A Facile and Cost-Effective Method to Prepare a Robust Superhydrophobic RTV Silicone Coating

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Abstract: A facile and cost-effective layer-by-layer assembly method is reported for the fabrication of a superhydrophobic coating using room temperature vulcanized silicone rubber and silica particles of different diameters. The superhydrophobic coating shows a contact angle of 154.6° and a rolling angle of 9°. The coating exhibits excellent self-cleaning properties and corrosion resistance with good softness and mechanical durability. The sample maintains its good superhydrophobicity, even after a series of tests by knife scratching, cutting, and hammering. The fabrication method has great advantages in terms of efficiency, cost, simple procedure and conditions. Its wide application in different substrates and the use of environment-friendly materials are also beneficial to the large-scale production of the coating.

Keywords: superhydrophobic; coating; RTV silicone; layer-by-layer assembly

1. Introduction

Inspired by the lotus leaf effect and rose effect, scientists discovered superhydrophobic surfaces. Superhydrophobic surfaces have a very wide range of applications due to their unique wettability, such as self-cleaning [1–3], liquid non-destructive transportation [4–6], drag reduction [7–9], corrosion resistance [10,11] and anti-icing [12,13], etc., which has caused widespread interest in the scientific community and industry. Artificial superhydrophobic surfaces have been fabricated by a variety of methods, including chemical vapor deposition [14,15], machining [16,17], femtosecond laser [18,19], lithography [20,21] and spraying [22,23] etc. The industrial application of superhydrophobic surfaces has been handicapped by the complex processing techniques, high-standard processing environments and expensive materials required. In addition, the fluorine-containing substances and acidic or alkaline solutions used in the preparation process cause serious environmental problems. Researchers have also found that the microstructure and chemical composition of superhydrophobic surfaces have a huge influence on their wettability, which changes with the microstructure on the surface [24,25]. The fragile nature of the microstructure can also be another obstacle for the application of superhydrophobic surfaces. Meanwhile in practical applications, a superhydrophobic surface is not always required on a flat surface, and maybe twisted or bent, for example, when the coated substrate is flexible, such as in the case of a fabric. Under external force, the substrate always deforms, causing damage to the microstructure and performance of the superhydrophobic surface. Therefore, to prepare a flexible superhydrophobic surface with a solid microstructure in a cheap and eco-friendly way can be crucial for the industrial production of the superhydrophobic surface.

One-component room temperature vulcanized (RTV) silicone rubber can be quickly cured into an elastomer in the air at different temperatures and humidity. Therefore, RTV silicone rubber has played an important part in the condensation of liquid silicone rubber. The cured silicone rubber has good chemical stability, hydrophobicity, electrical insulation, and resilience. It is widely applied in molds, construction and electronic appliances [26,27]. Due to its excellent properties, RTV silicone rubber can be used as a carrier and protective
layer against deformation in the microstructure. The simple operating environment and low price of RTV silicone rubber also benefit the preparation of superhydrophobic surfaces. The good adaptability of RTV silicone rubber to different substrate surfaces also expands the application range of the coating.

In this paper, we present a facile and low-cost method for preparing a superhydrophobic coating. A flexible superhydrophobic coating was prepared by using room temperature vulcanized silicone rubber as the main material and silica particles with different diameters by a layer-by-layer assembly method. The prepared sample can not only remove contaminants on the surface through water droplets, but can also resist bending, friction, hammering and knife cutting. This method can be applied to different substrates and does not require complex equipment, a strict production environment or expensive materials. The preparation process is environmentally friendly as it does not involve heating, fluorine-containing materials or acidic/alkaline solutions. Therefore, this facile and low-cost method has great potential to be mass-produced and applied in various fields.

2. Materials and Methods

2.1. Materials

The reagents and materials used in the experiment are as follows: RTV silicone adhesive (Shanghai Mingsheng Rubber Company, Shanghai, China), silica particles (240 mesh, diameter ~61 μm, Gongyi Lanzhirun Water Purification Material Sales Co., Ltd., Zhengzhou, China), silica nanoparticles (diameter ~40 nm, Shanghai Yuanjiang Chemical Co., Ltd., Shanghai, China), n-hexane (Xilong Scientific Co., Ltd. Guangzhou, China), absolute ethanol (Shandong Fuyu Chemical Co., Ltd., Dongying, China), glass slide (Jiangsu Feizhou Glass Plastic Co., Ltd., Taizhou, China) and copper foil (thickness about 0.05 mm, Shanghai Hujiang Hardware Tool Factory). A commercial household hairdryer (EH5246, Panasonic, Osaka, Japan, rated power: 1100 W) was used to dry samples. The water used in the experiment was deionized water.

2.2. Methods

The preparation process of the superhydrophobic coating is shown in Scheme 1. Silica nanoparticles and ethanol were added to the beaker and sonicated for 25 min to form a 0.5 wt.% silica-ethanol suspension (SES). To dilute the RTV silicone rubber adhesive, n-hexane was added to a beaker containing the RTV silicone rubber adhesive, and the 2 wt.% RTV silicone rubber adhesive solution (RSRAS) was formed after stirring.

![Scheme 1. Preparation of superhydrophobic coating. RSRAS: RTV silicone rubber adhesive solution; SES: silica–ethanol suspension.](image)

A thin layer of pure RTV silicone rubber adhesive was applied to the surface of the substrate to form the first layer, and then a layer of silica particles was immediately covered on the adhesive surface. After this, the adhesive was cured in the atmosphere for 1 h, causing it to become a silicone rubber. The sample was then immersed in ethanol to remove
excess silica particles. The cleaned sample was taken out and dried with a hair dryer to form the second layer. The dried sample was immersed in RSRAS for 5 s and then taken out and dried with a hair dryer. After repeating this cycle twice, the third layer was formed. The processed sample was immersed in SES for 5 s and then taken out and dried with a hair dryer. After six rounds of this cycle, the fourth layer was formed. The preparation method of the fifth layer was the same as that of the third layer. The sample was immersed in RSRAS for 5 s and then taken out and dried with a hair dryer. After repeating this cycle twice, a super-hydrophobic coating was formed, called coating C.

To investigate the influence of microparticles and nanoparticles on the hydrophobicity of the coating, coating A and coating B were prepared separately as comparative experiments. The first three layers of the coating were prepared according to the preparation method of coating C to form coating A. The substrate covered by the first layer was immersed in the SES for 5 s and then taken out and dried with a hair dryer. After six cycles of this cycle, the fourth layer was formed. The treated sample was immersed in RSRAS for 5 s and then taken out and dried with a hair dryer. After repeating this cycle twice, the fifth layer was formed and called coating B. Coatings of different compositions are shown in Table 1. In the experiment, if the coating number is not specified, coating C is taken as the test object.

| Coating | 1st Layer | 2nd Layer | 3rd Layer | 4th Layer | 5th Layer |
|---------|-----------|-----------|-----------|-----------|-----------|
| Coating A | RTV Silicone | Silica particles | RTV Silicone | - | - |
| Coating B | RTV Silicone | - | - | Silica nanoparticles | RTV Silicone |
| Coating C | RTV Silicone | Silica particles | RTV Silicone | Silica nanoparticles | RTV Silicone |

### 2.3. Characterizations

The surface morphology of the different coatings was observed by scanning electron microscopy (SEM, SIGMA, Carl Zeiss AG, Oberkochen, Germany). The chemical composition of the coating was determined by Fourier transform infrared spectroscopy (FTIR). The corrosion resistance of the coating in 3.5 wt.% NaCl aqueous solution was evaluated by an electrochemical workstation (CS2350H, Wuhan CorrTest Instruments Corp., Ltd., Wuhan, China). The static contact angle (CA) and sliding angle (SA) were measured by a contact angle measuring instrument (SDC-200, Sindin Precision Instrument Co., Ltd., Dongguan, China). A total of 6 µL water was used to measure the contact angle at six different locations on the coating surface.

### 3. Results and Discussion

#### 3.1. Wettability

Figure 1a presents that the CA of coating A is only 136.5°, therefore, coating A exhibits hydrophobicity. Compared with coating A, the CA of coating B is 156.4°, and the SA of coating A is 7°, hence coating A is superhydrophobic (Figure 1b). As shown in Figure 1c, the water droplets maintain a spherical shape on the glass slide and copper foil with the prepared coating C. The CA of the glass slide covered with coating C is 154.6° ± 3° and the SA is 9°, which indicates the superhydrophobicity of the coating (Figure 1d,e). The copper foil substrate covered with coating C also has superhydrophobicity, and its contact angle and rolling angle are 156° and 8.9°, respectively. As shown in Figure 1f, water from the needle tube refracts after reaching the coating surface, which shows the repellency of the superhydrophobic coating to the liquid and the resistance of the coating surface to water flow impact. The copper foil and glass slide covered with a superhydrophobic coating were placed in water, and a silver mirror appeared on the coating surface, proving that air was trapped in the microstructure of the coating surface (Figure 1g,h).
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Figure 1. (a) Contact angle of coating A. (b) Contact angle of coating B. (c) Digital image of water droplets on the surface of copper foil and glass slide covered by coating C. (d) The contact angle (CA) of the glass slide surface covered by coating C. (e) The sliding angle (SA) of the glass slide surface covered by coating C. (f) Water jet reflection on the glass slide surface covered by coating C. (g) Digital photo of glass slide covered by coating C in water. (h) Digital photo of copper foil covered by coating C in water.

3.2. Surface Morphology

To study the differences in the surface morphology of the coatings with different compositions and their effect on the surface CA, we compared the SEM images of coating A, coating B and coating C. As shown in Figure 2a,b, the surface of coating A is covered by a layer of micron-sized silica particles, the stacking of which forms a large number of voids. By observing high-magnification images, the surface of these micron-sized particles is relatively smooth, so even if voids are formed, they cannot trap enough air. Figure 2c,d is the SEM image of coating B. Compared with coating A, coating B has a smoother surface. The surface of coating B is composed of a large number of silica nanoparticle agglomerates stacked on top of each other. The structure is formed by the repeated immersion in the silica ethanol suspension. It can be seen from the high magnification image that the surface of this structure is quite rough, which is very beneficial to the improvement of hydrophobicity. Figure 2e,f is the SEM image of coating C. It can be seen that the surface of coating C is rougher than that of coating B. Through high-multiple images, it can be seen that micron-level silica particles on the surface are covered with a large number of silica nanoparticles. Research by Feng et al. proved that superhydrophobic surfaces with large contact angles and small sliding angles need to be formed under the cooperation of micro and nanostructures, that is, water droplets tend to move on the surface [28]. Since the surface of coating A only has a micron structure, it can only obtain a larger CA but not a low SA. Compared to coating A, the micro-particles covered by the nano-particles constitute a micro-nano structure, which makes water droplets on the surface of coating C have a high CA and a low SA. In addition, compared with coating B, silica nanoparticles attached to the surface of rough micron silica particles can protect the structure composed of silica nanoparticles and improve the stability of the coating.

3.3. Chemical Composition

Figure 3 shows the typical vulcanization procedure of RTV silicone rubber. The vulcanization reaction between polydimethylsiloxane (the capping group is hydroxyl) and ethyl orthosilicate (cross-linking agent) is catalyzed by dibutyltin dilaurate (DBTDL) to produce RTV silicone rubber and ethanol. The chemical composition of the superhydrophobic coating is determined by FTIR. The FTIR spectra of coating C and the pure and cured RTV silicone rubber is shown in Figure 4. By comparing the FTIR spectra, it was found that their characteristic absorption peaks are basically the same, which proves that a vulcanization reaction occurred on the substrate and RTV silicone rubber is generated. The absorption peaks at 1257 and 2964 cm$^{-1}$ are attributed to the skeleton vibration of the -CH$_3$ group. The absorption peaks at 1008 and 786 cm$^{-1}$ are attributed to the symmetric stretching
vibrations of Si–O–Si, and the absorption peaks at 446 and 469 cm\(^{-1}\) are attributed to the bending vibrations of Si–O–Si.

![Figure 3. (a,b) SEM image of coating A. (c,d) SEM image of coating B. (e,f) SEM image of coating C.](image)

**Figure 2.** (a,b) SEM image of coating A. (c,d) SEM image of coating B. (e,f) SEM image of coating C.

![Vulcanization reaction](image)

**Figure 3.** Vulcanization reaction of Room temperature vulcanized silicone. DBTDL: dibutyltin dilaurate.
Corrosion resistance is one of the important characteristics of the superhydrophobic surface. Electrochemical impedance spectroscopy (EIS) is used to compare the difference between polished copper foil and copper foil with a superhydrophobic coating [29,30]. Referring to the test method of Zhu et al. [31], using an electrochemical workstation (CS2350H, Wuhan CorrTest Instruments Corp., Ltd., Wuhan, China), at room temperature, through a standard three-electrode system, the corrosion behavior of different samples in 3.5 wt.% NaCl aqueous solution was tested. It can be seen from the Nyquist spectra, that the radius of the capacitive reactance arc of the superhydrophobic coating is much larger than that of the bare copper (Figure 5a). The larger the radius of the capacitive reactance arc is, the greater the resistance is in the process of charge transfer. As shown in Figure 5b, the Bode spectra provides further evidence. The $|Z|$ value of the superhydrophobic coating is eight orders of magnitude larger than that of the bare copper. To explain the corrosion resistance of the coating more intuitively, Table 2 shows the values of the components corresponding to the equivalent circuit diagram obtained by the fitting software (ZView 2, Solartron, West Sussex, UK), according to the equivalent circuit diagram in Figure 5c. CPE1 and CPE2 are artificially assumed to be alternatives for pure capacitive elements, and they are used to improve the results of the fitting. When the dimensionless index $n$ is equal to 1, the constant phase angle element is pure capacitance. $R_s$ and $R_1/R_2$ are solution resistance and charge transfer resistance, respectively. As shown in Table 2, the values of $R_s$, $R_1$ and $R_2$ of copper foil with superhydrophobic coating are far greater than the corresponding values of polished copper foil, which proves that the surface of the superhydrophobic coating is subject to greater impedance in the process of electron transfer than the polished copper foil. For metal substrates, the higher the electron transfer impedance is, the stronger the corrosion resistance of the metal is, confirming that the corrosion resistance of copper foil with superhydrophobic coating is greater than that of the polished copper foil. In other words, the superhydrophobic coating can effectively improve the corrosion resistance of the substrate.

![Figure 4. FTIR spectra of coating C and RTV silicone.](image-url)
3.5. Durability

Durability is an important criterion for evaluating whether superhydrophobic surfaces can be used in practice. Jia et al. synthesized PDMS-SiO$_2$ nanoparticles through a high-temperature dehydration reaction and then prepared a super-hydrophobic coating, but this coating tends to fail in harsh environments [32]. In this study, we tested the durability of the prepared superhydrophobic coating through a series of experiments. In order to study the stability of the superhydrophobic coating when the substrate is deformed, a bending test was carried out on the copper foil with a superhydrophobic coating. As shown in Figure 6a–c, when the copper foil is bent to form a concave surface, the water droplet rolls off quickly on the sample surface. The same phenomenon occurs on the sample bent into a convex surface. Dripping from any position, water droplets immediately roll off from the sample surface (Figure 6d–f). To further investigate the stability of the sample, we first bend the sample into a concave surface and then a convex surface to form a cycle. After 200 cycles, the superhydrophobic surface is still fixed on the copper substrate, and the average contact angle of the surface remains above 150° (Figure 7).

Figure 5. Electrochemical impedance spectroscopy (EIS) and equivalent circuit diagram of polished copper foil and coated copper foil in 3.5 wt.% Nacl solution. (a) Nyquist (b) Bode (c) equivalent circuit diagram.

Table 2. Values corresponding to equivalent circuit diagram components.

| Sample   | $R_s$  | CPE$_1$ | $n_1$ | $R_1$    | CPE$_2$ | $n_2$ | $R_2$  |
|----------|--------|---------|-------|----------|---------|-------|--------|
| Bare     | 5.476  | $9.751 \times 10^{-3}$ | 0.7238 | 267      | $1.558 \times 10^{-3}$ | 0.68  | 1.375  |
| Coating  | 198.5  | $9.137 \times 10^{-9}$ | 1     | $1.501 \times 10^6$ | $3.017 \times 10^{-9}$ | 1     | $9.574 \times 10^8$ |

Figure 6. (a–c) Water droplets rolling off from the sample surface bent into a concave surface. (d–f) Water droplets rolling off from the sample surface bent into a convex surface.
Water droplets still roll off the surface quickly when dripping on the sample (Video S5). Table 3 summarizes the CA and SA of different coatings before and after different durability tests. Therefore, compared with coating A and coating B, coating C has superhydrophobicity and high durability.
Table 3. The CA and SA of coatings with different compositions after different durability tests.

| Substrate  | Coating | Durability Test | CA (Before Test) | CA (After Test) | SA (Before Test) | SA (After Test) |
|------------|---------|----------------|------------------|-----------------|------------------|-----------------|
| Glass slide| Coating B| Finger wiping   | 156.4°        | 126.6°         | 7.5°             | >10°            |
| Glass slide| Coating C| Finger wiping   | 153.8°        | 154.3°         | 8.6°             | 8.7°            |
| Glass slide| Coating C| Putty knife scratch | 153.7°     | 153.2°         | 8.9°             | 9.5°            |
| Copper foil| Coating C| Hammer          | 153.1°        | 151.9°         | 8.5°             | 9.1°            |
| Glass slide| Coating C| Utility knife cutting | 153.4°     | 153.8°         | 9.2°             | 8.8°            |
| Copper foil| Coating C| Screwdriver scratch | 156°        | 152.1°         | 8.9°             | 9.3°            |

3.6. Self-Cleaning Effect

To study the self-cleaning effect of the coating, chalk dusts are sprayed onto the surface of the coating. As shown in Figure 9a–f, when dropped onto the surface of the coating, water droplets roll forward under the action of gravity. Part of the chalk dust is absorbed and leaves the surface along with the water droplets. With the increase in water droplets, more chalk dust is taken away from the sample surface, leaving a channel without chalk dust on the sample surface.
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4. Conclusions

In this study, a strong and flexible superhydrophobic coating produced using RTV silicone adhesive and silica particles of different diameters was prepared. By comparing coatings with different compositions, it was found that coating C, which has a micro-nano hierarchical structure composed of silica particles and silica nanoparticles, is superior to coating A and coating B in terms of hydrophobicity and durability. The CA and SA of the coating surface were 154.6° and 9°, respectively. The prepared coating exhibited good corrosion resistance and self-cleaning properties. Through a series of mechanical durability tests, such as finger wiping, putty knife scraping, hammering and utility knife cutting, the coating was proven to have excellent mechanical durability. Moreover, the sample retained superhydrophobic both during and after bending. The preparation method of the superhydrophobic coating has great advantages in terms of efficiency, low-cost, and the simple experimental procedure and conditions involved. Its wide application in different substrates and the use of environmentally-friendly materials are also beneficial to the large-scale production of the coating.

**Supplementary Materials:** The following are available online at [https://www.mdpi.com/2079-6412/11/3/312/s1](https://www.mdpi.com/2079-6412/11/3/312/s1), Video S1: finger wiping test; Video S2: putty knife scratch test; Video S3: hammer test; Video S4: utility knife cutting test; Video S5: screwdriver scratch test.

**Author Contributions:** Conceptualization, J.Z.; investigation, K.L.; methodology, J.Z.; software, K.L.; writing—original draft, K.L.; writing—review & editing, J.Z. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research received no external funding.

**Institutional Review Board Statement:** Not applicable.

**Informed Consent Statement:** Not applicable.

**Data Availability Statement:** The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

**Conflicts of Interest:** The authors declare no conflict of interest.

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**Figure 9.** (a-f) Self-cleaning test.
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