

Fabrication of durable polytetrafluoroethylene superhydrophobic materials with recyclable and self-cleaning properties on various substrates

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Abstract Most of the artificial superhydrophobic surfaces easily lose their good water repellency once they are chemically or mechanically damaged, which seriously hinders their practical application. Durable superhydrophobic materials with recyclability, lowtemperature resistance, and self-cleaning properties were successfully prepared by employing silica particles and polytetrafluoroethylene via a simple dropcoating method. The as-prepared superhydrophobic materials not only show excellent superhydrophobicity but also display fascinating durability and chemical stability after a series of rigorous tests. Importantly, the debris scraped from the superhydrophobic materials can be recycled and easily reused to prepare new superhydrophobic materials. This facile strategy may pave the way for the scalable and recyclable robust superhydrophobic materials in practical applications.

Keywords Superhydrophobic, Polytetrafluoroethylene, Mechanically durable, Self-cleaning

Introduction

Nature always surprises us with its remarkable creatures. For instance, the lotus leaf surface always remains clean due to its super water repellency and favorable self-cleaning property.^{[1,2](#page-8-0)} Spherical water droplets, deposited on lotus leaf surface with a small tilt angle lower than 10° , can easily roll off and

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M. Qu e-mail: mnanqu@gmail.com simultaneously pick up the contaminants such as dust and dirt on the surface, and this interesting phenomenon is known as the "lotus effect." $3,4$ Inspired by the unique water-repellent surfaces of the lotus leaf and the water strider's leg in the natural world, $5,6$ superhydrophobic surfaces have attracted intensive attention in both fundamental research and industrial applications in the past two decades.⁷⁻⁹ A large number of artificial superhydrophobic materials are fabricated by different methods and have applications in various fields, such as anticorrosion, $10,11$ self-cleaning,^{[12,13](#page-8-0)} antimicrobial,^{14,15} antifouling,^{[16,17](#page-8-0)} and oil/ water separation.^{[18–20](#page-8-0)} To achieve the superhydrophobicity, surface topography and surface energy are two crucial factors. $21,22$ Generally, most of the artificial superhydrophobic surfaces easily lose their good water repellency once they are chemically or mechanically damaged, which seriously hinders their practical application.²

To overcome these shortcomings, many methods were developed, $28,29$ such as fabricating the superhydrophobic surfaces with brilliant self-cleaning prop- erty^{30} erty^{30} erty^{30} and constructing robust hierarchical structures on the superwettable surface. $31-34$ However, these methods still rely on special conditions or treatments and thus are difficult to realize for large-scale applications. In this respect, fabricating recyclable and robust superhydrophobic materials with a facile strategy is desirable in practical application. Polytetrafluoroethylene (PTFE) possesses excellent properties in chemical stability, thermal resistance, low water absorption, potential biocompatibility, and good mechanical strength.³⁵⁻³⁸ Thus, it is a promising material for the preparation of high-mechanical-strength superhydrophobic materials.

In this study, we present a facile process to prepare durable superhydrophobic materials with good chemical and mechanical stability. The prepared superhy-

drophobic material is composed of micro/nanostructures throughout the thickness of the material and can now be considered a true superhydrophobic ''material,'' not just the usual superhydrophobic ''surface.'' In addition, material fragments can be easily recycled and further utilized to prepare a new superhydrophobic material. Moreover, the fabrication of the durable superhydrophobic material was versatile and costefficient and the resultant materials can be drop-coated on a variety of substrates. This superhydrophobic material has good durability and has potential application prospects in the fields of coating industry and environmental emergency.

Materials and methods

Materials

PTFE nanoparticles (TF-9207) were purchased from Minnesota Mining and Manufacturing Corporation. Diethoxydimethylsilane (DEDMS) was purchased from Sigma-Aldrich. Tetraethyl orthosilicate (TEOS) was purchased from TCI Shanghai. Polyvinylpyrrolidone (PVP) was purchased from Shanghai Lanji Biology. Hydrochloric acid (HCl), sulfuric acid, isopropanol, and absolute ethyl alcohol were analytical grade. The deionized water was used in the preparation of acid water. All chemicals were used as received without further purification.

Fabrication of the durable superhydrophobic materials

Silica sol was prepared by mixing 6 mL of TEOS, 2 mL of acid water ($pH = 2$, composed of 50 µL HCl, and 55 mL deionized water), and 10 mL of absolute ethyl alcohol. The as-prepared mixture was reacted at 60° C in a water bath for 4 h undermagnetic stirring, and after that, the acid silica sol was obtained. Then 10 g of isopropanol, 1 g of DEDMS, and 1 g of sulfuric acid were added into the acid silica sol. After reacting under magnetic agitation at room temperature for 30 s, the mixture was placed at room temperature for 30 min before use.

The wear-resistant superhydrophobic materials were synthesized by a sol–gel strategy. First of all, PTFE nanoparticles were dried at 120° C for 5 h to remove water before use. Then 1.5 g of PTFE nanoparticles was ultrasonically dispersed in 3.6 mL of absolute ethyl alcohol. Then obtained acid silica sol was added to the white PTFE nanoparticle suspension solution and stirred for 30 min. After that, 0.15 g of PVP was added to the mixture with magnetic stirring for 30 min. Finally, the prefabricated silane modifier was added to the suspension, stirring for another 4 h. The resulting suspension was carefully coated on clean glass slides with a plastic head dropper. Afterward, the samples were cured at 80° C for 2 h.

Characterization

The microstructures of the materials were observed by scanning electron microscopy (SEM, JEOL JSM-6460LV) operated at 10 kV. Before the SEM investigations, all the samples were coated with gold cluster. The water contact angles (WCAs) and sliding angles (SAs) were performed by an instrument (JC2000DM, China) at ambient temperature. The volumes of probing liquids were approximately $5 \mu L$ for the WCA measurement. Each contact angle reported was an average value of five independent measurements on different positions. The images of liquid droplets on the surface were captured by a digital camera (Nikon D3200). The chemical compositions of the materials were examined by Fourier transform infrared spectrophotometer (FTIR, PerkinElmer FTIR System 2000) and X-ray photoelectron spectrometer (XPS, K-Alpha of Thermo Electron Corporation). The mechanical stability of superhydrophobic samples was evaluated by water droplets impacting, tape adhesion test, and abrasion test. Furthermore, variations of the WCAs and SAs for the superhydrophobic surface were measured as functions of pH values of water droplet and immersion time in water.

Results and discussion

The optical photographs of water droplets (dyed by methyl blue) on the as-prepared superhydrophobic materials coated on various substrates are shown in Fig. [1.](#page-2-0) As shown in Fig. [1](#page-2-0)a[–1](#page-2-0)d, all these substrates can be successfully coated with the superhydrophobic materials, and the CAs of water are all higher than 150° . The water droplets almost cannot stay on asprepared material surface and easily roll down the sample surface when there is a slight tilt of the surfaces. These results indicate that superhydrophobic materials were successfully obtained. Moreover, to indicate the extremely low adhesion force between water droplet and the prepared superhydrophobic surface (Fig. [1](#page-2-0)d), adhesion test, contacting, and leaving motion of the water droplet dragged by needle tip were designed.

One of the challenges of practical application is the durability of the superhydrophobic materials. Most superhydrophobic materials will easily lose their superhydrophobicity under harsh conditions, such as mechanical abrasion and even a slight finger press.^{[39,40](#page-8-0)} Hence, water impact test and tape peeling test are carried out to investigate the mechanical durability of the superhydrophobic materials. During the process of water impacting, the distance between where the water droplets began to drop and the surface of the materials was about 0.5 m. As shown in Fig. [2](#page-2-0)a, the WCA still maintained 150.5° after impinging 3 h. Figure [2](#page-2-0)b shows WCA of as-prepared surface with different times of the peeling test cycles, where just a slight decrease in the WCA value was observed, and the WCA of the

material surface remained above 150°. These test results indicate that the superhydrophobic materials have excellent mechanical durability.

PTFE was chosen to fabricate durable superhydrophobic materials due to its excellent chemical stability, acid/alkali resistance, and high lubrication.

Fig. 1: Images of water droplets (dyed with methyl blue) with different sizes on the as-prepared superhydrophobic materials coated on various substrates: (a) glass slide, (b) corrugated board, (c) sponge, and (d) ceiling. (e) The bottom row is adhesion test. Insets in (a–d): the WCAs of water droplets on the corresponding surfaces are 153°, 151°, 151° and 153°, respectively

Figure [3](#page-3-0) schematically illustrates the preparation process of durable superhydrophobic materials. To begin, the silica was deposited onto the surface of PTFE $(SiO₂@PTFE)$ under continuous stirring to obtain the stable rough composite structures. Organosiloxane is a promising candidate for the preparation of wearresistant superhydrophobic materials, thanks to its polymerization to enhance the mechanical durability and chemical stability. The organic polymers were generated after the hydrolysis and polymerization of DEDMS under acidic condition. Moreover, the condensation reaction can occur between hydrolysate of DEDMS and the Si–OH of silica particles in which the DEDMS can endow the surface with low free energy. After a certain amount of $SiO₂@PTFE$ was added to the organosiloxane solution, the mixture was dropcoated onto various substrates and the wear-resistant superhydrophobic materials were obtained after curing.

To further study the mechanical durability of the superhydrophobic surfaces, abrasion tests were carried out.^{[41](#page-8-0)} Sandpaper (600#) was used as the abrasion surface, and with the superhydrophobic surfaces placed facedown, the abrasion material had a sliding speed of 3 cm/s. (The applied loading was 250 g, and the contact area was 25 mm \times 25 mm between the coated surface and sandpaper.) With the increase in abrasion length, WCA and SA variation on the asprepared surface are shown in Fig. [4a](#page-3-0). At the beginning of the abrasion test, the WCA increased remarkably, which attributed to the roughness of the surface increasing after sandpaper friction. When the abrasion length reached 200 cm, the material was largely consumed and part of the glass substrate was exposed, and thus the WCA began to decrease. After 250 cm of abrasion, even the materials almost wore off from the glass slide, the surface still maintained superhydrophobicity, and the WCA was 152°. These results confirm

Fig. 2: Durability tests (a) Evolution of WCAs and SAs on the as-prepared material surface under water dripping. (b) WCAs and SAs of the as-prepared superhydrophobic materials surface repeatedly torn by the adhesive tape. Insets: (a) images of the WCA of as-prepared surface after impinging different time with water drops; (b) images of water drops on the asprepared surface and the WCA of superhydrophobic surface after tape adhesion test

Fig. 3: Schematic illustration of the fabrication of superhydrophobic materials

Fig. 4: (a) WCAs and SAs measured after abrasion on the as-prepared surface. (b) Influence of immersion time, (c) influence of pH values of water droplets, and (d) influence of low temperature to WCAs and SAs of the superhydrophobic surfaces. Insets of (b) show the picture of diluted blue ink on the superhydrophobic surface after drying and rubbing. Insets of (c) show the different pH value liquids on the as-prepared superhydrophobic material (blue water droplets dyed with methyl blue, orange water droplets dyed with methyl orange, and red water droplets dyed with red ink)

that the as-prepared materials have excellent durability.

In daily life, superhydrophobic material will be exposed to diverse harsh conditions. In order to study the stability of the superhydrophobic surfaces, some tests were carried out. As shown in Fig. 4b, the WCAs and SAs of the superhydrophobic surfaces vary as the immersion time in water. The air layer disappears, the Cassie state transforms into Wenzel state after superhydrophobic surfaces are immersed in water 4 days, and the WCA slightly decreases from 158° to 153° and SA increases from 1° to 3° . After continuous immersion for more than 5 days in water, the superhydrophobicity was lost. Due to the irregular movement of water molecules, more and more water entered the micro/nanostructure of the prepared superhydrophobic material. After the microwater deposition reached a certain level, the superhydropho-

Fig. 5: SEM images at different magnifications of: (a, b) PTFE particles; and (c, d) the modified superhydrophobic materials. The scale bars represent (a, c) 5 μ m and (b, d) 2 μ m

Fig. 6: SEM images of the as-prepared superhydrophobic materials after 150 cm of abrasion. The scale bars represent (a) 20μ m, (b) 5 μm, and (c) 2 μm

Fig. 7: FTIR spectra of the unmodified PTFE particles (a) and the modified superhydrophobic particles (b)

Fig. 8: XPS survey spectra of (a) PTFE particles, (b) SiO2@PTFE particles, and (c) the modified superhydrophobic materials

bic performance of the material was reduced until lost. After being removed from water and drying at 80° C for 1 h before being rubbed with sandpaper (600#) for 25 cm, the superhydrophobicity of the as-prepared material was recovered. With the drying process, water escaped from the micro/nanostructure, so that the surface energy of the prepared material was reduced, and its superhydrophobic performance was restored again. The influence of pH on the as-prepared material was examined (Fig. [4c](#page-3-0)). The water droplets with different pH values were adjusted via HCl and NaOH and stayed on the sample surface for 1 min before the test. Obviously, the as-prepared surface could maintain superhydrophobicity in the pH values ranging from 1 to 13 because all WCAs were larger than 156° and SAs were below 4° . The experimental data

demonstrated that the superhydrophobic material had good chemical stability and that it could resist strong acid and strong alkali environments. The low-temperature resistance of the samples was evaluated by exposing it at -6° C. The CAs of the surface as a function of time are presented in Fig. [4d](#page-3-0), and the CAs were measured every 24 h. It can be seen that the WCA of the sample surface can be maintained at 150^o after placing in low-temperature environment for 12 days, indicating that the material has good lowtemperature resistance.

The appropriate micro/nanoscale rough structure is essential for constructing superhydrophobic materials. The microscopic morphology of the untreated and modified material surfaces was characterized carefully by SEM, as shown in Fig. [5.](#page-4-0) Figure [5a](#page-4-0) shows that the PTFE particles with different sizes distribute uniformly on the coating. Figure [5](#page-4-0)b shows the magnified graphic of the rough structures and shows that the PTFE particles converged together to form the micro/nanoscale hierarchical structures. In the preparation process, there are amounts of $SiO₂$ nanoparticles generated by adding TEOS under alkaline conditions, and after adding the PVP, the $SiO₂$ nanoparticles irregularly accumulate on kaolin surfaces to form micro/nanoscale hierarchical structures. Figures [5](#page-4-0)c and [5](#page-4-0)d show the surface morphologies of the durable superhydrophobic materials, and there are many micropores on the surface because of irregular accumulation of particles. The resulting micro/nanoscale roughness and micropores structure could be the main reason that the asprepared materials have such outstanding superhydrophobicity.

In order to further investigate the mechanical properties of the materials, sandpaper abrasion test was used. The samples were placed facedown on the sandpaper (600#) and abraded under a specific pressure (4000 Pa). The SEM with three different magnifications was performed to observe the surface morphology of the worn surface. Figures [6a](#page-4-0) and [6b](#page-4-0) show the low-magnification SEM images of the sample after 150 cm of abrasion, and there are apparent scratches on the surface. Figure [6c](#page-4-0) shows a higher magnification image of material surface after abrasion. It shows that the scratches were composed of porous and rough structures with micro-/nanoscale, which is the key reason that materials still have superhydrophobicity after abrasion.

The wetting behavior of surfaces is not only controlled by surface morphologies but also controlled by chemical composition of the surface. The chemical composition of the material deposited on glass substrate was investigated by FTIR spectroscopy. As shown in Fig. 7, all characteristic absorption peaks were observed in the range of 400–4000 $\rm cm^{-1}$. For the bare PTFE particles (Fig. 7a), the peak at 1148.6 cm⁻¹ was attributed to C-F stretching vibrations, and the peak at 722.43 cm^{-1} was assigned to the connection of more than four $CF₂$. As for modified superhydrophobic materials (Fig. 7b), the new absorption band at

Fig. 9: Self-cleaning investigation. (a) The deionized water dyed by methyl blue repellence experiment. (b) The images of water droplets cleaning the contaminated surfaces. (c) The coated glass slide remains clean when immersed in the muddy water

2960 cm^{-1} corresponded to the stretching vibration of the $-CH_3$ group. The adsorption bands at 1100– 1000 cm-¹ were assigned to the Si–O–Si stretching vibration, which demonstrated that the silane was successfully grafted to the SiO_2 @PTFE. The peak observed at 1647.28 cm⁻¹ was the C=O bond, and the broad band at around 3262.11 cm^{-1} was attributed to -OH, which indicated the formation of intermolecular hydrogen bonding between PVP and part of the hydrolytic DEDMS. These results indicate that PVP was successfully assembled onto the composite materials.

X-ray photoelectron spectroscopy analysis further confirms the chemical composition of the materials. Figure [8](#page-5-0) shows the XPS spectrum of bare PTFE (Fig. [8a](#page-5-0)), $SiO₂@PTFE$ (Fig. [8](#page-5-0)b), and the modified superhydrophobic materials (Fig. [8](#page-5-0)c). Figure [7](#page-5-0)a shows C1s and F1s peaks are detected which are symbol elements in PTFE particles. Compared with Fig. [8a](#page-5-0), the new peaks of O1s and Si2p were observed from the addition of the acidic silica sol, as shown in Fig. [8](#page-5-0)b. Figure [8c](#page-5-0) shows the XPS spectra of the as-prepared superhydrophobic materials. The F1s and C1s peaks in Fig. [8c](#page-5-0) are weaker than that of Fig. [8](#page-5-0)a, and these results are attributed to the modification effect of the DEDMS.

As shown in Fig. 9a, the deionized water dyed by methyl blue rolled down from the superhydrophobic surface without any traces because of low adhesion between water and material surface. Furthermore, the self-cleaning effect of superhydrophobic surface was investigated through methyl blue powder as contaminant (Fig. 9b). Hydrophilic methyl blue powder was placed on the material surface with a slight inclination angle. When water droplets (about 0.5 mL) were dripped onto the sample surface, hydrophilic methyl blue could easily dissolve in water and then roll down the surface. As shown in Fig. 9c, the as-prepared

Fig. 10: Schematic of the recycling process. The initial coating was scraped off by a blade, grounded into powders, dissolved into the solvent of ethanol at ambient temperature, and then drop-coating onto a glass substrate and dried to form the superhydrophobic material. The water droplet stayed spherical (CA = 156°) on recycled coating and rolled off easily

superhydrophobic surface could remain clean even if it was immersed into the muddy water. The micro/nanoscale hierarchical and porous structure of the materials could trap lots of air, which contributed to preventing the dirt from penetrating into the surface. Combined with the self-cleaning ability above, it can be concluded that the as-prepared materials possess an excellent self-cleaning property under different environments.

Superhydrophobic materials with excellent durability and chemical stability have attracted increasing attention from researchers. However, there is little research reported on any recyclable superhydrophobic materials. It is necessary to prepare the recycled superhydrophobic materials to overcome any potential problems caused by the low-surface-energy materials. $42,43$ Figure 10 demonstrates the recycling progress. The coating debris was scraped down by a blade and collected in a mortar, and the superhydrophobic powder was obtained after grinding. Then the powder was placed into ethanol and stirred at room temperature for 2 h. The resultant suspension was dropcoated onto a substrate via a glue dropper and then dried at 80° C for 2 h. Finally, reconstructed superhydrophobic material was obtained, and the water droplet remained spherical $(CA = 156^{\circ})$ on the recycled surface and rolled off easily $(SA = 2^{\circ})$. Compared with the initially obtained materials, the WCA and SA of the reconstructed superhydrophobic materials did not change significantly, indicating that the as-prepared superhydrophobic materials could be completely recycled and reused. The prepared superhydrophobic material has recyclable properties because crushing and grinding did not destroy the superhydrophobic structure and its surface properties at the micro/nanosize but only destroyed its macroscopic deposition

form. The ground and crushed powder was dried and dispersed in ethanol. After recoating, it was dried and then self-assembled into a new superhydrophobic material.

Conclusion

In summary, we have developed a simple, cost-effective, and environmentally friendly method to fabricate durable, chemically stable, and recyclable superhydrophobic material by drop-coating. The fabricated materials can be easily coated onto various substrates, which will expand its application range. In addition, the as-prepared superhydrophobic materials possess excellent durability, which can maintain superhydrophobicity after 250 cm sandpaper abrasion with a load of 250 g. The superhydrophobic materials also exhibit fascinating chemical stability after a series of rigorous tests, such as excellent resistance toward corrosion liquids, low temperature, and water dripping, and even after immersion in water for 7 days, they still have superhydrophobicity. Importantly, the debris scraped from the superhydrophobic materials can be recycled and easily reused to prepare the superhydrophobic materials. These properties will greatly accelerate and broaden the real applications of superhydrophobic surfaces.

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Conflict of interest The authors declare no conflict of interest.

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