



Enhancing Road Durability and Safety: A Study on Silica-Based Superhydrophobic Coating for Cement Surfaces in Road Construction

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Abstract

This study explores the application of a silica-based superhydrophobic coating to enhance the durability and safety of cement surfaces in road construction. The coating formulation comprises triethoxy(octyl)silane, nano-silica particles, a coupling agent, epoxy resin, and diethylenetriamine. The preparation method involves spraying the solution onto the cement surface, offering a significant advantage in terms of reduced preparation time compared to traditional methods. The coating achieves a high-water contact angle (WCA) of 161°, exhibiting excellent water-repellent properties and significantly reducing surface degradation. Scanning electron micrograph (SEM) and Fourier transform infrared spectroscopy (FTIR) were also performed revealing the presence of Si-O-Si bonds, which are typical of siloxane structures. Additionally, a blade scratch test demonstrated the mechanical robustness of the coating. Even after scraping the surface, the coating retained its superhydrophobic properties, evidenced by a WCA of 151°, indicating that the coating can withstand mechanical damage while maintaining its water-repellent functionality. The results highlight the potential of this innovative coating to improve road longevity and safety by effectively mitigating water-related damage.

Keywords: Superhydrophobic coatings; Silica nanoparticles; Triethoxy(octyl)silane; Water contact angle; Road construction.

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1. Introduction

Concrete, renowned for its robustness and longevity in the construction sector, is susceptible to strength degradation from water infiltration.^[1] One of the inherent drawbacks of concrete is its susceptibility to environmental factors such as water infiltration, freeze-thaw cycles, and chemical attacks. These factors can significantly degrade the performance and longevity of concrete roads, leading to increased maintenance costs and reduced service life.

Hydrophilicity of concrete explains by its composition,

because it typically consists of coarse aggregate, sand, water, and cement, all of which are hydrophilic. Consequently, water and corrosive ions can easily infiltrate the interior of concrete and as a result corrode the reinforcing rebar, reducing its strength.^[2-5] More critically, the expansion caused by rebar corrosion can further damage the concrete structure, leading to bursting, flaking, and a loss of load-bearing capacity. Since water serves as the medium for ion transport during this corrosion process, the main measure to extend the lifespan of concrete is to prevent water from penetrating it.^[6]

To address these challenges, innovative surface treatments and coatings have been developed to enhance the durability and functionality of concrete. Over the past few years superhydrophobic coatings for building materials garnered significant attention due to their anti-fouling,^[7] corrosion-resistant,^[8] and ice-repellent properties.^[9,10] Among these, superhydrophobic nano SiO₂-based coatings have emerged as a promising solution, where SiO₂ nanoparticles play a critical

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role in achieving the desired hydrophobic properties.^[11,12] Superhydrophobic materials are characterized by a contact angle greater than 150° and a sliding angle less than 10°.^[13] Inspired by the water-repellent properties of lotus leaf,^[14] butterfly wings,^[15] superhydrophobic coatings have been effectively utilized in applications such as aircraft,^[16,17] metal surfaces,^[18] oil/water separation^[19] and power transmission systems,^[20] Enhanced Oil Recovery (EOR),^[21,22] construction materials.^[23-25] In the realm of deicing for cement pavements, the superhydrophobic coating can be directly sprayed onto the surface without altering the structure of cement.^[26] It is effective through three main approaches: repelling incoming water droplets before they freeze,^[27] inhibiting ice formation during freezing,^[28] and reducing ice-pavement adhesion after freezing,^[29] all while requiring minimal energy consumption. Hydrophobicity is influenced by the chemical characteristics of the materials, also known as surface energy, as well as a hierarchical topographical or surface roughness structure on the nano to micro scale.^[30]

Nanoparticles are often used to generate the necessary roughness for superhydrophobic surfaces^[31] and this is explained by the fact that the presence of air trapped on this rough surface can minimize the contact between solid and water, a characteristic known as the Cassie-Baxter state.^[32,33] This is crucial in road construction, where water repellency can prevent water infiltration, thus reducing the risks of road surface deterioration due to freeze-thaw cycles and water-induced erosion.^[34,35] Role of SiO₂ nanoparticles in the preparation of superhydrophobic coatings in road construction is in such qualities as surface roughness enhancement, durability and mechanical stability, self-cleaning properties.^[36-41] Based on this, SiO₂ nanoparticles are used to create the micro- and nanoscale roughness necessary for superhydrophobicity.

For instance, Alfieri *et al.* demonstrated the creation of a hybrid sol consisting of TiO₂ and SiO₂ nanoparticles, treated with a fluoroalkyl-functional water-based oligosiloxane in an aqueous solution, and applied onto bricks. Despite the surface hydrophobicity of this TiO₂-SiO₂ coating diminishing under UV exposure, it regained its hydrophobic properties in the absence of light, while maintaining its photocatalytic capabilities.^[42]

She *et al.*, established superhydrophobic surfaces (SHS) on concrete by directly spraying nano-silica gel treated with low surface-energy surfactants.^[43] This surface treatment can also be applied to microscopic cracks in concrete, which enhances its durability. Coatings can confer superhydrophobic properties onto concrete.

Silicon-based compounds such as silanes, siloxanes are

commonly employed as water-repellent agents in the construction industry.^[44,45] The alkyl groups within these compounds notably diminish the interaction between concrete and water. Herb *et al.*, revealed that superhydrophobic coating on concrete can be obtained by being immersed in the alkyltrialkoxysilane solution for 24 hours to facilitate the creation of intricate siloxane compounds, specifically silsesquioxanes.^[46] Sobolev and Batrakov^[47] employed admixtures based on polyethyl hydrosiloxane and polymethyl hydrosiloxane, which emit hydrogen to generate hydrophobic spaces within the concrete.

This paper explores the development and application of superhydrophobic nano SiO₂-based coatings on cement used in road construction. It examines the principles of superhydrophobicity, the synthesis and characterization of nano SiO₂ coatings, and their impact on the properties of concrete pavements. This paper aims to contribute to the advancement of innovative solutions for durable and resilient road infrastructure.

2. Materials and methods

2.1 Chemicals

Silica nanoparticles (Sigma-Aldrich, nanopowder spherical, porous, 5 - 15 nm particle size (TEM), 99.5% trace metal basis, MW: 60.08 g/mol), Triethoxy (octyl)silane (TEOS) (Sigma-Aldrich, technical, 97%. MW: 276.49 g/mol), Acetone (Sigma-Aldrich, ACS reagent, ≥ 99.5%. MW: 58.08 g/mol), Methanol (Sigma-Aldrich, for HPLC, ≥ 99.9%. MW: 32.04 g/mol), 3-glycidyloxy(propyl)octylsilane (Sigma-Aldrich, ≥ 98%. MW: 236.34 g/mol), DER 736 Epoxy Resin (Sigma-Aldrich, d: 1.14 g/mol), Diethylenetriamine (Sigma-Aldrich, ≥ 98%. MW: 236.34 g/mol), Poly(ethylene)glycol (PEG). The chemicals were used without further purification.

2.2 Superhydrophobic coating preparation

Superhydrophobic coating for concrete was prepared by a simple method as shown in Fig. 1. Triethoxy (octyl)silane (3ml), methanol (0.6ml) were mixed with similar amount of acetone and 3-glycidyloxy(propyl)octylsilane (3-GPTMS) - 0.3 ml. Subsequently, epoxy resin DER 736 and diethylenetriamine were added to the solution in ratio 2:1 to ensure the adhesiveness on substrate. Superhydrophobic SiO₂ nanoparticles were prepared by the methodology as described by Suiindik *et al.*^[48] Before adding the modified superhydrophobic SiO₂ nanoparticles which provide hydrophobicity and roughness on the surface, the main solution was stirred for 15 minutes at the temperature of 50 °C and continued stirring for additional 30 minutes with nanoparticles to obtain the superhydrophobic solution.

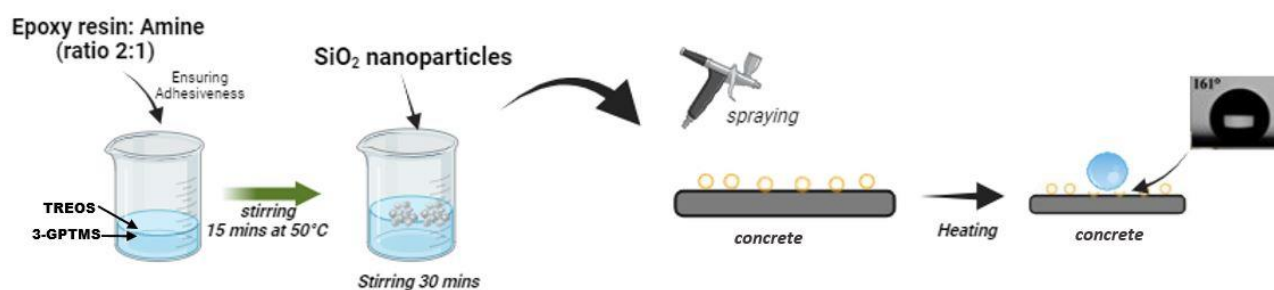


Fig. 1 Superhydrophobic coating preparation.

Thereafter, the prepared SHS was sprayed on the cement samples with pressure 200 kPa for 5 seconds keeping distance 15 cm between airbrush and substrate. The coated cement samples were left for 24 hours at room temperature and then dried for 2 hours at temperature 150 °C to activate hydrophobic properties of the components.

2.3 Materials characterization

The contact and sliding angles of water droplets on the samples were measured using the optical contact angle device (OCA 15 EC, Neurtek Instruments) at room temperature. The volume of water droplet was 10 µl. The surface micro-morphologies of the samples were observed using a scanning electron microscope (SEM) (JEOL JSM-IT200(LA)). The elemental composition and chemical groups of the sample surfaces were examined using FT-IR spectrometer (Nicolet iS10).

3. Results and discussion

3.1 Characterization

Figure 2 illustrates the FTIR characterization of the

superhydrophobic coating and shows various peaks corresponding to different functional groups present in the sample. The peak observed at 445 cm⁻¹ was associated with Si-O-Si bond, which is typical in siloxane structures,^[49] while the band at 783 cm⁻¹ is indicative of the presence of silicon-carbon bonds, often found in organosilicon compounds.^[50] Additionally, the peak at 1055 cm⁻¹ corresponds to the stretching vibrations of carbon-oxygen bonds (-CO). Furthermore, the absorption peak at 1454 cm⁻¹ (-CH₂) was assigned to the vibrations of methylene (-CH₂) groups, commonly present in hydrocarbon chains. The peak at 2921 cm⁻¹ is associated with the asymmetric stretching vibrations of methyl (-CH₃) groups.^[51] Additionally, the peaks observed at 3293 cm⁻¹ represents the stretching vibrations of hydroxyl (-OH) groups and it is typically broad due to hydrogen bonding. The FTIR spectrum therefore shows the presence of siloxane (Si-O-Si), organosilicon (Si-C), carbon-oxygen (C-O), methylene (-CH₂), methyl (-CH₃), and hydroxyl (-OH) groups, indicating a complex mixture of organic and silicon-based compounds.

Figure 3 shows the scanning electron microscope image of

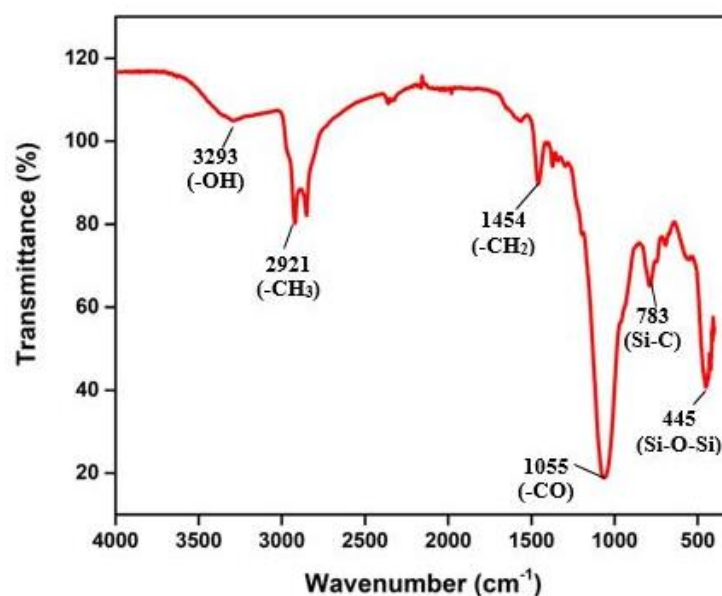


Fig. 2 FTIR characterization of the superhydrophobic coating.

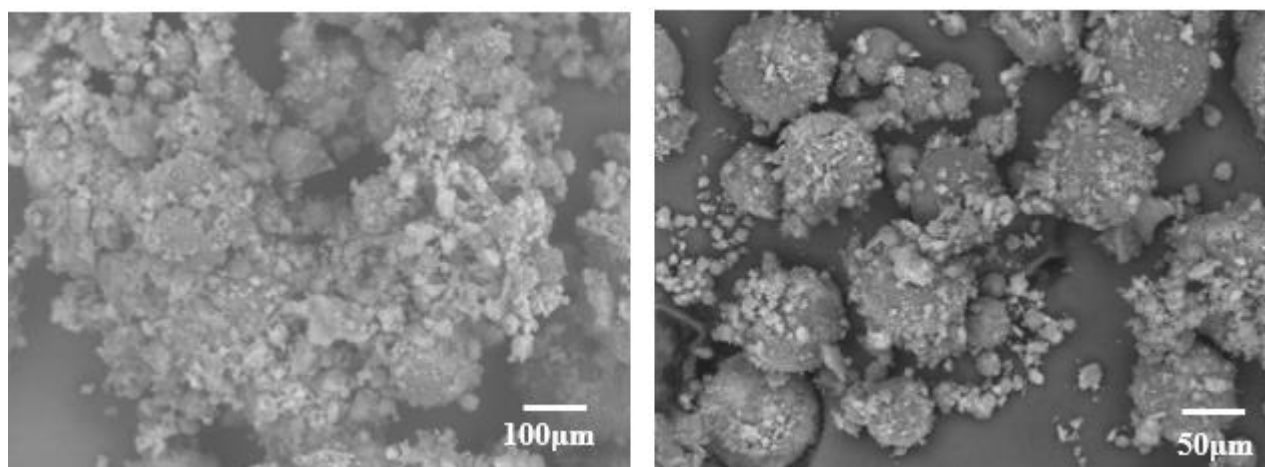


Fig. 3 SEM images revealing the morphology of the particles.

the superhydrophobic precipitate as aggregates of spherical or irregularly shaped particles. These particles are likely SiO_2 nanoparticles, which are a common component in SH solutions due to their ability to create a rough surface that enhances hydrophobic properties.

A standard sessile drop method was used to measure the WCA, where a calibrated micro-syringe was used to deposit a predetermined volume of distilled water on the cement surface. The angle formed at the water-microfiber interface was then

measured using a goniometer (OCA 15 EC, Neurtek Instruments). An amount of 10 μl water droplet was applied to the sample and the WCA was measured immediately. The WCA with water is crucial for understanding surface wettability. As we can see in Fig. 4, the superhydrophobic coated cement had a CA of 161° . This extreme superhydrophobicity allows water droplets to effortlessly detach from the concrete surface without leaving any residue, showcasing a characteristic water bouncing process, as

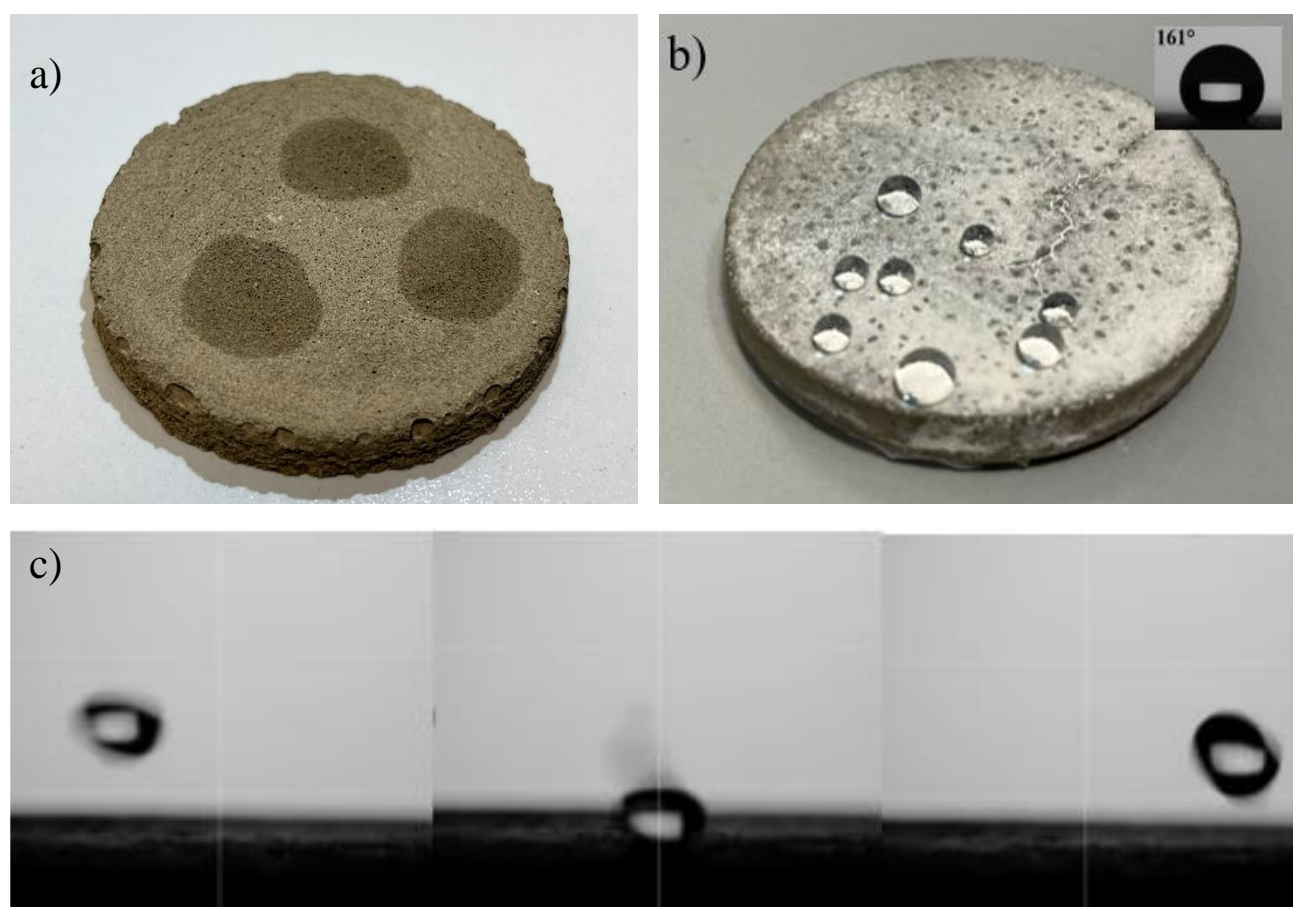


Fig. 4 Results of WCA showing (a) uncoated sample, (b) SH coated cement sample, and (c) Dynamic bouncing processes of water droplets on the superhydrophobic concrete (Video 1. is given in the supplementary material).

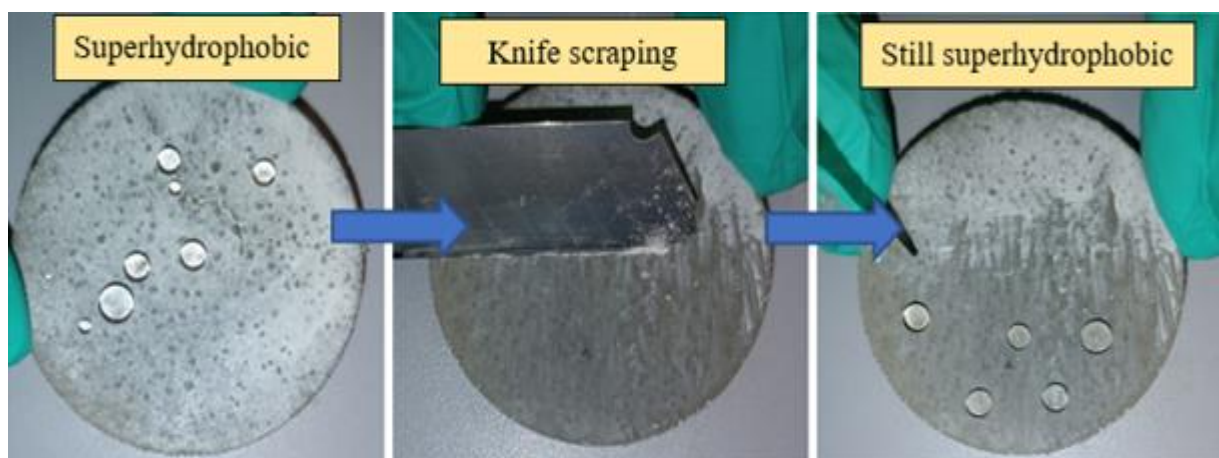


Fig. 5 The blade scratch processes of the SH-concrete coating.

illustrated in Fig. 4 and Video 1.

3.2 Mechanical surface strength test

A blade scratch test was conducted to thoroughly assess the mechanical strength and durability of the superhydrophobic (SH) concrete coating, as depicted in Fig. 5. During this test, a sharp utility blade was used to scrape the surface of the coating in a direction perpendicular to the blade's edge. The primary objective of this test was to evaluate how well the superhydrophobic properties of the coating could withstand such mechanical stress. By intentionally creating scratches and imperfections on the surface, the test aimed to determine if the SH coating could maintain its water-repellent characteristics despite the inflicted damage. The test also allowed for the observation of any changes in surface morphology or the shedding of particles, which could indicate the extent of the coating's resistance to mechanical forces.

The effectiveness of the coating was further analyzed by measuring the water contact angle on the damaged areas. The retention of a high WCA after the scratch test would demonstrate the robustness of the superhydrophobic effect, confirming that the coating remains functional and effective even when subjected to harsh conditions. This test is crucial in validating the coating's suitability for practical applications where durability and long-term performance are essential.

The superhydrophobic effect was specifically assessed by observing the point at which noticeable powder shedding occurred from the surface. Despite the deliberate damage inflicted by the blade, the surface continued to exhibit its superhydrophobic properties. This was evidenced by the formation of nearly spherical water droplets on the scratched surface. The water contact angle measured on this damaged surface was 151° , which is characteristic of a SH surface, as shown in Fig. 6. This result demonstrates that even after undergoing the blade scratch test, the coating retained its

superhydrophobic characteristics.

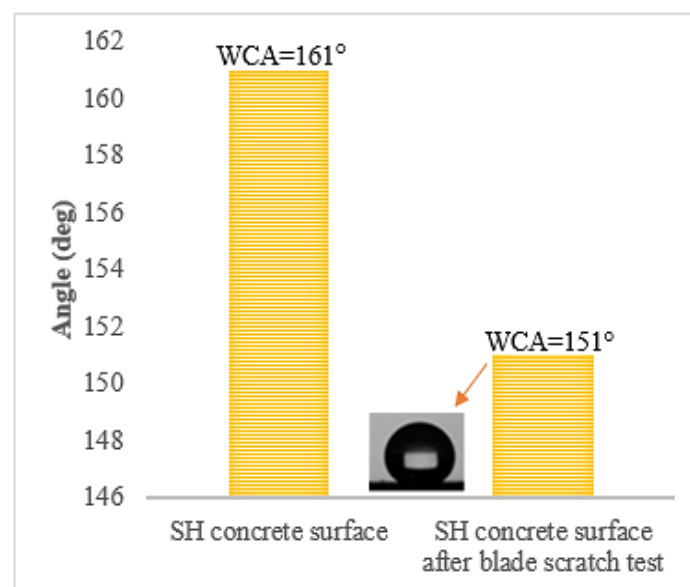


Fig. 6 The variation of WCAs of the SH coated concrete before and after blade scratch tests.

4. Conclusions

In summary, we have prepared a superhydrophobic SiO₂-based coating on a cement surface, which exhibits self-cleaning and water-repellent properties. TREOS was used as the silane coupling agent because, for areas with heavy mechanical wear, a TREOS-based coating might offer better long-term performance. Additionally, the preparation time of the SH solution was only 45 minutes, which is shorter compared to traditional methods and can be economically beneficial. The water contact angle of the coated cement surface was 161° , and after a blade scratching mechanical test, it remained superhydrophobicity with a WCA of 151° demonstrating the mechanical robustness of the coating and indicating that the coating can withstand mechanical damage while maintaining its water-repellent functionality. Moreover,

the dynamic bouncing of water droplets on the superhydrophobic concrete demonstrated the self-cleaning properties of the surface. FTIR spectroscopy was also performed revealing the presence of Si-O-Si bonds typical of siloxane structures. Overall, the SH coating exhibits excellent chemical composition, surface morphology, wettability, and mechanical strength, making it a promising candidate for applications requiring durable superhydrophobic surfaces. SH-coated cement can be used for roofs of buildings, road pavements, and other building materials, providing long-term durability, safety, and economic benefits.

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Conflict of Interest

There is no conflict of interest.

Supporting Information

Not applicable.

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