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REVIEW

An overview of surface with controllable wettability for microfluidic system, intelligent cleaning, water harvesting, and surface protection

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Abstract
Wettability is an important property of material surfaces, in which surfaces with controllable wettability have important application prospects in anti-corrosion, anti-fog, anti-icing, self-cleaning, fluid drag reduction, oil-water separation and water collection, etc. In this paper, the development of surface controllable wettability was reviewed from several aspects. The first consisted of methods used to change the chemical compositions and microstructures of material surfaces by conditions like light, temperature, pH, and electric potential as external stimuli. On the other hand, the development processes utilized for preparing different types of wettability surfaces by means of reactive ion etching, photolithography, particle hybridization, and nanopattern growth were summarized. Based on the analysis of the current research status of the controllable wettability surface of materials, the problems which need to be solved in the future are put forward.

Keywords Superhydrophobic · Superhydrophilic · Controllable wettability · Smart surfaces

1 Introduction

Surface wettability is a basic property of solid materials. In recent years, the materials with special wettability have been used in many applications like oil/water separation [1, 2], self-cleaning [3, 4], water collection from fog and condensation [5, 6], anti-icing [7], anti-fogging [8], drag reduction [9], and anti-corrosion [10]. Nowadays, more research has been focused on the reversible conversion of two or more wettability behaviors on the same surface, so that the materials can better meet the needs of practical applications. Wu et al. [11] prepared a variety of asymmetric hybrid grids with different directional water transport behaviors (Fig. 1a). Among them, the asymmetric hybrid grid with bidirectional Janus structure greatly improved the rate of water collection. These hybrid meshes have huge potential applications for alleviating water shortages. Esmaeilzadeh et al. [12] fabricated new type of nanocomposites with selective superhydrophobic, superoleophobic-superhydrophilic, and superamphiphobic surfaces (Fig. 1b). This will provide a variety of practical applications in different areas, including condensate banking removal in gas reservoirs or oil-water mixture separation. Tran and Chun [13] created superhydrophobic metal surfaces with adjustable water adhesion (Fig. 1c). These surfaces can be used for complex practical applications such as controlled microdroplet transportation and microfluidic systems. Fu et al. [14] invented an iodine-oxidized diene-based rubbers polymer coating (Fig. 1d). After oxidation, this polymer coating changed from hydrophobic to amphiphilic, with stronger ice resistance. This coating is full of possibilities in the practical application of anti-icing in various fields such as roofing, wire cable. He et al. [15] demonstrated flexible and superwettable bands with superhydrophobic-superhydrophilic microarrays (Fig. 1e), which can be used for sweat sampling and analysis. Such wearable band-based

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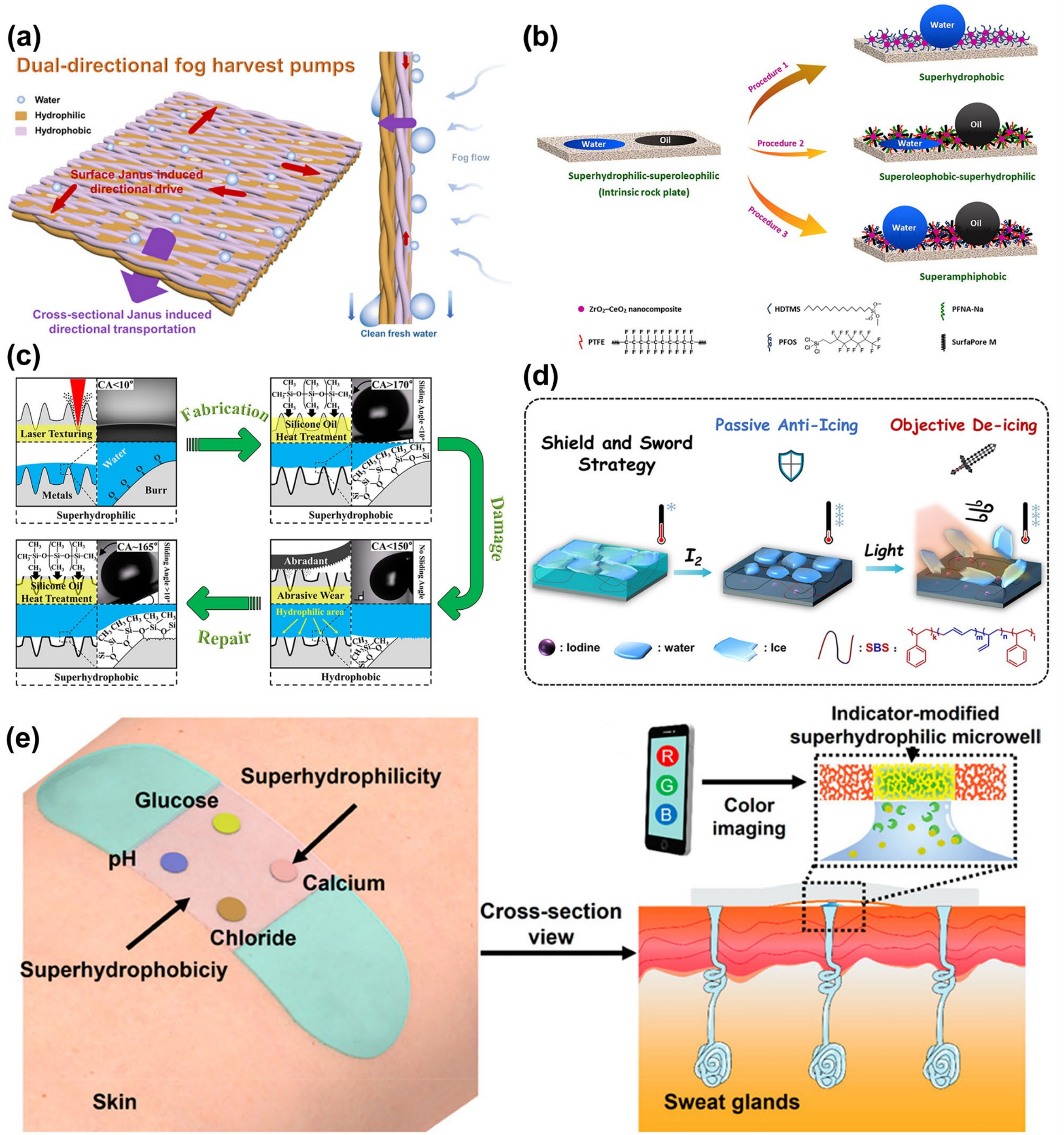


Fig. 1 **a** Schematic diagram of the dual-directional Janus pump with the Janus and hybrid working mode. Adapted with permission from Wu et al. [11]; Copyright American Chemical Society. **b** Schematic illustration of preparing new type of nanocomposites with selective superhydrophobic, superoleophobic-superhydrophilic, and superamphiphobic surfaces. Adapted with permission from Esmaeilzadeh et al. [12]; Copyright American Chemical Society. **c** Schematic diagram of superhydrophobic surface fabrication, damage by abrasion, and the repair process with the surface chemistry, surface morphology, and associated wetting behavior. Adapted with permission from Tran and Chun [13]; Copyright American Chemical Society. **d** Schematic illustration of the "sword and shield" anti-icing strategy based on iodine-oxidized diene-based rubbers. Adapted with permission from Fu et al. [14]; Copyright American Chemical Society. **e** Concept of superwettable and flexible bands as a platform toward sweat sampling and monitoring. Adapted with permission from He et al. [15]; Copyright American Chemical Society

biosensors provide a promising route for emerging personal health care and clinical monitoring fields.

The purpose of this paper is to provide researchers with an expanded idea for preparing surfaces with special wettability. Firstly, the methods to regulate the wettability of materials by adjusting the surface chemical composition and changing the surface microarray morphology are introduced in detail, and the controlling mechanism is revealed so as to understand the influence of microstructure and chemical properties on the change of wettability. In addition, we focus on the strategy of regulating the wettability of materials by combining shape memory polymers (SMP), and develop new ways to control the wettability through different functional structures and molecular designs so as to meet a variety of applications in different situations. Finally, several methods for developing different types of wettability surfaces are systematically introduced.

2 Control of surface wettability based on responsive changes in surface chemical components

2.1 Light as external stimulus

Illumination is widely used as an external stimulus to regulate the wettability of the material surface. The principle is based on the material surface that must contain light-sensitive components. Under the stimulation of light, the surface free energy of the material would change, thereby switching the surface wettability [16]. Rosario et al. [17] found spiropyran able of achieving reversible changes between ring-opening and ring-closing under UV and visible light irradiation, thereby realizing the reversible switching of the surface contact angle (Fig. 2a). Similarly, by introducing photoresponsive azobenzene into the substrate, Jiang et al. [18] realized a reversible change in wettability using the mutual transformation between the *cis*- and *trans*- structures of photosensitive oxides, such as titanium dioxide (TiO_2), vanadium pentoxide (V_2O_5), and zinc oxide (ZnO) may exhibit two different states with or without light stimulation (Fig. 2b). The surface wettability can be controlled to a large extent using light, thermal, and chemical stability. Feng et al. [19] prepared a TiO_2 nanofilm with a special structure (Fig. 2c). The surface of the film underwent a reversible transition from superhydrophobicity to superhydrophilicity after being irradiated by UV light to yield good reversible wettability control performance. Lim et al. [20] manufactured a rose-shaped nanostructured V_2O_5 film with a reversible switch between superhydrophilic and superhydrophobic by UV light irradiation and dark storage (Fig. 2d). Qu et al. [21] reported an intelligent nano-V_2O_5/octadecylamine-coated copper network (nano-V_2O_5/ODA) with cooperative response characteristics (Fig. 2e). The intelligent network achieved light-controlled water purification, as well as removed oil phase and soluble pollutants at the same time. Degli Esposti et al. [22] obtained poly(methyl methacrylate) nanocomposite porous membranes modified with ZnO nanoparticles (ZnONPs). The as-prepared in situ ZnONPs transferred their typical UV-adjustable wetting behavior to the porous polymer structure to yield a light-adjustable wettability material.

2.2 Temperature as external stimulus

Temperature-responsive controllable wettability materials have attracted increasing attention due to their useful properties. For example, Shang et al. [23] prepared a new photothermal responsive coating (PTRC) by mixing thermally responsive poly (styrene-co-n-isopropyl acrylamide) colloidal spheres and ferrosoferric oxide (Fe_3O_4) nanoparticles with polysiloxane emulsions followed by casting on the substrate (Fig. 3a). The oil adhesion of the coating surface can be remotely adjusted by near-infrared radiation (NIR). The local surface temperature rose with NIR light irradiation, resulting in sharp changes in oil adhesion from low adhesion rolling state to high adhesion pinning states in a very short time, while adjacent regions not exposed to NIR retained their original wettability. Thus, the NIR controllable property could achieve selective manipulation of material wettability and lossless transport of oil droplets on photothermal responsive coating. To solve problems linked to ultrafiltration membrane contamination, Yaghoubi and Parsa [24] designed multiwalled carbon nanotubes and poly (N-isopropylacrylamide) (MWCNT-PNIPAAm) nanocomposite membrane based on thermally responsive and hydrophilic polymers. Note that PNIPAAm is a heat-sensitive polymer. At low temperatures (lower than lower critical solution temperature (LCST), 25 °C), PNIPAAm was in its hydrophilic state with an extended chain structure. As the temperature rose above LCST, PNIPAAm became hydrophobic, and the polymer changed from a chain structure to a spherical structure. The variations in the state of the nanocomposite material improved the antifouling performance of the ultrafiltration membrane. Similarly, Ou et al. [25] synthesized a heat-responsive polymer film with switching between superhydrophilicity and superhydrophobicity by evenly coating PNIPAAm hydrogel on the surface of thermoplastic polyurethane (TPU) microfibers (Fig. 3b). The as-obtained TPU-PNIPAAm membrane separated 1 wt% silicone oil-water emulsion at room temperature with an efficiency ≥ 99.26%. The TPU-PNIPAAm membrane separated 99 wt% silicone oil-water emulsion at 45 °C with an efficiency ≥ 99.85%. Thermal response textile is a new kind of textile with wide application potential in various fields. Wu et al.

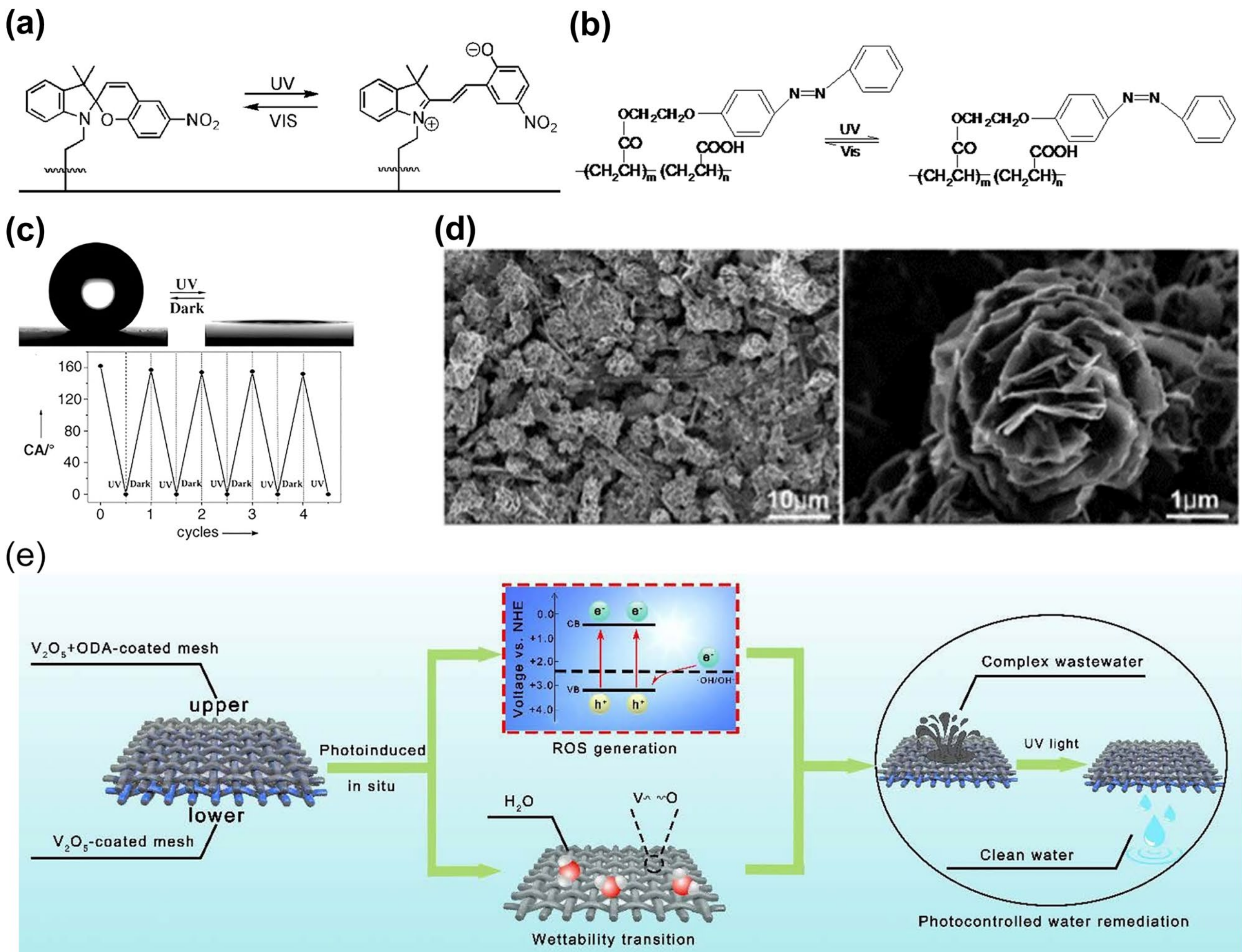


Fig. 2 **a** Diagram of surface-bound spiropyrans switching between non-polar (left) and polar (right) forms. Adapted with permission from Rosario et al. [17]; Copyright American Chemical Society. **b** Structural switching diagram of azobenzene under UV and Vis irradiation. Adapted with permission from Jiang et al. [18]; Copyright Royal Society of Chemistry. **c** Photos of TiO_2 nanocrystals before and after UV irradiation and period diagram of wettability transition. Adapted with permission from Feng et al. [19]; Copyright 2005 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim. **d** SEM image of a rose-garden-like nanostructured V_2O_5 substrate and individual rose-like spherical structure. Adapted with permission from Lim et al. [20]; Copyright Royal Society of Chemistry. **e** Schematic diagram showing the mechanism of V_2O_5/ODA coating network and corresponding auxiliary network participating in photocontrolled water restoration. Adapted with permission from Qu et al. [21]; Copyright Royal Society of Chemistry

[26] developed a simple method to manufacture polyester fabric by firstly bringing the active group to the surface by hydrolysis of polyester, and then using group (-NH_2) generated on the polyester fabric surface as the grafting site for growing PNIPAAm. The thermal-responsive wettability of polyester PNIPAAm was studied by measuring the water contact angle and the swelling rate at different temperatures. The temperature rise changed PNIPAAm grafted polyester fabric from hydrophilic to hydrophobic. The contact angle increased from 0 to about 120°. The process was reversible with a transition temperature of about 35 °C. Overall, the preparation of heat-responsive polyester fabric with intelligent cleaning looks promising for future applications.

2.3 pH as external stimulus

The effect of pH on the wettability of material surface mainly depends on the gain and loss of acid group, leading to changes to regulate the surface wettability. Cheng et al. [27] first chemically gold-plated the surface before modifying it with 1-dodecamethiol, 11-amino-undecane-1-thiol, and 12-mercaptodecanoic acid through self-assembly (SAM) coating (Fig. 3c). After treatment under acidic conditions, most of the amino groups on the material surface became protonated and covered by the hydrate layer, resulting in super hydrophilicity on the entire surface. As pH value increased from 1 to 13, the protonated amino groups became deprotonated and the methyl groups dominated, forming a

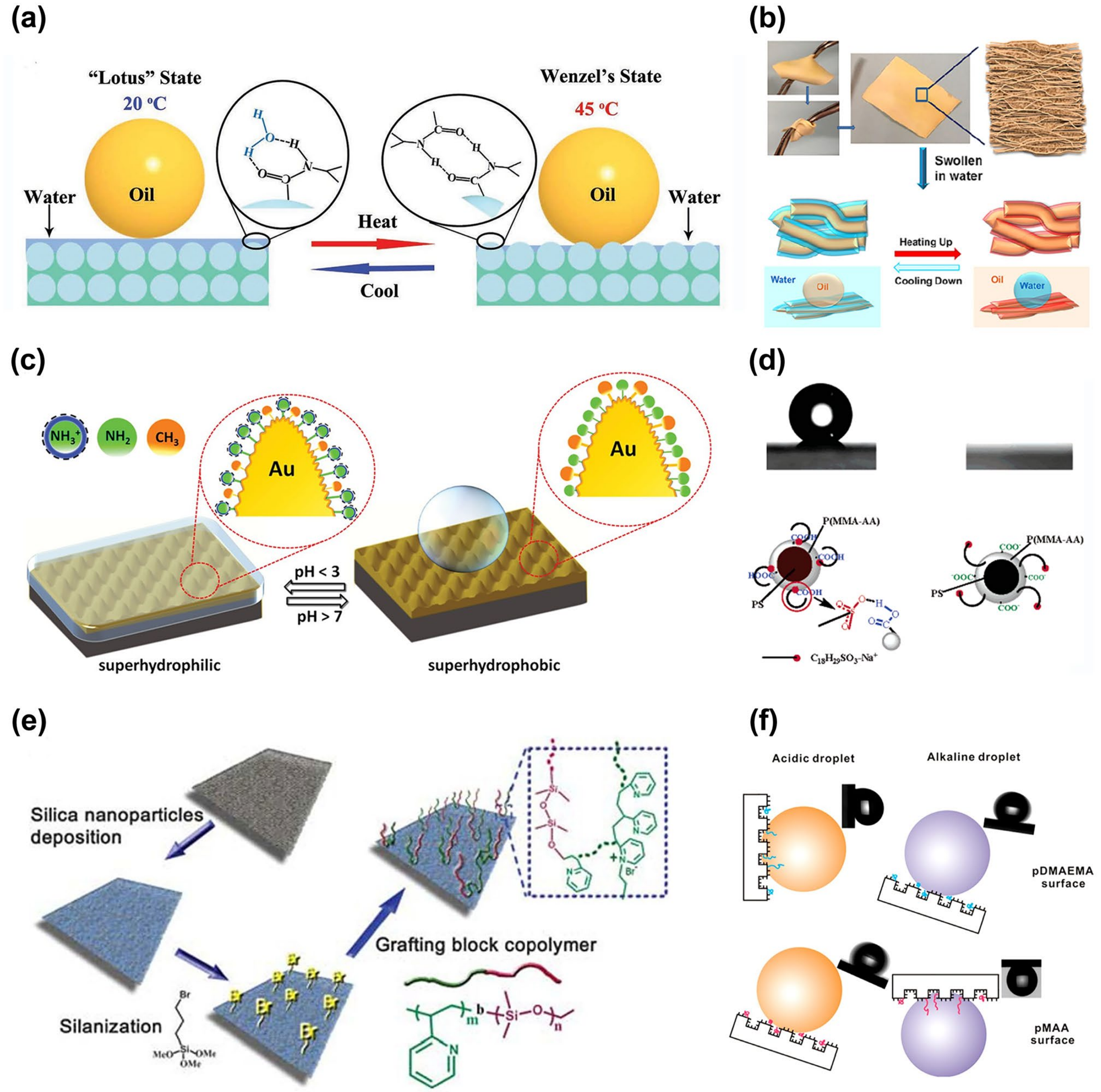


Fig. 3 **a** Schematic illustration of the prepared thermal response coating with adjustable underwater oil adhesion. Adapted with permission from Shang et al. [23]; Copyright 2019 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim. **b** Schematic diagram of twisting test of TPU-PNIPAAm composite membrane and switching wettability of TPU-PNIPAAm membrane at different temperatures. Adapted with permission from Ou et al. [25]; Copyright American Chemical Society. **c** Variation in surface wettability with pH value after modification with SAM. Adapted with permission from Cheng et al. [27]; Copyright 2013 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim. **d** Photos of the shape of water droplets on films with pH 6.0 and 12.0, and a schematic diagram of the structure of latex spheres. Adapted with permission from Wang et al. [28]; Copyright American Chemical Society. **e** Schematic diagram of preparation process of a switchable surface on a polyurethane sponge. Adapted with permission from Zhang et al. [29]; Copyright 2012 Springer Nature. **f** Schematic diagram of the adhesion transition of surfaces grafted with acid and alkali, respectively. Adapted with permission from Wu et al. [30]; Copyright American Chemical Society

superhydrophobic surface and realizing the transformation between superhydrophobicity and superhydrophilicity. Wang et al. [28] prepared poly(styrene-methyl methacrylate-acrylic acid) thin films with changes in surface wettability due to the presence or absence of hydrogen bonds between COOH and $SO_3^-Na^+$ at different pH values (Fig. 3d). Accordingly, the emulsifier sodium dodecylbenzenesulfonate (SDBS) appeared in two different conformations. At pH 6.0, the head group of SDBS represented the surface of the latex sphere, resulting in an almost inverted form of SDBS. After the introduction of $NH_3{\cdot}H_2O$ into the latex suspension, the hydrogen bond became inhibited. Also, the strong electrostatic repulsion between COO^- and SO_3^- facilitated the exposure of the hydrophilic groups COO^- and $SO_3^-Na^+$ to air due to the deprotonation of the carboxyl group to COO^-, thereby improving the hydrophilicity of the film. Currently, more research has been focused on better applications in daily life. In this view, Zhang et al. [29] investigated a grafting strategy for block copolymers using pH-responsive poly(2-vinylpyridine) and oleophilic hydrophobic poly(dimethylsiloxane) to prepare polyurethane sponges (Fig. 3e). In an acidic aqueous medium (pH = 2.0), the prepared polyurethane sponge repealed oil owing to water occupying the pores of the sponge. After washing the sponge with water (pH 6.5) and drying by nitrogen flow, the sponge was put back into the aqueous medium to recover its super lipophilicity to be used again for the selective removal of oil from the water. In addition to applications in oil-water separation, Wu et al. [30] grafted two pH-sensitive polymers, namely poly(dimethyl aminoethyl methacrylate) and poly(methacrylate), onto anodic alumina substrates to yield two pH-sensitive surfaces (Fig. 3f). By adjusting the swelling states of the two polymers, water droplets can roll off or adhere to the surface thanks to the presence of different adhesion forces. The adhesion forces also affect the boundary slip, where large slip lengths can be obtained by changing the fluid pH. The superhydrophobic surfaces with large slip lengths and responsive properties have significant applications in the development of biological devices.

2.4 Electric potential as external stimulus

The electrical potential (EP) stimulation can be used to regulate the wettability of the material surface through external stimulation, thereby a promising technique for adjusting the wettability and flow properties of water droplets on the material surface due to its fast response, non-abrasiveness, and easy application can be got. Thus, EP stimulation is widely applied in many fields, including microfluidics, drag reduction systems, controllable oil-water separation, intelligent filtration, microreactors, and microfluidic devices. For example, Isaksson et al. [31] reported an electronic wettability switch (EWS) for a solid-state electrochemical device based on two conjugated polymers (polyaniline doped with dodecyl benzene sulfonic acid and 3-hexylthiophene polymer). By switching the redox state of the conjugated polymer, the wettability of the polymer film can effectively be controlled, and the electronic transition of the wettability can be adjusted. Krupenkin et al. [32] applied electrowetting to nanostructured surfaces, where electrowetting was used to control “local” contact angles by dynamically changing the interaction force between the liquid and nanostructures (Fig. 4a). This provided new perspectives for liquid-solid interface physics research to further promote the development of new applications. Zheng et al. [33] proposed a strategy to prepare polyaniline nanofiber network by emulsion polymerization to realize oil-water separation induced by an electric field (Fig. 4b). Under the stimulation of 160 V, the superhydrophobicity of the micro-nanoscale polyaniline net can be transformed into hydrophilicity, and the selective filtration of water can be achieved at 170 V. Therefore, the separation of oil and water can be achieved under electric fields as long as the capillary electrical pressure was greater than the hydrostatic pressure of the membrane on water. The polyaniline mesh with low underwater oil adhesion and corrosion resistance may be employed for the effective control of oil and water separation even under harsh actual environments. Zhao et al. [34] reported a manganese dioxide nano-superhydrophobic film, which can immobilize water droplets on the film by applying a small direct current bias even with a large contact angle. The nearly spherical water droplets can be fixed on the substrate even for the inverted substrate. The in situ wettability regulation produced fast, efficient, and environmentally friendly smart materials with adjustable properties for biotechnology use.

2.5 Multiple external stimuli

In addition to common wettable surfaces responding to light, temperature, pH, and EP, ion-responsive surfaces [35, 36] and solvent-responsive surfaces [37, 38] could also play a key role. However, such surfaces only respond to one external stimulus while single-stimulus-responsive materials cannot adapt well to complex and changing environments. To solve this problem, some scholars have explored some multi-responsive materials. For example, Xia et al. [39] developed a dual-responsive material with good sensitivity to both temperature and pH by preparing poly (N-isopropyl acrylamide-co-acrylic acid) copolymer films on silicon substrates (Fig. 4c). The LCST of the copolymer can be adjusted according to the pH value. Under external stimuli, the film can reversibly be switched between superhydrophilic and superhydrophobic due to the reversible change in the hydrogen bond between the two components and water. The reversible conversion can be around 10 °C narrower in temperature range and relatively wider in pH range. Such a dual response characteristic resulted from the combined effect of surface chemical composition changes and surface roughness. The method of controlling wettability by changing temperature and pH has important application prospects in many fields, including microfluidic switches, drug delivery, and surfactants. Liu et al. [40] grafted stimulus-responsive

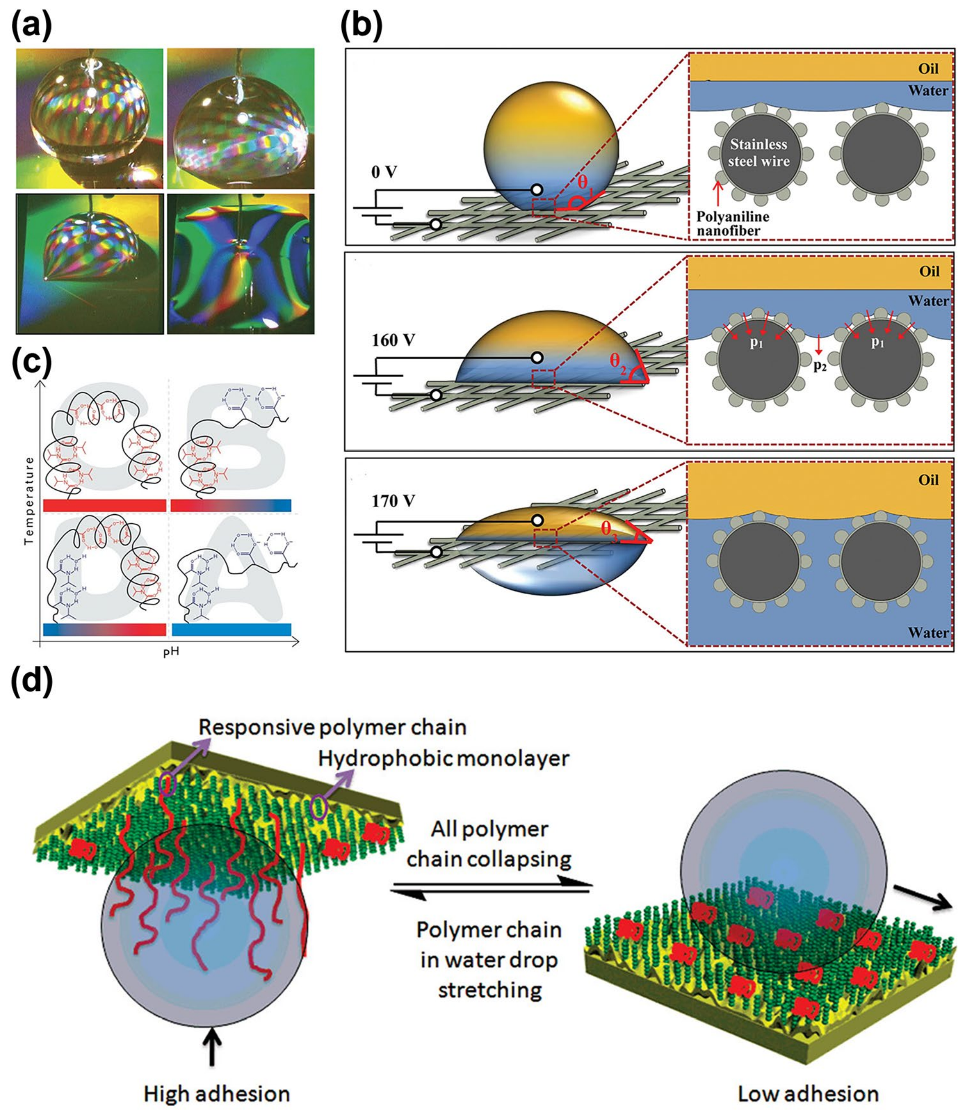


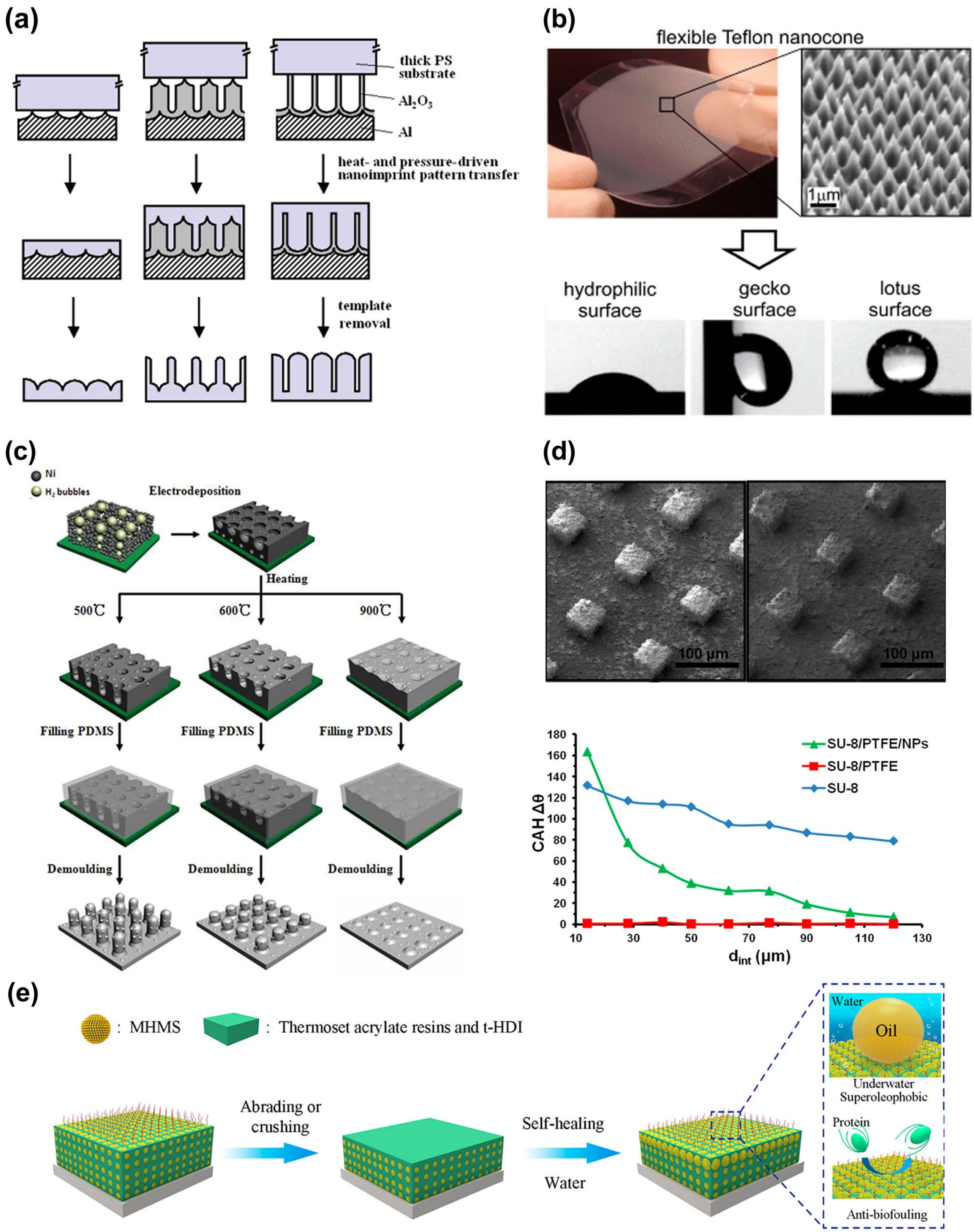
Fig. 4 **a** Photograph of droplet transitions between different wetting states via electrical induction. Adapted with permission from Krupenkin et al. [32]; Copyright American Chemical Society. **b** The schematic diagram of the electric field-induced oil-water separation process based on polyaniline mesh and corresponding schematic diagram of liquid-solid contact amplification. Adapted with permission from Zheng et al. [33]; Copyright 2016 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim. **c** Variation in hydrogen bond between copolymer and water. Adapted with permission from Xia et al. [39]; Copyright 2006 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim. **d** Mechanism diagram of hydrophobic molecule-modified Al_2O_3 substrate inducing reversible adhesion of water droplets. Adapted with permission from Liu et al. [40]; Copyright American Chemical Society

polymers (PNIPAAm and poly(dimethyl aminoethyl methacrylate)) on anodic alumina (Al_2O_3) substrates with irregular micro and nano surface topography (Fig. 4d). The spherical droplet can reversibly be switched between the pinning and rolling states due to changes in temperature, pH, and electrolyte. The author also established a general way to reversibly switch droplet mobility on a rough surface containing a polymer chain. In other words, the folding of the polymer chain resulted in non-wettable surface, thereby droplets rolling off the surface. However, the polymer in the swollen state resulted in strong interactions between the molecular chains and the droplet enough to prevent the droplet from moving on the surface. The latter can be useful in fields, such as microdroplet motion, smart coating, and self-cleaning surfaces. Huang et al. [41] studied a class of stimuli-responsive hydrogels that swell or contract under specific external stimuli, capable of reversibly changing from superhydrophobic to superhydrophilic. The obtained hydrogel responded to various stimuli, such as temperature, pH, electric, magnetic, light, pressure, gases, ions, alcohol, and many different types of biomolecules. The wettability was controlled by adjusting the intensity of external stimuli.

3 Control of surface wettability by surface microstructure

3.1 Wettability control of different types of surfaces

The geometric structure of the material surface and arrangement of micro and nanostructures would directly impact the movement of liquid droplets on the material surface, thereby changing the surface wettability. In this regard, Lee et al. [42] utilized the nano-patterned aluminum plate as the replication template in hot-pressing driven nano-imprint pattern transfer to study the surface wettability (Fig. 5a). Their


(a)
thick PS substrate
Al_2O_3
Al
heat- and pressure-driven nanoimprint pattern transfer
template removal
(b)
flexible Teflon nanocone
1μm
hydrophilic surface
gecko surface
lotus surface
(c)
Ni
H_2 bubbles
Electrodeposition
Heating
500℃
600℃
900℃
Filling PDMS
Demoulding
(d)
100 μm
100 μm
SU-8/PTFE/NPs
SU-8/PTFE
SU-8
CAH Δθ
d_{int} (μm)
(e)
: MHMS
: Thermoset acrylate resins and t-HDI
Abrading or crushing
Self-healing
Water
Water
Oil
Underwater Superoleophobic
Protein
Anti-biofouling

◂**Fig. 5** **a** Schematic diagram of heat and pressure-driven nanoimprint pattern transfer process. Adapted with permission from Lee et al. [42]; Copyright American Chemical Society. **b** High-magnification SEM image of the surface of PTFE nanocone array. Adapted with permission from Toma et al. [44]; Copyright American Chemical Society. **c** Schematic diagram of the preparation process of superhydrophobic PDMS film. Adapted with permission from Zhang et al. [43]; Copyright Royal Society of Chemistry. **d** SEM images of SU-8/PTFE, SU-8/PTFE/NPs micro-pillars and CAH values for the SU-8, SU-8/PTFE and SU-8/PTFE/NPs substrates for different column spacing. Adapted with permission from Milionis et al. [46]; Copyright American Chemical Society. **e** Working principle of the self-repairing underwater superoleophobic/antifouling coating. Adapted with permission from Chen et al. [47]; Copyright American Chemical Society

data revealed that surface wettability can be controlled by applying nano-level roughness on the polystyrene substrate. Zhang et al. [43] used the electrodeposition method to prepare underwater ultra-lipophilic nickel (Ni) and nickel oxide (NiO) surfaces with controllable morphology to clarify the influence of surface morphology on adhesion. Toma et al. [44] prepared a tunable flexible nanocone surface with hydrophobicity/hydrophilicity by a simple two-step process in which a colloidal monolayer of polystyrene beads was etched on Teflon film by oxygen plasma and controlled the surface wettability by nanocone array size and various surface modifications (Fig. 5b). The surface of the synthesized polytetrafluoroethylene (PTFE) nanocone array possessed good hydrophobicity and adhesion, with contact angle related to the aspect ratio and sharpness of the nanocone. After the modification of PTFE nanocone with gold nanoparticle films (AuNPs) to form a layered nanostructure, the surface was transformed into a superhydrophobic surface. Zhang et al. [45] designed biomimetic polydimethylsiloxane (PDMS) films with different hierarchical structures (Fig. 5c). The superhydrophobicity of the films can be adjusted by controlling the microstructure of the film. Milionis et al. [46] created surfaces with combined micron, submicron, and nanoscale roughness by using a lithographically structured SU-8 microcolumn as a substrate for continuous spray deposition of PTFE submicron particles and hydrophobic coated iron oxide colloidal nanoparticles (NPs) (Fig. 5d). SU-8/PTFE/NPs microcolumn showed adjustable contact angle hysteresis (CAH), which decreased with the increase of column spacing. Thus, self-cleaning surfaces with localized highly adhesion regions can be fabricated for localized deposition and controlled evaporation of droplets. Such surfaces can be excellent candidate for the development of smart surfaces, biotechnology, and novel antifouling materials. Chen et al. [47] successfully prepared a self-healing coating modified with hydrophilic polymer chain for the first time (Fig. 5e). The obtained material did not only show good underwater superhydrophobicity, but also superior anti-biological pollution performance. Moreover, the structure maintained its original anti-biological pollution ability even when the surface was damaged.

3.2 Reversible wettability control on same surface

Current research dealing with controllable wettability has been focused on controlling certain special wettability, and only a handful of studies have been devoted to the reversible conversion between different wettability behaviors on the same surface. In this view, Tian et al. [48] constructed a composite interface by introducing magnetic fluid based on silicon oil into ZnO nanoarrays (Fig. 6a). The wettability can be adjusted by dynamic deformation between smooth and rough microstructures under alternating magnetic fields, achieving a fast responsive intelligent interface for controllable liquid transport. Yang et al. [49] prepared a new superhydrophobic material with magnetic induction properties by spraying and magnetic-field-directed SAM (Fig. 6b). The material surface consisted of tightly packed magnetorheological elastomer (MRE) micropillars, which can alternate between folded and erected states under an external magnetic field. In the absence of a magnetic field, the MRE micropillars under droplet load tended to bend, resulting in high surface adhesion. Under the applied external magnetic field, the MRE micropillars became hard and vertically oriented, in which the vertical MRE micropillars can significantly reduce the contact area between the surface and the droplet, making the surface superhydrophobic.

The magnetically responsive surface has reversible wettability and fast magnetic response performance, providing an opportunity for the development of intelligent controllable devices. So far, the controllable wettability of thin films has attracted considerable attention. For example, Lee et al. [50] prepared MRE films composed of polydimethylsiloxane and carbonyl fluoride iron particles at different concentrations (Fig. 6c). Both the wettability and adhesion of the film can be adjusted by applying a magnetic field. The water contact angle can be changed from 100° (without a magnetic field) to 160° (with a magnetic field) and the sliding angle of water varied from 10° (with magnetic field) to 180° (without magnetic field).

Zhang et al. [51] reported an elastic polyamide membrane with a triangular network structure (Fig. 6d). The reversible switch between superhydrophobic and superhydrophilic is caused by the change of the average side-length of the triangular net-like structure after biaxial stretching and unloading, as well as the change of the surface tension of the water droplet. The excellent elasticity of the film prolonged the reversible change between superhydrophobicity and superhydrophilicity by more than 20 times.

Biomimetic superhydrophobic materials have attracted increasing attention due to their unique functions with special wetting properties. A typical example is lotus leaves, which present self-cleaning properties. Also, the

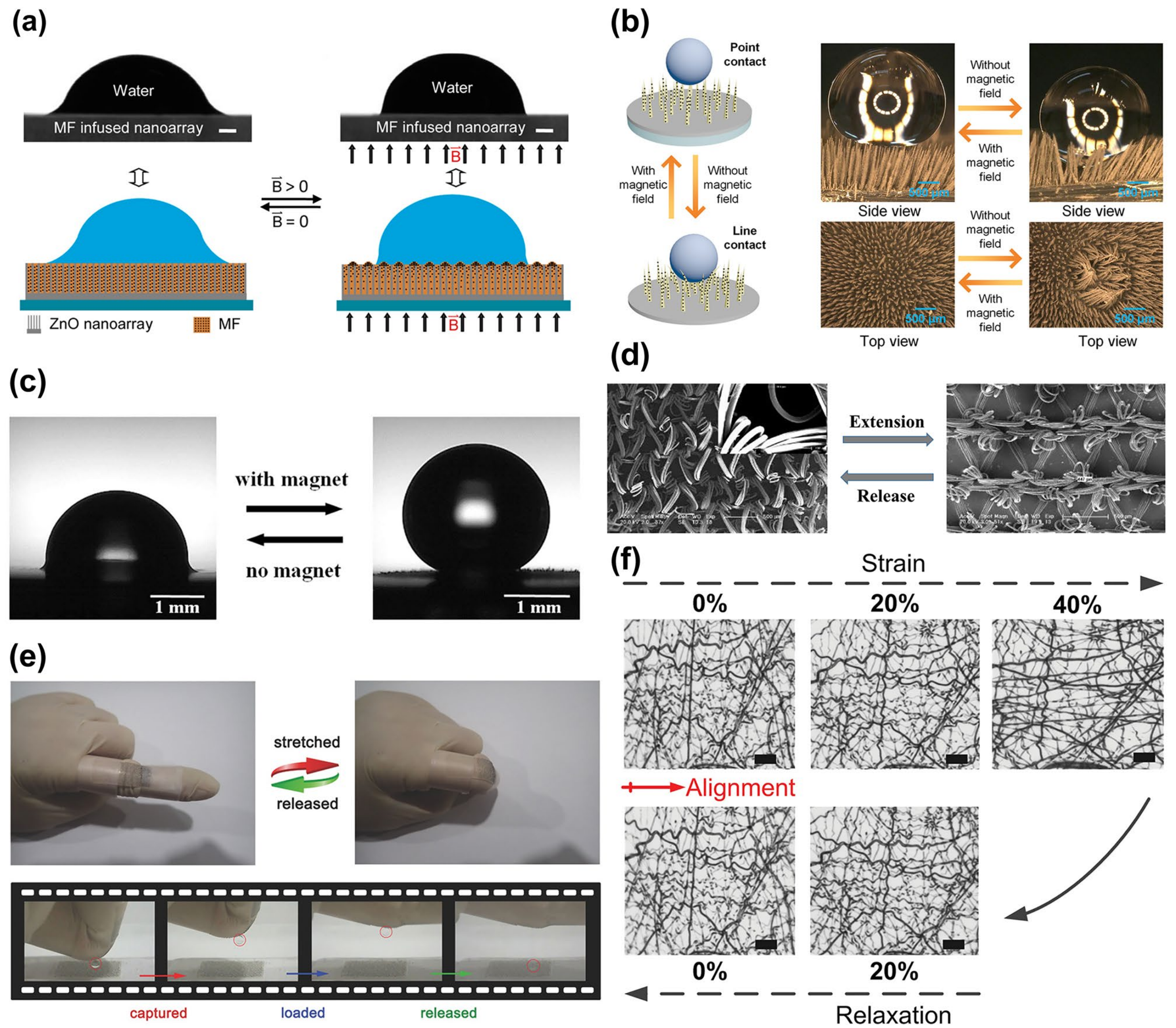


Fig. 6 **a** Schematic diagram of magnetic response wetting behavior on ZnO nanocomposite interface. Adapted with permission from Tian et al. [48]; Copyright American Chemical Society. **b** Scheme for reversible switching of the wettability and adhesion properties of the magnetically responsive superhydrophobic surface by on/off switching of magnetic field and optical microscope images showing the stiffness tunability of the MRE micropillars under an external magnet field. Adapted with permission from Yang et al. [49]; Copyright American Chemical Society. **c** Optical microscopy image of water droplets on MRE with or without magnetic field. Adapted with permission from Lee et al. [50]; Copyright American Chemical Society. **d** Schematic diagram of switch between superhydrophobicity and superhydrophilicity of elastic polyamide film. Adapted with permission from Zhang et al. [51]; Copyright 2005 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim. **e** Pressure exerted and released on the wearable surface by human movement. Adapted with permission from Wang et al. [52]; Copyright 2018 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim. **f** Optical microscopy image of complete strain-relaxation cycle. Adapted with permission from Wong et al. [53]; Copyright 2015 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim

one-dimensional arrangement of micro-nano papillae on rice leaves provides surface anisotropic wettability, and the directional arrangement of microstructures on butterfly wings leads to directional liquid-solid adhesion. Wang et al. [52] reported a skin-like superhydrophobic elastic surface that can switch between lotus leaf and rose petal shape (Fig. 6e). The direct laser writing (DLW) technology was used for the large-scale manufacturing of the skin conformal surface according to the pre-programmed skin texture on the elastomer substrate. Surface topography can be finely tuned rapidly and reversibly by simple stretching to control surface wettability with simple body movements. Similarly, Wong et al. [53] combined the polystyrene nanofiber layer with the soft elastic substrate (Fig. 6f). The anisotropic stretching and self-relaxation of the elastic substrate led to reversible changes resulting in a series of superhydrophobic wetting states on the surface. The reversible dynamic adjustment of wettability and adhesion between shapes of lotus leaves and rose petals can be achieved through continuous stretch-relaxation cycles. This bottom-up design provided a flexible platform for the fabrication of materials with controlled wettability, useful for applications in flexible microelectronics and tissue engineering.

3.3 Application of SMPs in smart surface

3.3.1 Application of SMPs in static surface wettability regulation

Based on the shape memory effect (SME) of polymers, the static wettability of surfaces can be adjusted subtly by controlling surface microscopic morphologies, such as nanoscale and microscale structures, as well as hierarchical micro and nanostructures. Various strategies combining responsive molecules with shape memory microstructures have been proposed. Li et al. [54] used polycaprolactone and allyl alcohol as substrates and Fe_3O_4 nanoparticles as a magnetic response source to prepare biodegradable nanocomposite substrates with good SME under hot water and alternating magnetic field (Fig. 7a). The state of the surface microcolumn can easily be adjusted by changing the ambient temperature.

Since the synergistic effect of dynamic changes in surface topography and the resulting restoring force could control the cell behavior, the method looks promising for the design and fabrication of scaffolds close to human tissues or organs. Lv et al. [55] studied the superhydrophobic surface made of epoxy-based SMP with specific shape memory layered micro and nanostructures applied for rewritable platforms (Fig. 7b). The surface was prepared by using SMP to replicate hierarchical structured template, and its micro/nanostructure can be reversibly controlled between permanent and temporary shapes. As a result, surfaces can acquire different wetting properties. In addition, with the help of the special memory ability of micro/nanostructures, by designing microstructure-depended patterns, the surface can be used as rewritable platforms for droplet storage chips, overcoming the drawback that structural patterns cannot be reprogrammed.

Controlled transitions in wettability behavior can also be achieved by selective surface treatments on polymer columnar arrays to create multilayer surfaces with switchable wettability. Laursen et al. [56] performed the surface treatment with trichloromethanesilane, polydimethylsiloxane, and polydopamine on acrylate-based synthetic columnar arrays to create a multilayer surface with switchable wettability (Fig. 7c). The polymer array formed a rigid upright column at low temperatures, while a flexible column with easy bending was formed at high temperatures. The static contact angle was used to characterize the polymer array. The hydrophobic surface was exposed when the column was supported, while the hydrophilic surface was exposed when the column was bent. The surface could restore its original condition by removing the external force and readjusting to low-temperature operating conditions.

3.3.2 Application of SMPs in dynamic surface wettability regulation

Based on the static wetting characteristics, the dynamic wettability can be regulated by adjusting the microstructure. García-Huete et al. [57] fabricated shape memory microcolumns by laser etching crosslinked polycyclooctene (PCO), and changed the contact angle through the shape memory

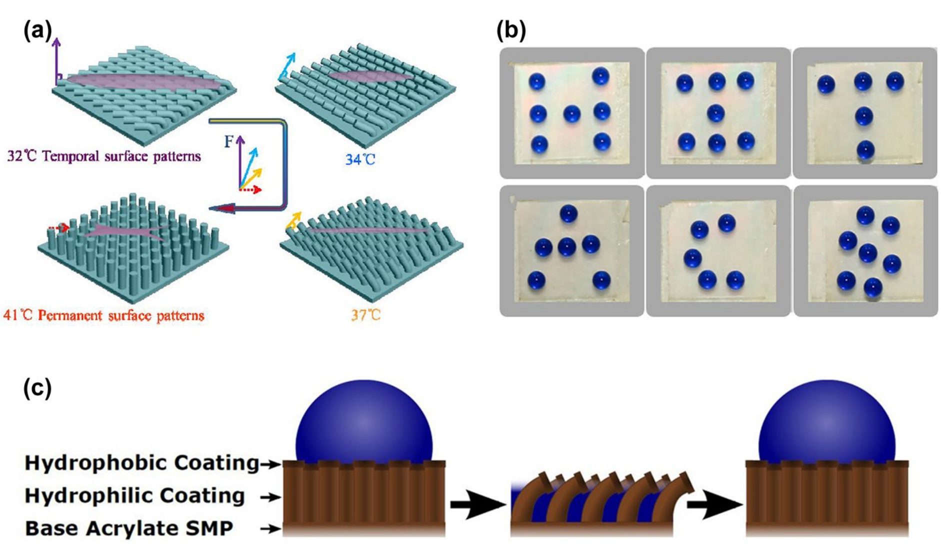


Fig. 7 **a** Schematic diagram of dynamic changes in surface microarray caused by heating (F stands for restoring force). Adapted with permission from Li et al. [54]; Copyright Royal Society of Chemistry. **b** Rewritable platform for related pattern function chip. Adapted with permission from Lv et al. [55]; Copyright American Chemical Society. **c** Schematic diagram of switchable wettability surface structure. Adapted with permission from Laursen et al. [56]; Copyright 2016 Wiley Periodicals, Inc

characteristics of PCO microcolumns under heating conditions (Fig. 8a). The surface contact angle of the deformed microcolumn decreased from 136 to 124° due to the reduction in roughness. However, the original surface structure can be restored by thermal induction, thereby restoring the original hydrophobicity. Han et al. [58] constructed a hydrophilic surface with microstructure on the styrene SMP (S-SMP) substrate by deformation of the metal film (Fig. 8b). Reversible changes in wettability were achieved by controlling the temperature. The water contact angle of the original S-SMP surface was estimated to 85°, and the contact angles of the deformed surface and recovered surface were 25° and 85°, respectively. The resulting surface would thus have potential application in microfluidic switching and controlled cell capture and release.

In addition to controlling the nanoscale structure, the droplet sliding can also be controlled by adjusting the microscale column structure. The change in the shape of the microscale column structure cannot only increase or decrease the contact area between the solid and liquid reversibly, but also control the sliding and pinning state of liquid droplets. Chen and Yang [59] used SMP to produce a recyclable inclined column through the copying process to yield different wettability inclined and recovered states (Fig. 8c). The water droplets on the deformed SMP column showed Wenzel wetting, while the droplets on the straight column displayed Cassie-Baxter state with a limited sliding angle. Based on the static contact angle and dynamic sliding angle characterization results, the SMP column array in the deformed state and the original state illustrated different wettability features. These characteristics were used to design a surface for drainage. Park and Kim [60] designed a nanostructured array of SMP columns with a periodic square lattice. The precise control of droplet motion was achieved by applying external forces on the structured SMP surface. For axial compression without bending, the authors limited the aspect ratio of the struts to 0.8 while increasing the temperature to glass transition temperature (T_g) (80 °C) and applied pressure to create different wetting conditions for the structured SMPs surface. Wang et al. [61] obtained a superhydrophobic shape memory surface by pasting a columnar structure of superhydrophobic polyurethane layer on a shape memory polyurethane-cellulose nanofiber (PU-CNF) layer. The good shape memory performance of its underlying layer facilitated the reversible SME of the surface strut microstructure during stretching and recovery (Fig. 8d). A 3μL water droplet showed a high contact angle of about 151° on the PU−CNF surface and remained in the

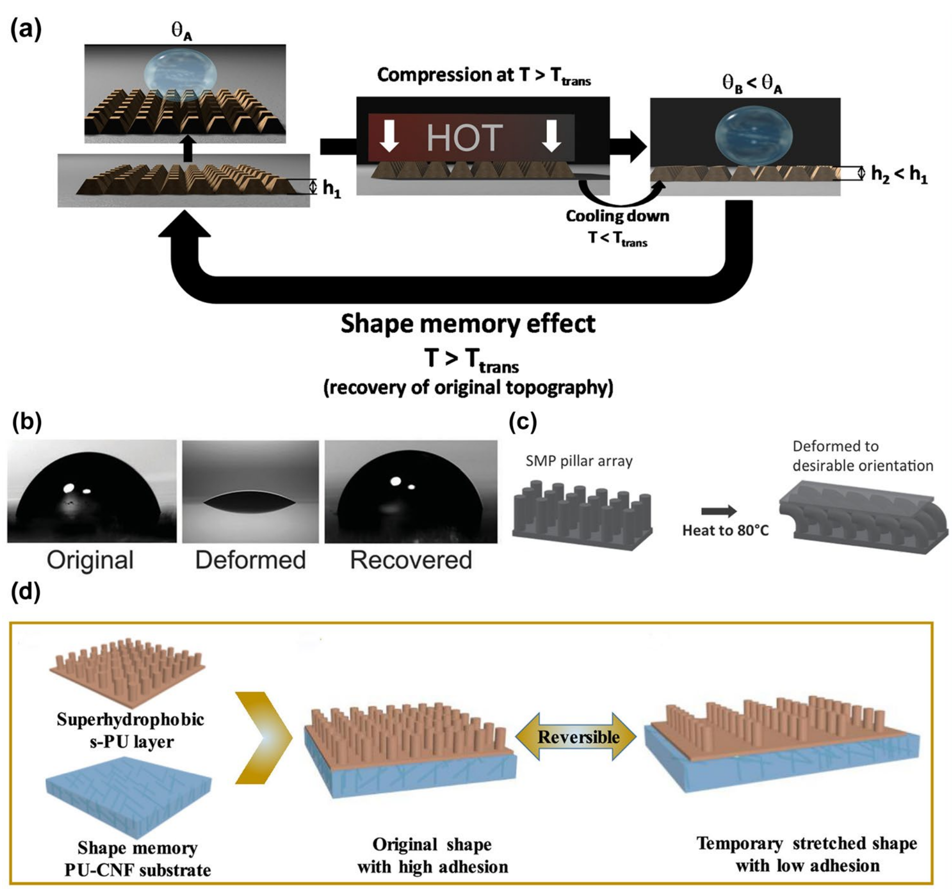


Fig. 8 **a** Schematic representation of the switching wettability of the original θ_A restored by heating and compressing the contact angle from θ_A to θ_B. Adapted with permission from García-Huete et al. [57],Open Access, MDPI. **b** Changes in contact angle of SMP surface in original, deformed, and restored state, respectively. Adapted with permission from Han et al. [58]; Copyright Royal Society of Chemistry. **c** Schematic diagram of deformation process of SMP column array. Adapted with permission from Chen and Yang [59]; Copyright 2013 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim. **d** Schematic diagram of superhydrophobic shape memory adhesion surface and its microstructure changes. Adapted with permission from Wang et al. [61]; Copyright American Chemical Society

pin state to the surface after flipping, indicating a highly viscous and superhydrophobic surface. After wetting the underlying PU−CNF layer with water, the droplets were released after being stretched by an external force. Based on the excellent shape memory performance of PU−CNF, temporary shapes can be restored to permanent shapes with higher adhesion.

Another dynamic wettability has to do with the bouncing behavior of droplets on superhydrophobic surfaces. This attracted increasing attention in recent years owing to important applications in outdoor equipment anti-icing, wire protection, aircraft wings, and other fields. The traditional static microstructure surface can only provide constant bounce characteristics but cannot change the contact time and bounce shape. However, subtly adjusting the surface microstructure on superhydrophobic SMP surfaces would not only endow the material surface with different bouncing properties, but also provide bouncing properties for droplets, such as bouncing shape, solid-liquid contact time, and bouncing direction. Song et al. [62] systematically studied the influence of the size of superhydrophobic column array on the droplet rebound dynamics (Fig. 9a). On an array of superhydrophobic pillars with a diameter of 1.05 mm, a height of 0.8 mm, and a pitch of 0.25 mm, the authors observed a typical pancake bounce phenomenon. They also proposed a facile replication spray method to fabricate a series of superhydrophobic pillars on a large scale to yield surfaces with a pancake effect, effectively reducing the adhesion during the replication process. Since the column spacing greatly influenced the bounce dynamics, the contact time and bounce shape can be reversibly regulated as long as the tilt degree of the polymer column was adjusted. Cheng et al. [63] prepared SMP surfaces by polymerizing bisphenol A epoxy resin, octylamine, and M-xylene diamine on micro and nanostructured templates (Fig. 9b). By dynamically controlling the microstructure to simulate the lotus leaf structure and rice leaf structure, the SMP wetting state can be reversibly adjusted between superhydrophobic isotropy and anisotropy. The SME of the polymer induced a surface with repeated memory behavior and different microstructure, finally achieving an intelligent

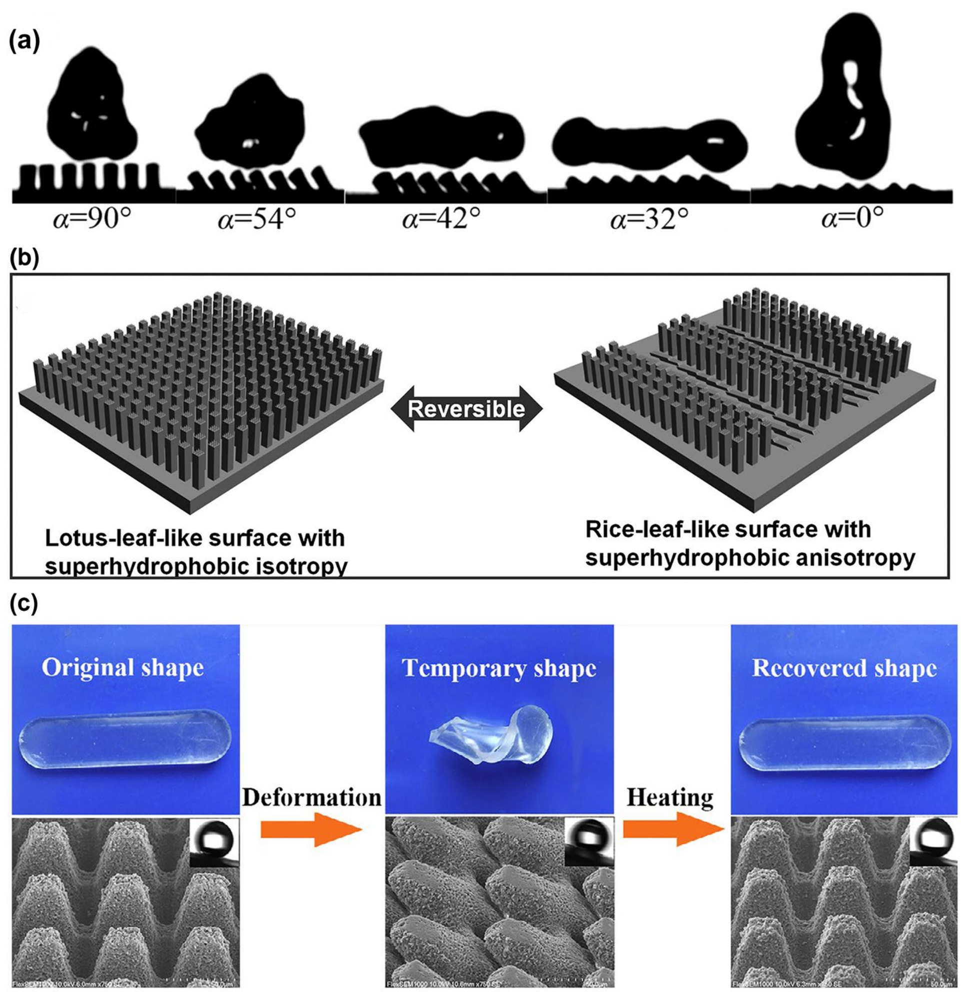


Fig. 9 **a** The rebound shape of water droplets at the moment of separation on a superhydrophobic column array with different inclination angles. Adapted with permission from Song et al. [62]; Copyright American Chemical Society. **b** Schematic diagram of surface microstructure change between lotus leaf and rice leaf structures. Adapted with permission from Cheng et al. [63]; Copyright 2017 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim. **c** Photos of SMPs film at the original shape, fixed temporary shape, and restored shape. Adapted with permission from Bai et al. [64]; Copyright 2019 Elsevier

transition between two wetting states. The material has potential applications in many other fields, such as biological separation and microfluidic devices.

Though intelligent wetting control can be achieved by adjusting the nanoscale or microscale surface structures, the above-resulting surface structures are not optimal since both natural selection and experimental studies demonstrated the perfect suitability of layered micro and nanostructures for superhydrophobic surfaces. Meanwhile, the high dimensions described above are not mechanically stable for dynamic structure control. To avoid such problems, researchers proposed a strategy to replace the single nanoscale or microscale structure by adjusting the layered micro or nanoscale structure. In this respect, Bai et al. [64] used femtosecond laser to prepare multilayer microcolumn arrays on the SMP surface followed by modification with fluoroalkyl silanes (Fig. 9c). The contact angle and sliding angle of water droplets on the surface with the graded microcolumn structure were estimated to 153.5 ± 0.6° and 7 ± 0.5°, showing good low adhesion and superhydrophobicity. After tilting the microcolumn to one side by the external load, the contact angle of water droplets on the surface decreased to about 142°, while the sliding angle increased to about 20°. The significant SME of SMPs, surface topography, and wettability were fully recovered by a simple heating process repeated for at least 10 cycles.

4 Preparation methods of controllable wettability surface

4.1 Reactive ion etching technology

Reactive ion etching technology is a dry etching process with not only strong anisotropy but also high selectivity, suitable for etching through chemical reactions initiated by ions. However, the etching process can cause severe damage and contamination of workpieces coupled with difficulty in forming very fine patterns. Hou et al. [65] patterned a silicon oxide layer by standard lithography technology followed by advanced oxide dry etching (Fig. 10a). Using the second lithography and oxide dry etching, the excess silicon dioxide (SiO_2) edges at the top of the microcolumn formed by etching were removed. A modified deep reactive ion etching (DRIE) process was then used to fabricate homogeneous nanolfibers between the side wall and the microcolumn. After the DRIE process, a hydrophobic fluoride film was deposited on the nanolayer to achieve overall superhydrophobicity, while the fluoride on top of SiO_2 was selectively removed by buffering oxide etching to form hydrophilic spots on the surface. This approach was employed to develop biomimetic hybrid surfaces with high wetting contrast enabling seamless integration of membrane condensation and droplet condensation modes. Garimella et al. [66] treated P-type silicon wafers with hydrofluoric acid for 2 min in

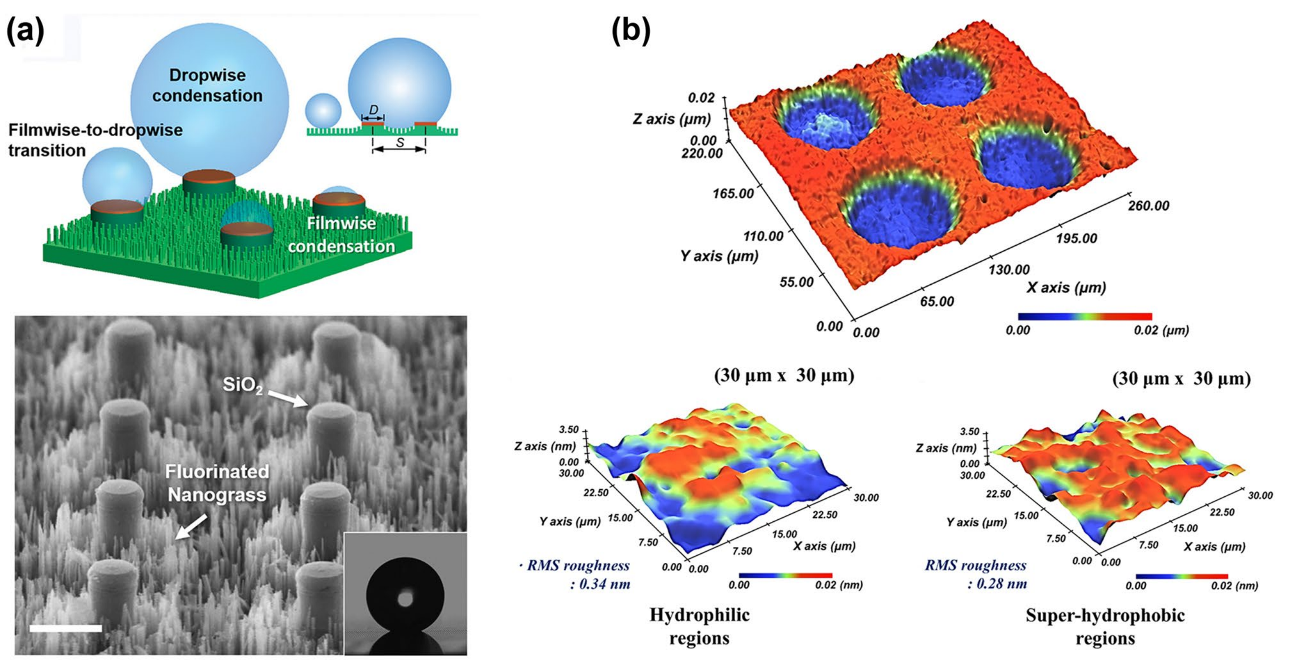


Fig. 10 **a** The non-uniform wettability of the mixed surface and the SEM image of the mixed nano-silicon surface. Adapted with permission from Hou et al. [65]; Copyright American Chemical Society. **b** A scan of the selectively hydrophilic modified superhydrophobic surface, hydrophilic region and superhydrophobic region. Adapted with permission from Lee et al. [67]; Copyright 2016 Elsevier

an acid mask. The natural oxide layer was then removed by UV light and ozone treatment. A photoresist was used for standard lithography through a rotary coater and contact printer. After the deposition of polytetrafluoroethylene (PTFE) layer on the sample by sputtering for 10 min, the photoresist layer was stripped off with acetone to expose the hydrophilic region and yield patterns with different wettability regions. The condensation and post-condensation movements of water droplets on the surfaces with different wetting abilities revealed the gathering of droplets condensed in the hydrophobic area into the hydrophilic drainage channels when water droplets condensed to a certain size, thereby renewing the dry hydrophobic area. Therefore, the surfaces can better improve the water collection efficiency. Lee et al. [67] prepared selective surfaces with dual wettability (hydrophilic and superhydrophobic) (Fig. 10b). After the preparation of superhydrophobic surface (contact angle > 150°) by radio frequency atmospheric plasma system, the active alcohol was deposited on the superhydrophobic surface by electrohydrodynamic (EHD) jet to carry out selective hydrophilic modification (contact angle about 70°). The EHD jet precisely controlled the droplet volume and facilitated the preparation of hydrophilic point arrays on superhydrophobic surfaces. Using the above steps, discrete patterns with selective hydrophilic properties were formed on the superhydrophobic surface.

4.2 Photolithography technology

Lithography is a process in which an image is copied from a mask to a substrate by a photoresist illuminated by a light source. The basic steps of the method consist of using UV light to irradiate the substrate with a photoresist through a mask so that the photoresist at the exposed sites will produce chemical changes. The photoresist of exposed or non-exposed parts is then dissolved by imaging technology, and the pattern is copied on the photoresist film. Finally, the image is copied onto the substrate by etching technology. Cho et al. [68] fabricated hierarchical polymer microstructure by a novel method (Fig. 11a). A highly ordered concave anodic aluminum oxide (AAO) pattern was first prepared by anodic oxidation. Various types of layered structures were then prepared by lithography and soft lithography using highly stable epoxy resin micropatterns as masks. After hydrophobic treatment of the surface, the selective nucleation and growth of water droplets in the grooves between the patterns were observed. Moazzam et al. [69] used desert beetles as inspiration to create biomimetic synthetic surfaces (Fig. 11b). The SU-8 super-hydrophilic surface with a contact angle as low as 10° was formed on the surface of SU-8 within 5 s by applying polydopamine coating. The SU-8 substrate was then immersed in a newly prepared solution (pH 8.5) containing dopamine hydrochloride (2 mg/ml) and polyvinyl imine (1000 MW, 2 mg/ml) for 4 h by polydopamine coating method. A raised structure was next fabricated on the SU-8 surface by lithography, and the geometry on the mask was transferred to the SU-8 photoresist surface by negative lithography. The method can be used to improve the fog capture rate using negative lithography to form bumps on surfaces followed by the formation of hydrophilic bumps on the hydrophobic surface together with the polydopamine solution coating.

4.3 Particle hybridization method

Owing to their high droplet nucleation efficiency, hybrid wetting surfaces have been developed for heat exchange [70], water harvesting [71], oil-water separation [72], and microfluidic control [73]. However, some difficulties still need to be overcome before simple, actionable, low-cost, and large-scale applications. For controllable wettability surfaces prepared by particle hybrid method, the droplet peeling effect and water harvesting efficiency of superhydrophobic surfaces would improve along with surface thermal efficiencies. Wang et al. [74] proposed a new method for sprayable mixed wetting coatings by adding hydrophilic substances (such as monodisperse nano-SiO_2 or silica powder) to a homogeneous superhydrophobic coating containing chain-like nano-SiO_2 (Fig. 12a). The comparison of the nucleation, growth, aggregation, and ejection behaviors of dewdrops on mixed wetting surfaces of different hydrophilic particles through conventional condensation tests revealed superhydrophobic particles allowing droplets to merge and separate from the surface quickly. The reason for this had to do with the hydrophilic particles on the mixed wetting surface, which can quickly capture water vapor from the air. Therefore, the mixed wetting surface containing superhydrophobic and hydrophilic particles possessed higher water harvesting efficiency.

In recent years, numerous studies have focused on preparing surfaces with different wettabilities by mixing various particles with SiO_2 particles. Zeng [75] used lithography technology to prepare four kinds of silicon wafers with different sizes and spacings. Next, they selected three kinds of particles (boron nitride (BN), silicon carbide (SiC), and molybdenum disulfide (MoS_2)) with different wettabilities to surface treat the nano-SiO_2 superhydrophobic coating and obtain a new type of superhydrophobic film. The surface contact angle reached more than 150° and the rolling angle was less than 10°. The solution can be sprayed on glass and aluminum plates to form uneven wet surfaces with mixed particles. Wang et al. [76] prepared an ultra-wettable coating by mixing superhydrophobic and superhydrophilic (TiO_2-SiO_2) nanoparticles modified with fluoroalkyl silane. The irradiation of the surface with UV light produced a hybrid super-wetting surface with superhydrophobicity and superhydrophilicity (Fig. 12b). The surface combined

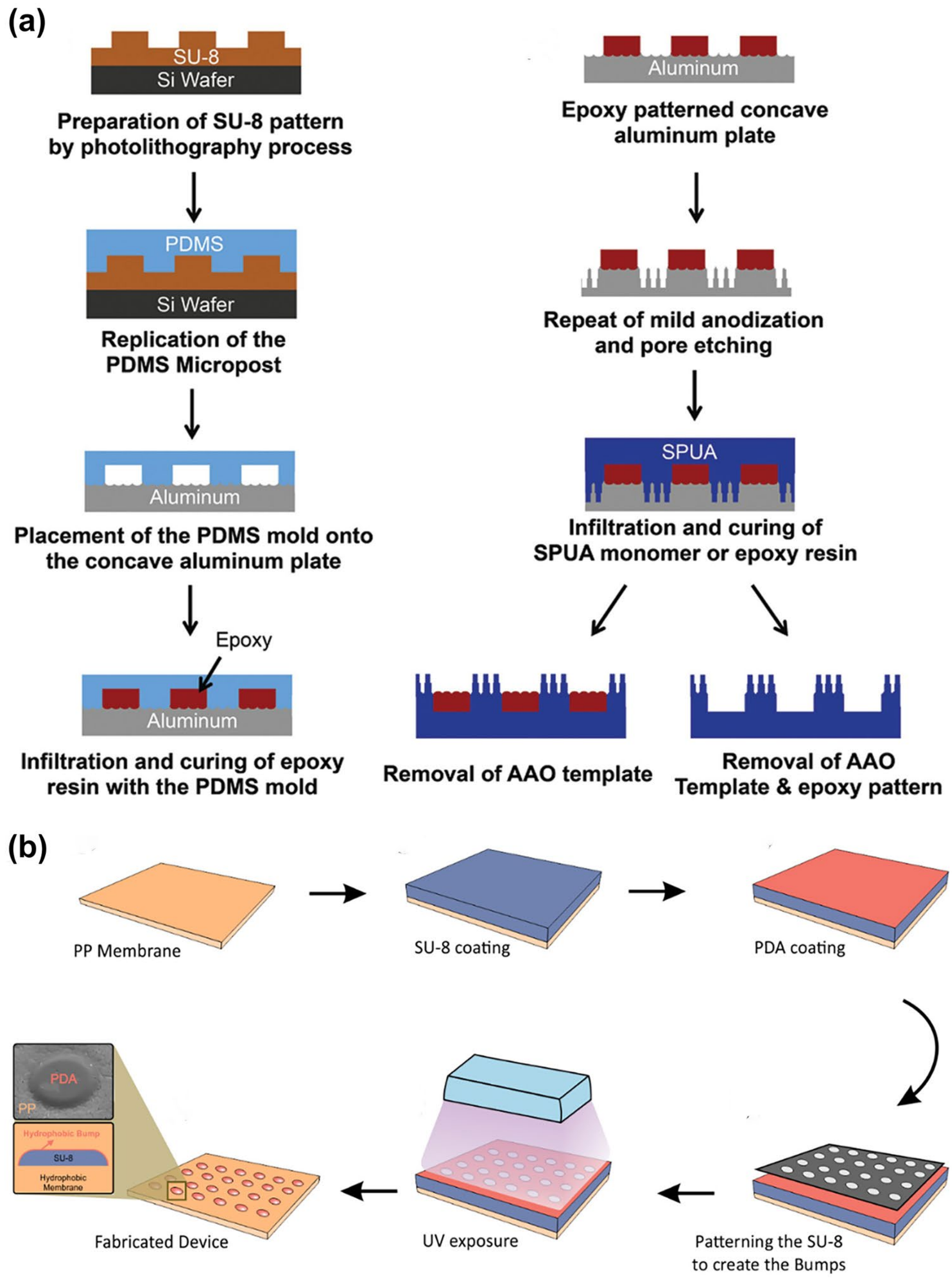


Fig. 11 **a** Process diagram of preparation of epoxy pattern (left) and layered polymer structure (right) on an aluminum plate. Adapted with permission from Cho et al. [68]; Copyright 2015 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim. **b** Manufacturing schematic diagram of bionic water collector equipment. Adapted with permission from Moazzam et al. [69]; Copyright 2017 Elsevier

superhydrophobic and superhydrophilic nanoparticles, showing excellent water and mist collection performance. The water collection efficiency can be optimized by controlling the UV irradiation time to yield surfaces with good condensation performance, efficient water collection performance, and good wetting stability.

4.4 Nano-pattern growth method

A patterned surface refers to the superhydrophilic area required for production on superhydrophobic surfaces. The treated area often has special properties, while the unprocessed area was still superhydrophobic. Tian et al. [77] developed a tightly packed ZnO nano-needle through wet chemical crystal growth and hydrophobic treatment. The synthesized nanostructure surface showed significant self-propulsion performance of condensed droplets after modification with low-surface energy fluorosilane molecules. The property was attributed to the liquid adhesion of the tightly packed nanoneedles, as well as the excess surface energy released by aggregation of tiny droplets ensuring coalesced droplets propel themselves. The findings are of great significance for the development of new self-propelled droplet condensing surfaces for applications in

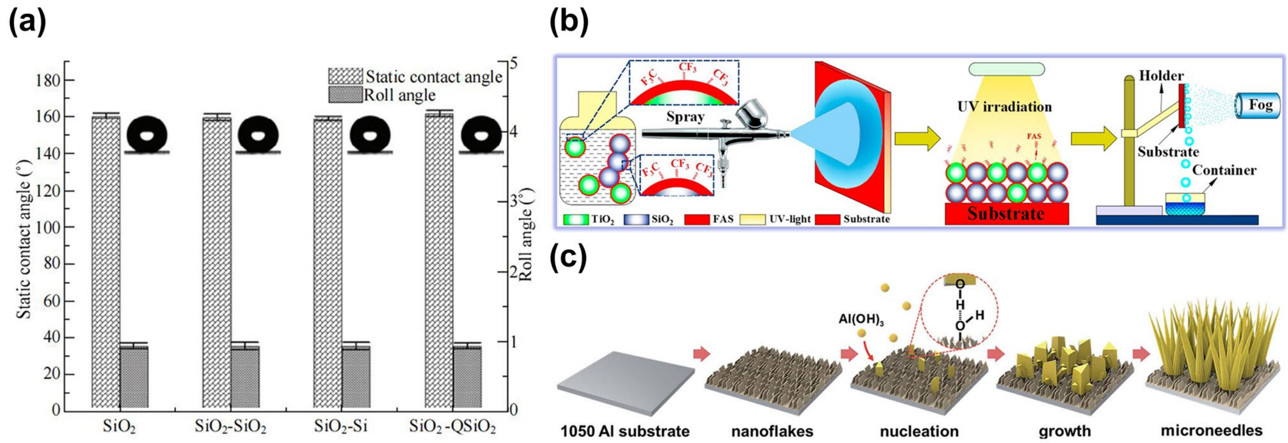


Fig. 12 **a** Static contact angles and rolling angles on differently coated surfaces. Adapted with permission from Wang et al. [74]; Copyright 2018 Springer Nature. **b** Schematic diagram of preparation procedure of hybrid super-wetting surface and water collection. Adapted with permission from Wang et al. [76]; Copyright 2018 Elsevier. **c** Aluminum hydroxide microneedle array formed on an aluminum substrate. Adapted with permission from Cho et al. [78]; Copyright Royal Society of Chemistry

highly efficient heat transfer equipment, such as moisture-proof, self-cleaning, and supercooled antifreeze agents. Cho et al. [78] developed a simple hydrothermal method to prepare β-Al(OH)$_3$ microcapsule array structure with layered morphology to form highly wettable surfaces and efficient air–water collection devices (Fig. 12c). The formation mechanism of the microstructure consisted of nucleation of β-Al(OH)$_3$ on γ-AlOOH nanoparticles to grow the crystal structure. The author also investigated the morphological development and growth mechanism of β-Al(OH)$_3$ microstructure. And the data revealed a crystal structure tending to grow upward with the increase in reaction time. Also, the crystal structure underwent uniform nucleation on the surface and expanded outward. The microcapsules showed a layered surface structure due to epitaxial growth, playing an important role in enhancing wettability and water harvesting properties. Wang [79] used the electrochemical anodic etching method to etch aluminum-based materials and yield surfaces with wettability. The fluorosilane solution was used to modify the prepared aluminum-based superhydrophilic material and effectively reduce its surface energy, thereby obtaining a superhydrophobic surface material. The superhydrophobicity of aluminum base was processed by a carving machine to yield different wettabilities. The contact angles of the droplet were measured as 71° on the conventional aluminum material, 3° on the superhydrophilic aluminum sheet, and about 152° on the surface of the superhydrophobic aluminum sheet. In addition, researchers also studied the controlled evaporation of droplets on patterning wettable surfaces [80], directional bouncing [81], directional transport [82, 83] and other functions.

5 Summary

In recent years, the research on extreme wettability of material surface, such as superhydrophobic, superhydrophilic, and superhydrophobic/superhydrophilic pattern wettability, has attracted the attention of many researchers. However, overall, the research on the regulation technology and application of surface wettability is still in the laboratory stage, far from reaching the requirements of industrial application. The main problems are as follows:

1. Although there are many methods to control the surface wettability of materials, these techniques still have some shortcomings. For example, the equipment required is expensive; modified reagents used to reduce surface energy are not environmentally friendly; the experimental method has strict requirements on the operating environment; the operation is not repeatable and so on. Therefore, it is an important task to explore the new preparation technology, simplify the preparation process, and reduce the preparation cost.
2. Another major factor restricting the practical application of materials with special wettability is that the structures required by the special wettability of the material surface are often very fragile. After physical friction or chemical erosion, the structures will be destroyed, thus losing their special wettability.
3. With the continuous development of science and technology, people have higher requirements for the functionality of materials. Therefore, it is one of the development trends to develop more intelligent and multi-functional materials with controllable wettability surfaces.

To sum up, it is of great significance to simplify the preparation process, develop the surface preparation method with simple operation steps and low cost, solve the stability problem of special wettability surface, and conduct the application research and data accumulation of wettability surface.

Author contribution Xiaoli Bai contributed to the design of the article. The manuscript was written by Junyu Song and Ruixin Shi, Hassan Algadi, and Deepak Sridhar are responsible for the modification of the article and the authorization of reference pictures. All authors read and approved the final manuscript.

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Data availability Because of it is a review manuscript, data are reviewed from the related scientific research and related references are written in the references list.

Declarations

Ethical approval This article is based on previously conducted studies and does not contain any new studies with human participants or animals performed by any of the authors.

Consent for publication All authors approved the paper submission.

Conflict of interest The authors declare no competing interests.

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