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Study on Superhydrophobic Coatings for Concrete Surfaces

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Abstract: ***Concrete is one of the most versatile and important materials in civil engineering constructions. It is being used from ordinary platforms to many mega- structures. Durability of this important material is still a matter of great concern among civil engineers all over the world. Durability of concrete is affected mostly by intrusion of aggressive species from the immediate environment. Therefore it is an absolute necessary to block the pores, voids, capillaries, interconnecting pores and other surface defects of concrete surfaces. This can be achieved by proper coatings, particularly by superhydrophobic coatings (lotus leaf coating). In this study superhydrophobicity is achieved by using Silane, Siloxane, Silicone as binder with Nanosilica, Silica fume, Micro silica, TiO_2, etc. Twenty eight different coatings were formulated and applied on concrete surfaces to study the hydrophobicity and superhydrophobicity. The best coatings were selected based on contact angles and roll on of water droplets and they never stick to the concrete surface. These coated surfaces are photo and video graphed and well established and discussed in detail.***

Keywords: Hydrophobic, Superhydrophobic, Durability, Coatings, Nanosilica, Pores

1. Introduction

Concrete plays a vital part in our daily life. Concrete also plays an important role in construction field because it can be cast in to virtually any shape or form, which allows for design freedom and an almost infinite variety of applications. But, there occurs penetration of water and other aggressive materials in to the concrete due to its porous nature which in turn causes expansion, cracking, scaling and crumbling in concrete. This paper mainly describes to reduce absorption of water and other materials into concrete and also to increase the durability of concrete. There were several methods to make the concrete as the repellent one, in which superhydrophobic coating is the best. Both superhydrophobic and hydrophobic coatings were developed in this work. These coatings were applied on concrete surface by brush.

Different combinations of coatings were formulated using the materials such as silane, siloxane, etc., with and without additives such as nanosilica, silica fume, TiO_2, ZnO. Applying of such coatings, make the concrete as rough surface (due to formation of pillar and groove like structures on concrete surface. This rough surface is the main factor which makes the water droplets to flow out of the surface, thereby avoiding the penetration of water into the concrete which in turn makes it more durable.

In this paper, superhydrophobic and hydrophobic surfaces were created on different specimens such as square tiles, roof tiles and hollow core disc. And these coated specimens were subjected to different tests such as screening (by flowing of water on concrete surface), water droplet test, droplet absorption test, water absorption (core) test, contact angle measurement. By these methods the best superhydrophobic coatings were selected in this work.

2. Principle Aspects of Superhydrophobic

The hydrophobicity is the term which is defined as the ability of material to repel water and it depends on the contact angle value. The term contact angle denotes the angle between a drop of water and the surface of the material. Depends on the contact angle value the nature of surface of the material is classified in to three as shown in the fig 1.1.

1) If contact angle is greater than 90^0, then it is **hydrophobicity (>90^0)**.
2) If angle is less than 30^0 then it is termed as **hydrophilic (<30^0).**
3) If contact angle is greater than 150^0, then it is **superhydrophobic (>150^0).**

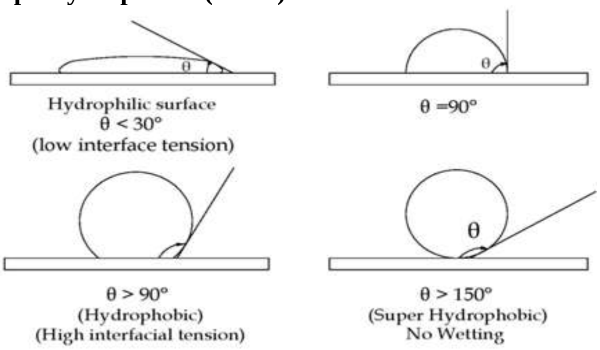

Figure 1.1: Hydrophobic, Superhydrophobic and Hydrophilic

3. Experimental Investigation

3.1 Materials

In order to investigate the effect of superhydrophobic coating, different type of specimens were cast and different types of combination of chemicals were selected for the purpose of coating on the specimen surface. The selected

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coatings include the type of water-repellent materials (silane, silicone emulsion, siloxane, etc,).

3.1.1 Preparation of water droplet test/droplet absorption/water contact angle specimens

For water droplet test, square tile of size 100x100x10mm were cast with cement mortar ratio of 1:3 and water cement ratio of 0.4. Twenty-eight numbers of square tiles were cast, cured and coated with different combinations. Droplet absorption test was developed in CSIR-CECRI. Roof tiles of size 225x225x15mm were selected and coated for water droplet test.

3.1.2 Preparation of specimen for water absorption test (core test)

Hollow core specimen of size 83x50mm with inner core size 40x25mm was cast with cement mortar ratio of 1:3 and water cement ratio of 0.4. Specimen was coated with different combination of chemicals. Water absorption (core) test was also developed in CSIR-CECRI.

3.1.3 Preparation of coating combinations

Various combinations were selected for superhydrophobic coatings by using the materials such as Silane, Siloxane, Latex and Silicone emulsion with and without additives such as Nanosilica, silica fume, TiO_2, Al_2O_3, ZnO. Twenty eight numbers of coating combinations were developed by using the above mentioned chemicals and additives.

3.2 Methods

Different methods of test were conducted for coated specimens. And through which the results were obtained.

3.2.1 Chemical combinations

1. Silane
2. Silane + Nanosilica
3. Silane + Nanosilica + TiO_2
4. Silane + Nanosilica + Al_2O_3
5. Silane + Nanosilica + TiO_2 + Al_2O_3
6. Siloxane
7. Siloxane + Nanosilica
8. Siloxane + Nanosilica + TiO_2
9. Siloxane + Nanosilica + Al_2O_3
10. Siloxane + Nanosilica + TiO_2 + Al_2O_3
11. Silicone emulsion
12. Silicone emulsion + Nanosilica
13. Silicone emulsion + Nanosilica + TiO_2
14. Silicone emulsion + Nanosilica + Al_2O_3
15. Silicone emulsion + Nanosilica + TiO_2 + Al_2O_3
16. Silicone emulsion + Silane
17. Silicone emulsion + Silane + Nanosilica
18. Silicone emulsion + Silane + Nanosilica + TiO_2
19. Silicone emulsion + Silane + Nanosilica + Al_2O_3
20. Silicone emulsion + Silane + Nanosilica + TiO_2 + Al_2O_3
21. Silicone emulsion + Silica fume
22. Silicone emulsion + Silica fume + TiO_2
23. Silicone emulsion + Silica fume + TiO_2 + ZnO
24. Latex
25. Latex + Nanosilica
26. Latex + Nanosilica + TiO_2
27. Latex + Nanosilica + Al_2O_3
28. Latex + Nanosilica + TiO_2 + Al_2O_3

3.2.1 Water droplet test

The Coatings were applied on the surface of square specimens of size 100x100x10mm. After allowed it to dry, the water droplets were poured on the surface of the coated specimens. The coated specimens were visually observed for movement of water droplet on concrete surface, and the best coatings were screened through this test. Same test was carried out on the roof tiles with best coatings.

3.2.2 Droplet Absorption test

The best coatings from the water droplet test were selected for this test. The water droplets were poured on surface of the coated specimens and covered with closed bottles to avoid the evaporation of water droplets. The specimens were visually observed for 24 hours and the size of the water droplets was examined.

3.2.3 Water Absorption (core) test

Cylindrical disc specimens of size 83x50mm with hollow core at the center of size 40x25mm were selected and coated with best coatings. Dry weight of the coated specimens was noted initially and then the hollow core region was filled with water as shown in fig 3.1. The decrease in water level in the hollow core due to water absorption was noticed at regular intervals by refilling the hollow core for every1 hour up to 24 hours

Figure 3.1: Measuring of weight of dry and filed specimen

3.2.4 Contact Angle Measurement

The water contact angles [5] of the specimens were determined by using Optical contact angle OCA 35 Goniometer as shown in fig 3.2. The water droplet was poured on the concrete surface of the coated specimen by the instrument. The volume of droplet is 8μl.

Figure 3.2: Goniometer

3.2.5 Sorptivity Test (ASTM C 1585-13)

The cylindrical specimens of size 50 mm x 80 mm were cast for Sorptivity test. The specimens were coated with best coatings. The dry weight of the coated specimen was noted.

This test is conducted to measure the capacity of the coated specimen to absorb liquid by capillarity; the weight of the coated specimen was measured for 1 hour by using weighing machine.

3.2.6 Chloride Ion Diffusion Test(ASTMC C 1202-12)

This test method covers the determination of the electrical conductance of cement mortar to provide a rapid indication of its resistance to the penetration of chloride ions. The test method consists of monitoring the amount of electrical current passed through 50mm thick slices at 83mm diameter cylinder specimens during 6 hours period. A potential difference of 60 volt direct current is maintained across the ends of the specimen one of which immersed in a 3% Sodium Chloride solution and the other in a 0.1 M Sodium Hydroxide solution.

3.2.7 TAFEL Extrapolation Method

The lollipop specimens of size 40mm diameter and 65mm height were made with placing steel plates in the centre. These casted specimens were exposed to 3% Sodium Chloride solution. All the lollipop specimens were subjected to Tafel Extrapolation test for 0th, 5th, 10th, and 25th day. It is the method to calculate the corrosion rate.

4. Results and Discussions

4.1 General

Moisture and water penetration along with aggressive species into concrete/mortar will cause deterioration of concrete/mortar. The best and foremost protective mechanism against penetration of moisture or water is surface coating. Even some polymeric coatings may fail due to membrane absorption of water and as a consequence further penetration of water into the concrete. But, if it totally prevent the contact of moisture or water from the surface of concrete/mortar will totally reset in highly durable material with longer service life. Keeping this in mind, hydrophobic and superhydrophobic coatings were developed. This will form as a thin layer between the environment and the concrete/mortar. These coatings will provide top rough surface which will carry the droplets by not allowing the water to just come and contact with the surface. In other words, this will form as the surface like "LOTUS LEAF". So there the question of contact of water with the surface doesn't rise at all.

In this study, a twenty eight numbers of specially formulated coatings for superhydrophobic nature were developed and were subjected to various tests for its durability and applicability.

4.2 Water Droplet Test

The formulated superhydrophobic coatings were applied on (100 x 100 x 10 mm) sized square mortar panel and left for drying a day. A next day water droplets were placed on the horizontal surface of the coated panels and shacked horizontally. It is found that some coatings were absorbed the water droplet and many other coatings showed the droplets were rolling on the horizontal surface in the spherical form itself. This indicated high hydrophobicity and superhydrophobicity nature of the coatings number 2,4,11,12,13,14,15,17,18,19,20,21 and 23 (13 no's) as hydrophobicity and superhydrophobicity. The selected best coatings were showed in the table 4.1.

This test was conducted on burnt clay roof tiles and water was poured on the surface of the coated specimens. When water was poured, the ordinary uncoated roof tiles absorbed water in plenty, whereas the coated tiles did not absorb any water and it was expelling quickly.

4.3 Droplet Absorption Test

The best coatings (13 no's) selected were subjected to droplet absorption test. This test was conducted by placing a water droplet on the coated surface and a plastic bottle was covered a droplet with air tightness. It was allowed for a day for the water droplet to come in contact with the surface ad for further absorption by the mortar panel. It was absorbed even after 24 hours of exposure, the following coatings have never showed any loss of water or absorption if water droplet 17, 20, 14, 23, 18, 19 and 21 shown in table 4.2. These seven coatings were subjected to further studies. The ranking can be assessed as 17, 20 and 14 are the best.

Table 4.1: Results of Water Droplet Test

S. No	Actual Discription Number	Combinations
1	2	Silane + Nanosilica
2	4	Silane + Nanosilica + Al_2O_3
3	11	Silicone emulsion
4	12	Silicone emulsion + Nanosilica
5	13	Silicone emulsion + Nanosilica + TiO_2
6	14	Silicone emulsion + Nanosilica + Al_2O_3
7	15	Silicone emulsion + Nanosilica + TiO_2 + Al_2O_3
8	17	Silicone emulsion + Silane + Nanosilica
9	18	Silicone emulsion + Silane + Nanosilica+TiO_2
10	19	Silicone emulsion + Silane+Nanosilica+ Al_2O_3
11	20	Silicone emulsion+Silane+Nanosilica+TiO_2+Al_2O_3
12	21	Silicone emulsion + Silica fume
13	23	Silicone emulsion + Silica fume + TiO_2 + ZnO

Table 4.2: Results of Droplet Absorption Test

S. No	Actual Discription Number	Combinations
1	17	Silicone emulsion + Silane + Nanosilica
2	20	Silicone emulsion + Silane + Nanosilica + TiO2 + Al2O3
3	14	Silicone emulsion + Nanosilica + Al2O3
4	23	Silicone emulsion + Silica fume + TiO2 + ZnO
5	18	Silicone emulsion + Silane + Nanosilica + TiO2
6	19	Silicone emulsion + Silane + Nanosilica + Al2O3
7	21	Silicone emulsion + Silica fume

4.4 Water Absorption Test (Hollow Core)

This test has been conducted in a specially cast hollow core specimens, in which 83mm diameter X 50 mm thick circular discs were cast and centrally on one side 40mm diameter X 25mm depth core was made using a PVC pipe, this forms the hollow core. After this specimens were set, cured and dried. The selected coatings (7 no's) were applied inside the core on cylindrical surface and horizontal surface. Initially the core was filled with water and measured how much ml of water was required for filling the hollow core. Then, this specimen along with water allowed for water absorption in both vertical and radial directions.

After certain interval of time, the water was again filled to make it level in the core. So this quantity of water filled is equal to the quantity of water absorbed with that particular time. This was carried out to a period of 48 hours. The results were showed in table 6.3. Out of seven coatings, six coatings were selected as best and ranked them as shown in table 4.3.

4.5 Measurement of contact angle

Contact angle of a droplet determines the coating is hydrophobic or superhydrophobic in nature. The contact angle up to 150^0 from the horizontal surface is called hydrophobic and degree beyond 150^0 is called superhydrophobic. There were nine coatings which performed best in water droplet test were subjected to measurement of contact angle. The contact angle of a coating was measured using an instrument called "Goniometer".

The contact angle is measured by providing a water droplet of 8ml volume through an injection over the coated surface, and then the instrument is adjusted to measure the contact angle from the horizontal surface. The droplet is being displayed in the monitor with its contact angle on its left hand top corner. For each specimen 3-4 times repeated to arrive at mean value of contact angle as shown in table 4.4. The highest contact angle shows, the best is the coating.

Table 4.3: Result of Water Absorption Test (Hollow Core)

Time (hrs)	Quantity of Water Added IN ml						
	CONTROL	17	20	14	23	18	19
Initial	42	37	35	34	35	45	40
1	11	-	-	1	2	-	-
2	2	-	1	2	5	-	1
3	4	-	1	-	6	-	2
4	3	-	-	3	7	-	4
5	1	-	-	-	7	-	2
6	-	-	-	-	-	-	-
7	1	1	-	2	8	1	6
8	2	1	2	3	5	1	-
24	21	3	3	14	24	3	30

Table 4.4: Contact Angle Value

Specimen No	Contact Angle			Final Result
	Trial 1	Trial 2	Trial 3	
2	160.3	161.2	154.1	161.2
12	162.9	152.5	155.8	**162.9**
14	162.0	155.1	156.1	162.0
17	159.8	164.5	161.2	**164.5**
18	157.7	154.7	154.3	157.7
19	152.9	154.7	157.7	157.7
20	160.2	159.8	162.9	**162.9**
21	164.8	161.7	156.7	**164.8**
23	159.1	157.0	157.5	159.1

4.6 Sorptivity (ASTM C 1585-13)

Sorptivity test was conducted with the help of mortar specimens of size 50 mm diameters X 80mm height. One control specimen plus from bottom and side coated specimens were placed in 5mm depth of water in a glass tray. Before starting of the test, the coatings were dried completely and initial weight was measured with the help of the digital balance. The initial weight before placing in water is noted and this specimen were placed in water. Then after 5 minutes, this specimen is taken out and bottom portion is wiped with cotton and again measured in the digital balance. This test is conducted with certain time interval as mentioned in the table 4.5 up to a period of 60 minutes. The quantity of water absorbed during 60 minutes indicates the capacity of the material in capillary rise against gravity. The lower value showed the best material. Subsequently the coefficient of absorption of water (K_a) was calculated for all selected samples using the formula 4.1,

$$Ka = (Q/A)^2 \longrightarrow x\ (1/t) \qquad 4.1$$

Where,
Ka = Coefficient of water absorption
Q = Quantity of water absorbed by the oven dried specimen in time
T = 60 minutes
A= Total surface area of concrete through which water penetrates

This test, coating number 18, 20, 19 and 21 are the best. This is quite good in agreement with contact angle measurement. The graphical representation of Sorptivity results are shown in Fig 4.1

Table 4.5 Sorptivity test result and Coefficient of water absorption (K_a) for selected coatings

Specimens No	Time Interval (Final - Initial) (in ml)	K_a ($x10^{-12}$)
17	4.92	1744
20	0.14	1.410
14	0.73	38.40
23	1.93	26.83
18	0.13	1.220
19	0.23	2.080
21	0.53	20.20

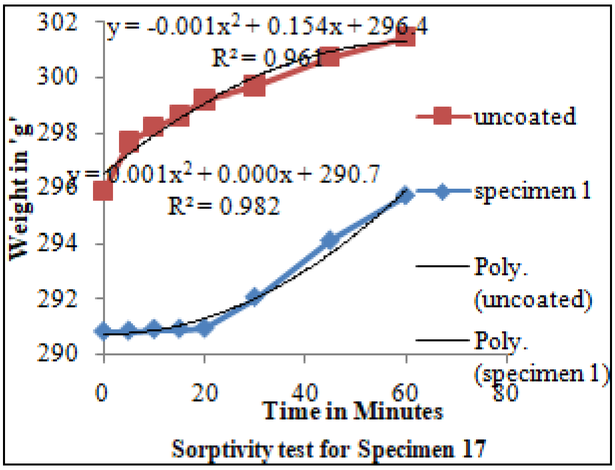

Figure 4.1: Sorptivity graph for Specimen 17

4.7 Chloride ion diffusion test (ASTMC C 1202-12)

Chloride ion penetration test has been conducted as per ASTMC C (1202-12). This test comprises of two PVC cells are of each capacity around 900ml. A disc mortar specimen one side coated with selected coatings is placed in the two PVC cells. And in each cell compartment titanium metal electrode are placed. The coated face is facing the compartment is filled with 0.1 molar NaOH solution.

The concrete specimen and the PVC cells are tightly connected without any water leak using M-Seal. The Sodium Chloride cell is connected to negative terminal and other compartment with NaOH solution is connected to positive terminal. A voltage of 60V DC is applied across the cell and the cell current is measured separately for each cells and recorded for computation of charge passed. The current is applied using power source. The test is conducted for 6 hours duration.

At a time period of every 30 minutes, the cell current is measured and recorded by using high impedance multimeter. The corresponding computation of charge passed is given in the table 4.6.

It is found from the results coating number 17 has 97 coulombs and for 20 has 121 coulombs and for 14 has 143 coulombs as against uncoated specimen as the value of 2916 coulombs. From this test, no 17 is found to be best.

Table 4.6 Results for chloride ion diffusion

Specimen No	Charge passed (in coulombs)	Remarks	Ranking
Control	2916	Moderate	-
17	97	Negligible	I
20	121	Very low	II
18	134	Very low	III
14	143	Very low	IV
23	156	Very low	V
21	162	Very low	VI
19	248	Very low	VII

4.8 TAFEL Extrapolation

This test carried out with the help of electrochemical analyzer called "AUTOLAB" attached with three electrode system configuration of electrochemical cell. The three electrodes are working electrode (WE), counter electrode (CE) and reference electrode (RE) (Ag, Agcl). And 3.5% Nacl solution was used as an electrolyte. A cylindrical mortar specimen fully coated with selected coatings (17, 20) with centrally placed 10mm tar steel as WE, a circular perforated stainless steel plate was used as CE. This three electrodes were connected to AUTOLAB (AUTOLAB is galvano static/potential static) instrument. A built-in software works for drawing anodic and cathodic curves on selection of tangent points on anodic and cathodic tangents are drawn by the computer and E_{corr} and I_{corr} values are automatically generated in addition to current density (A/cm^2) and corrosion rate, R (mm/yr).

This test was conducted for one control specimen and two coated specimens namely 17 and 20. The experimental setup is shown in fig 4.2. And the result of the test is presented in table 4.7. It is understood, coating no 17 found to posses lowest corrosion rate of 9.839 x 10^{-2} mm/yr as against the corrosion rate of control specimen 5.466 x 10^{-1} mm/yr on 25th day. To conclude, in almost all the test the coating no 17 found to be the best among the 28 formulated coatings.

Table 4.7: Corrosion Rate for best coatings

Day	Specimen	I_{corr} (A/cm^2) X 10^{-5}	Corrosion Rate, R (mm/yr) X 10^{-2}
0th	Control	1.527	4.997
	17	0.193	0.633
	20	1.421	4.650
5th	Control	16.86	55.16
	17	1.826	5.975
	20	1.943	6.945
10th	Control	34.47	112.8
	17	4.045	13.24
	20	4.704	15.39
25th	Control	16.71	54.66
	17	3.007	9.839
	20	4.678	3.967

Figure 4.2: Experimental setup of TAFEL Extrapolation

1. Conclusion

- By screening test (Water Droplet Test), it is found that specimens number 2, 4, 11, 12, 13, 14, 15, 17, 18, 19, 20, 21 and 23 (13 no's) act as hydrophobic and superhydrophobic. In these coatings, the water droplets were rolled on the horizontal surface. These indicate the development of hydrophobic and superhydrophobic nature on the coated surface.
- The seven coatings (17, 20, 14, 23, 18, 19 and 21) have never showed any loss or absorption of water droplet in Droplet Absorption Test. These seven coatings were subjected to further studies.
- Out of seven coatings from Droplet Absorption Test, six coatings (17, 20, 14, 23, 18 and 19) were selected as best in Hollow Core Absorption Test.
- The contact angle was measured for selected best coatings. The highest contact angle value shows the best coating. The selected best coatings show the contact angle greater than 150^0, this indicate the surface is superhydrophobic in nature.
- The specimen numbers 18, 20, 19 and 21 taken as the best coatings from the Sorptivity test. This is quite good in agreement with contact angle measurement values.
- From the Chloride ion diffusion test, the specimen numbers 17, 20 and 14 are found to be good.
- From TAFEL Extrapolation graph, it is understood that coating number 17 found to posses lowest corrosion rate.
- The combination of **silicone emulsion** and **silane** with additives such as **Nanosilica, TiO_2, Silica fume** and **Al_2O_3** showed good results as superhydrophobic nature.
- To conclude, in almost all the test the coating number 17 found to be the best among the 28 formulated coatings.

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