PAPER

Fast fabrication of silicone-modified polyurethane/SiO\textsubscript{2} composite superhydrophobic coating with excellent anti-icing and self-cleaning behaviour

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Abstract

The superhydrophobic coating has many excellent properties, but due to the limitation of poor surface mechanical stability and sophisticated preparation engineering, it is challenging to realize large-area application. In this study, a robust superhydrophobic coating with excellent performance was prepared by a simple and fast method. The modified silica nanoparticles was sprayed on the hydrophobic polyurethane coating, and the superhydrophobic surface with a water contact angle of 160° and sliding angle of 2° was successfully prepared after curing under UV lamp for 3 min. The coating can be applied to the surface of all kinds of substrates, and it still has a static water contact angle of 150° after 400 cycles of friction on 500 mesh sandpaper. Furthermore, the coating with verified self-cleaning and anti-icing properties exhibits superhydrophobicity to strong acid and alkali solutions. This simple, fast and environmentally friendly preparation method is expected to realize the wide application of superhydrophobic coatings.

1. Introduction

The surfaces of natural animals and plants like butterfly wings and lotus leaves provide inspirations in the manufacture of artificial superhydrophobic surfaces, which possess a water contact angle (WCA) greater than 150° and a sliding angle (SA) less than 10° [1]. In the past two decades, tremendous effort has been devoted to prepare multifunctional superhydrophobic surface, due to the potential applications including anti-icing [2], anti-fouling [3], oil/water separation [4], anti-corrosion [5], and self-cleaning [6]. Previous research has established that superhydrophobicity is attributed to low surface energy surface and micro and nanometer roughness structure [7–9]. Among them, roughness structure is more critical in fabricating superhydrophobic surface, due to this roughness structures can hold air in its cavities, thereby reducing the contact area between the droplet and the surface. However, the unique hierarchical structure is usually fragile and can be easily destroyed, resulting in a sharp decrease in WCA and loss of superhydrophobicity.

To date, two main methods have been proven effective to improve the mechanical durability of superhydrophobic coatings. One solution is fabricating dual-scale roughness structures on the substrate, due to the microscale roughness structures provide protection for the fragile nanometre protrusions during mechanical abrasion [10]. Chao H et al manufactured a superhydrophobic coating combining both micro- and nano- structures. Compared with the nano-scale roughness superhydrophobic coating, the coating with dual-scale hierarchical structures shown better wear-resistance in abrasion test [11]. However, complicated manufacturing processes restrict the practical application of the hierarchical roughness superhydrophobic coatings.
On the other hand, the mechanical durability of superhydrophobic coatings can also be improved by strengthening the adhesion between the roughness structures and the substrate. Some film-forming polymers such as epoxy resin, polydimethylsiloxane (PDMS), and polyurethane are often added in the coating, bonding the micro and nanometer structures to the substrate [12–14]. Tang Y et al. constructed a high performance superhydrophobic coating with excellent abrasion resistance using a mixture of polyurethane/molybdenum disulfide (MoS2) [15]. Xue C et al. fabricated a durable and robust superhydrophobic surface by spraying the suspensions of polystyrene (PS)/silica nanoparticles and PDMS [16]. In addition to blending with nanoparticles to make durable superhydrophobic coatings, the film-forming polymers can also act as a layer of binders to adhere robust micro and nanometer structures to the substrate. Manca M et al. prepared durable superhydrophobic surfaces by bonding trimethylsiloxane modified silica nanoparticles onto the organosilica matrix, which showed an excellent water repellency [17].

In this study, we demonstrated a feasible method to prepare a mechanically robust superhydrophobic coating by covalently bonding modified silica nanoparticles to the polyurethane layer. First, hydrophobic polyurethane prepolymer and modified silica nanoparticles suspension were sprayed on the substrates respectively to form a double-layer structure. Then, the interfacial crosslinking of IEM-SiO2 and Si-PUA and the self-crosslinking Si-PUA were initiated by 1173 under a UV lamp. Due to the roughness structures constructed by modified silica nanoparticles, the superhydrophobic coating has a WCA of 160°. Meanwhile, the superhydrophobicity was maintained after 400 cycles of friction in the coating wear-resistance test, showing good mechanical properties. In addition, this mechanically robust double-layer coating with verified self-cleaning and anti-icing properties also enhances the adhesion of the coating to various substrates, which is conducive to the industrial application of the superhydrophobic surface.

2. Experimental

2.1. Materials
Pentaerythritol triacrylate (PETA), isophorone diisocyanate (IPDI) and 2-Isocyanatoethyl methacrylate (IEM) were supplied by Maclean Biochemical Technology Co., Ltd (Shanghai, China). 2-Hydroxy-2-methylpropiophenone (1173) and dibutyltin dilaurate (DBTDL) were provided by Shanghai Titan Technology Co., Ltd (China). Silica nanoparticles (30 nm) and ethyl acetate were purchased from Aladdin Reagent Co., Ltd (China). Hydroxy-terminated polydimethylsiloxane (PDMS-OH) was purchased from Shanghai Resin Factory Co., Ltd (China).

2.2. Synthesis of modified silica nanoparticles
The silica nanoparticles (1 g), which has been dried in a vacuum oven at 200 °C for 2 h, were dispersed in 50 ml ethyl acetate. Then, IEM (3 g) and DBTDL (0.02 g) were added in the SiO2 suspension stirring at 70 °C for 4 h. Finally, IEM modified silica nanoparticles (IEM-SiO2) were obtained after washing and drying. The synthetic route of IEM-SiO2 is shown in figure 1.

2.3. Synthesis of silicone-modified polyurethane prepolymer
As shown in figure 2, the preparation of the Si-PUA prepolymer is divided into two parts. First, the reaction of PDMS-OH and IPDI (2:1:1 in molar ratio) was initiated by the DBTDL under the N2 atmosphere at 45 °C. The first part reaction last for 3 h. In the second part, the temperature was raised to 65 °C. Then PETA was added dropwise to isocyanate-terminated polyurethane (PDMS-NCO) in a molar ratio of NCO:OH = 1:1. Silicone-modified polyurethane acrylic (Si-PUA) prepolymer was obtained after 3 h.

2.4. Preparation of IEM-SiO2/Si-PUA superhydrophobic coating
The preparation process of IEM-SiO2/Si-PUA coating is shown in figure 3. First, Si-PUA prepolymer and 1173 (mass ratio of 97:3) were dissolved in ethyl acetate to make a homogenous resin solution with mass loading of 20 wt%. Then the Si-PUA prepolymer solution was sprayed onto the cleaned aluminum substrate. After 5 min, IEM-SiO2/1173 (mass ratio of 97:3) solution with the content of 1–4 wt% was sprayed onto the modified polyurethane layer, respectively. The interfacial crosslinking of IEM-SiO2 and Si-PUA and the self-crosslinking Si-PUA were initiated by 1173 under a UV lamp. After 3 min, a mechanically robust superhydrophobic coating was prepared.

2.5. Characterization
FTIR spectrum of the IEM-SiO2 and Si-PUA prepolymer were recorded on an infrared spectrometer (FTIR, Nicolet, USA) between 500 cm−1 and 4000 cm−1 at 25 °C. The thermal stability of silica nanoparticles was characterized by the comprehensive thermal analyzer (TG, NETZSCH, Germany). Surface morphology of
superhydrophobic coating was recorded by an atomic force microscope (AFM) and a scanning electron microscope (SEM, Zeiss, Germany), respectively. Surface wettability of coatings was measured by a JC2000D contact angle meter (Zhongchen, Shanghai, China) with a 5 μl water droplet at 5 different locations on the coating. In the abrasion test, the as-prepared coating (5 cm × 5 cm) with a load of 100 g was placed on 500 mesh sandpaper, and moving 20 cm along the ruler on the sandpaper was recorded as one cycle of friction.
3. Results and discussion

3.1. Analyses of IEM-SiO$_2$

IEM-SiO$_2$ were investigated with FTIR spectral analyses, and the results are exhibited in figure 4(a). Both spectrums show a strong absorption peak at 1105 cm$^{-1}$, belonging to the stretching vibration of Si–O–Si. The peak assigned to stretching vibration of –OH is observed at 3430 cm$^{-1}$ [18]. Compared with the silica nanoparticles, there is an additional peak at 2963 cm$^{-1}$ on the IEM-SiO$_2$ spectra, which is attributed to the tensile vibration –CH$_3$. Meanwhile, some characteristic absorption peaks of IEM appear at 1730 cm$^{-1}$ and 1526 cm$^{-1}$, which are assigned to the vibration of –COO– and N–H, respectively. Furthermore, the peak embodied the vibration of the C=C structure is observed at 1637 cm$^{-1}$. Thus, those additional peaks confirm IEM was successfully introduced into the surface of silica nanoparticles.

TG analysis can further verify the successful preparation of IEM-SiO$_2$. As shown in figure 4(b), silica nanoparticles without any modification have only one weight loss before 150 $^\circ$C, which mainly due to the evaporation of the adsorbed water [19]. Apart from a weight loss about 2.6% before 150 $^\circ$C, there is another weight loss about 9.9% between 250 $^\circ$C–550 $^\circ$C in the TG curve of IEM-SiO$_2$. The second weight loss between 250 $^\circ$C and 550 $^\circ$C is mostly caused by the decomposition of the IEM structure on the surface of nanoparticles.

3.2. Analyses of Si-PUA

As shown in figure 5, a new peak at 2270 cm$^{-1}$ correspond to the N=C=O is clearly observed on the spectrum of the PDMS-NCO. Meantime, the stretching vibration peak of C=O and N–H appear at 1724 cm$^{-1}$ and 1535 cm$^{-1}$, respectively, indicating the successful preparation of PDMS-NCO. Compared with the PDMS-NCO spectrum, the peak embodied the vibration of the C=C structure is observed at 1637 cm$^{-1}$ on the Si-PUA spectrum, which proved the successful preparation of Si-PUA prepolymer.

3.3. Surface morphologies

Figure 6 shows the surface morphology of the Si-PUA/IEM-SiO$_2$ coatings with the IEM-SiO$_2$ contents ranging from 0 wt% to 4 wt%. The coating without any IEM-SiO$_2$ has a smooth surface. With the addition of 1wt% modified silica nanoparticles, the coating surface was composed of protrusions and flat resin, indicating that the
quantity of silica nanoparticles is insufficient to form roughness structures. When the content of silica nanoparticles was more than 2 wt%, IEM-SiO$_2$ were uniformly dispersed and wholly covered the Si-PUA layer. Meanwhile, the surface roughness of the Si-PUA/IEM-SiO$_2$ coatings continued to grow with the increase of IEM-SiO$_2$ content. Through the further enlarged image (figure 6(f)), it can be observed that many cavities were formed, which can hold air and reduce the contact area between the water droplets and the surface.

The micro and nanometer roughness structures of the coating was further verified on AFM. As a 3D morphology image of the prepared coating (figure 7) shown, there are evenly distributed protrusions with lots of gaps formed by aggregation of silica nanoparticles, constructing micro and nanometer roughness structures.

3.4. Surface wettability
The variation of wettability of the Si-PUA/IEM-SiO$_2$ coatings with the IEM-SiO$_2$ contents ranging from 0 wt% to 4 wt% is shown in figure 8. It was found that the wettability of Si-PUA/IEM-SiO$_2$ coating was affected by the contents of IEM modified silica nanoparticles. The WCA of the coating surface drastically increases from 105° without any silica nanoparticles to 140° with only 1wt% IEM-SiO$_2$. Meanwhile, the hydrophobicity of the
IEM-SiO₂/Si-PUA coating was constantly improved with the increasing of the IEM-SiO₂ content. Especially, spray-coating 4 wt% modified silica nanoparticles, the WCA of the surface was as high as 160°, and SA was 2°. Although the higher the content of IEM-SiO₂, the better the hydrophobic performance of the coating, excessive IEM-SiO₂ will make the nanoparticle layer on the surface too thick, which will weaken the mechanical properties. Furthermore, the superhydrophobic coatings not only have excellent superhydrophobicity, but also
have good adhesion to various substrates, as shown in figure 8(b). Besides aluminium, the prepared Si-PUA/IEM-SiO2 coating also can be formed on a variety of substrates, including plastic, paper and glass.

3.5. Mechanical properties

As shown in figure 9, a sandpaper friction test was utilized to examine the wear-resistance of the Si-PUA/IEM-SiO2 superhydrophobic coating on the aluminium substrate [20]. The change in the WCA of the coating is shown in figure 10(a). In the first 50 cycles of abrasion, there is a 5° drop in WCA of the Si-PUA/IEM-SiO2 coating. Furthermore, the WCA would not change much as the friction cycle increases. Due to the silica nanoparticles anchored on the resin layer have a strong resistance to abrasion, the superhydrophobic surface maintained a WCA of 150° and a SA of 9° after 400 cycles of friction. Meanwhile, a superhydrophobic coating composed of silica nanoparticles without modification and silicone modified polyurethane was prepared to examine the enhanced wear-resistance. By contrast, unmodified silica nanoparticles easily detached from the surface of the superhydrophobic coating under friction, causing a sharp decrease in WCA. The comparison of these two superhydrophobic coatings proves that enhancing the adhesion between modified silica nanoparticles and polymer through covalent bond is an effective method to increase mechanical durability of superhydrophobic coating.

Besides surface abrasion damages, shedding of the roughness structures also severely limits the industrial application of superhydrophobic coating [21]. In this study, we examined the adhesion between the Si-PUA/IEM-SiO2 coating and the substrate by placing the coating in the environment of ultrasonic vibration. The WCA of the coating slightly decreased in the first 40 min, due to some silica nanoparticles not covalently bonded on the surface of the resin layer detached from the surface. As time continues to increase, the WCA and SA of the surface did not change significantly. Meanwhile, the coating maintained superhydrophobicity after 100 min of vibration in an ultrasonic environment. In order to further examine the interfacial adhesion of the coatings onto the aluminium substrate, we take a cross-cut test according to ASTM D3359 for IEM-SiO2/Si-PUA superhydrophobic coating. A cross hatch cutter was used to cut the coating followed by tearing off with a 3 M 610 tape to check the adhesion to the substrate. The results are shown in figure 11. After cutting with a cut-off knife and peeling off with 3 M 610 tape respectively, the coating remained intact, which indicates a high level of adhesion of the coating onto the aluminium substrate.

For large-scale applications of superhydrophobic coatings, durability has always been a concern [22]. In the durability test of the as-prepared coating, the aluminium sheets coated with the Si-PUA/IEM-SiO2 superhydrophobic coating were immersed in strong acid solution (pH = 1), water (pH = 7) and strong alkaline solution (pH = 14), respectively. The coatings were taken out and dried after 24 h of immersion. Compared with the superhydrophobic coating placed in the air, the surface of the immersed coatings had no change, and all maintained a WCA greater than 150°. These results indicate that the coating not only has excellent durability but also protect the substrate from extreme environment. Furthermore, the superhydrophobic coating has a WCA of greater than 150° for water droplets with a pH range of 1 to 14, which proves that the coating is superhydrophobic to both acid and alkali solvents, as shown in figure 12(a).

3.6. Self-cleaning test

Water droplets can roll down on inclined lotus leaves and take away the dust along the way, keeping lotus leaves dirt-free. This is the so-called self-cleaning [23–25]. To examined self-cleaning property of the Si-PUA/IEM-SiO2 superhydrophobic coating, about 50 mg carbon black considered as dirt were placed on the uncoated aluminium and the coated aluminium respectively. As shown in figure 13, some water drops cannot
even roll off on tilt uncoated aluminium. Meantime, carbon black moistened by water droplets tightly adhered to the surface of uncoated aluminium. By contrast, the carbon black on the coated aluminium was removed entirely by fast rolling water drops, confirming excellent self-cleaning property of the as-prepared surface.

Figure 10. (a) The variation of WCA and SA in the wear-resistance test. (b) WCA and SA of the coating after ultrasonic vibration.

Figure 11. The cross-cut test for IEM-SiO₂/Si-PUA superhydrophobic coating.
3.7. Anti-icing test

Ice formation on the exposed materials or equipment may lead to severe problems in many sectors, including power transmission, wind turbines and aeroplane [26]. Many studies have proven that superhydrophobic coatings can reduce ice adhesion and delay icing, making superhydrophobic coatings an attractive candidate for anti-icing applications [27–29]. In this study, we also examined the anti-icing performance of the Si-PUA/IEM-SiO₂ coating by measuring the freezing time of the super-cooled water droplets in cold
environment. First, the blank aluminium sheet and the aluminium with coating were placed in an environment of $-10\,^\circ\text{C}$ in advance for 20 min. Then 30 $\mu\text{l}$ of water with a temperature close to 0 $^\circ\text{C}$ was placed on the surfaces of coated aluminium and uncoated aluminium respectively. As shown in figure 14, the freezing time of supercooled water droplets on the as-prepared coating is much longer than the exposed aluminium (uncoated aluminium: 5 min, coated aluminium: 37 min), confirming that the Si-PUA/IEM-SiO$_2$ coating can delay icing. This may be attributed to the air in cavities hinders the transfer of heat and roughness structures supplies less contact area for ice formation [30].

4. Conclusion

In this study, we successfully prepared a modified silicon dioxide/polyurethane (IEM-SiO$_2$/Si-PUA) superhydrophobic coating with excellent performance in combination with the light-curing technology. The coating shows superhydrophobic property when applied to the surface of plastic, paper, aluminium and glass. The coating also shows excellent mechanical properties. It still has a static water contact angle of 150$^\circ$ after 400 cycles of friction and remains superhydrophobic after 100 min of ultrasonic vibration. Meanwhile, the coating still exhibited excellent hydrophobic performance after soaking for 24 h in the environment of strong acid and strong base, respectively. Besides, this robust superhydrophobic coating with verified self-cleaning and anti-icing properties can further expand its application range.

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