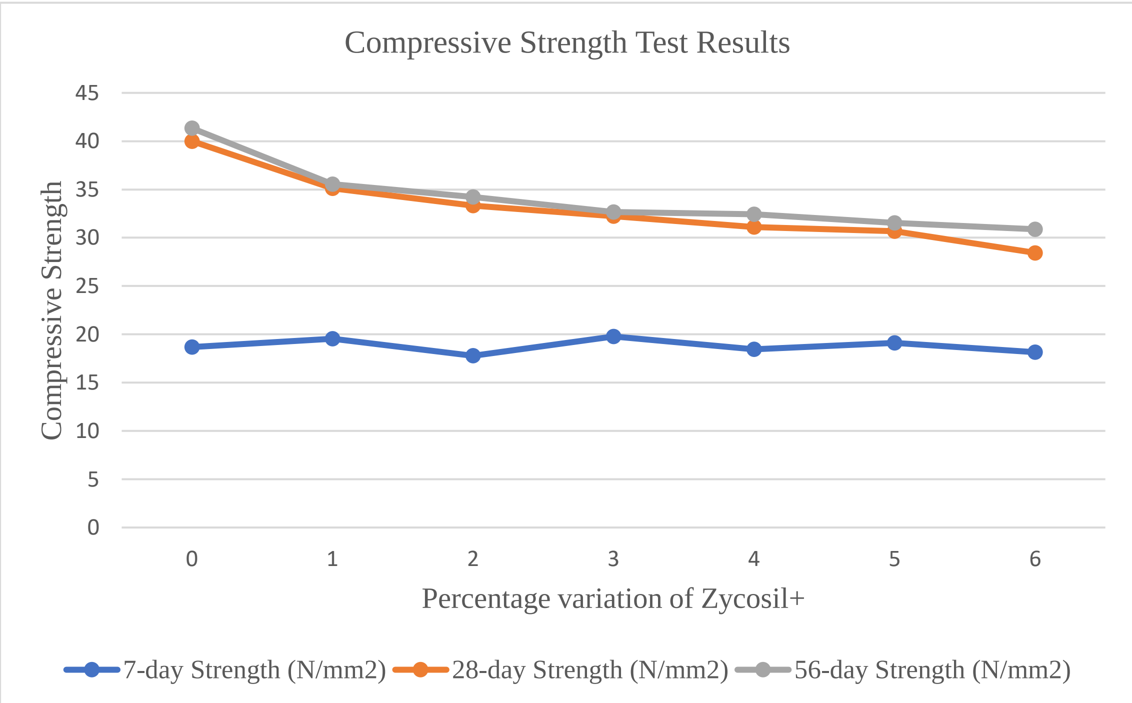


**Figure 10.** Compressive Strength Test Results for different percentage variation of admixed Zycosil+

### *3.2. SPLIT TENSILE STRENGTH*

The split tensile strength values of concrete mixtures at various curing ages are shown in Table 4. There is no significant change in the tensile strength of various mixes based on the percentage of the silane. The results of the various ages showed that there is no particular trend found in the tensile strength of the concrete mixes with a different percentage variation of silane.

**Table 4.** 7-day, 28-day and 56-day Tensile Strength

| Percentage of Silane | 7-day Strength (N/mm2) | 28-day Strength (N/mm2) | 56-day Strength (N/mm2) |
|---|---|---|---|
| **0** | 2.16 | 3.70 | 3.84 |
| **1** | 2.54 | 2.86 | 2.94 |
| **2** | 1.97 | 2.60 | 2.60 |
| **3** | 2.45 | 2.49 | 2.49 |
| **4** | 2.38 | 2.46 | 2.46 |
| **5** | 2.26 | 2.25 | 2.43 |
| **6** | 2.16 | 2.15 | 2.11 |
| **Surface coated samples** | 2.14 | 3.65 | 3.81 |

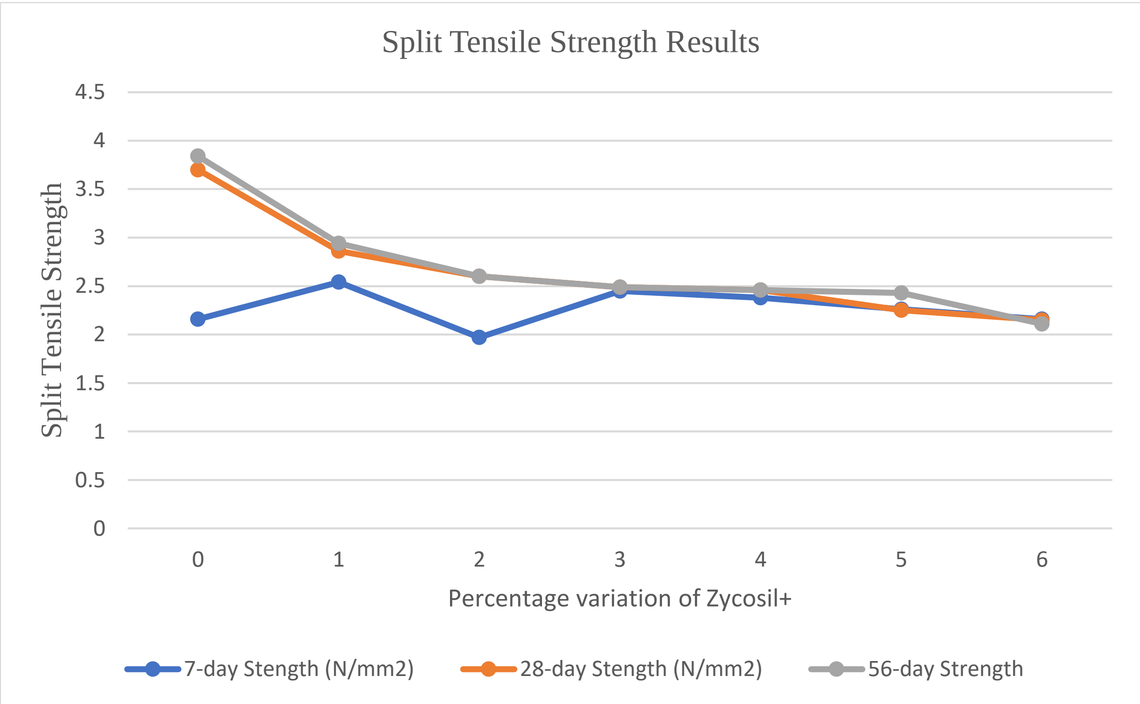


**Figure 11.** Split Tensile Strength Results

## *3.3. SORPTIVITY TEST*

No particular pattern was observed in the sorptivity values of the various mixes. The values obtained are shown in Table 5.

**Table 5.** Sorptivity value

| Percentage of silane | Change in the mass (in g) | Absorption (in mm) |
|---|---|---|
| **0** | 16 | 20.38 |
| **1** | 18 | 22.90 |
| **2** | 14 | 17.83 |
| **3** | 19 | 24.20 |
| **4** | 13 | 16.56 |
| **5** | 20 | 25.46 |
| **6** | 15 | 18.35 |

## *3.4. SESSILE DROP TEST*

The WCA values obtained in the integral hydrophobic modification technique are shown in Table 6. It is observed that the WCA values for all integrally modified samples are less than 90o, indicating that the concrete is hydrophilic. For the surface modification method, the contact angle is found to be more than 90o, indicating that the surface of the concrete shows hydrophobicity. For the double and triple coating of the silane, the WCA values increased slightly, as shown in Table 7. Further, the ratio of the Zycosil+, Zycoprime, and water is changed to see the change in WCA values; but the change in the water contact angle is not very significant.

**Table 6.** WCA for integral modification

| Percentage of silane | WCA |
|---|---|
| **0** | 45.1º |
| **1** | 44.3º |
| **2** | 46.2º |
| **3** | 50.6º |
| **4** | 37.9º |
| **5** | 40.3º |
| **6** | 38.3 º |

**Table 7.** WCA for surface coating on specimens

| S.No. | Type of coating | WCA |
|---|---|---|
| **1** | Single coating | 150.2º |
| **2** | Double coating | 153.5 º |
| **3** | Triple coating | 155.3 º |

**Table 8.** WCA for surface coating- Ratio variation

| S.No | Zycosil+:Zycoprime:Water | WCA |
|---|---|---|
| 1 | 0.5:2:20 | 156.9° |
| 2 | 1:2:20 | 150.2° |
| 3 | 2:2:20 | 157.1° |
| 4 | 3:2:20 | 153.5° |

## 4. CONCLUSION

The water ingress in the concrete is the cause behind all major durability issues, which is the inspiration behind the current experimental investigation. Here, an organosilane-based commercial compound is used to induce hydrophobicity in the concrete by both integral hydrophobic modification as well as surface modification. In the case of the integral hydrophobic modification, the WCA achieved is less than 90˚ for all silane percentages indicating that the concrete is still hydrophilic after silane modification. On the other hand, the WCA for the hydrophobic surface method is found to be greater than 150˚ indicative of concrete surface showing superhydrophobicity. A WCA as high as 157.1˚ was successfully obtained by surface modification for a Zycosil+: Zycoprime: Water ratio of 2:2:20. It was observed that the surface-coated samples exhibit self-cleaning properties with high WCA values, and the WCA values are found to increase with a double and a triple coating, although the increase was not very significant.

The results of the experimental investigation reported a haphazard trend for the 7 days compressive strength and tensile strength of all mixes with different percentage variations of the silane compound, whereas the 28-day and the 56-day compressive and tensile strength of various mixes decreased compared to the control sample and this decrease is directly proportional to the amount of silane incorporated in the concrete. This can be attributed to the fact that the silane-based admixtures interfere with the hydration reactions of cement composites. In the case of sorptivity, the change in values for the various mixes is not significant compared to the control samples.

Hence, from this study, it can be concluded that satisfactory results were not obtained from the point of view of hydrophobicity for integral hydrophobic modification, and the concrete, after modification, still remained hydrophilic. Whereas the surface modification method proved highly effective, yielding a superhydrophobic self-cleaning concrete surface, and thus it can be used effectively to protect concrete from water ingress and improve its durability of the concrete.

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This work was carried out in collaboration among all authors.

CONFLICT OF INTEREST

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