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RESEARCH ARTICLE

Fabrication of Superhydrophobic Coating by Spraying PDMS-SiO_2 Suspension for Self-Cleaning Application

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ABSTRACT: Superhydrophobic surfaces that mimic lotus leaf surfaces have been widely studied due to their unique self-cleaning ability. Herein, we have fabricated a self-cleaning superhydrophobic coating on a glass substrate using the facile sol-gel spray coating method. The silica particles were synthesized via the sol-gel method in acidic conditions. A self-cleaning superhydrophobic coating was achieved by spraying a suspension of synthesized SiO_2 particles and PDMS on a glass substrate at room temperature. The incorporation of SiO_2 particles in the PDMS matrix resulted in the formation of a rough structure on the glass surface. This surface exhibited excellent superhydrophobic properties with a high water contact angle (WCA) of 154° and a low sliding angle (SA) of 4°. In addition, the prepared samples showed strong mechanical resistance against various tests such as multiple adhesive tape peeling, sandpaper abrasion, and pencil hardness. As a result, the developed superhydrophobic coating holds great potential for self-cleaning applications on a larger scale.

Keywords: SiO_2-PDMS suspension, self-cleaning, superhydrophobic, spray coating

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1. INTRODUCTION

The Lotus leaf is renowned for its water-repellent properties, which are widely regarded for their exceptional self-cleaning abilities [1]. The superhydrophobic surface is defined by it's wetting character, which exhibits a static water contact angle (WCA) greater than 150° and a sliding angle (SA) smaller than 10° [2]. The superhydrophobicity on the lotus leaf surface occurs due to the presence of a thin waxy layer on micro-scale papillae [3]. Therefore, a geometrically rough surface structure and low surface energy materials is essential for fabricating a superhydrophobic surface on a solid substrate [4, 5]. The superhydrophobic coating is esteemed as a promising application across diverse domains in self-cleaning [6-8], oil−water separation [9, 10], corrosion protection [11, 12], and anti-icing [13, 14]. Number of techniques have been documented for producing superhydrophobic surfaces, including dip-coating [15], sol-gel method [16], and chemical vapor deposition [17], spray coating [18]. For example, researchers [17] have fabricated a translucent and superhydrophobic PDMS coating on a glass substrate by incorporating SiO_2 nanoparticles through an aerosol-assisted chemical vapor deposition method. Liu and co-workers [19] have prepared transparent superhydrophobic coating with excellent durability and chemical stability by dipping the glass substrate into the PDMS/SiO_2 composite. From a materials perspective, PDMS is the best option in coating research for its fluorine-free composition, excellent chemical stability, notable adhesion with the substrate, and hydrophobic nature [20]. Since the

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addition of SiO_2 nanoparticles in PDMS improves the hydrophobicity and mechanical stability of coatings [21]. Li et al. [22] have fabricated a robust superhydrophobic coating on a glass substrate by spraying a suspension of epoxy resin, SiO_2 nanoparticles, and hexadecyltrimethoxysilane (HDTMS). Guo et al. [21] have synthesized robust superhydrophobic coating using a suspension of polydimethylsiloxane (PDMS), and epoxy resin (EP) on substrates through facile spray-coating method. Superhydrophobic coatings possess a fine surface structure that tends to degrade upon mechanical contact, thus limiting their applications for long-term usage. Therefore, there is an urgent need to develop highly robust superhydrophobic coatings that can withstand wear and tear to ensure their long-lasting applications.

In this work, we proposed a simple spray-coating method to fabricate superhydrophobic coating on the glass substrate using SiO_2-PDMS composite for self-cleaning application. Initially, silica nanoparticles were synthesized by sol-gel process using tetraethylorthosilicate (TEOS) as a precursor. The synthesized silica nanoparticles were mixed in a certain ratio with PDMS solution, and stirred using a magnetic stirrer to get a homogeneous suspension. The prepared homogeneous suspension was sprayed on a glass substrate using a spray gun. The coating exhibits rough structure due to incorportaion of SiO_2 particles in PDMS matrix. The composite coating demostrated excellent superhydrophobicity and self-cleaning performance along with strong mechanical resistance.

2. EXPERIMENTAL DETAILS

2.1. Chemicals and materials

Tetraethyl orthosilicate (TEOS, 98%) was purchased from Sigma Aldrich (Bangalore, India). Polydimethylsiloxane (PDMS, viscosity 5 cSt) was procured from Sigma-Aldrich (St. Louis, MO, USA). Nitric acid (HNO_3), Ethanol (anhydrous, 99.9%), Hexane were bought from Spectrochem PVT. LTD (Mumbai, India). The micro-Glass substrates (75 ×25 ×1.35 mm^3) were obtained from Blue star, Polar Industrial Corporation (Mumbai, India). Distilled water was prepared in the laboratory and used as it is.

2.2. Synthesis of Silica particles

The silica nanoparticles were synthesized via sol-gel process using TEOS as silica source. In the typical synthesis process, aqueous acidic solution of HNO_3 was added dropwise in the TEOS-ethanol solution. The molar ratio of TEOS:ethanol: H_2O: HNO_3 was 1:2.67:2:0.67. The prepared solution was stirred for 1 h and left overnight for gelation. An opaque gel was kept in hot air oven at 150 ℃ for 12 h to dry gel completely. The dried gel was rigorously grounded with mortar and pestle to achieve fine silica powder. Finally, the silica particles kept in hot air oven at 100 ℃ for 24 h to completely evaporate the solvent residue.

2.3. Preparation of SiO_2-PDMS superhydrophobic coating

The glass substrates were thoroughly cleaned by sequential ultrasonication in 0.1 M HCl, 0.1 M NH_4OH, ethanol and distilled water for 10 min each. After cleaning, the glass substrates were kept in a hot air oven at 60 ℃ for 10 min and were then used for the fabrication process. To prepare the coating solution, 1 v/v% PDMS was dissolved in 20 mL of hexane under magnetic stirring for 1 h at 500 rpm. Synthesized silica particles were then added to the solution, which was stirred and ultrasonicated for 1 h each to obtain uniform dispersion of silica nanoparticles in the coating solution. The solution was sprayed on the glass substrate using a manual spray gun (Pilot Spray Gun 64 HSN: 8424) at a distance of approximately 15 cm through its nozzle orifice diameter of 1.6 mm. The spray coated glass substrates were then kept in a hot air oven at 150 ℃ for 2 h. To optimize the coating, the concentration of silica particles was varied at 0.6, 0.8, and 1 wt%, and labeled as PS1, PS2, and PS3, respectively.

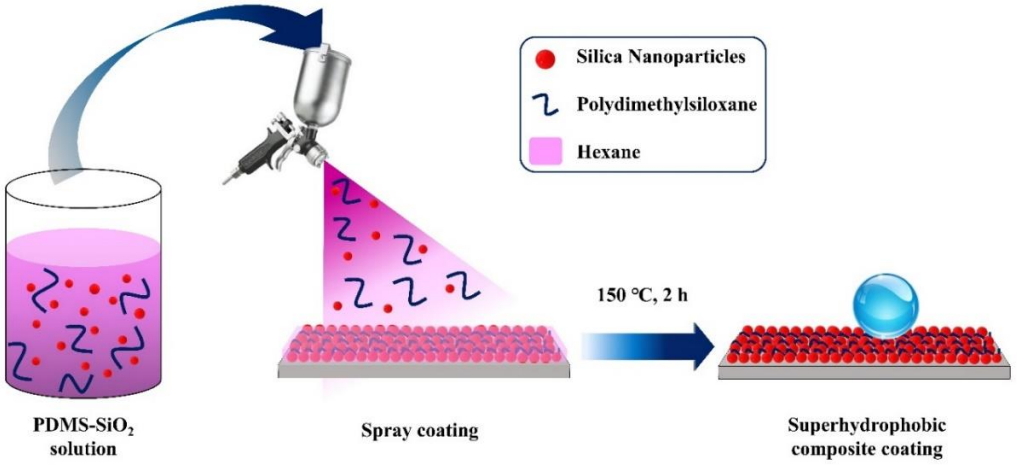

Fig. 1. Schematic of the fabrication of self-cleaning superhydrophobic coating on glass substrate.

2.4. Characterization

A Scanning Electron Microscope (SEM, JEOL, JSM-7610F, Tokyo, Japan) was used to investigate the surface structure of the prepared coatings. The surface roughness was determined using a Stylus profiler (Mitutoyo, SJ 210, Sakado, Japan). The average roughness value was determined by recorded at three different places. Chemical bonding was determined by Fourier Transform Infrared (FTIR) spectroscopy. The WCA and SA were measured at three different places on the samples using a contact angle meter (HO-IAD-CAM-01, Holmarc Opto-Mechatronics Pvt. Ltd., Kochi, India). The average value of the WCA and SA of samples were noted. The mechanical durability of the coatings will be evaluated by adhesive tape test, sandpaper abrasion test and adjustable pencil hardness tester (BGD 505, Biuged). The self-cleaning properties of the coating was determined using chalk powder as dust particles.

3. RESULTS AND DISCUSSION

3.1. Surface morphology and wettability

The surface morphology of the coatings is significantly influenced by the concentration of silica particles, which directly impacts their wettability. Scanning electron microscopy (SEM) micrographs presented in Figure 2(a-f) illustrate the surface structure of PDMS coatings with varying concentrations of silica particles (PS1, PS2, and PS3). The incorporation of SiO_2 into the PDMS matrix results in particle agglomeration, which then distributes across the glass surface during the deposition process. This distribution leads to the formation of a rough surface structure on the coatings, which is critical for achieving desired wetting properties.

For the PS1 sample, the SEM images reveal a relatively smoother surface with minimal roughness, which correlates with a water contact angle (WCA) of 138° and no sliding angle (SA). In contrast, the PS2 and PS3 samples show more pronounced surface roughness due to higher concentrations of SiO2 particles. The roughness of the PS2 sample, with an average value of 0.015 µm, combined with the intrinsic low surface energy of PDMS, significantly enhances its superhydrophobicity. The PS2 sample exhibited a WCA of 154° and an SA of 4°, indicating excellent water repellency. Similarly, the PS3 sample displayed a WCA of 151° and an SA of 7°, though slightly less effective than PS2.

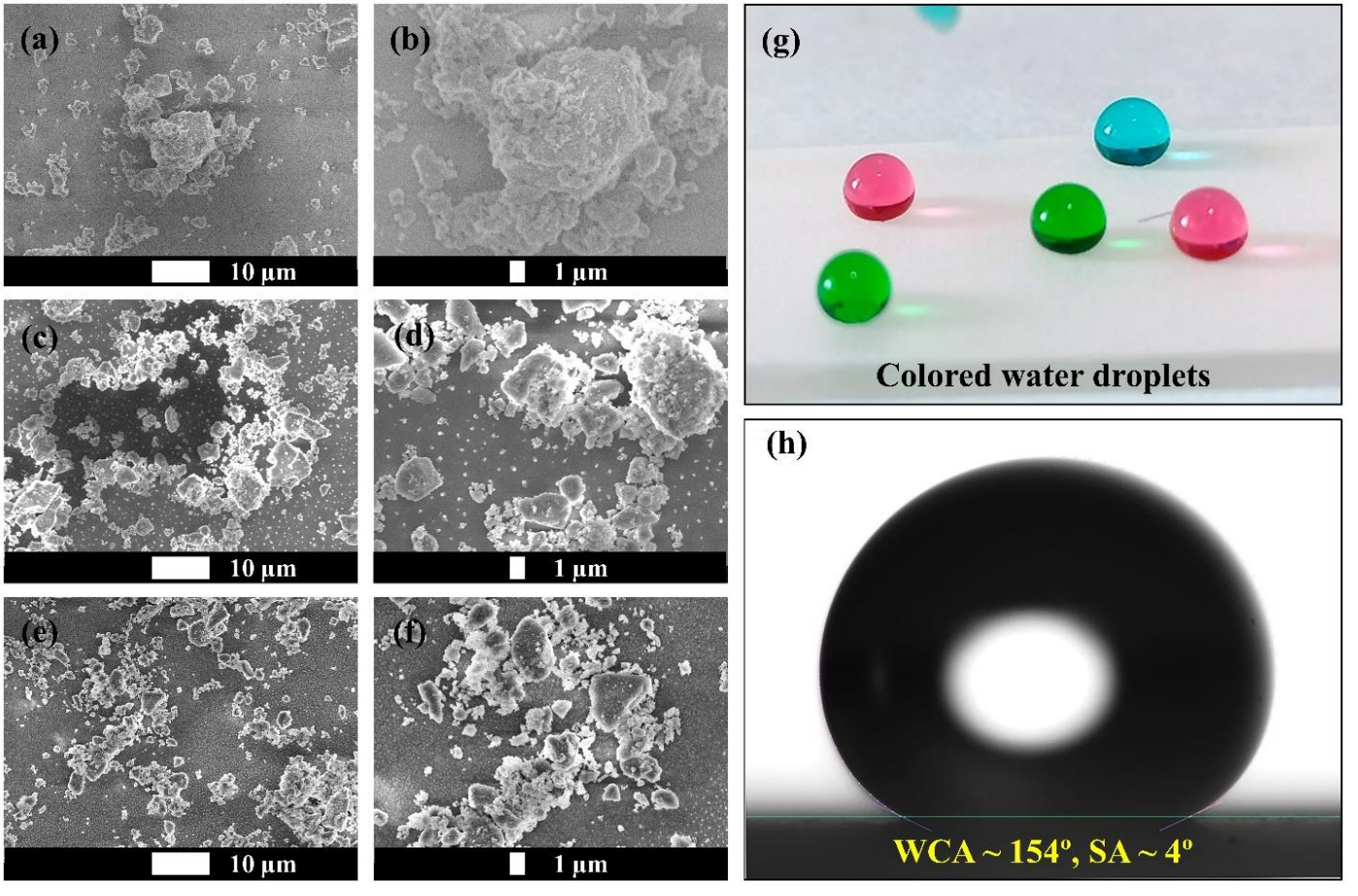

Fig. 2. SEM micrographs of (a, b) PS1, (c, d) PS2 and (e, f) PS3 with lower and higher magnification. (g) Optical image of colored water droplets on the PS2 sample. (h) Water contact image of the PS2 sample.

The improvement in wettability is attributed to the synergistic effect of the surface roughness created by the aggregated SiO_2 particles and the hydrophobic nature of the PDMS. The rough microstructure traps air pockets, reducing the solid-liquid contact area and allowing water droplets to bead up and roll off the surface easily. Figure 2(g) provides an optical image of colored water droplets on the PS2 sample, demonstrating the superhydrophobic effect visually. Additionally, Figure 2(h) shows the measured WCA on the PS2 sample, further confirming its superior wetting properties.

3.2. Chemical composition

The chemical composition of the PS2 sample was analyzed using Fourier-transform infrared (FT-IR) spectroscopy to understand the bonding and structure of the composite material. The FT-IR spectra, depicted in Figure 3, reveal several characteristic peaks that confirm the presence of PDMS and silica particles in the coating. The spectrum shows strong peaks in the range of 1100-470 cm^{-1}, which are attributed to the composite coating's primary constituents. The peak at 1571 cm^{-1} is indicative of Si-CH_3 bonding, a characteristic feature of the PDMS matrix. The prominent peak at 1033 cm^{-1}corresponds to the asymmetric stretching vibration of the Si – O – Si bond, which is a hallmark of the silica network. Additionally, peaks at 2913 cm^{-1} and 3192 cm^{-1} are associated with the C-H asymmetric and symmetric vibrations, respectively, further confirming the presence of organic components from PDMS [23].

These spectral features collectively affirm the successful integration of silica particles into the PDMS matrix. The Si–O–Si network contributes to the structural integrity and mechanical robustness of the coating, while the Si-CH_3 and C-H bonds enhance its hydrophobic properties [24]. The chemical bonding confirmed by FT-IR analysis is crucial for the coating's performance, ensuring that the composite material maintains its structural stability and desired superhydrophobic characteristics under various environmental conditions. The FT-IR spectra provide a comprehensive understanding of the chemical composition of the PS2 sample, highlighting the effective incorporation of silica particles within the PDMS matrix. This integration is vital for achieving the desired mechanical and hydrophobic properties essential for the coating's practical applications.

3.3. Mechanical stability

To evaluate the practical applicability in outdoor exposure, it is essential to determine the mechanical stability of the prepared samples. For this purpose, adhesive tape peeling test [25], sandpaper abrasion test [26], and pencil hardness test [27] were carried out. The adhesiveness of the prepared coating toward the substrate was evaluated by the adhesive tape test. An adhesive tape was applied to the PS2 sample, and a metallic disc weighing 20 g was rolled on it for 1 minute to remove the air gap between the substrate and tape. Afterward, the tape was torn off from the substrate, which was considered a cycle. The WCAs were measured after each cycle. It was observed that the WCA decreased to 113.72° after three cycles, indicating some detachment of the coating material, which adhered to the tape. The variation of WCA after each cycle is depicted in Figure 4(a), and the optical photograph of the experimental setup for the adhesive tape peeling test is shown in the inset of Figure 4(a).

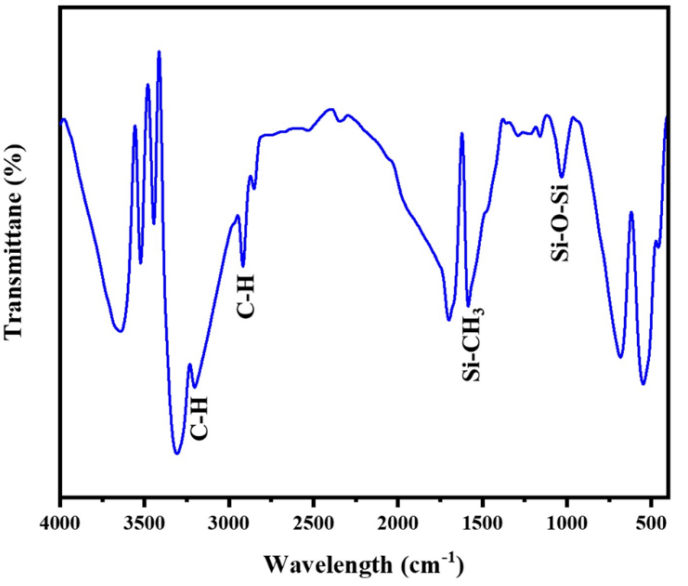

Fig. 3. FT-IR spectra of the PS2 sample.

In another mechanical stability testing method, the coated surface of the PS2 sample was placed on sandpaper (grit number 1500) with a 20 g weight loaded on top (Figure 4b). For one cycle of sandpaper abrasion, the sample was dragged for 10 cm at a normal speed. After each cycle, the WCA was recorded to evaluate the mechanical stability of the coating. Unfortunately, the WCA was reduced to 106.73° after ten cycles. Post-test, the coated surface from the sample appeared partially removed and observed on the sandpaper surface. The reduction in WCA after each cycle is depicted in Figure 4b, along with an optical image of the experimental setup in the inset.

During the pencil hardness test, a 6B pencil tip was used with a pressure of 500 g applied to the sample. The tester was dragged across the coating, and it was observed that the dragged area underwent complete removal, leaving no discernible coated layer (as shown in Figure 4c).

3.4. Self-cleaning performance

An intrinsic property of the self-cleaning superhydrophobic surface is its ability to repel water droplets, causing them to roll off the surface and carry away contaminants. To assess the self-cleaning performance of the PS2 sample, chalk powder was uniformly sprinkled over the surface.

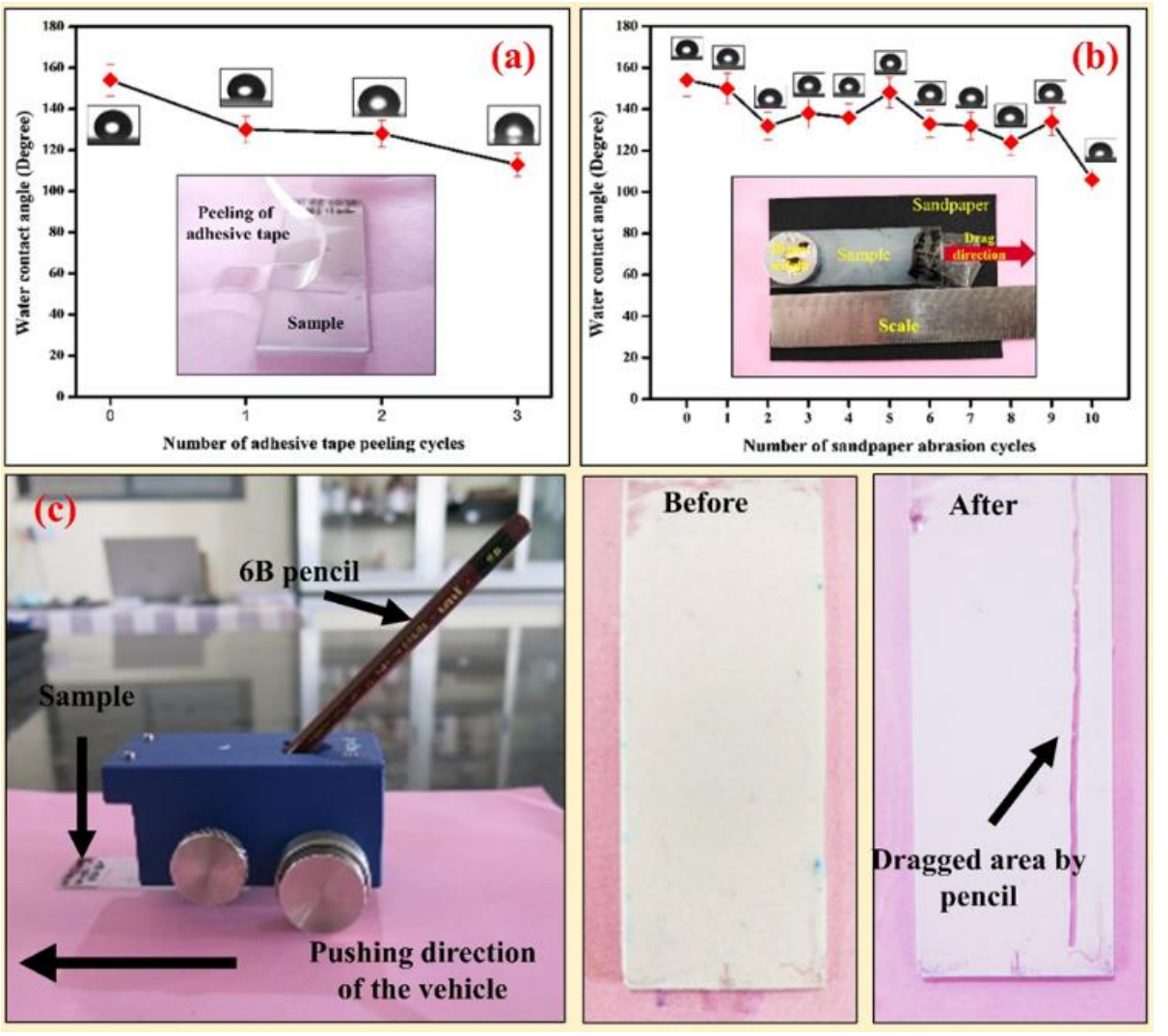

Fig. 4. (a) An adhesive tape, (b) sandpaper abrasion test and (c) pencil hardness test conducted on PS-2 sample.

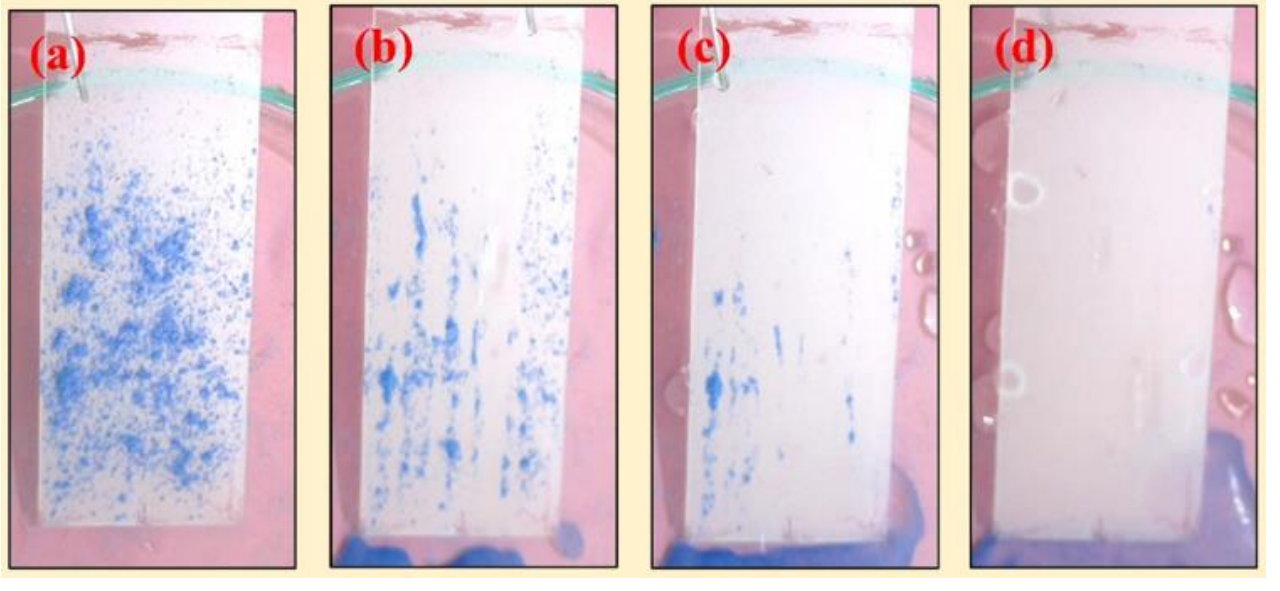

Fig. 5. (a - d) Self-cleaning performance of the PS2 sample against blue chalk powder.

Following this, water droplets were gently applied to the dust-contaminated surface using a syringe. The water droplets, upon contact with the surface, immediately picked up the chalk particles due to the low adhesion between the surface and the contaminants. This interaction caused the water droplets to bead up and roll off the surface, effectively removing the chalk powder and leaving behind a clean and uncontaminated surface.

The process was repeated multiple times to ensure consistency and reliability of the self-cleaning performance. As illustrated in Figure 5(a-d), the sequence of images captures the progression of the water droplets as they collect and remove the chalk powder from the surface. The coating exhibited exceptional self-cleaning properties, demonstrating its capability to maintain cleanliness with minimal effort. This performance can be attributed to the high water contact angle (WCA) and low sliding angle (SA) of the coating, which facilitate the easy rolling off of water droplets, thereby ensuring efficient removal of contaminants. The robust self-cleaning ability of the PS2 sample highlights its potential for applications in environments where maintaining surface cleanliness is critical, such as in outdoor structures, automotive surfaces, and electronic devices.

4. CONCLUSION

In conclusion, we have successfully fabricated a self-cleaning superhydrophobic coating on a glass substrate using a facile sol-gel spray coating method. The incorporation of silica (SiO_2) particles into a polydimethylsiloxane (PDMS) matrix resulted in the formation of a rough surface structure, which is essential for achieving superhydrophobic properties. The synthesized coating exhibited a high water contact angle (WCA) of 154°, indicating excellent water repellency, and a low sliding angle (SA) of 4°, confirming its superior self-cleaning capability. Moreover, the mechanical durability of the coating was rigorously tested through multiple adhesive tape peeling, sandpaper abrasion, and pencil hardness tests. The results demonstrated that the coating maintained its superhydrophobic properties and structural integrity under these conditions, indicating strong mechanical resistance. Additionally, the self-cleaning performance was evaluated using chalk powder, and the coating effectively repelled contaminants without losing its superhydrophobic properties. The study highlights the potential of this superhydrophobic coating for practical applications, particularly in environments where maintaining cleanliness and minimizing contamination are crucial. The scalability and simplicity of the spray coating method further enhance its applicability for large-scale production. This research opens new avenues for developing advanced self-cleaning surfaces, which can significantly benefit industries such as construction, automotive, and consumer electronics by reducing maintenance costs and improving product longevity.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests.

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