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Superhydrophobic surface on aeronautical materials via the deposition of nanoparticles and a PDMS seal

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Abstract
This paper reports a stable superhydrophobic surface on resin composites through the coprecipitation process of hydroxyethyl cellulose and modified Zn-particles, subsequently with the use of PDMS seal. Surface morphologies and chemical compositions are investigated with SEM, EDS, and FT-IR. As expected, the prepared surface has the water repellency with the contact angle of 153° and the sliding angle of 1°, respectively. According to the comparison of different specimens when after the liquid impingement test, it can be reasonably demonstrated that appropriate PDMS seal improves the stability with regard to superhydrophobic surfaces. When superhydrophobic surface is soaked chronically and impinged strongly by water, its water repellency is going to reduce. However, further surface modification introduced by combustion leads to excellent water repellency again. And the sliding angle value of further modified surface is below 1°. The preparation of superhydrophobic surface is also applicable to a paper. This superhydrophobic paper is less flammable than the untreated paper. In addition, the as-prepared superhydrophobic surface exhibits good self-cleaning ability towards avoiding different types of contaminants.

1 Introduction

Superhydrophobic phenomenon is generally trend as the static contact angle of above 150° and the sliding angle of below 5° [1]. This wetting behavior is widely concerned by the research community due to the versatile applications. The popular topic of superhydrophobic applications mainly includes anti-fouling, anti-icing, anti-corrosion, anti-bacteria, and oil–water separation [2–6]. In addition, resin composite is the representative among all the aeronautical materials. It exhibits the light in weight, good mechanical properties and potential applications of blocking electromagnetic wave [7, 8]. Hence, it is going to display significant engineering values by introducing a functional superhydrophobic surface on resin composites. This type of surface can enrich the applications of resin composites.

The real-time applications of superhydrophobic surface are still limited by its stability. Two key factors, the micro-nanostructure and low surface energy, are generally accepted to create an artificial superhydrophobic surface [9, 10]. Chemical etching technique [11, 12] is a time-saving and flexible method for achieving a superhydrophobic surface. However, superhydrophobic surface by chemical etching technique is prone to lose water repellency due to the fragility of micro-nanostructure. Laser ablation technique is reported towards creating a strongly superhydrophobic micro-nanostructure [13, 14]. However, superhydrophobic surface hardly remains the low sliding angle when after an abrasion such as finger pressing multiple times. Electrochemical method [15, 16] can obtain a relatively stable superhydrophobic surface. But this method is difficulty to work in the large-scale operation, allowing for enormous energy consumption and complex process flow. Among all the preparations of superhydrophobic surface, it is a simple and effective way to obtain a stable superhydrophobic surface through the deposition of modified nanoparticles.

Up till now, perfluorinated modification has been the widely accepted method for obtaining the superhydrophobic nanoparticles [17, 18]. After that, superhydrophobic nanoparticles are generally added into the solution of epoxy resin [19, 20], PDMS [21, 22], and any other thermoset resins. Subsequently, superhydrophobic surface is fabricated on different substrates through the deposition or spraying. In fact, the method above still has the challenging issue on high cost from the perfluorinated modification. And this preparation is difficulty to obtain the surface with the sliding angle

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of below 1°. According to the demonstrations of previous researches [23–26], low sliding angle is the key to exhibit the anti-icing, self-cleaning, and even any other properties. In addition, very few researches reported a superhydrophobic surface on the substrate of resin composites. It can be explained that resin composites are so difficult to be pretreated that chemical and laser etching technique play a minor role in the water repellency. Our strategy is through an organosilicon polymer such as PDMS towards creating a stable superhydrophobic surface. PDMS with low surface energy may replace epoxy resins and achieve the similarly durable results. Stearic acid (STA) modification is conformed to the environmental sustainability and low on cost for practical applications [27, 28]. Meanwhile, zinc particles exhibit the flame-resistant property [29, 30] and the electromagnetic wave shield [31, 32]. Adding the hydroxyethyl cellulose and using a PDMS seal are conducive to improve the stability of superhydrophobic surface.

In this paper, our group fabricates a stable superhydrophobic surface on resin composites through a coprecipitation process of hydroxyethyl cellulose and modified Zn-particles, subsequently with the use of PDMS seal. The prepared surface exhibits the excellent water repellency. Meanwhile, combustion process leads to the further modification for the surface which is destroyed by the liquid immersion and impingement. This further surface modification results to the excellent water repellency with the sliding angle of below 1°. In addition, the as-prepared surface displays a degree of flame retardancy and good self-cleaning ability.

2 Experimental section

2.1 Materials

Hydrochloric acid (37 wt%), hydrogen peroxide (30 wt% in water), ethanol (99%), N-hexane (98%), and tetrahydrofuran (THF) were purchased from Shanghai Lingfeng Chemical Reagent Co., Ltd., China. Stearic acid (STA), zinc oxide (ZnO), and hydroxyethyl cellulose were obtained from Shanghai Mairuier Chemical Reagent Co., Ltd., China. PDMS (Sylgard 184 and the curing agent) was used from Dow Corning Corporation. Resin composite materials were provided by Beijing Institute of Aeronautical Material, China.

2.2 Fabrication of superhydrophobic surface

The superhydrophobic surface was obtained by three procedures, as shown in Fig. 1a. The resin composite was treated with sandpaper and then cleaned with deionized water. After that, the resin specimen was first immersed into the mixed solution (the volume ratio was 10:3 for HCl and H_2O_2) for 6 min. Subsequently, this pretreated specimen was rinsed with ethanol and deionized water, followed by drying with a stream of nitrogen.

Then, the pretreated specimen was deposited into the Zn–STA suspension (0.142 g of STA was dispersed into 15 mL of N-hexane to gain a STA solution. Under a magnetic stirring, 2 g of ZnO was added into the STA solution at ambient temperature. After stirring for 30 min, 0.1 g of hydroxyethyl cellulose was added into the Zn–STA solution after that sequentially stirring for 10 min.). After the deposition for 15 min, the specimen with covered by modified Zn-particles was dried at ambient temperature for 10–12 min.

Finally, 1 g of Sylgard 184 and 0.1 g of curing agent were dispersed into 30 mL of THF to gain a sealing solution. The specimen by the deposition above was dipped into the sealing solution for 30 s. After that, the sealed specimen was allowed to dry under the vacuum at 60 °C for 2 h and 100 °C for 1 h, respectively.

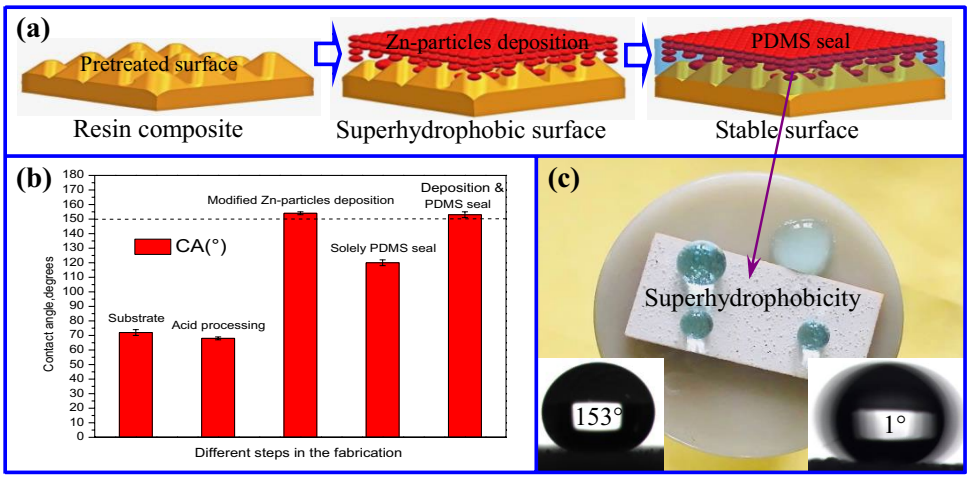

Fig. 1 **a** Scheme of the procedure to fabricate a superhydrophobic surface on resin composites. **b** Changes in wetting behaviors during the fabrication procedure. **c** Wetting behaviors of as-prepared superhydrophobic surface

2.3 Characterizations and measurements

Field-emission scanning electron microscopy (FE-SEM, FEI, Quanta 250F) with an energy-dispersive spectroscopy (EDS, Oxford AZtech x-max 80) was employed to analyze the surface morphologies and element compositions. Fourier transform infrared spectrum (FTIR) was recorded on the Ncxus670 Transform Infrared Spectrometer (Nicolet IS-10) in the range 4000–400 cm^{-1}. Thermal gravimetric analyses was carried out by a TGA/SDTA 851e instrument using a dynamic heating rate of 10 °C min^{-1} under an atmosphere of air. Surface wetting behaviors (the contact angle and the sliding angle) were measured with a contact-angle measurement instrument (JC2000D) by a contact of 5μL deionized water onto the specimen surface. The sliding angle was evaluated by tilting an inclination until a water droplet can roll off the specimen surface. Averages from six measurements are reported in the paper. Liquid impingement test was carried out under a water jet of above 50 kPa. With the definite nozzle diameter, jet height, and specimen tilted angle, the damaged situation of different specimen surfaces was recorded by a camera at the different liquid impingement time. The change of contact angle on the superhydrophobic surface was recorded with the time evolution of this surface immersed in water. The grinding clays (weight: 20 g) were added into 50 mL of deionized water to gain a clay solution. Self-cleaning performance of superhydrophobic surface was evaluated by soaking in the clay solution under the magnetic stirring. The as-prepared surface was further scratched by the uniform arrangement with a 1 mm of scratch gap. After that, the powders of cigarette ash were purposely covered onto the superhydrophobic surface. Such surface was tilted at a 12° of inclination [26, 33, 34] to test the ability of self-driven dust removal.

3 Results and discussion

3.1 Surface wetting behaviors

Figure 1 shows the procedure to fabricate the superhydrophobic surface on resin composites and indicates the change of wetting behaviors during the procedure. It can be seen from Fig. 1b that the contact angle value was 72° on the substrate of resin composites. When the surface of resin composites was deposited by modified Zn-particles, the contact angle was on the rise rapidly and the surface would indicate the water repellency. As expected, the as-prepared surface by the deposition combined with PDMS seal exhibited super hydrophobicity. However, it was difficult to create superhydrophobic surface through the PDMS seal solely. The contact angle value was 120° with regard to the surface by PDMS seal solely. Therefore, modified Zn-particles are the key to result the super hydrophobicity. As can be observed in Fig. 1c, the as-prepared superhydrophobic surface exhibited the contact angle of approximately 153° and the sliding angle of 1°, respectively.

3.2 Surface morphologies and chemical compositions

It can be seen from Fig. 2a that modified Zn-particles are covered irregularly on the prepared surface and these particles connect to each other. Per particle size is at the region of below 350 nm. EDS analysis in Fig. 2b was carried out to investigate the chemical compositions of superhydrophobic surface. The EDS results indicate that there are C, O, Si, and Zn elements on the prepared superhydrophobic surface. Importantly, existing C element on this superhydrophobic surface may be caused by STA modification to ZnO powders. Si element can be detected on this superhydrophobic surface mainly due to the surface sealed by PDMS already [35]. To further confirm the main factor for fabricating the water-repellent surface, the characterization of FT-IR was employed to analyze the change of functional groups. The FT-IR results of different surface materials are shown in Fig. 2c. The adsorption bands at 2915 cm^{-1}, 2848 cm^{-1}, and 1698 cm^{-1} correspond to the $–CH_3$ group, $–CH_2$ group and C=O group, respectively, which attributes to pure STA. By contrast, ZnO powders are without C–H adsorption bands. As expected, the adsorption band of C=O stretching vibration is shifted from at 1698 cm^{-1} to 1537 cm^{-1} [27] when after the STA modification to ZnO powders. And the alkyl chain of STA is showed on the prepared surface. The results above indicate that ZnO powders were chemically grafted with STA successfully, which leads to the excellent water repellency [19]. Furthermore, the functional groups have no change with regard to the specimen by further PDMS seal. It can be reasonably concluded that modified Zn-particles display the main factor for creating the water-repellent surface, which is highly consist with the results of Fig. 1b.

3.3 Superhydrophobic surface stability and combustion processes

Based on the previous researches [36–38], using the liquid impingement test is a simple and fast strategy to investigate the stability of superhydrophobic surface. However, the

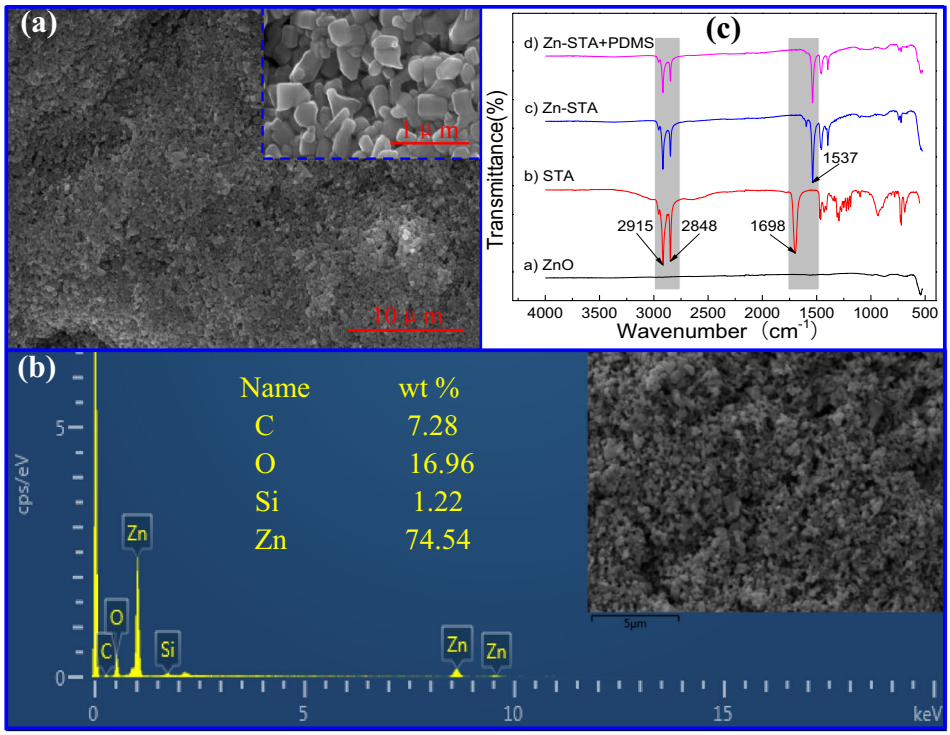

Fig. 2 **a** SEM images of as-prepared superhydrophobic surface with the different magnifications. **b** EDS data with different element compositions on the superhydrophobic surface. **c** FT-IR data of different surface materials

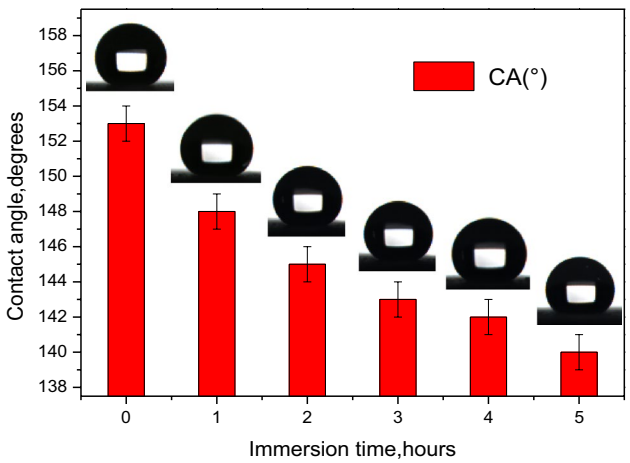

Fig. 3 Time evolution of contact angle on the as-prepared superhydrophobic surface by continuous water immersion

contact angle of superhydrophobic surface also reduces with the time evolution by continuous water immersion [39–41]. It can be observed in Fig. 3 that the water contact angle of superhydrophobic surface exhibited the downward tendency with the water immersion time increased. The contact angle value reduced to 140° on the superhydrophobic surface by 5 h of water immersion. But the modified Zn-particles on this hydrophobic surface were still covered onto the substrate of resin composites. The results of liquid impingement test in Fig. 4a can further illustrate the stability of as-prepared superhydrophobic surface. The surface prepared by modified Zn-particles deposition solely was damaged when this surface after the reported liquid impingement test for 1 min. With regard to our prepared surface by further PDMS seal, the modified Zn-particles were covered onto the substrate surface completely despite after the liquid impingement test for a long time. But the contact angle reduced to 143° when this sealed surface after the liquid impingement test for 2 min. According to the comparison of two specimens above, it can be reasonably demonstrated that appropriate PDMS seal improved the stability of superhydrophobic surface. In addition, this hydrophobic surface by liquid impingement could change to the superhydrophobic when after combustion (Fig. 4b). Carbon soot introduced by incomplete combustion resulted to the further modification for the hydrophobic surface [42, 43]. The sliding angle value of further modified surface by carbon soot was below 1°, indicating the excellent water repellency. SEM images are shown in Fig. 4c, d, corresponding to the liquid impinged surface and the further modified surface, respectively. The presence of carbon soot was confirmed on the further modified surface in the comparison of two types of surface morphologies.

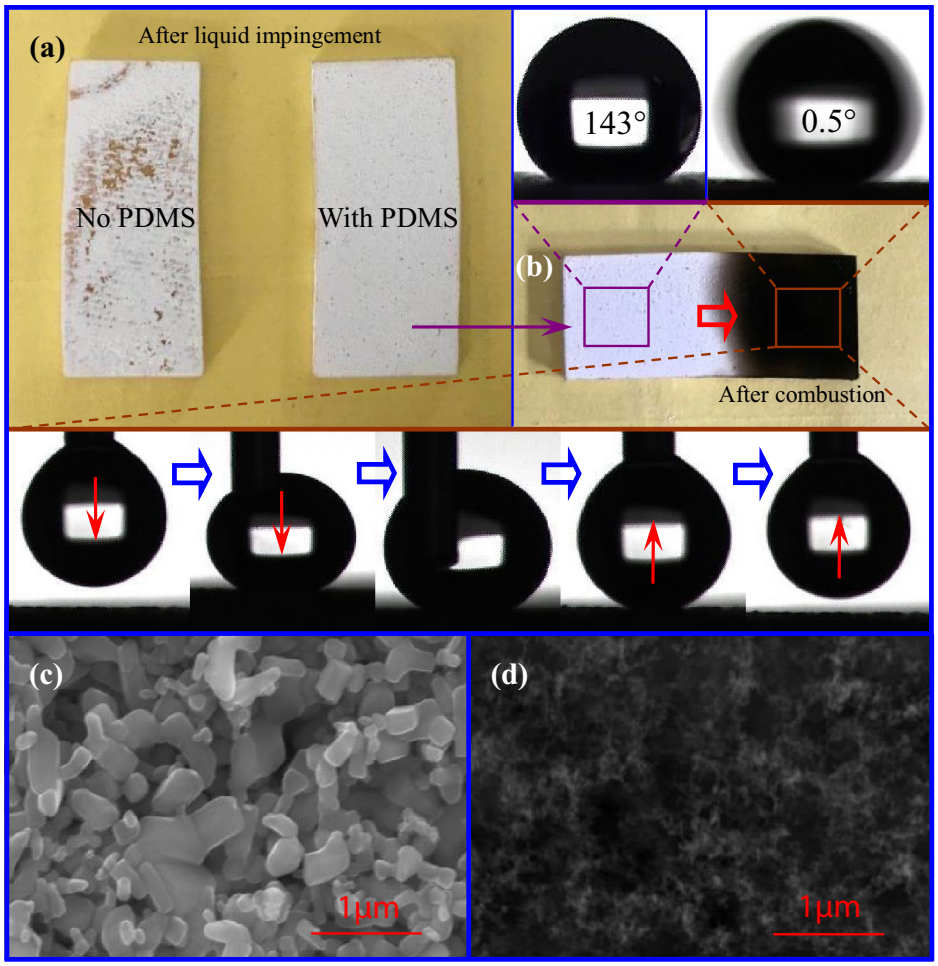

Fig. 4 **a** Stability of superhydrophobic surface respectively without or with the PDMS seal when after the liquid impingement test. **b** Changes in wetting behaviors on the PDMS sealed superhydrophobic surface by liquid impingement when before and after the combustion processes. SEM images of as-prepared superhydrophobic surface (**c**) by 50 kPa liquid impingement, and **d** further modified by combustion

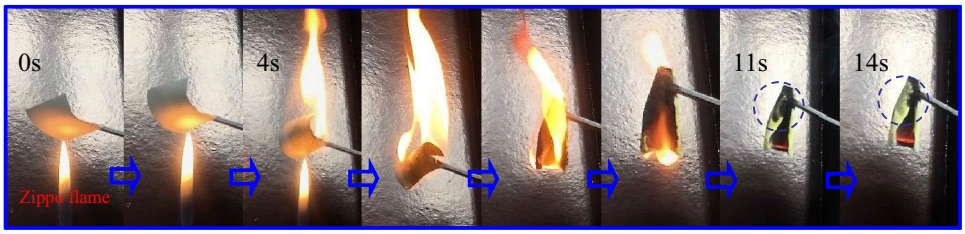

Fig. 5 Superhydrophobic paper with prepared in accordance with the experimental section and investigation of its nonflammable situation

3.4 Nonflammable property of superhydrophobic surface

As can be observed in Fig. 5, the preparation of superhydrophobic surface is also applicable to the substrate of paper to clearly illustrate the flame-resistant situation of superhydrophobic surface. This superhydrophobic paper was exposed to the zippo flame and self-extinguished when after the combustion for 11 s. With the heat increased by fire, the untreated surface of paper was ignited easily at 4 s when compared to the prepared superhydrophobic surface. Along with time lengthening, the untreated surface was burnt to residues. The analysis of thermal stability by showing in TG curves can further demonstrate the nonflammable property of superhydrophobic surface [29, 30, 44, 45]. It can be seen from Fig. 6 that inorganic ZnO powder displayed

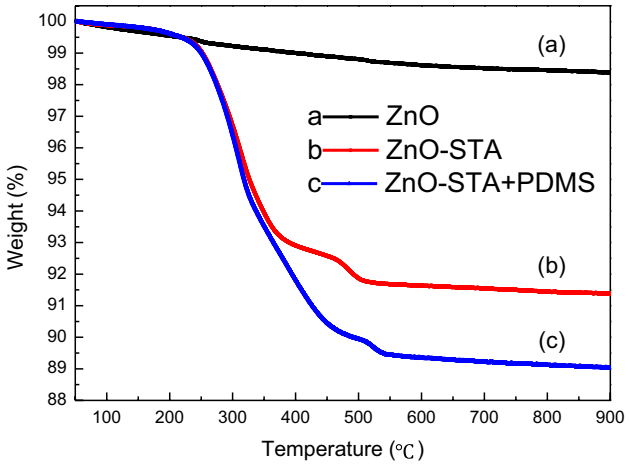

Fig. 6 TG curves of different surface materials on the as-prepared superhydrophobic surface. **a** Pure ZnO nanoparticles. **b** ZnO powders with chemical grafted by STA. **c** The modified Zn-particles combined with PDMS seal

excellent thermal stability. Its weight loss was below 2% until 900 °C due to its nonflammable property. The obvious weight loss at about 300 °C was due to the decomposition of STA caused in the superhydrophobic modification process. The superhydrophobic surface by the deposition combined with further PDMS seal (Fig. 6c) delayed the temperature of complete decomposition to approximately 550 °C, when compared to the surface by the deposition of modified Zn-particles solely (Fig. 6b). And the residue weight percentage of as-prepared superhydrophobic surface (Fig. 6c) was 89% at 900 °C. According to the results above, it can be reasonable concluded that the prepared superhydrophobic paper was less flammable than the untreated paper and this surface indicated a degree of flame retardancy.

3.5 Self-cleaning performance of superhydrophobic surface

Protective coatings are exposed inevitably in a dirty environment among the outdoor applications. Liquid contaminants can roll-off instead of sliding on the surface. And solid contaminants on the surface are carried away when the liquid rolled. This promising property [46–49] is called "self-cleaning". Self-cleaning performance of our prepared superhydrophobic surface is evaluated by soaking in a clay solution, as shown in Fig. 7a. This tested method could further simulate the real-time situation when compared to that of method by a dripped dirty droplet onto the superhydrophobic surface [50–52]. As expected, the as-prepared superhydrophobic surface exhibited good self-cleaning performance. No visible contaminants could be accumulated on this superhydrophobic surface. It can be explained that the adhesion force of dust on superhydrophobic surface is small [53, 54]. When the water droplets rolled on the superhydrophobic surface, the purposely covered powders were carried away from this surface, despite on the scratched superhydrophobic surface (Fig. 7b). Concluded in these results can indicate that the prepared superhydrophobic surface displays good self-cleaning ability towards avoiding liquid and solid contaminants.

4 Conclusions

In summary, our group obtains a superhydrophobic surface on resin composites via the deposition of modified Zn-particles combined with the PDMS seal. This prepared surface exhibits good water repellency and stability. When superhydrophobic surface is soaked chronically and impinged strongly by water, its water repellency is going to reduce. However, further surface modification introduced by combustion leads to excellent water repellency. Additionally, the preparation of superhydrophobic surface is also applicable to many material surfaces such as a paper. This superhydrophobic paper is less flammable than the untreated paper. In addition, such superhydrophobic surface shows good self-cleaning ability towards avoiding liquid and solid contaminants.

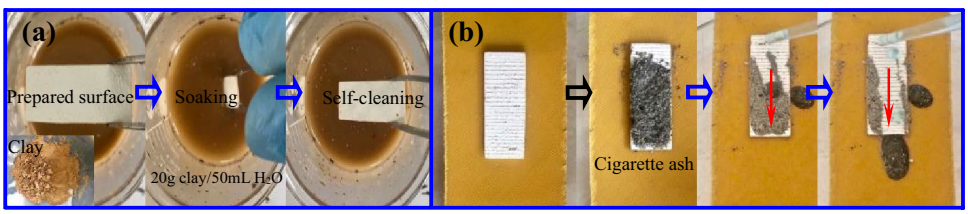

Fig. 7 Self-cleaning ability of as-prepared superhydrophobic surface towards avoiding different types of contaminants

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