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Research Article **Open Access**

Fabrication of Superhydrophobic Coating (SiO_2/PDMS) by a Simple Method

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Abstract

A simple and economical method for producing a superhydrophobic surface on a glass substrate is investigated. The surface composes of silica particles synthesized via a sol-gel method with an average particle size of 69.33 nm. Organosilan PDMS (FS-1200 silicon sealant) was used to reduce the surface energy of particles, which were then coated onto a glass substrate by dip coating. After coating, the substrate was dried for one hour at 60°C in an oven to remove excess solvent. XRD, FE-SEM, FTIR, and contact angle (CA) measurement techniques were used to characterize silica particles and coated surfaces. The water contact angle (WCA) of pure SiO_2 (NPs) was 86° indicating its hydrophilic qualities, while the contact angle of a superhydrophobic surface was 156°.

Keywords: SiO_2 nanoparticles, superhydrophobic, silicon sealant, sol-gel method.

الخلاصة

في هذا العمل استخدام طريقة بسيطة وغير مكلفة لاعداد سطح طارد للماء (سطح عديم البلل) مركب من جسيمات ثاني اوكسيد السليكون النانوية (السليكا) والتي تم تحضيرها بطريقة السول-جل .حيث كان معدل حجم الجسيمات (69.33 نانومتر)، تم استخدام السيليكون العضوي لتقليل الطاقة السطحية للجسيمات النانوية .يتم الحصول على السطح الطارد للماء بعد طلاء الشريحة الزجاجية بطريقة الغمر وتجفيف بدرجة حرارة (60 سليليزية) داخل الفرن .تم دراسة الخواص التركيبة للجسيمات النانوية والسطح المحضر باستخدام حيود الاشعة السينية (XRD) والمجهر الالكتروني الماسح (FE-SEM) ومطياف الاشعة تحت الحمراء (FTIR) وقياس زاوية الاتصال (CA) لكل من الجسيمات السليكا النانوي النقية والسطح الطارد للماء المحضر,حيث كانت زاوية الاتصال الماء مع الجسيمات النانوية النقية (86°)، والتي تشير الى خواص محبة للماء بينما كانت زاوية الاتصال السطح الطارد للماء (156°).

INTRODUCTION

Wettability, a fundamental surface property of solid materials, is often assessed by the determination of the contact angle (CA) using specialized instrumentation [1]. Therefore, a significant water contact angle (WCA) over (150°) and a small slide angle (SA) below (10°) are frequently distinguishing characteristics of superhydrophobic surfaces [2]. In general, wetting behavior can be classified into four different regimes on the basis of water contact angle (WCA). WCA in the range of (0°<θ<10°), (10°<θ<90°), (90°<θ<150°), and (150°<θ<180°) can be termed as superhydrophilic, hydrophilic, hydrophobic, and superhydrophobic, respectively [3].

The idea of superhydrophobic surface Inspired by nature, for Example: lotus leaves, groundnut leaf, rose petal, poplar leaf, Salvinia molesta floating leaves, butterfly wing, fish scale, water strider, mosquito's compound eyes, and gecko's feet [4,5]. Lotus leaves are the most well-known examples of a superhydrophobic phenomenon in nature known as the lotus effect, using scanning electron microscopy (SEM), Barthlott and Ehler first investigated the self-cleaning ability of lotus leaves in 1977. They found that the waxy, hydrophobic epicuticular crystalloids on the lotus leaf surface are responsible for the self-cleaning phenomenon. These patterns provide the leaf surface with low surface energy and dual micro and Nano scale roughness [6].

The wetting qualities of solids are primarily determined by topographical features and chemical composition [7]. Hence, the design of a superhydrophobic surface necessitates the consideration of two crucial parameters: (1) a low surface energy and (2) the presence of micro-nano-scale hierarchical roughness in the solid substrate [8]. Therefore, the production of superhydrophobicity is often achieved by two methods: the creation of hierarchical structures, namely micro and nanostructures, on hydrophobic substrates or the chemical modification of a hierarchically structured surface using a material with low surface energy.

Superhydrophobic surfaces have become more popular in recent years because they are used in a wide range of industry and biological uses, such as anti-icing, anti-corrosion, self-cleaning, drag-reducing [9,10], oil-water separation [11]. Anti-bacteria and blood repellent [12], energy harvest [13].

A variety of methods may be used to create a superhydrophobic surface, such as chemical vapour deposition (CVD) [14], electro-spinning, templates [15], lithography, sol–gel [16], self-assembly, electrochemical, plasma etching, dip coating [17], spray coating [18] and hydrothermal method [19]. In contrast, several types of substrates may be used for the production of superhydrophobic coatings, including sawdust, cotton, copper mesh, sponge, stainless steel mesh, glass, and filter paper, among others [20]. Many inorganic nanomaterials with organic modifiers were utilized in the preparation of superhydrophobic coatings such as SiO_2, TiO_2, ZnO and Al_2O_3 to enhance the surface roughness and reduce the surface free energy [11][21]. Silica-based coatings offer multiple advantages, including fabrication simplicity, cost-effectiveness, enhanced adhesion, thermal stability, mechanical stability, and easy structural regulation [22]. Silicon dioxide (SiO_2), or silica, is used in a wide variety of applications, such as reflective and non-reflective coatings, dielectric mirrors, beam splitters, bandpass filters, polarizers, and many more.; the tire industry; rubber; glass; cement; concrete; ceramics; textiles; paper; cosmetics; electronics; paints; films; toothpaste; adsorbents; cordierite; and aluminosilicates [23-25]. As a result, it has gained immense popularity and is extensively employed in the field of coating technology, particularly for the preparation of superhydrophobic silica coatings.

Combining inorganic nanomaterials with hydrophobic organosilane compounds like polydimethylsiloxane (PDMS) decreases surface energy. Silicone, carbon, hydrogen, and oxygen combine to form the elastomers known as silicone rubber and silicon sealant based on polydimethylsiloxane (PDMS). It has low surface energy, making it a kind of material [26][27]. Xiao Gong et.

al. fabrication of superhydrophobic coated as self-cleaning on different substrates utilize silica nanoparticle and modified with (PDMS) by spray method, the water contact angle (156.4º) and sliding angle less than (5º) [28]. Eun Jin Park et. al. prepares superhydrophobic of SiO_2 (NPs) coated with (PDMS) by dip coting method on different substrates the contact angle (160º) [29]. In the present work we have synthesized and characterized SiO_2(NPs) for preparing superhydrophobic surface coating on glass substrate by simple, low cost and dip coating method.

MATERIALS AND METHODS

Sodium Silicate ($Na_2SiO_3.6H_2O$) from Thomas Baker, India, hydrochloric acid (HCl 35%) from Thomas Baker, India. N, N-Dimethylformamide (DMF) from scharlou, spin, and commercial silicone sealant (FS- 1200) and Glass slides.

Synthesis of SiO_2 by Sol-gel method

An initial sodium silicate solution was prepared by dissolving (1 g) of sodium silicate in (10 ml) of distilled water (D.W) at (80ºC) and stirred for (10min.). A viscous gel was produced after adding (28 ml) of (HCl) drop-wise to a sodium silicate solution placed on a magnetic stirrer (at 60 °C) for one hour, The product is then centrifuged with distilled water until it lacks of Cl-iones. The final product was then dried in a furnace at (100 °C) for (2 hours) and calcined at (650 °C) for two hours, as shown in Figure 1.

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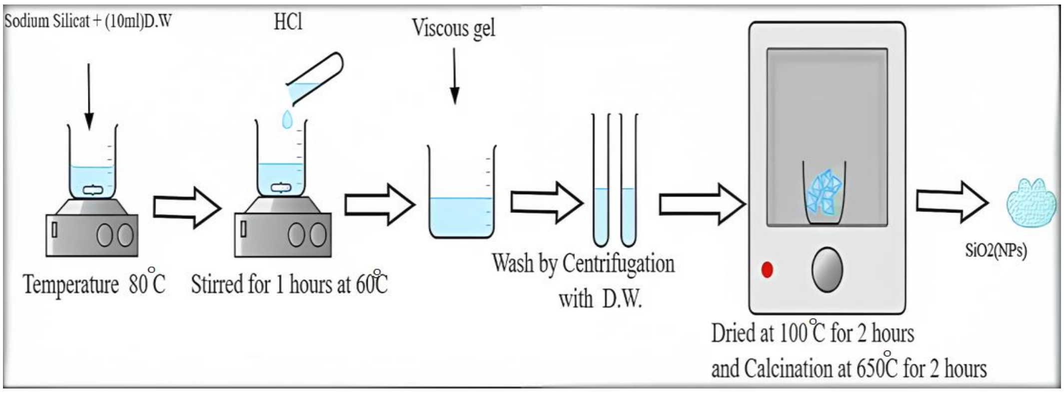

Figure 1. Schematic digram for the preparation of SiO_2 (NPs) by Sol-gel method.

Preparation of Superhydrophobic Surface

To remove contaminants, the glass substrate was washed with deionized water (D.W.) and ethanol in an ultrasonic container for 5 minutes. (0.2g) of SiO_2 was stirred into 10 ml of (DMF) for 30 minutes. Afterwards, add (0.6g) of silicon sealant solution, and stirred the mixture for an additional 30 minutes. The substrate was dipped twice for (2 min.) in the final solution. The coated substrate was dried for (1 hour) at 60°C. as show in Figure 2

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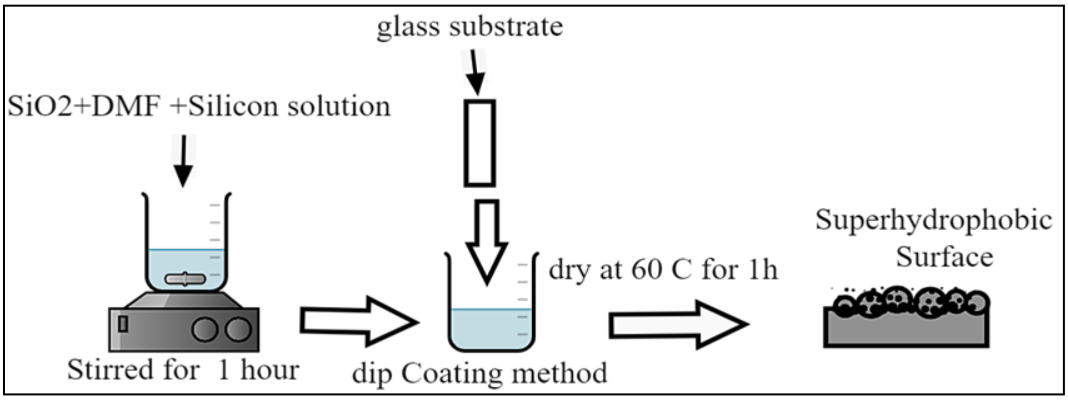

Figure 2. scheme diagram for the synthesis of superhydrophobic surfaces.

Characterization

The X-ray diffraction of the powder was obtained using an X-ray diffractometer at room temperature, (XRD- 6000 Shimadzu) equipped. Field Emission Scanning Electron Microscopy (FE-SEM) The samples were examined by "(Zeiss SIGMA VP-FESEM)" at an acceleration voltage of 10 kV. FTIR analysis. was used Fourier transform infrared spectroscopy (Biotech Engineering Management). In the wavenumber domain, the instrument operates (400-4000) cm^{-1}. A contact angle measuring was used (digital microscope 500x-1000x magnification ratio, China) at room temperature, and used droplets of (5μL) Deionized water. Image j software was used to measure the contact angle.

RESULTS AND DISCUSSION

XRD of SiO_2 (NPs)

The XRD patterns of SiO_2 synthesis by sol-gel method is shown in the Figure 3. The XRD of silica nanoparticles exhibits a broad peak that starts at 2θ (20.11°) and ends at 2θ (23.15°) This broad peak indicates to the very small size of crystallites called semi-crystalline [30, 31], which centered at (22.65°) which refers to reduced crystallinity degree of product. From the XRD analysis data for synthesis powder (SiO_2 by sol-gel), a few peaks with low intensities appear as crystalline at diffraction angles (12.53°, 17.96°, 26.95°, 27.42°, and 29.39°) corresponding to crystal planes (002), (022), (114), (024), and (233). This peak matches well with the reference code no. (98-017-0482) for the cubic crystal structure of the zeolite phase of SiO_2. This result good agreement with work [32].

This diffraction peaks indicates to complete inner structure of the powder and convert to crystal structure due to heat treatment at (650ºC) for (2h) [33][34]. The average crystalline size D (nm) is 46.8 nm.

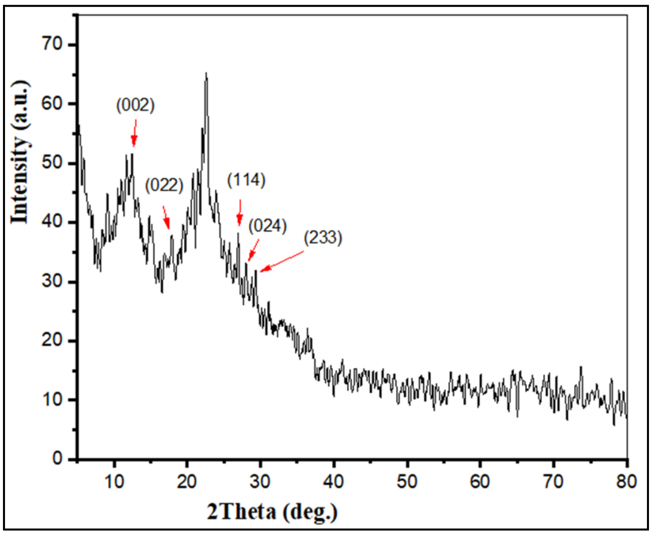

Figure 3. XRD pattern of SiO_2(NPs) synthesis by sol –gel method.

Surface Morphology

Figure 4 (a and b) shows the FE-SEM image of SiO_2 (NPs) synthesis by sol-gel method and cancelation at 650ºC for 2 h, the particles agglomeration in flower like shape. Average size of particles is (69.33nm). Figure 4 (c and d) indicate to superhydrophobic coating on glass substrate prepared when silica (NPs) modified with silicon sealant solution indicates very roughness and the agglomeration particles was uniform. In addition, this surface roughness was composed of numerous gaps that enhance superhydrophobic surface. The silica particles modified with silicon sealant lead to silica particles collect in the form of longitudinal agglomerations in addition to aggregations resembling flower like with average size (225nm). This architecture allows for the storage of air, which reduces the surface area of contact between water droplets and the coating and so increases the contact angle. Therefore, water droplets are able to maintain their spherical shape, resulting in surfaces that are extremely hydrophobic. The transition from the Wenzel model to the Cassie model is also accomplished [35].

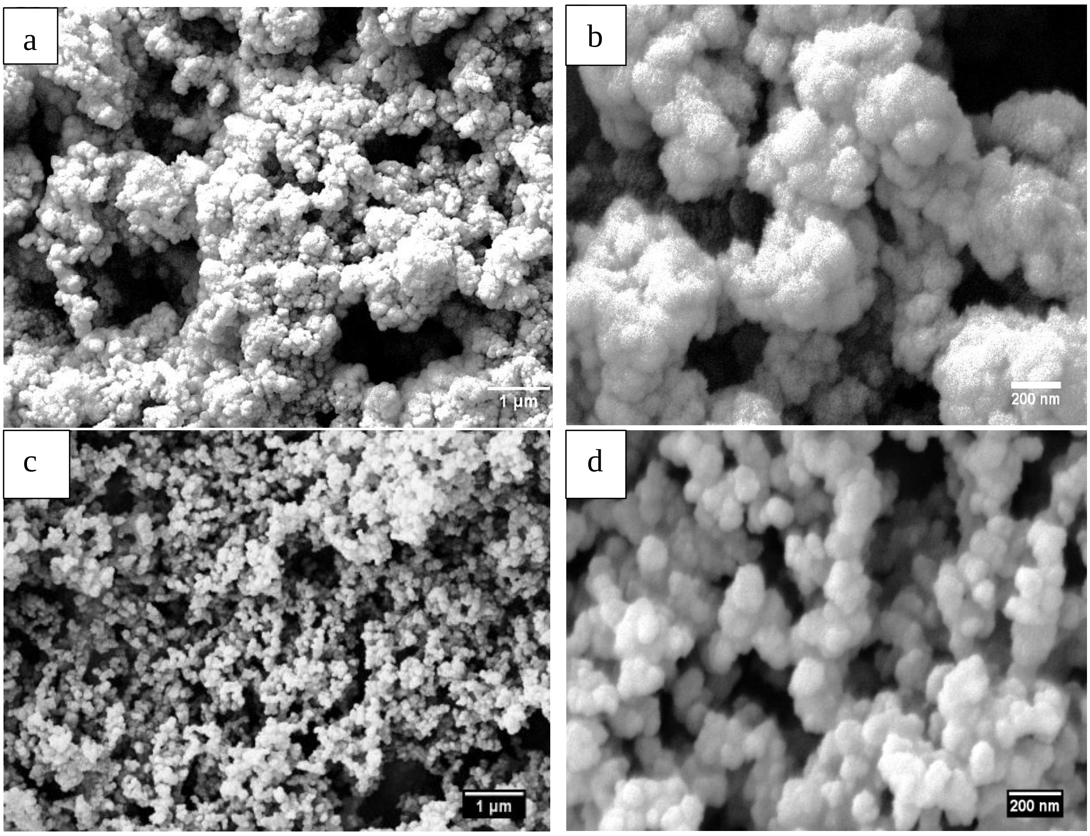

Figure 4. FESEM photo of (a,b) pure SiO_2 (NPs), (c,d) SiO_2 with silicon sealant.

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FTIR analysis

To corroborate the presence of SiO_2, FTIR analysis was used to characterize the sample Figure 5. For SiO_2(NPs) synthesis by sol-gel method FTIR spectrum in range of (4000- 400 cm^{-1}) it can be seen asymmetric and symmetric stretching vibration modes in the (Si-O-Si) network was linked to the band at about (457 cm^{-1}) [36]. The weak band at (834cm^{-1}) is due to symmetry stretching vibration of (Si-O) [37], as a result of a difference in the preparation steps of nanoparticles in terms of the raw materials involved in the preparation and a difference in the preparation and annealing temperatures, the values of the bond peaks are not identical, but they are very close to the results of previous work. The broad band at (1064 cm^{-1}) which is assigned asymmetric stretching vibration of (Si-O-Si) [10]. the band at (971 cm^{-1}) is ascribe to the (Si-OH) bending vibration, also the bands at range between (3850-3753 cm^{-1}) attributed to (Si-OH) asymmetry stretching of (OH) group. The band at (1716 cm^{-1}) was made up of the OH stretching vibration of silanol or water molecules that had stuck to the surface of silica [38]. The band at (1541cm^{-1}) and (2360cm^{-1}) is associated to (Si-C) stretching vibration [39]. In the FTIR spectra of SiO_2(NPs) modified by silicon sealant solution, the band at (3781cm^{-1}) denoted to OH group. (2982cm^{-1}) and band (2342cm^{-1}) attribute to C-H stretching vibration in CH_3 and CH_2 respectively. the band at (1648 cm^{-1}) revere to bending of (O-H) group band. the band at (1258 cm^{-1}) and strong sharp band at (789cm^{-1}) represent Si-C stretching in Si–CH_3 functional group. The strong beak at (1008cm^{-1}) attribute symmetric stretching vibration of Si-O-Si band, the band at (449cm^{-1}) denoted to bending vibration of Si- OH [40][41].

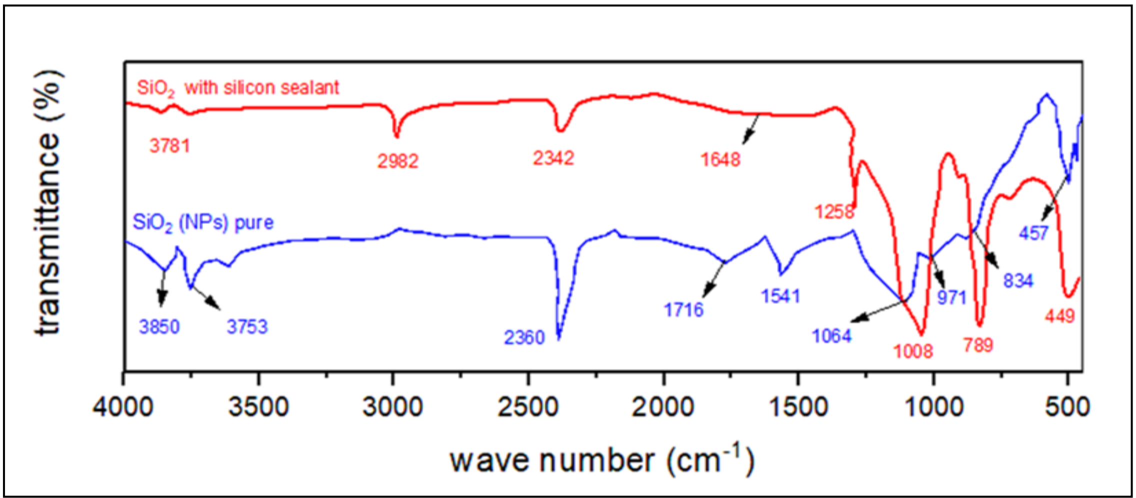

Figure 5. FTIR of pure (SiO_2) and SiO_2 modified with silicon sealant.

Wettability of coating surface

Figure (6a) shows the water contact angles of pure SiO_2 (NPs) synthesis by sol-gel method is (86º) represented the silica particles hydrophilic. While the (WCA) of silicon sealant (101.27º) indicate to hydrophobic materials in nature [42], figure (6b).

The contact angle of prepared surface (SiO_2/silicon sealant) coated on glass substrate exhibited as superhydrophobic surface with WCA (156º) figure (6c). High surface roughness (hierarchical micro-nanostructure) and low surface energy are necessary for superhydrophobic properties, from FE-SEM image of surface Figure (3c-d) shows increasing roughness surface due to existing nanoparticles of SiO_2 combination with low surface energy also existing gapes between cluster make trapped air which is benefit to reduce the contact area of drop water on surface and Non-adhesion of a drop of water on surface .In this case the coated surface agreement with Cassie-Baxter Model of wetting surface[43].

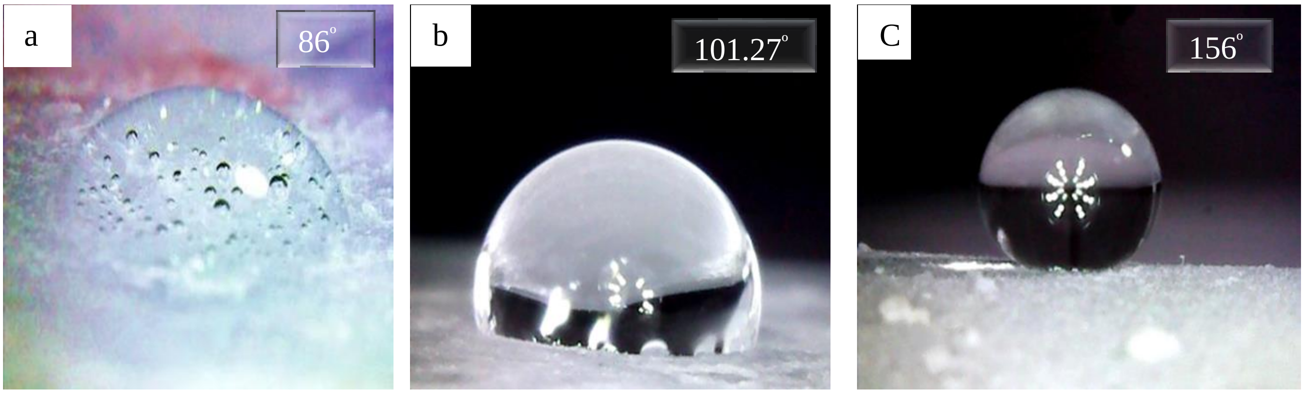

Figure 6. water contact angle of (a) pure SiO_2 (NPs), (b) pure silicon sealant elastomers. (c) superhydrophobic surface on glass .

CONCLUSIONS

In summary, a simple and effective method for fabricating a superhydrophobic surface involves employing SiO_2 (NPs) prepared by the sol-gel method. Through XRD analysis, we note that they are semi-crystalline with some diffraction peaks that indicate crystallinity with the average crystalline size (D = 46.8 nm). Silica nanoparticles (69.33 nm) are assembled in a flower-like shape as in the FE-SEM result. Polymer PDMS (FS-1200 silicon sealant) was used to reduce the surface energy of the silica nanoparticles and work to bond the particles with each other, as we notice an increase in the size of the clusters reaching (225 nm) with the presence of bonding with the polymer, as shown in the spectrum (FTIR analysis). The coating prepared by the dip coating method on a glass slide has the properties of a superhydrophobic surface with a high contact angle of up to (156º).

Disclosure and Conflict of Interest: The authors declare that they have no conflicts of interest.

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