



Article A Facile Method to Prepare Superhydrophobic Coatings for Various Substrates

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Abstract: In this study, the superhydrophobic composite coatings for self-cleaning were fabricated by mixing fluorine resin and two kinds of nanoparticles of carbon nanotubes (CNTs) and SiO₂. When the mass ratio of CNTs to SiO₂ is 2:3 and the added amount of nanoparticles is 75 wt. %, the superhydrophobic composite coatings with a water contact angle of 156.8° show the best self-cleaning property and 3.6° of contact angle hysteresis. Furthermore, the superhydrophobic composite coatings demonstrate good properties such as chemical resistance, thermal stability, and mechanical stability. The superhydrophobic composite coatings could be used for oil/water separation and could be applied to various substrate surfaces such as glass plates, cloth, board, steel plate, PVC plate, and so on. The superhydrophobic composite coatings show practical value in many fields because of their low cost and large area preparation.

Keywords: superhydrophobic; fluorine resin; composite; robust; self-cleaning

1. Introduction

Due to their wide applications in self-cleaning, oil/water separation, corrosion resistance, anti-icing, anti-fogging, and anti-bacterial, superhydrophobic surfaces have attracted much attention over the last ten years [1-4]. A superhydrophobic surface is characterized by a water contact angle (WCA) over 150° and a sliding angle lower than 10° [5]. It is widely known that two key factors play important roles in preparing superhydrophobic surfaces: suitable multi-scale surface roughness and low surface free energy [6]. Many ways have been reported to fabricate superhydrophobic surfaces in recent years, such as chemical vapor deposition [7,8], plasma etching [9,10], hydrothermal synthesis [11,12], electrospinning [13,14], dip coating [15,16], sol-gel method [17,18], templating [19,20], and so on. There are many ways to prepare superhydrophobic surfaces, but most methods usually involve specialized equipment or multistep procedures, high cost, inconvenience, and the inability to fabricate a large area of superhydrophobic surfaces, which limits their practical uses. Simplifying the preparation procedure will greatly benefit the practical use of superhydrophobic surfaces. Facile, low cost, and suitable for large area preparation of superhydrophobic surfaces is the goal of scientific researchers in this field. Moreover, the very common problem of artificial superhydrophobic surfaces is poor mechanical properties. Recently, many researchers have attempted to solve the problem.

At present, although several researchers have chosen to fabricate fluorine-free superhydrophobic surfaces due to environmental problems [21], it is undeniable that the excellent performance of fluorine-containing materials [22–24], such as excellent weather resistance, high chemical stability, extraordinary water repellency, oil repellency, stain resistance, and so on. Jia et al. have prepared a superhydrophobic composite membrane



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). of (polyvinylidene fluoride-co-hexafluoropropylene)/(polystyrene) via a novel hybrid electrospin-electrospray process [25]. Cui et al. prepared the superhydrophobic coatings using nanofibrous poly(vinylidene fluoride)/stearic acid [13]. Nanoparticles of SiO₂, TiO₂, ZnO, and carbon nanotubes (CNTs) are often applied to make surface roughness to fabricate superhydrophobic coatings [26–29]. One-dimensional CNTs with hydrophobic property and high aspect ratio are suitable to construct superhydrophobic surfaces [30]. SiO₂ nanoparticles are also widely used in superhydrophobic surfaces [31–33].

We propose a facile way to prepare superhydrophobic composite coatings by integrating CNTs/SiO₂ nano-additives on fluorine resin in this study. The two nanoparticles of hydrophobic silica and carbon nanotubes were used to prepare superhydrophobic coatings. The surface energy of SiO₂ was low because their surfaces were modified by alkyl groups. The addition of hydrophobic nano-silica can reduce contact angle hysteresis and increase the self-cleaning property of the superhydrophobic composite coating. The superhydrophobic coatings exhibited good properties such as chemical, thermal, and mechanical stability. Moreover, the superhydrophobic composite coatings could be applied to oil/water separation and could be applied to various substrate surfaces. The superhydrophobic composite coatings show practical value in many fields because of their low-cost and large-scale preparation.

2. Materials and Methods

2.1. Materials

Fluorine resin (CF-803) and curing agent were purchased from Shanghai De Yude Trading Co., Ltd. (Shanghai, China). CF-803 is a solvent-based two-component fluorocarbon resin that is a product of the copolymerization of chlorotrifluoroethylene with vinyl ester and other functional monomers. Silica sol (xylene) with a particle size of approximately 20 nm (30 wt. %) was supplied by Wuhan Green Chemical Technology Co., Ltd. (Wuhan, China). CNTs were purchased from Qingdao Haoxin New Energy Technology Co., Ltd. (Qingdao, China). Butyl acetate was purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Cloths, boards, steel plates, and PVC (polyvinyl chloride) plates were purchased through Taobao online shopping. Glass plates (7.5 cm × 2.5 cm) were purchased from CITOTEST[®] Scientific.

2.2. Preparation of the Coatings

A dispersion containing fluorine resin, curing agent, and butyl acetate was prepared by 30 min of magnetic stirring. Then, a mixed solution was obtained by mixing a certain ratio of silica sol (xylene), CNTs, and the as-prepared fluorine resin solution by 10 min of ultrasonic dispersion and 30 min of magnetic stirring. The total solid content of the mixed solution was regulated to 2 wt. %. All substrates (cloths, boards, steel plates, PVC, and glass plates) were ultrasonically cleaned in the detergent solution, rinsed with deionized water, and dried in air dry oven at 40 °C as the preparative. About 1.5 mL of the mixed solution was dripped onto the glass plate through a pipette. The coatings were dried for 12 h at room temperature.

Below was an optimal formula (1.5:1 of the mass ratio of silica sol to CNTs nanoparticles, 75 wt. % of total nanoparticles): fluorine resin (0.180 g) and curing agent (0.018 g) were homogeneously mixed with butyl acetate (14.32 g) by 30 min of magnetic stirring. Then, the above solution was mixed with silica sol (1.20 g) and CNTs (0.24 g), then there was 10 min of ultrasonic dispersion and 30 min of magnetic stirring. The mixed solution was dripped onto every substrate through a pipette. The composite coatings were dried for 12 h at room temperature. Note here that the silica sol used in this study was a solution of 30 % solid content.

2.3. Characterization

KRÜSS DSA 100 (Hamburg, Germany) was used to test water contact angle (WCA). WCA was measured at room temperature through 5 μ L water drop and the result was

an average of 6 fresh points. Advancing and receding contact angles were measured through the growth and shrinkage of a drop, respectively. Cloths, boards, steel plates, and PVC plates were used as substrates to investigate the superhydrophobic coatings on different substrates. The chemical resistance of the superhydrophobic composite coating was tested by dipping the coating into HCl (pH = 2), NaOH (pH = 12), and 3.5 wt. %NaCl solution, respectively. Field emission scanning electron microscopy (FESEM, Nova NanoSEM400) was used to characterize the surface topography of the coatings. Thermal gravimetric analysis (TGA) was performed on NETZSCH F1 Libra® (TGA209F1D-0315-L) under air atmosphere from room temperature to 600 °C with 10 °C/min of a constant heating rate. The superhydrophobic composite coatings were placed in an electricity heat drum wind drying oven at 200 °C for 3 days to further test the thermal stability of the superhydrophobic composite coatings. Mechanical stability was measured by sandpaperabrasion and the experimental steps were as follows: (1) the sample was placed face-down to sandpaper (standard sandpaper, grit no. 1000); (2) moved for 15 cm along the ruler under a weight of 100 g; (3) the sample was rotated by 90° (face to the sandpaper) and moved for 15 cm; and (4) the operation was repeated 3 times. The method of the self-cleaning test of the superhydrophobic composite coatings was as follows: (1) river sand as the model contamination was placed to the surfaces of the superhydrophobic composite coatings; (2) the water droplets were dripped; and (3) the river sand was removed with water drops rolling off the surfaces of the superhydrophobic composite coatings. A camera was used to record the self-cleaning behavior of the superhydrophobic composite coatings.

3. Results

3.1. Preparation and Characterization of the Superhydrophobic Coatings

The wettability of the composite coatings influenced with the SiO₂ content was shown in Figure 1. The mass fraction of CNTs in each of these coatings was fixed at 30%. With increasing SiO₂ nanoparticles content, the WCA of the composite coating improved gradually. At the same time, the contact angle hysteresis of the composite coating decreased gradually. When the total mass of the nanoparticles accounted for 75% of the coatings and the mass ratio of CNTs to SiO_2 was 2:3, the superhydrophobic composite coatings with a WCA of 156.8° and contact angle hysteresis of 3.6° were fabricated through mixing fluorine resin and two kinds of nanoparticles of CNTs and SiO₂. In fact, the hydrophobic property of the composite coating could be improved slightly by further improving the amount of SiO_2 nanoparticles used. However, it was not suitable to further increase the SiO₂ nanoparticles content considering the surface state and mechanical strength of the composite coatings. In this study, the optimal formula is that the mass ratio of CNTs to SiO_2 is 2:3, and the loading of nanoparticles is 75 wt. %. Unless otherwise stated, the following superhydrophobic composite coating refers to the coating in this formulation. Many methods for preparing superhydrophobic surfaces are based on specific substrates. The dependence on substrates is due to the need for substrates to provide all or part of the roughness. For example, the superhydrophobicity of metal surfaces can be achieved by chemical etching and post-hydrophobic molecular modification [34]. If the superhydrophobicity of a surface does not depend on the substrate, then the superhydrophobic coatings can be used more widely. In this study, the superhydrophobic coatings can be used for various substrates. The superhydrophobic composite coatings coated on cloth, board, steel plate, and PVC plate are shown in Figure 2. In Figure 2, the superhydrophobic composite coating is suitable for a variety of substrates. The superhydrophobic composite coatings were dipped into three aqueous solutions for three days to investigate their chemical stability. The photographs of the experiment are displayed in Figure 3. The superhydrophobic composite coating could still maintain its hydrophobicity after being soaked in HCl (pH = 2), NaOH (pH = 12) and 3.5 wt. % NaCl solution for three days. This result shows that the superhydrophobic composite coating has good chemical stability. The surface topography of the composite coatings are displayed in Figure 4. The CNTs are clearly visible in Figure 4a–c. In Figure 4d, to the composite of silica nanoparticles and fluorine resin, a lesser degree of surface roughness of the composite coating can be observed. The mass fraction of silica nanoparticles is 75%. The surface roughness of the composite coating was significantly improved with an increase in the use of CNTs. Compared to adding CNTs alone, the composite application of silica nanoparticles and CNTs can further improve the hydrophobicity of the coating and reduce the contact angle hysteresis. The difference between the three images (Figure 4a–c) is not obvious because the silica nanoparticles have little change on the surface morphology of the composite coatings. This may be because silica nanoparticles have good compatibility with fluorine resin due to the large number of alkyl groups on its surface. Therefore, the silica nanoparticles have little change on the composite coatings.



Figure 1. Relation between weight ratio of SiO₂, water contact angles, and contact angle hysteresis. The mass fraction of CNTs in each of these coatings was 30%. The inserts were 5 μ L water drop profiles on the surface of the composite coatings.



Figure 2. The superhydrophobic composite coatings coated on cloth (**a**), board (**b**), steel plate (**c**), and PVC plate (**d**). Water drops were dyed blue by methylene blue, dyed orange by coffee, and dyed red by methyl orange in acids.



Figure 3. The superhydrophobic composite coatings against water droplets after dipping into (**a1–a3**) HCl (pH = 2), (**b1–b3**) NaOH (pH = 12), and (**c1–c3**) 3.5 wt. % NaCl aqueous solution for three days.



Figure 4. Surface topography of the coatings with same CNTs of 30 wt. % and different SiO₂ nanoparticles contents: (**a**) 0 wt. %, (**b**) 15 wt. %, (**c**) 45 wt. %; (**d**) surface topography of the coatings with SiO₂ nanoparticles of 75 wt. %.

3.2. Thermal Stability Properties of the Superhydrophobic Coatings

High thermal stability is very important for superhydrophobic coatings to their practical application. In this study, two methods were applied to test the thermal stability of the superhydrophobic composite coating. The results of the TGA test in air atmosphere are displayed in Figure 5. As shown in Figure 5, CNTs showed the highest thermal stability. Fluorine resins were almost completely weightless at 600 °C (Figure 5a). The addition of nano-additives slightly increases the thermal stability of the composite coatings. These composite coatings have almost no weight loss up to 250 °C. As Figure 5c shows, the superhydrophobic composite coatings lost about 5 wt. % at around 310 °C. The superhydrophobic composite coatings were processed by 3 days of heat treatment at 200 °C to further test their heat resistance. The photos of the superhydrophobic composite coatings before and after heat treatment are exhibited in Figure 6. From Figure 6d, the composite coatings are still very hydrophobic with WCA of 154.5°. These two kinds of test results show that the superhydrophobic composite coatings have good thermal stability.



Figure 5. The TGA curves: (a) fluorine resin; the coatings with same CNTs of 30 wt. % and different SiO₂ nanoparticles contents: (b) 0 wt. %; (c)15 wt. %; (d) 45 wt. %; (e) the coatings with SiO₂ nanoparticles of 75 wt. %.



Figure 6. (a) The picture of the superhydrophobic composite coatings; (c) the picture of the superhydrophobic composite coatings after 3 days of heat treatment at 200 $^{\circ}$ C; (b,d) the picture of water drops on the composite coatings of (a,c), respectively. Water drops were dyed blue by methylene blue, dyed orange by coffee, and dyed red by methyl orange in acids.

3.3. Mechanical Property of the Superhydrophobic Composite Coatings

The mechanical robustness of the superhydrophobic composite coating was examined by an abrasion test. As can be seen in Figure 7, the superhydrophobic composite coating had partially peeled off after the abrasion test. The test had little effect on the hydrophobicity of the composite coatings. This result of the surface robustness test of the superhydrophobic composite coatings reflects the good mechanical stability of them.





Figure 7. The experimental pictures of sandpaper-abrasion, the superhydrophobic composite coating: (a) test preparation; (b) under a weight at 100 g was placed face-down to sandpaper; (c) after the test; (d) the pictures of water drops sitting on the tested composite coatings. Blue water drops were dyed by methylene blue.

3.4. The Self-Cleaning Property of the Superhydrophobic Composite Coatings

The self-cleaning property plays an important function to the superhydrophobic composite coatings in the practical application. As illustrated in Figure 8, the self-cleaning test of the superhydrophobic composite coatings was measured by contaminating with river sands. The river sands were taken away by water drops rolling. The surface of the superhydrophobic composite coatings became clean after a short time, which suggested that they could be applied for self-cleaning. The superhydrophobic composite coatings could also be applied to oil/water separation and the oil/water separation results are shown in Figure S1.



Figure 8. Pictures of self-cleaning test: (**a**) the superhydrophobic composite coating with river sands to be tested; (**b**,**c**) pictures in the test.

4. Conclusions

In conclusion, we successfully prepared superhydrophobic composite coatings by mixing CNTs/SiO₂ nanoparticles and fluorine resin. The superhydrophobic composite coatings with WCA of 156.8° and contact angle hysteresis of 3.6° show the best self-cleaning property. Moreover, the superhydrophobic composite coatings exhibited good properties such as chemical stability, thermal stability, and mechanical stability. Moreover, the superhydrophobic composite coatings substrate surfaces such as glass plates, cloth, board, steel plate, PVC plate, and so on. Therefore, the superhydrophobic composite coatings have great potential for practical applications.

Supplementary Materials: The following are available online at https://www.mdpi.com/article/10 .3390/app12031240/s1, Figure S1: The images illustrate oil/water separation of the superhydrophobic coatings. Water droplets were dyed blue by methylene blue. DCM was dyed red by oil red O.

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