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RESEARCH ARTICLE

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

Sagar S. Ingole [1], Akshay R. Jundle [1], Pradip P. Gaikwad [1], Rutuja A. Ekunde [1], Rajaram S. Sutar [2], Sanjay S. Latthe [3,*]

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 addition of SiO_2 nanoparticles in PDMS improves the

[1] Self-cleaning Research Laboratory, Department of Physics, Raje Ramrao Mahavidyalaya, Jath 416404, (Affiliated to Shivaji University, Kolhapur), Maharashtra, India.
[2] College of Chemistry and Molecular Science, Henan University, Kaifeng 475004, P. R. China.
[3] Self-cleaning Research Laboratory, Department of Physics, Vivekanand College (Autonomous), Kolhapur 416003, (Affiliated to Shivaji University, Kolhapur), Maharashtra, India.
* Author to whom correspondence should be addressed: latthes@gmail.com (Sanjay S. Latthe)

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.

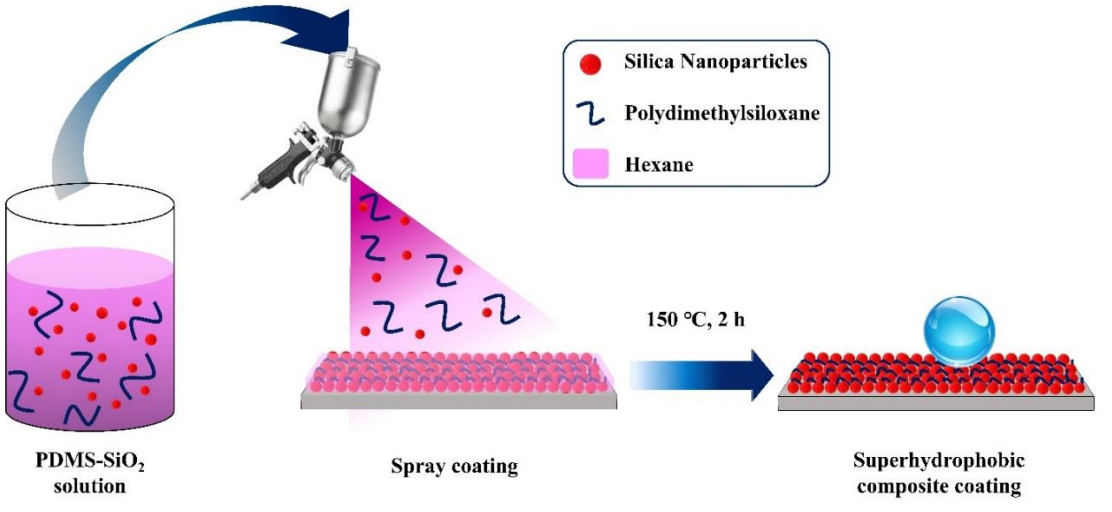

Fig. 1. Schematic of the fabrication of self-cleaning superhydrophobic coating on glass substrate.

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 structure of coatings is significantly impacted by the concentration of silica particles. The SEM micrographs shown in Figure 2(a-f) depict the surface structure of PDMS coatings with varying concentrations of silica particles (PS1, PS2, and PS3). In particular, the incorporation of SiO_2 in PDMS matrix leads to agglomeration and eventual distribution on the glass surface during the deposition process. This results in the creation of a rough structure on the coating surface, which in turn affects the wetting property of the coating. A PS2 sample exhibited average surface roughness values of 0.015 µm. The roughness created by the aggregated SiO_2 particles, combined with the low surface energy of PDMS, improves the super-hydrophobicity of the coatings. A PS1 exhibited WCA of 138° with no SA on the other hand, PS2, and PS3 sample revealed a WCA of 154°, 151° and SA of 4°, 7° , respectively. Figure 2(g) depicts the optical image of color dyed water droplets on the surface of the PS2 sample and an image of WCA on PS2 depicted in Figure 2(h).

3.2. Chemical composition

The FT-IR spectroscopy was utilized to study the chemical bonding of the PS2 sample. In Figure 3, it is evident that strong peaks in the range of 1100-470 cm^{-1} are attributed to the composite coating [23]. Si-CH_3 bonding is responsible for the peak at 1571 cm^{-1}, while the peak at 1033 cm^{-1} is assigned to the asymmetric stretching vibration of Si–O–Si bond. The peaks located at 2913 cm^{-1} and 3192 cm^{-1} are attributed to C-H asymmetric and symmetric vibrations, respectively. Thus, the FTIR spectra confirms the composition of PDMS and silica particles [24].

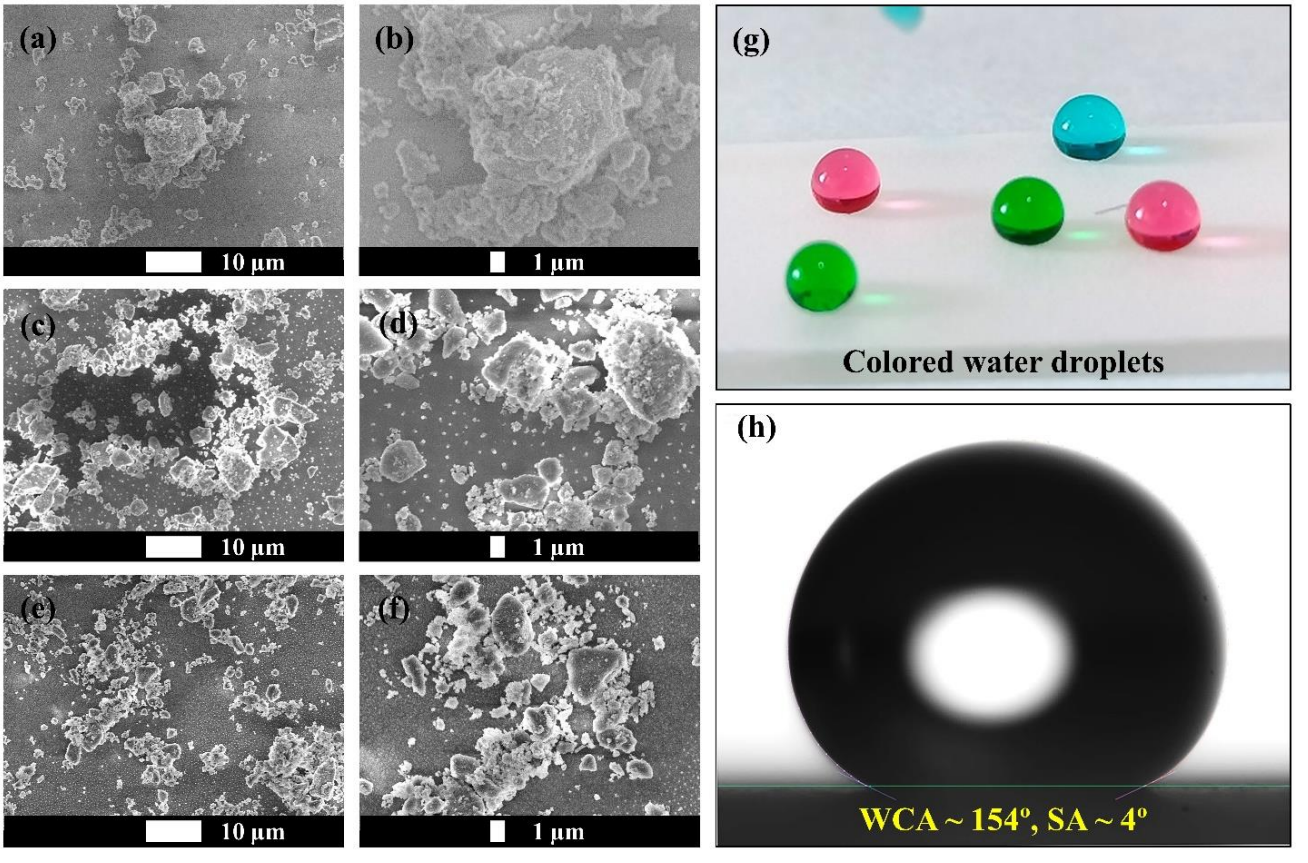

Fig. 2. SEM micrographs of (a, b) PS1, (c, d) PS2 and (e, f) PS3 with lower and higher magnification. (g) Optical image of colored water droplets on the PS2 sample. (h) Water contact image of the PS2 sample.

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], pencil hardness test [27] were carried out. An adhesiveness of the prepared coating toward the substrate was evaluated by adhesive tape test. An adhesive tape was applied on PS2 sample and a metallic disc of weight of 20 g was rolled on it for 1 min to remove the air gap between substrate and tape. After that, the tape was tear off from the substrate and considered as a cycle. The WCAs were measured after each cycle. It was observed that the WCA decreased to 113.72° after three cycles. The detached coating material was stick to the adhesive tape. The variation of WCA after each cycle was depicted in Figure 4(a). The optical photograph of the experimental of adhesive tape peeling test was in inset of Figure 4(a).

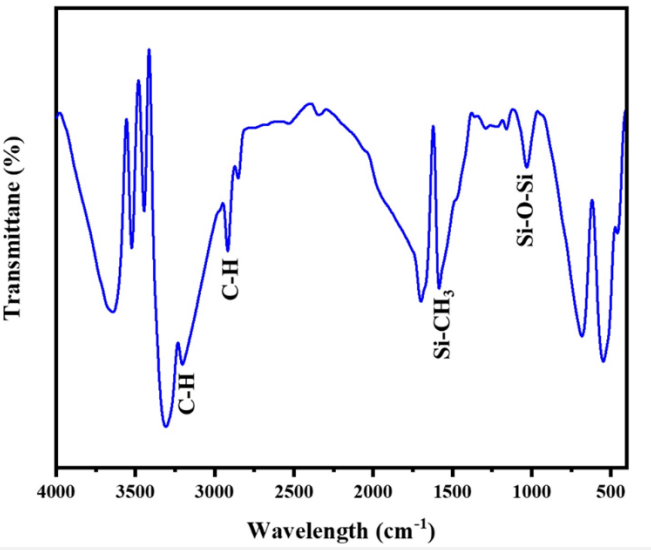

Fig. 3. FT-IR spectra of the PS2 sample.

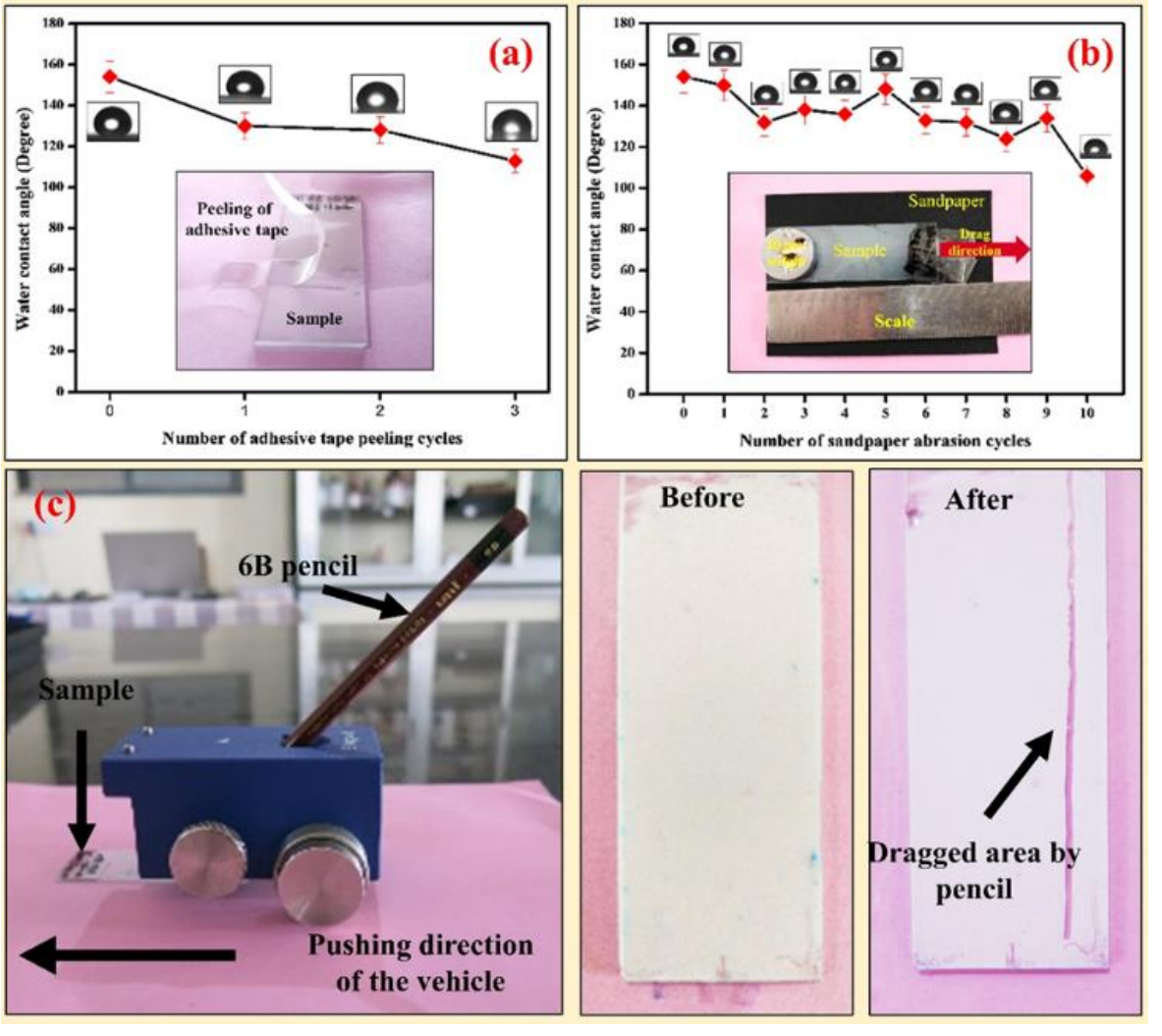

Fig. 4. (a) An adhesive tape, (b) sandpaper abrasion test and (c) pencil hardness test conducted on PS-2 sample.

In another mechanical stability testing method, the coated surface of the PS2 sample was placed on the sandpaper (grit number 1500) and 20 g of weight was loaded (Figure 4b). For one cycle of sandpaper abrassion, the sample was dragged for 10 cm with normal speed. After each cycle the WCA was recorded to evalute mechanical stability of coating.

Unfortunatly, the WCA was reduced to 106.73° after ten cycles. After test, the coated surface from the sample was seemed as partially removed and observed on the surface of the sandpaper. The reduction the WCA after each cycle is depicted in Figure 4b as well as an optical image of the experimental setup of testing is shown in inset. During the pencil hardness test, a 6B pencil tip was used with pressure of 500 gm onto the sample. The tester was drove onto the coating. It was observed that the dragged area had undergone complete removal and there was no discernible coated layer left (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. In order to assess the self-cleaning performance of the PS2 sample, chalk powder was sprinkled on the sample and water droplets were applied using a syringe on the dust-contaminated surface. The water droplets collected chalk particles and rolled off the surface, leaving behind a clean surface (as illustrated in figure 5(a-d)). The coating exhibited exceptional self-cleaning properties against the chalk powder.

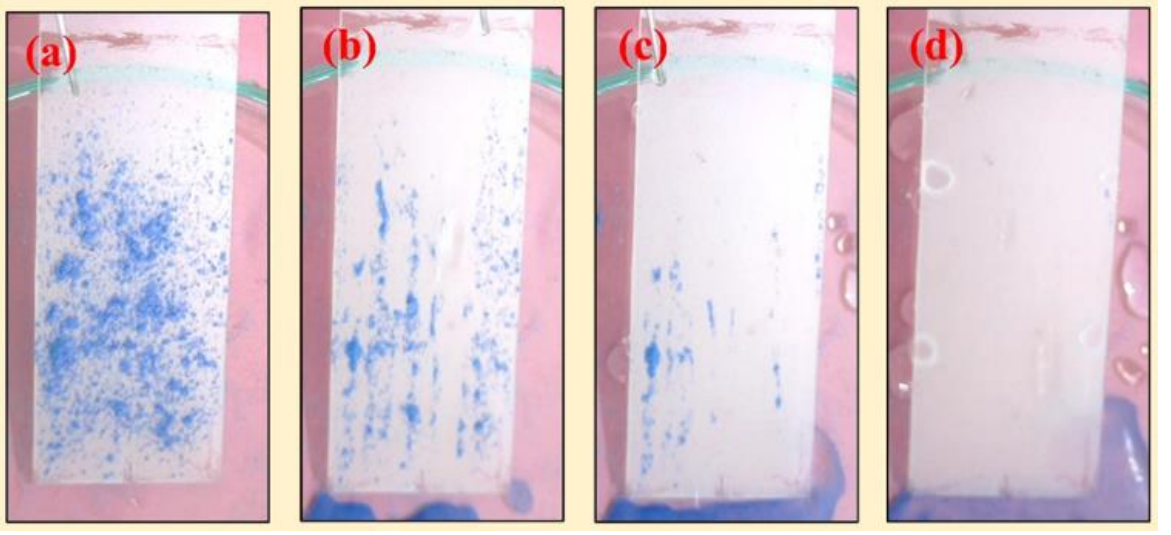

Fig. 5. (a - d) Self-cleaning performance of the PS2 sample against blue chalk powder.

4. CONCLUSION

A facile spray coating technique was used to fabricated self-cleaning superhydrophobic coating. The as-prepared superhydrophobic coating exhibited high WCA of 154° and low SA of 4°. The as-prepared coating was exhibited excellent self-cleaning performance against chalk powder without losing its self-cleaning properties. In addition, the sample exhibited good stability against various mechanical tests including adhesive tape peeling, sandpaper abrasion, and pencil hardness.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests.

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