Materials Research Express

PAPER

Facile preparations of superhydrophobic coatings with self-cleaning, mechanical durability, anticorrosion and easy-repairable properties

Yanlong Zhan1,2∗, Sirong Yu1,2∗, Alidad Amirfazli1, Abdul Rahim Siddiqui2 and Wen Li1,2∗

1 School of Materials Science and Engineering, China University of Petroleum (East China), Qingdao 266580, People’s Republic of China
2 School of Materials and Engineering, Jiangsu University of Technology, Changzhou 213001, People’s Republic of China

∗ Authors to whom any correspondence should be addressed.

E-mail: lxfzzl@126.com

Keywords: superhydrophobic coatings, self-cleaning, mechanical durability, anticorrosion, easy-repairable

Abstract

A facile and scalable way was developed to successfully prepare multifunctional superhydrophobic coatings (SHCs) by a one-step spraying method. The SHCs could be quickly coated on various substrates on a large scale and cured at room temperature. The as-prepared SHCs exhibited outstanding self-cleaning ability and chemical stability, and special optical properties underwater such as the silver-mirror and black-hole effects. Moreover, the SHCs had excellent anticorrosion property with a protection efficiency of 98.7% for the bare 2a12 Al alloys. Furthermore, the SHCs exhibited excellent mechanical durability and good adhesion strength to the substrate after mechanical abrasion test against 1000 grit SiC sandpaper for 1.0 m at the applied pressure of 5 kPa and scratch test. Additionally, the superhydrophobicity of SHCs could be regenerated by a simple spraying repair process after some severe abrasions, indicating a strong easy-repairable property. The present study therefore suggests that such versatile SHCs are promising for various practical applications in different fields.

1. Introduction

Since the first super-water-repellent surface was prepared [1] and the self-cleaning mechanism of the lotus effect was revealed [2], SHCs with a contact angles higher than 150° and a rolling angle less than 10° originated from all kinds of natural plants, animals and insects have drawn widespread attention [3]. In recent decades, a large number of artificial SHCs have been prepared due to their importance in basic research and potential industrial applications. For instance, the SHCs can be applied to water-repellent and self-cleaning for various surfaces [4], anti-icing in aircraft and power network [5], anti-drug in fluid transportation [6], directional water-collecting and transportation [7], antifogging for glasses [8], increasing buoyancy for boats [9], anti-corrosion for all kinds of metals and alloys [10], oil-water separation for oil-contained wastewater [11, 12], microfluidic chip [13], antibiofouling for marine and underwater equipment [14], antibacterial in biological and medical sciences [15], and energy conversion and storage [16, 17], and so forth. Based on the existing experimental research and theoretical basis, there are three conventional strategies for preparing SHCs. One is to construct appropriate rough structure on the surface of low surface energy material, the other is to modify the rough structure surface with low surface energy material, and the third is to directly endow the surface with rough structure and low surface energy material [18]. Up to now, a plenty of approaches have been successfully developed to prepare SHCs, such as template method [19], hydrothermal synthesis method [20], physical/chemical vapor deposition [21], electro-deposition method [22], electrospraying method [23], chemical etching [24], sol-gel method [25], solution-immersion method [26], lithography method [27], magnetron sputtering method [28], laser micromachining [29], and electrochemical method [30, 31], etc. However, most methods of manufacturing SHCs are too complicated, expensive and time-consuming, which is not conducive to large-scale implementation in practical applications. Therefore, it is urgent to develop straightforward, high-efficiency and inexpensive methods for preparation of SHCs. Spraying method is simple and time-saving, which can realize the
commercial production on a large scale without being restricted by substrates. In this work, the SHCs were facilely fabricated by one-step spraying method onto different kinds of substrates.

In addition to the bottleneck of the lack of large-scale preparation methods, the poor mechanical properties of SHCs are also the main factors that restrict their extensively practical applications. Although different SHCs have been prepared by different methods, due to their poor mechanical properties and surface stability, there are few reports on the practical application of SHCs. Some SHCs are very fragile and can be destroyed by just a slight touch and scratch. Undoubtedly, obtaining excellent mechanical wear resistance and stability of the SHCs is an urgent requirement for its practical applications. In addition, good corrosion resistance is also very important for metals and alloys. In some extreme service environments, the damage of the coatings will inevitably lead to the failure of the surface functions. Therefore, the easy-repairable property is essential to prolong the service life of the SHCs. To this day, a few literatures have studied the mechanical durability and easy-repairable property of the as-prepared SHCs.

In consequence, there is no doubt that excellent mechanical durability, anticorrosion and easy-repairable properties are very significant for the SHCs in practical applications. In this work, a facile and scalable way was deployed to successfully fabricate SHCs by one-step spraying method. The SHCs could be quickly coated on various substrates on a large scale and was cured at room temperature without any thermal treatments. The as-prepared SHCs had outstanding self-cleaning ability, good chemical stability in acidic and alkaline environments and special optical properties underwater. Moreover, the SHCs exhibited excellent anticorrosion property for 2a12 Al alloy and mechanical durability. In addition, the SHCs presented easy-repairable property, after severe abrasion, the damaged area could regenerate superhydrophobicity by a simple spraying repair process. The above-mentioned characteristics eliminated the main obstacles to the practical application of SHCs.

2. Experimental

2.1. Materials and equipment
Fluorocarbon resin (FEVE) was purchased from Anhui Linghu Paint Co., Ltd, China. Zinc oxide (ZnO with average particle size of about 2 μm), silicon oxide (SiO2 with average particle size of about 50 nm), hydrochloric acid, acetone, anhydrous ethanol, sodium hydroxide and hexadecanoic acid were purchased from Sinopharm Chemical Reagent Co., Ltd, China. All chemical reagents are analytical reagent. 2a12 Al alloy, Al and FPA alloy were purchased from Lanxiang Aluminum (Shanghai) Co., Ltd, China. Ceramic chips, wood, fiber cloth, sandpaper and weights were purchased in the market. Ultrapure water was produced by laboratory. Magnetic stirring apparatus was purchased from Shanghai Mei Yingpu instrument and meter manufacturing Co., Ltd, China. Spraying apparatus was purchased from EKOH industries incorporation, China.

2.2. Preparations of SHCs
Firstly, 2a12 Al alloy, Al and FPA alloy were polished with 500 grit sandpaper to remove the oxide layer and construct a certain roughness to increase the adhesion strength between the coatings and substrate. Then, 2a12 Al alloy, Al, FPA alloy, ceramic chips, wood and fiber cloth were cleaned with acetone, anhydrous ethanol and ultra pure water respectively and then dried. The next step, FEVE, curing agent, ZnO, SiO2 and hexadecanoic acid were uniformly mixed at a ratio of 20:4:1:1:1 by magnetic stirring for about 1 h. The diluent was added according to the degree of viscosity. Following, the uniform mixtures were respectively sprayed onto above-mentioned different substrates with a pressure of 0.3 MPa and a spray gun with a nozzle diameter of about 0.5 mm. The spraying distance between the spray gun and the substrates maintained about 30 cm. Finally, the coatings were cured and dried at ambient temperature for several hours. After these processes, the SHCs were obtained (as shown in figure 1).

2.3. Characterization techniques
The surface morphology and structures were surveyed by field emission scanning electron microscope (FESEM, JEOL, JSM-7800F, Japan) under vacuum environment at an accelerating voltage of 10 kV. The chemical compositions were analyzed by x-ray photoelectron spectroscopy (XPS, Thermo Scientific ESCALAB 250Xi, USA) with Al-Kα irradiation and all binding energies were calibrated using the adventitious carbon (C 1s: 284.8 eV) as the reference. The wettability was characterized by water droplet contact angles and rolling angles measured by employing contact angle measuring instrument (Easydrop, Krüss, Germany) with automatic liquid dispensing system. The contact angles and rolling angles were obtained by averaging values measured with an ultra-pure water droplet of 10 μl at five different positions on each sample surface. The corrosion resistance was investigated by electrochemical workstation (Corrtest Instrument, CS300X, China) according to potentiodynamic
polarization method. The scratch test was carried out by using scratch tester (Modern Environmental Engineering Technology, ZHY, China).

3. Results and discussions

3.1. Wettability, morphology, and chemical compositions

The wettability of the selected research subjects was shown in figures 2(a) and (b), they were basically hydrophilic (the contact angles of 2a12 Al alloy, Al, FPA alloy, ceramic chips were less than 90°) or superhydrophilic (the contact angles of wood and fabric were 0°, the water droplets spread out completely on the surface). After being coated, they all became superhydrophobic surfaces. The maximum contact angle of SHCs on 2a12 Al alloy surface was 165.8°, the rolling angle was around 2.8°, which proved that the as-prepared SHCs possessed excellent static and dynamic hydrophobic properties. This kind of SHCs was universal and not limited by the specific substrates, which could be applied to the surface of a variety of materials such as metal, alloy, ceramics, wood, fiber, etc.

SEM images of the SHCs at different magnification times were shown in figure 3. As shown in figure 3(a), the surface morphology of SHCs was composed of many ravines and hills-like structures. The surface of ravines and hills-like structures were covered with a good deal of ZnO and SiO2 micro-nano particles, as shown in figure 3(b). It could be seen from figure 3(c) that the ZnO and SiO2 micro-nano particles were wrapped up and implanted in the FEVE matrix. As shown, a rod-shaped structure with a larger size with the ZnO was presented, while a smaller granular structure with the SiO2 was presented. As depicted in figure 3(d), the scale of ZnO and SiO2 was between tens of nanometers and several micrometers, and they interlaced each other to form a coral structure. The double hierarchical morphology provided an essential structure for the formation of superhydrophobic state.

The XPS spectra of the SHCs were shown in figure 4. The XPS spectra confirmed the existence of C, O, F, Zn and Si. It could be seen that the SHCs presented strong signals of C 1s, O 1s, Zn 2p and Si 2p, and a very weak signal of F 1s. The Zn 2p peaks located at 1022.0 eV and 1045.0 eV were assigned to the zinc atom of ZnO. The Si 2p peak located at 103.3 eV was assigned to the silicon atom of SiO2 [36]. The weak signal of F 1s proved that the fluorine content in fluorocarbon resin was relatively low. Figure 4(g) showed the XPS high-resolution spectrum of C 1s. The peak at 284.6 eV was assigned to the carbon atom of C–C and C–H. The peak at 286.1 eV was assigned to the carbon atom of C–OH. The peak at 287.6 eV was assigned to the carbon atom of C=O. The peak at 289.1 eV was assigned to the carbon atom of O–C=O. The C 1s peak locating at 290.2 eV was assigned to the carbon atom of –CF2– [37]. These results demonstrated that ZnO, SiO2 and hexadecanoic acid were not involved in the chemical reaction. It can be concluded that ZnO and SiO2 were used as additives to increase the surface microcosmic roughness. Here it should be indicated that in the present work, fluorocarbon resin not only acted as a matrix but also played a role in reducing the surface energy of the system. Because fluorocarbon resin was a mixture, there was no specific value for the surface energy. Nevertheless, it can be determined that the surface energy could be less than 50 mN m⁻¹. The weak XPS signal proved that the fluorine content in fluorocarbon resin was relatively low. Hence, in order to further reduce the surface energy of the system, the hexadecanoic acid with a surface energy of about 33 mN m⁻¹ was used as low surface energy substance to further reduce the free energy of the coating system.
3.2. Self-cleaning effect

The lotus leaf can get out of silt without staining because of its self-cleaning effect. The SHCs with self-cleaning ability are of great significance for its extensively practical applications. The self-cleaning effect of the as-prepared SHCs on 2a12 Al alloy substrate was studied by using 300 grits Fe$_3$O$_4$ powder as pollutant. The 2a12 Al alloy substrate without SHCs was used as a control experiment. Schematic diagram and processes of self-cleaning behavior of as-prepared SHCs was depicted in figure 5. The SHCs have a high static contact angle, a small dynamic rolling angle and a low adhesion force. Water droplets have a strong adhesion to most of dust that fall on the surface of the object in the actual situation. Due to the micro-nano hierarchical structure of the SHCs, dust and other pollutants only contact with the peak of the micro/nano-scale surface (as shown in figure 5(i)), resulting in a smaller van der waals force between pollutants and SHCs compared with the normal surfaces because of a point contact rather than surface contact [29, 38]. This self-cleaning effect may also be the result of the high capillary force generated by the water droplets and the weak adhesion of the powder particles to the SHCs [39]. So the water droplets are easy to take away dust and other pollutants attached to the surface. It was obvious that the spherical water droplets could roll rapidly and remove the Fe$_3$O$_4$ powders on as-prepared SHCs even if the Fe$_3$O$_4$ powders had stronger adhesion to the surface and heavier mass compared to dust contaminants, resulting in a fresh and clean surface (as shown in figures 5(a)–(f)). As a contrast, the water accumulated on the substrate without SHCs and could not carry away the contaminants (as shown in figures 5(g)–(h)). The self-cleaning process depicted that the contaminated surface became clean after the water droplet rolled off the surface. Therefore, it can be concluded that the as-prepared SHCs can protect substrates from being contaminated in practical applications.

Figure 2. Contact angles of SHCs on different substrates: (a) optical photos and (b) contact angles of water droplets on coated and uncoated surfaces of different substrates.
Figure 3. SEM images of SHCs at different magnification times (a) 250 × (Inset was image of water droplet on the surface); (b) 5000 ×; (c) 25000 ×; (d) 50000 ×.

Figure 4. XPS spectra of the SHCs: (a) XPS survey spectra; (b) C 1s; (c) O 1s; (d) F 1s; (e) Zn 2p; (f) Si 2p; and (g) high-resolution of C 1s.
3.3. Chemical stability and optical property

The application environment of SHCs is usually more complicated, and facing different degree of acidic and alkaline environments. Figure 6(a) showed the variations of the contact angles and rolling angles of the SHCs on 2a12 Al alloy substrates with the pH of the droplet. The different pH value of the water droplet was adjusted by hydrochloric acid and sodium hydroxide. When the water droplets were strong acid and alkali, the superhydrophobicity decreased slightly, which might be due to the presence and exposure of ZnO and SiO2 in the coating system. Even so, it was obvious that the prepared surface still maintained superhydrophobicity in the pH range of 1.0 to 13.0. The results showed that the prepared SHCs had good chemical stability in both acidic and alkaline environments.

The SHCs can be applied not only in the atmospheric environment but also in the underwater environment. Hence, their stability and other characteristics in underwater environments are equally important. When the prepared SHCs on 2a12 Al alloy substrates were immersed in water and viewed at a different angle, some interesting and striking phenomena were observed. As shown in figures 6(b) and (c), the surface appeared as a ‘silver-mirror’ effect and ‘black-hole’ effect, respectively. When the prepared SHCs was inserted into water at about 30° from the vertical and 45° from the horizontal, the silver-mirror phenomenon could be observed (as shown in figure 6(b) in red triangle region). For a superhydrophobic surface immersed in water, the so-called silver-mirror effect has been reported. In the present work, the surface appeared as a silver mirror, when viewed at an inclined angle, this mirror-like phenomenon was still kept due to the Cassie-Baxter state [40]. As a result, the trapped air layer between water and the SHCs could effectively prevent surface wetting the underwater. In addition, because the refractive index of water is greater than that of air, when the angle of light is appropriate, the interface between water and trapped air of SHCs can produce total reflection phenomenon [41]. The underwater SHCs may be innovatively used to make liquid-core optical fibers. However, to our knowledge, it was the first time that this kind optical property of black-hole effect had been reported about superhydrophobic surfaces. When the prepared superhydrophobic sample was inserted vertically and at 45° from the horizontal into water, black-hole phenomenon could be observed (as shown in figure 6(c) in red triangle region). The surface of the underwater part presented black, and the light seemed to be absorbed by the SHCs acted as a black hole. This phenomenon is also understandable. Because the micro-nano rough structure and the trapped air of the SHCs worked together, the light was incident at a certain angle and scattered and refracted, but could not be reflected into the viewing angle and appeared black. This novel optical phenomenon of SHCs may be able to be
applied to the invisibility of various underwater equipments. The result suggested that the immersion in water would not greatly affect the surface wettability states. In other words, the as-prepared coatings can maintain superhydrophobicity as it was immersed in water. In addition, the as-prepared SHCs presented amazing optical properties, which broadened the application ranges of SHCs.

3.4. Anticorrosion property

Metal and alloy materials and their products are often faced with serious corrosion problems. Therefore, the anticorrosion property of metal-based SHCs is a critical factor to determine the practical application in various fields. The electrochemical workstation is usually used to study the corrosion resistance of SHCs based on the potential dynamic polarization method. The potentiodynamic polarization curves of the bare 2a12 Al substrate and superhydrophobic 2a12 Al surface measured in 3.5 wt% NaCl solution were depicted in figure 7. The corrosion potential ($E_{corr}$) and corrosion current density ($I_{corr}$) obtained by Tafel extrapolation from the potential polarization curve were shown in table 1. In a typical polarization curve, the lower the corrosion current density is, the lower the corrosion rate and the better the corrosion resistance is $^{39, 42}$. As well, the more positive the corrosion potential is, the better the corrosion resistance is $^{43}$. Consistent with theoretical results, experimental results proved that the superhydrophobic 2a12 Al surface had much better corrosion resistance than the bare 2a12 Al substrate. It could be noted that $I_{corr}$ of the superhydrophobic 2a12 Al surface decreased by 2 orders of magnitude compared to that of the bare 2a12 Al substrate, which meant that the SHCs provided very effective protection against 2a12 Al corrosion. In addition, $E_{corr}$ of the superhydrophobic 2a12 Al surface was more positive than that of the bare 2a12 Al surface (as shown in figure 7 and table 1).

In addition to theoretical qualitative analysis, quantitative analysis was performed. The protection efficiency (PE) of Al alloy can be calculated by following formula $^{44, 45}$:

$$PE = \left(1 - \frac{i}{i_0}\right) \times 100\%$$

Where $i$ represents the corrosion current density of the Al alloy after coating, and $i_0$ represents the corrosion current density of the bare Al alloy. The larger the value of PE is, the better the corrosion resistance of the surface prepared under certain conditions is. According to this formula, the protection efficiency of superhydrophobic 2a12 Al surface was calculated to be 98.7%. Hence, it could be concluded that the as-prepared SHCs possessed a good anticorrosion protection for the bare 2a12 Al alloy. The excellent anticorrosion performance of SHCs is

| Sample                  | $E_{corr}$ (mV) | $I_{corr}$ (A cm$^{-2}$) |
|------------------------|-----------------|-------------------------|
| bare 2a12 Al substrate | $-845.5$        | $1.04 \times 10^{-5}$   |
| superhydrophobic 2a12 Al surface | $-605.8$        | $1.39 \times 10^{-7}$   |

Figure 7. Potentiodynamic polarization curves measured in 3.5 wt% NaCl solution for the bare 2a12 Al substrate and superhydrophobic 2a12 Al surface.
mainly attributed to the superhydrophobic performance. On the one hand, the air retained in the micro-nano structure maintained a stable air/liquid interface and inhibited the corrosion of the corrosive medium. On the other hand, the reduced surface area with a low surface energy greatly slowed down the reaction rate of the corrosive medium [46]. The corrosive tests demonstrated that the as-prepared SHCs provided excellent anticorrosive properties, and such versatile SHCs can be applied to the corrosion protection of other various metals and alloys.

3.5. Mechanical durability
The mechanical stability is of great significance to the practical application of SHCs, and it determines the service life of SHCs in practical use. In this work, the mechanical abrasion test and scratch test were employed to assess the mechanical durability of the as-prepared SHCs on 2a12 Al alloy substrates. Mechanical abrasion tests were generally utilized to be an effective method to assess the abrasion resistance of the SHCs [45]. Therefore, the similar method was took to implement abrasion tests by using 1000 grit SiC sandpaper as an abrasion surface, and a pressure of 5 kPa was applied to the sample, a speed of 5 mm s\(^{-1}\) was carried out to move (as shown in figures 8(a)–(b)). The mechanical abrasion test of SHCs with contact angle of 165.8° was implemented at different abrasion distance. The test result showed that the surface still presented superhydrophobicity with a contact angle of 151.5° and a rolling angle of 9.8° after abrasion for 1.0 m at pressure of 5 kPa (as shown in figure 8(c)). Figure 9 displayed the SEM images of SHCs after abrasion for 1.0 m at pressure of 5 kPa. As shown in figures 9(a)–(b), a few groove-like scratches were observed on the abrasion surface, the surface was slightly damaged by the sandpaper abrasion and the surface roughness had a certain decline. Even so, it could be seen from figures 9(c)–(d) that the surface still had a good micro-nano hierarchical structure. As a result, the surface still remained superhydrophobicity (see illustration in figure 9(a)).

The scratch test was carried out to evaluate mechanical adhesion strength of SHCs. The test method referred to the implementation of the international standard ISO 12137-2. The tip radius of the pointed stylus was 0.5 mm, and the applied load was 200 g. The experimental process of the scratch test of the SHCs was depicted in figure 10. The experimental result displayed that the SHCs did not fall off in a large area, and it also demonstrated that the surface still remained superhydrophobicity after destructive scratch test. The FEVE matrix had strong mechanical strength and adhesive strength and could form a tough polymer matrix to maintain a durable microstructure. In addition, ZnO and SiO\(_2\) micro-nano particles had good mechanical stability and wear resistance, they were coupled with each other to show good mechanical properties. It could be concluded that as-prepared SHCs possessed excellent mechanical durability and good adhesion strength to substrate.

3.6. Easy-repairable property
Generally, the SHCs are faced with the problem of lacking mechanical strength and are easily damaged in actual use. The limited mechanical durability is considered to be one of the main bottlenecks in advancing the practical application of SHCs. In addition to improving the abrasion resistance of the SHCs, the research and development of easy-repairable surfaces is another effective way to deal with the problem of inevitable mechanical damage [47, 48]. Such SHCs can easily repair the loss of superhydrophobicity and renovate superhydrophobicity. Generally, the easy-repairable SHCs are prepared by integrating inorganic particles and organic compounds. After being damaged by an external influence such as abrasion, scratching, and other damages, the superhydrophobicity can be renovated by a straightforward retreatment process such as soaking.
Figure 9. SEM images of the SHCs after abrasion for 1.0 m at applied pressure of 5.0 kPa at different magnification times (a) 500× (Inset was image of water droplet on the surface); (b) 2000×; (c) 10000×; (d) 30000×.

Figure 10. Experimental process of the scratch test of the SHCs on 2a12 Al alloy substrates: (a) before the scratch tests; (b) after the scratch tests.
spraying, and remodification, etc. Superhydrophobic performance are determined by surface micro-nano structure and chemical composition, so the principle for any repairs is to reconstruct surface roughness structure and endow low surface energy to the coatings [5]. In the case of unavoidable damage under harsh conditions of use, the novel easy-repairable property of the SHCs is of great significance for practical applications.

Even though the as-prepared SHCs exhibited good mechanical abrasive resistance under normal use and abrasion, it still lost the superhydrophobic properties under severe abrasion conditions. After conventional abrasion, the rough structure and low surface energy substances of SHCs on 2a12 Al alloy substrates were partially destroyed (as shown in figure 11(a)). However, after severe abrasion, the rough structure and low surface energy substances of SHCs were almost completely destroyed (as shown in figure 11(b)). Interestingly, due to the simplicity and efficiency of the preparation method of this study, it provided an opportunity for the construction of regenerated SHCs. After a simple spraying repair process, the damaged area reappeared superhydrophobicity (as shown in figure 11(c)). The rough structure and low surface energy substances of the surface were reconstructed. The easy-repairable feature initiates a new approach to extend the practical application life of SHCs, and will bring a wide range of new applications in various fields.

4. Conclusions

The main bottlenecks in the practical application of SHCs are the inextensibility of the preparation methods and the inability to adapt to the complex application environment. In this work, a facile and scalable way was deployed to successfully fabricate multifunctional SHCs by one-step spraying method. The SHCs could be quickly coated on various substrates on a large scale. The as-prepared SHCs demonstrated outstanding self-cleaning ability, chemical stability in acidic and alkaline environments, and special optical properties underwater. Moreover, the as-prepared SHCs had excellent anticorrosion property with a protection efficiency of 98.7% for the bare 2a12 Al substrate. Furthermore, the SHCs exhibited excellent mechanical durability and good adhesion strength to the substrate. In addition, the as-prepared SHCs presented easy-repairable property, after severe abrasion, the damaged area could regenerate superhydrophobicity by a simple spraying repair process. Such scalable and versatile SHCs provided valuable researches and prospective practical applications on various fields.

Figure 11. Schematic illustration of abrasion and repairing processes of SHCs on 2a12 Al alloy substrates: (a) normal abrasion; (b) severe abrasion; (c) after repairing.
Acknowledgments

The work was supported by the National Natural Science Foundation of China (52073127), National Natural Science Foundation of Jiangsu Province (BK20191478, BK20191039), and Changzhou Sci & Tech Program (CZZ20190020 and CM20193004).

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

ORCID iDs

Wen Li https://orcid.org/0000-0001-8100-2275

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