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Springtail-inspired omniphobic slippery membrane with nano-concave re-entrant structures for membrane distillation

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Omniphobic membranes, due to their exceptional properties, have drawn significant attention for overcoming the bottleneck in membrane distillation (MD) technology. This study demonstrates an innovative method for fabricating an omniphobic membrane that is simple and facile compared to other methods such as wet/dry etching and photolithography. The surface morphology of springtails was imitated using electrospraying technique to coat a polyvinylidene fluoride substrate with concave-shaped polystyrene beads that were successfully developed by controlling the electrical traction (voltage) and air resistance (humidity). Then, the lipid coating of springtail surfaces was mimicked by dip-coating the membrane in a low-toxicity short-chain perfluoropolyether lubricant. The concave structure's tiny air pockets increased membrane hydrophobicity significantly, indicated by the fact that the first round of water bouncing took only 16.3 ms. Finally, in MD treatment of seawater containing 1.0 mM sodium dodecyl sulfate, the optimized omniphobic membrane maintained a stable 99.9% salt rejection rate.

Increasing water scarcity issues and stringent discharge regulations have necessitated the consideration of unconventional water resources, such as seawater, hypersaline water, and wastewater, calling for emerging technologies to successfully augment water supplies. As a thermally driven membrane-based separation process, membrane distillation (MD) is a promising technology for treating hypersaline waters from a variety of industries that cannot be treated by pressuredriven reverse osmosis and traditional technologies^{1–3}. MD is a thermal-driven process that separates volatile and non-volatile

substances by using a hydrophobic porous membrane for selective water purification and salt rejection^{4,5}. In an MD system, vaporized water in the hot feed side crosses through the porous membrane to the permeate side, and the non-volatile pollutants are intercepted and rejected effectively by the hydrophobic membrane^{6,7}, whose hydrophobicity and anti-wetting properties are the key factors for ensuring a high rejection rate. However, when it comes to the treatment of wastewater or seawater containing low surface tension pollutants, the development of MD membrane technology has been facing a

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bottleneck⁸⁻¹⁰. MD membranes are prone to wetting when treating wastewater with low surface tension, which causes them to lose their function of separating liquid and water vapor and ultimately suffer a rapid drop in rejection rate. A wide range of typical wastewaters have the ability to cause membrane wetting and the crystallization of concentrated brine¹¹. In particular, MD membranes are most vulnerable to surfactants in shale gas wastewater¹², trace oils in wastewater from petrochemical plants¹³, organic solvents contained in the semiconductor industry, and pharmaceutical wastewater¹⁴. To tackle such complex wastewaters containing low surface tension pollutants, recent research has been turning their attention to developing omniphobic membranes with exceptional anti-wetting properties.

The strategies for making omniphobic membranes with stronger anti-wetting capacities have often turned to nature for inspiration. The surface of lotus leaves, for example, inspired the creation of superhydrophobic surfaces with highly rough micropillar re-entrant microstructure coupled with a low surface energy lubricant coating¹⁵. Likewise, the observation of Springtail's skin opened a floodgate of scientists developing omniphobic surfaces. Springtails store air within their outer skin to breathe underwater¹⁶. The protective layer of air in the springtail's skin surface is made up of nano-sized, concave-shaped double re-entrant structures combined with a low surface energy lipid layer, creating pockets to store air effectively and endowing strong resistance to falling droplets^{17,18}. While both superhydrophobic and omniphobic membranes require re-entrant structures to enhance the overall roughness of the membrane surface and further modification to reduce the surface energy, what makes a membrane omniphobic, as opposed to superhydrophobic, is the concave topography of the reentrant microstructures on the omniphobic membrane's surface. The excellent anti-wetting ability of these concave double re-entrant structures can be explained by the Cassie-Baxter model, which indicates that the non-wettability of the rough surface is due to the presence of tiny air pockets at the bottom of the interface. Therefore, the key to creating an omniphobic membrane is to build a double reentrant structure with concave topography, that is, a cross-sectional solid area that gradually decreases from top to bottom. The smaller geometric angle between the concave structure's profile texture and the horizontal line compared to the equilibrium contact angle (CA) triggers the formation of upward capillary force and a stable Cassie -Baxter state, enabling the repulsion of liquid with low surface energy.

The most common method used for fabricating omniphobic membranes at the present stage is to build a rough surface using nanoparticles, then combining long molecule chain fluoridation additives to reduce the surface energy^{19,20}. Lin et al. fabricated an omniphobic membrane by coating silica nanoparticles on a hydrophilic glass fiber membrane through electrical attraction and applying further modification by long-chain fluorinated alkyl silane (FAS)²¹. Chen et al. successfully fabricated an omniphobic hollow fiber membrane through a chemical bath deposition of ZnO nanoparticles and modification by FAS17²². Lu et al. modified nanosized polyhedral oligomeric silsesquioxane (POSS) with 1H.1H.2H.2H-perfluorodecanethiol under UV light to build a crosslink, then mixed polyvinylidene fluoride (PVDF) to create an electrospun omniphobic membrane²³. However, there arose an urgent need for a new fabrication method for developing omniphobic membranes without the long-chain perfluorocarbons when the U.S. Food and Drug Administration (FDA) banned the use of long-chain perfluorinated compounds in a number of areas, including oil-proof packaging materials²⁴, after the widespread utilization of long-chain perfluorocarbon additives led to the detection of fluorine-containing pollutants in water bodies that are harmful to human health and the environment^{25,26}. Moreover, long-chain perfluorocarbons contain eight or more carbon atoms that are resistant to breaking down, thereby persisting for hundreds of years and spreading throughout the earth once released into the environment^{27,28}. In place of long-chain perfluorocarbons, other short-chain fluorinated polymers and low-surface energy lubricants have been used to reduce the surface energy of solid surfaces. Cao et al., bioinspired by lotus, fabricated a slippery surface by covering silica nanoparticles with a polydimethylsiloxane (PDMS) lubricant and coating them on a glass slide using dip-coating technology²⁹. Ma et al. modified a nano-textured alumina gel membrane by infusing a perfluoropolyether (PFPE) lubricant on the top surface to achieve omniphobic properties³⁰. At present, PFPE is widely applied as biomaterial for cell culture and tissue engineering owing to its excellent hydrophobicity and low-toxicity³¹⁻³³.

In terms of the specific technique used to fabricate omniphobic membranes, electrohydrodynamic techniques, such as electrospinning and electrospraying, have been preferred by many scientists owing to their simplicity, low cost, and high controllability over a wide variety of spinnable materials^{34–36}. Hou et al. fabricated an omniphobic cellulose membrane by electrospinning, mixing silica nanoparticles with cellulose nanofiber and fluorinated by perfluorocarbons additives³⁷. Wu et al. also used electrospinning technology, coupled with fluorination using perfluorocarbons additives by vapor deposition technology, to fabricate a polyvinylidene fluoride-cohexafluoropropylene (PVDF-co-HFP) nanofiber membrane that has omniphobic properties³⁸. Compared with membranes prepared by other fabrication processes, the larger porosity of electrospun membranes is more conducive for improving water flux during an MD operation³⁹.

For these reasons, this study employs electrospraying technology to prepare the concave-structured coating layer using a polymer chosen based on the facility of constructing porous beads^{40,41} through the principle of phase separation under high humidity. Specifically, this study tailored a commercial PVDF (C-PVDF) membrane by coating a layer of uniform concave-shaped re-entrant-structured beads that mimic springtail surfaces using electrospraying technology, followed by low surface energy PFPE lubricant dip-coating to endow omniphobicity. Polystyrene (PS) was chosen as the polymer employed in the electrospinning process for building the porous bionic springtail surface concave re-entrant structure. To enhance the connection between the PS nano-beads and the substrate membrane and attain robust surface stability, tetraethoxysilane (TEOS) was mixed with PS and cross-linked with the NaOH-treated C-PVDF membrane⁴². In the electrospraying process, we optimized the PS beads' morphology from a sphere shape to a concave re-entrant structure by adjusting the environmental humidity, voltage, and injection rate. Through the combined action of the electrical traction and air resistance, the porous polymeric beads formed under high voltage took on the desired concave structure imitating springtail surfaces^{43,44}. The size of the PS beads was reduced from the micron level to the nanometer level by pretreating the dope solution with high intensity ultrasonic processing. Finally, the optimized bionic springtail PS nano-concave membrane was applied to a desalination process to test its anti-wetting ability under different concentrations of sodium dodecyl sulfate (SDS).

Results and discussion

Study on the formation of concave topography during omniphobic membrane fabrication

The schematic diagram of PS bead formation with spherical and concave structure geometry during the electrospraying process is provided as Fig. 1. Overall, the process for forming PS beads with concave structure consisted of three parts: 1) the polymer solution droplet is emitted from a positively charged needle by electric traction force and flies towards the negatively charged collector; 2) as nanofibers are elongated apart and broken by the traction force of electric field, air instantly enters to form hollow structured PS beads; 3) the air resistance force causes the porous PS bead to bend and deform to take on a



Fig. 1 | Mechanism of PS bead formation. The schematic diagram of the PS bead formation with spherical and concave geometry during electrospraying process.

concave structure. Through the simulation derivation, as shown in Supplementary Fig. 1, it was observed that the depth of the concave structure *L* has a linear relationship with the square of applied voltage. The degree of the concave structure deepens with the increase of applied voltage during the electrospraying process in a high-humidity environment. However, in a low-humidity environment with heating treatment during the electrospraying process, non-porous PS beads are produced, which are hollow and filled with air. The air resistance cannot overcome the atmospheric pressure to bend the air-filled PS beads, so spherical PS beads are produced.

When the relative humidity is low, thereby having less moisture in the air to mix with the PS jet flow, sphere-shaped PS beads with nonporous surfaces were formed as shown in Fig. 2a. In comparison, in a high humidity environment (i.e., more moisture mixing with the PS jet flow), the resulting beads take on a porous surface structure through the phase separation mechanism, and during their flight to the collector, they take on a concave shape due to the combined action of the electrical traction under the high voltage electric field and air resistance. Accordingly, it was difficult to find PS beads that are concaved in the direction opposite to the beads' flying direction as shown in Fig. 2 and the higher magnification SEM images of coating layer surface morphology are shown in the insets in Fig. 2a–d.

Also, as a higher voltage results in a higher flight speed of the PS bead, producing greater air resistance, increasing the applied voltage during electrospraying resulted in more obvious and deeper concave shapes as shown in Fig. 2c. Compared to general sphere-shaped beads, the porous, concave-shaped beads created a re-entrant structure capable of storing air, thereby forming a protective layer of air that more closely mimics springtail surfaces. The concave structure also reduced the surface contact area with liquid droplets, producing larger wetting resistance and significantly improving the membrane's hydrophobicity^{17,45}. From PS1 to PS3 membranes, higher humidity (PS1:25%, PS2:50%, PS3:75%) and applied voltage (PS1:14 kV, PS2:20 kV, PS3:26 kV) led to the creation of deeper concave re-entrant structures, which improved the water CA from 149.2° (PS1) to 156.2° (PS3) as shown in Fig. 2e.

The initial concave re-entrant PS beads were relatively larger (micron) in size compared with the nanosized concave re-entrant structure of springtail surfaces. Therefore, high-power ultrasonic pretreatment was introduced in this study to break the long molecular chain of the PS polymer and reduce the size of the PS beads as shown in Fig. 2d. The average diameter of the PS4 beads (92.8 nm) was much smaller than the PS1 beads without ultrasonic processing (526.4 nm) as shown in Supplementary Fig. 2. Meanwhile, a significant improvement in surface roughness was observed when the PS beads' diameters became smaller, as smaller beads produce more contact sites with the substrate membrane. The improvement in surface roughness directly translated into enhancements in surface hydrophobicity: the PS4 membrane, made with PS4 beads treated with ultrasonic processing, achieved the highest water CA of 167.5°. Moreover, due to the crosslink reaction between the TEOS-doped PS beads and the NaOH-treated substrate membrane, surface stability was also significantly enhanced as shown in Fig. 2f and the inset. The results of the abrasion test (Supplementary Fig. 3, 4) show that the water CA of the membrane without NaOH treatment started to decrease after only 30 cycles of the abrasion test, while the membrane after NaOH treatment maintained a stable water CA even after 120 cycles. However, although the optimized nanosized PS beads with concave re-entrant structures contributed greatly to achieving high hydrophobicity and stability, further surface modification to reduce the membranes' surface energy was necessary to achieve omniphobic properties.

Instead of conventionally combining long-chain perfluorocarbons with nanoparticles for membrane surface modification, dip-coating technology was applied to tailor a lubricant layer on the concave structure of the nanosized PS beads using PFPE lubricant, which is a short-chain fluoropolymer, as a low surface energy additive. Similar to the molecular structure of hydrocarbon lubricants, the fluorine atoms (C-F bond) of PFPE triggers the replacement of hydrogen atoms (C-H bond) in the substrate. Dipcoating was selected as the main technology for lubricant coating due to its simple configuration and fast operation. In the dipcoating process, the PS membranes were fixed on a moving holder and dropped at uniform speed until they are fully immersed in lubricant oil for a set time. Then, the lubricated PS membranes were taken out to dry in an oven overnight. The lubricant immersion time is the factor that decides the thickness of the coating layer on the membrane surface⁴⁶. As shown in Fig. 3a, the SEM images of membranes that were immersed in lubricant oil for one minute show a thick coating layer on the membranes covering and hiding the concave re-entrant structures. The higher magnification SEM images of the lubricant coating layer surface morphology are shown in the insets in Fig. 3a, b. The water CA of these membranes also dropped, confirming the destruction of the concave structures and the reduction of surface roughness. When the lubricant immersion time was reduced to 30 s, the highly rough nanosized concave reentrant structures were still clearly visible as shown in Fig. 3b, while



Fig. 2 | **The surface morphology, hydrophobicity and stability of membrane after coating the PS beads.** SEM images of the (**a**) PS1 (**b**) PS2 (**c**) PS3 (**d**) PS4 membranes surface morphology and the higher magnification SEM images is also included in the inset in (a–d). **e** The water CAs of employed membranes with surface energy information. **f** Results of the abrasion test and stability tests

performed on the membranes under different pH conditions (indicated in the inset). **g** Schematic depiction of the bionic concave PS beads coated omniphobic membrane fabrication process. Error bars represent the standard deviation from three independent measurements.



Fig. 3 | **The surface morphology and hydrophobicity of membranes after lubricant immersions.** SEM images of lubricant coated PS4 membranes after (**a**) 120 sec immersions (PS4L1) (**b**) 30 s immersions (PS4L2) and the higher magnification SEM images is also included in the inset in (**a**, **b**). **c** The CA test with DI water,

0.5 mM SDS, castor oil, and 75% ethanol on the C-PVDF, C-PVDFL2, PS4, and PS4L2 membranes. Error bars represent the standard deviation from three independent measurements. **d** The images of SDS droplets with different concentrations standing on the A. C-PVDF membrane, B. PS4 membrane, and C. PS4L2 membrane.

the lubricant coating effectively reduced the surface energy from 31.42 to 12.39 mN m⁻¹ and increased the water CA to 171.1°. Moreover, the CA of the lubricant-coated membrane was quite stable under the abrasion test, demonstrating its excellent surface stability. The lubricant was blocked from entering the membrane pores by the concave structured PS beads. Moreover, the lubricant mainly stayed inside the cave structure of the PS beads, so the membrane's permeability was not affected much by the dip-coating, as verified by the porosity measurement results: the porosity of the lubricant coated membrane was 87.4%, similar to that of the non-lubricated PS beads coated membrane (88.2%).

To verify our hypothesis that the electrosprayed nano-concave re-entrant structured PS beads with lubricant coating can imitate springtail surfaces to achieve omniphobic property, we conducted a CA test using synthesized solutions with different surface tensions. Four membranes were selected for the test: the substrate C-PVDF membrane, the C-PVDF membrane with lubricant coating, the optimized PS beads coated membrane (PS4), and the optimized PS beads coated membrane applied with lubricant coating for 60 s (PS4L2). The water CA of the PS4L1 membrane is shown in Supplementary Fig. 5. The C-PVDF membrane showed some antiwetting capacity for DI water with 0.5 mM SDS but underwent severe wetting when tested with cater oil and 75% ethanol solutions. The CAs of cater oil and 75% ethanol solutions on the PFPE lubricant coated C-PVDF membrane improved to 72.4° and 48.3°. In comparison, the PS4 membrane with nano-concave re-entrant structures had excellent superhydrophobicity (water CA: 167.5°) and showed improved CAs for 0.5 mM SDS, castor oil, and 75% ethanol solutions compared to the C-PVDF membrane. However, the CA of castor oil on the PS4 membrane still failed to meet the standard for omniphobic surfaces (i.e., the CA of oil on the membrane surface should be greater than 120°) because the surface energy of the PS4 membrane was still not low enough. The PS4L2 membrane, on the other hand, achieved omniphobic properties: the CAs of castor oil and 75% ethanol on the PS4L2 membrane reached 139.6° and 131.1°, respectively, as shown in Fig. 3c. To demonstrate the anti-wetting performance of the membranes for long-term underwater operation, the CAs of the membranes were measured again after 24 h of immersion in DI water, as well as 0.1 mM SDS, oil, and ethanol solutions as shown in Supplementary Fig. 6. The CAs of the membranes did not change significantly when immersed in water for 24 h. However, after 24 h of immersion in the 0.1 mM SDS solution, the water CA of the C-PVDF membranes decreased from 122.3° to 108.6°, proving that the membrane had begun to undergo wetting. The surface stability of membrane after lubricant coating was also tested, the results of which is provided in Supplementary Fig. 7.

We further tested how the CAs on the three membranes changed when SDS concentration was increased. The CAs on the C-PVDF and PS4 membranes demonstrated a significant decline with the increase in SDS concentration (Fig. 3d). The C-PVDF membrane was instantly wetted by the 1 mM SDS solution. Comparatively, the PS4L2 membrane exhibited the most robust anti-wetting capacity with a relatively stable CA for a wide range of SDS concentrations up to 1 mM. The test results verified that the PS4L2 membrane with nano-concave structures has enormous wetting resistance to low surface tension liquids. Also, the pore size of the substrate (0.22 μ m and 0.45 μ m) had a negligible effect on the PS4 membrane's surface hydrophobicity as shown in Supplementary Fig. 8.



Fig. 4 | The physical and chemical properties of employed membranes. a XRD, b ATR-FTIR spectra, c pore size distribution and LEP (indicated in the inset), and d Tensile Stress-Strain Curve of the C-PVDF, PS4, and PS4L2 membranes. Error bars represent the standard deviation from three independent measurements.

Physical and chemical properties analysis of employed membranes

The FTIR and XRD spectra of the C-PVDF, PS4, and PS4L2 membranes are shown in Fig. 4a, b. In Fig. 4a, the peak at 1185 cm⁻¹ can be attributed to the C-PVDF's C-C band⁵. The peaks at the wavenumbers of 872 and 838 correspond to the C-PVDF's CF stretching and asymmetrical stretching vibration^{23,47}. New peaks indicating the PS bead coating layer appeared in the FTIR spectra of the PS4 membrane. The peaks at 1488 and 1449 cm⁻¹ represent the aromatic C = C bond and the benzene ring⁴⁸, and the peaks of the C-H bond were observed at 754 and 701 cm⁻¹⁴⁹. The new absorption peak appearing at 1105 cm⁻¹ in the FTIR spectra of the PS4L4 membrane can be attributed to the C-F stretching of PFPE⁵⁰. The XRD patterns of the C-PVDF membrane is shown in Fig. 4b, where the diffraction peaks at 2 θ of 18.4° and 25.1° correspond to the α phase^{51,52}, and the peak at 20 of 20.4° is related to the β phase⁷. After coating PS beads and lubricant on the C-PVDF membrane, the diffraction peaks either weakened or became non-existent at 20.3°.

For MD, the membrane's pore diameter needs to be under a certain size (< 0.5 µm), since membrane wetting starts from the largest pores of the membrane to reduce the LEP and cause the wetting issue⁵³. The pore size distribution of the three membranes studied is presented in Fig. 4c. As can be seen from the diagram, the PS beads coating reduced the pore size of the PS4 membrane as the PS beads partially covered the membrane's pores. The lubricant coating layer further reduced the membrane pore size to 0.34 µm. The LEP of the PS4L2 membrane, which had the smallest pores and highest surface hydrophobicity out of the three membranes, was two times higher (2.46 bar) than the C-PVDF membrane as shown in the inset in Fig. 4c. Furthermore, as shown in Fig. 4d, the overall mechanical strength of the PS4 membrane was also enhanced due to the cross-linking of TEOS with the hydroxy function group on the C-PVDF. The lubricant coating layer enhanced the mechanical strength even further, acting like a sticky hydrogel after drying on the PS4L2 membrane. The porosity,

thermal conductivity, and Zeta potential information of the employed membranes are provided in Supplementary Fig. 9-10.

Investigation of the mechanism on achieving omniphobic surface

It has been reported that concave re-entrant structures play an important role in endowing superhydrophobicity and omniphobicity, as the air-storing pockets of these structures reduce the contact area between the liquid and the membrane surface effectively and improve surface hydrophobicity significantly¹⁶. In addition to this specific microstructure, the membrane's surface roughness and surface energy are also very important for building omniphobic properties. The concave structure of the PS beads became more defined and deeper from the PS1 to PS3 membrane, accompanied by increases in surface roughness from 0.88 to 1.47 µm (Fig. 5a-c). Furthermore, after the PS4 beads' sizes were maximally reduced (92.8 nm), the surface roughness of the PS4 membrane reached the highest value $(2.35 \,\mu\text{m})$ as shown in Fig. 5d. However, although higher surface hydrophobicity was achieved by increasing surface roughness, efforts to lower surface energy was still required to achieve omniphobic surfaces. The PFPE lubricant coating on the PS beads effectively reduced surface energy, although lubricant coating with excessive thickness could also destroy the nanosized concave re-entrant structures, as in the case of the PS4L1 membrane, in which the surface roughness was reduced to 0.48 µm (Fig. 5e). Lubricant dip-coating for 30s employed on the PS4L2 omniphobic membrane retained the re-entrant structures and achieved high surface roughness, as shown in Fig. 5f, and effectively reduced the surface energy to 12.39 mNm⁻¹. Consequently, the optimized PS4L2 membrane gained omniphobicity from its specific nanosized concave re-entrant structures, high roughness, and relatively low surface energy.

The effect of the concave re-entrant structures on surface hydrophobicity was analyzed systematically. First, as discussed above, surface roughness increased with the deepening of the concave



Fig. 5 | The surface roughness of employed membranes. Three-dimensional optical profile images of the (a) PS1, (b) PS2, (c) PS3, (d) PS4, (e) PS4L1, and (f) PS4L2 membranes.



Fig. 6 | **Interaction between water droplet with employed membranes surface.** Images of water bouncing with 0.5 mL water droplets on the five membranes (**a** PS1, **b** PS2, **c** PS3, **d** PS4, and **e** PS4L2) were recorded using a high-speed camera (Photron, frame rate of 3600 fps).

re-entrant structures on the membrane surface. More importantly, the springtail-inspired nano-concave re-entrant structures stored air to form a protective layer and reduce the contact area between droplets and the membrane surface, thereby endowing greater hydrophobicity. To elucidate the omniphobic mechanism of the nano-concave reentrant structures, a water bouncing test was carried out on the five different membranes: 0.5 mL water droplets were dropped from a needle 20 mm away from the membrane surface, and the bouncing phenomenon of the water droplets were recorded using a high-speed camera. The duration of contact between the water droplet and the membrane surface is an important indicator, where a shorter time required for the droplet to bounce indicates that there is a smaller contact area with lower adhesion force between the water droplet and the membrane surface. The height and number of water droplet bounces on the membrane surface indirectly reflect the repulsion intensity of the upward force generated by the stored air pockets.

The results of the water bouncing test showed that, in addition to the development of clearer and deeper concave re-entrant structures from the PS1 to PS4 membrane, more air pockets had formed on membrane surface, which reduced the water droplet's contact area. The contact time of the first round of water bouncing on the PS3 membrane was 18.2 ms, which is much shorter than that on the PS1 membrane (22.1 ms) as shown in Fig. 6. The PS4 nanosized beads increased the membrane's surface roughness and further shortened the contact time of the first round of water bouncing to 17.5 ms. The lubricant coating further reduced the adhesive force of the membrane surface, and the first round of the water drop bouncing was completed in the shortest time of 16.3 ms on the PS4L2 membrane. Figure 6b presents the number and height of water bouncing on the membranes. The PS4L2 membrane obtained the most number (four times) and greatest height of water bouncing as shown in Fig. 6c, owing to having the strongest upward force produced by the air pockets and the low surface adhesion by lubricant coating.

In addition to experimental observations, the relationship between omniphobic properties and the nano-concave re-entrant structures was further verified through theoretical models. In the Cassie-Baxter model, droplets are retained at the top of the solidliquid-gas interface, and the non-wettability of the rough surface is derived from tiny air pockets below the liquid droplets⁵⁴. The PS beads' nano-concave re-entrant structures cooperate with the low surface energy lubricant coating in suspending the liquid droplets on the top of the PS beads' edges (which are filled with air pockets below), which conforms to the Cassie-Baxter model. The Cassie-Baxter equation can be expressed as⁵⁵:

$$\cos\theta_a = f_a(\cos\theta_b) - f_b = f_a(\cos\theta_b + 1) - 1; f_a + f_b = 1$$

where θ_a and θ_b represent the CAs of rough and smooth surfaces, respectively, and f_a and f_b represent the area fractions of liquid droplets' contact with membrane surfaces and air pockets, respectively. A smooth membrane was prepared by phase inversion using the same dope solution, and its water CA was 117.2°, which was considered as θ_b . The other parameters were calculated as shown in Supplementary Table 1. As the nano-concave structures became clearer and deeper from the PS1 to PS4 membrane, the contact area between the water droplets and the membrane surface (f_a) decreased, but the contact area with the air pocket (f_b) increased, so shorter time was required to complete a round of water bouncing. Thus, we theoretically confirmed that the nano-concave re-entrant structures and low surface energy additive coating are both effective strategies for increasing membrane hydrophobicity and achieving omniphobic property. Moreover, based the membrane wetting simulation as shown in Supplementary Fig. 11, the water equilibrium contact angle θ is 172.3°, which is larger than the ψ , and thus, an upward capillary force is formed, resulting in a stable Cassie-Baxter state

The wetting resistance of lab-made omniphobic membrane in MD test

An MD test was conducted on three membranes (C-PVDF, PS4, and PS4L2) to test their anti-wetting capacity for treating real seawater containing different concentrations of SDS. In the MD test, the feed solution initially contained only real seawater, then 0.058 g SDS was added every 90 min. That is, the concentration of SDS on the feed side increased by 0.01 mM at 90 min intervals. When treating just real seawater, all three membranes showed stable flux and a 99.9% NaCl rejection rate. However, when the concentration of SDS was increased to 0.2 mM, the conductivity in permeate side started to rise rapidly and the flux began to decrease in the case of the C-PVDF membrane. These signs indicated that the C-PVDF membrane was wetted and that crystal precipitation had occurred in the inner pores of the membrane, thus causing membrane pore obstruction and flux decline¹³. In comparison, the PS4 membrane showed a high salt rejection rate (99.9%) and stable flux even when dealing with a 0.4 mM SDS feed solution, indicating improved antiwettability, which can be attributed to the formation of protective layer of air stored in the nanoconcave re-entrant structure. However, when the concentration of SDS was increased to 0.6 mM, membrane wetting occurred, resulting in a rapid decrease in water flux and salt rejection rate as shown in Fig. 7. Meanwhile, the PS4L2 omniphobic membrane achieved the best anti-wetting performance among the membranes due to its nano-concave re-entrant structures and low surface energy lubricant coating that enables the effective treatment of low surface tension wastewater or seawater. The PS4L2 membrane maintained a 99.9% salt rejection rate and stable flux even when the concentration of SDS was increased to 1 mM, which is a significant improvement even when comparing with previous studies as shown in Table 1. The salt rejection rate during the MD process with three of the membranes developed in this study is summarized in Supplementary Fig. 12. The MD test was repeated once again, and the results are provided in Supplementary Fig. 13.

Tests were performed to examine the membranes' anti-wetting properties against cationic and non-ionic surfactants and antifouling performance, the results of which are shown in Supplementary Fig. 14 and 15, respectively. After the MD test, most of the C-PVDF membrane's surface was covered with green-colored foulant as shown in Fig. 7b. In contrast, the surface of the PS4L2 membrane remained mostly exposed. The excellent fouling resistance of the PS4L2 membrane is attributed to its concave surface structure. which stores a large number of air pockets, thereby effectively reducing the contact area between the foulant and the membrane surface as shown in Fig. 7c. Moreover, due to reduced adhesion interaction, the foulant on the surface of the PS4L2 membrane can be easily washed away with simple water flushing, demonstrating the membrane's excellent regeneration ability. A fluid flow and particle tracing simulation of the interaction between nanostructure and feed solution was performed to understand concave surface microstructure's anti-wetting mechanism, as shown in Fig. 7d and Supplementary Fig. 16, 17. The frequency of particle adhesion on the membrane surface can be indicative of membrane wetting and fouling tendency. With higher adhesion force, the liquid flow tends to flow into the inside of membrane, making it easy to cause wetting issue. With lower adhesion force, the repulsive forces from air pocket stored in the concave structure dominate. thereby enhancing anti-wetting capacity. These results conclusively verified our hypothesis, and we were able to confirm that the PS nano-concave re-entrant structures coupled with the low surface energy lubricant coating can successfully achieve an omniphobic surface that can be applied for desalinating seawater with low surface tension.

In this study, we successfully fabricated a springtail-inspired nanoconcave re-entrant structured polystyrene (PS) membrane using electrospraying technology followed by lubricant dip-coating, which endowed the membrane with omniphobic properties. The main findings are summarized as follows:

- 1. Nano-Concave Structured PS Beads Coating: The nano-concave structured PS beads coating was prepared by controlling the relative humidity and applied voltage during the electrospraying process. It was observed that the depth of the concave structure has a linear relationship with the square of the applied voltage. Raising the relative humidity and applied voltage increased the concave depth of the PS beads, consequently enhancing the hydrophobicity of the membrane surface.
- 2. Lubricant Dip-Coating Process: An ultra-thin, robust coating layer was achieved by controlling the immersion time and incorporating condensation reactions for cross-linking during the PFPE lubricant dip-coating process. This allowed the concave nanostructures to be remained further reducing surface energy and achieving omniphobicity. The contact angles (CAs) of water and castor oil on the optimized omniphobic PS4L2 membrane were 171.1° and 139.6°, respectively.
- 3. Omniphobicity Mechanism: A water bouncing test and simulation elucidated the relationship between the nano-concave structures and omniphobicity. The air pockets stored in the nano-concave



Fig. 7 | **The MD performance and anti-wetting mechanism investigation. a** MD performance of the C-PVDF, PS4 and PS4L2 membranes. **b** The OCT images of employed membrane surface after MD test. **c** a schematic diagram of the nano-

concave re-entrant structured membrane's anti-wetting property. **d** The fluid flow and particle tracing simulation for investigating the interaction between nanos-tructure and feed solution.

structures reduced the contact area between liquid droplets and the membrane surface, thus enhancing surface omniphobicity.

4. Membrane Distillation (MD) Performance: In MD tests using real seawater containing sodium dodecyl sulfate (SDS), the

commercial PVDF (C-PVDF) membrane was quickly wetted by seawater containing 0.2 mM SDS. In contrast, the PS4L2 membrane maintained stable flux and a high salt rejection rate of 99.9% during the treatment of seawater with 1.0 mM SDS concentration.

No	Material	Fabrication Technique	MD performance	Ref
1	PVDF +perfluorocarbon	Electrospinning	No wetting until up to 0.4 mM SDS for 5 h	52
2	PVDF+SiNPs+ perfluorocarbon	Phase inversion	No wetting until up to 0.3 mM for 11.5 h	56
3	$PVDF + HFP+TiO_2 + perfluorocarbon$	Electrospinning + Self-roughening	No wetting until up to 0.5 mM SDS;	57
4	PVDF+ZnO+ perfluorocarbon	Chemical bath deposition	No wetting until up to 0.3 mM SDS for 3 h	58
5	PVDF + POSS+ perfluorocarbon	Electrospinning	No wetting until up to 0.3 mM SDS for 8 h	23
6	PVDF	Electrospinning + CF ₄	No wetting until up to 0.7 mM SDS for 10 h	59
7	PVDF	Sodium/naphthalene etching+silanization	No wetting until up to 0.4 mM SDS for 9 h	60
8	PVDF+Teflon AF 2400	Phase inversion	No wetting until up to 0.4 mM SDS for 9 h	61
9	PVDF+silica particles	Phase inversion	No wetting until up to 0.6 mM SDS for 7 h	62
10	PVDF+silica+ perfluorocarbon	Dip-coating	No wetting until up to 0.2 mM SDS for 6 h	63
11	PVDF + PS + PFPE lubricant	Electrospray+Dip-coating	No wetting until up to 1.0 mM SDS for 14 h	This study

Table 1 | A comparison of MD performance with omniphobic membranes reported in previous studies and this study

In conclusion, the developed PS4L2 membrane, with its nanoconcave structures and optimized surface coating, demonstrated significant potential for enhancing the performance and efficiency of MD processes, particularly in challenging environments.

Methods

Materials and chemicals

The dope solutions for electrospraying were prepared using polystyrene (PS, Mw = 192,000) as solutes in particle form and tetrahydrofuran (THF, \geq 99.9%) as solvents with various PS percentages, both purchased from Sigma-Aldrich. Tetraethoxysilane (TEOS, 99%) was employed as the cross-linking agent and was supplied by Shanghai Macklin Biochemical Co. Ltd., China. A perfluoropolyether (PFPE) lubricant (98%, DuPont Krytox GPL 103, surface tension = 17 mN/m) was utilized as the low surface energy additive. Commercial PVDF (C-PVDF) membranes supplied by Millipore with a mean pore size of 0.45 µm were used as the substrate. Sodium hydroxide (NaOH, \geq 97.0%, Sigma Aldrich) was mixed with DI water to pre-treat the C-PVDF membrane. Sodium dodecyl sulfate (SDS, \geq 98.5%, Sigma Aldrich) was added in the feed solution for the MD test to determine the antiwetting capacity of the employed membranes.

PS nano-concave omniphobic membrane fabrication

The PS nano-concave omniphobic membrane was prepared via the electrospraying process, which is illustrated in Supplementary Fig. 18. In the electrospraying process, the fiber becomes fractured due to tensile failure, causing air to rush in, and this influx of air creates a balance between the dope solution's surface tension and air expansion to shape the fractured fiber into sphere beads. Two parameters greatly affected the formation of the PS bead concave re-entrant structure, namely, relative humidity and applied voltage. Better bead formation was observed when applying higher voltage, while other parameters, such as dope solution viscosity, jet charge, and solution surface tension, significantly affected the morphology of the beads. The 5% PS dope solution was prepared by dissolving 0.5 g of PS particles in the THF solvent (9.5 mL), and 0.1 mL TEOS was added as the cross-linking agent. The dope solutions were stirred overnight at 65 °C to obtain a homogeneous mixture prior to electrospraying. The syringe containing the dope solution was fixed on the corresponding syringe pump. The capillary needles in the front of the syringe were connected with a high voltage (25 kV) supply. A 18 G needle with an inner diameter of 0.66 mm was used, and the distance between the syringe needle to the collector was maintained at 12 cm with an injection rate of 0.8 mL h⁻¹. A high relative humidity environment $(80 \pm 5\%)$ was maintained during the electrospraying process under room temperature. The C-PVDF membrane was attached to aluminum foil and thereafter fixed on the grounded cylinder collector. The PS bead was injected and collected on the base C-PVDF membrane. Upon completion of electrospraying, the PS coated membrane was instantaneously separated from the rolling collector and kept in an oven at 70 °C overnight. To reduce the surface energy further, the PFPE lubricant was coated on the surface of the PS4 membrane through dip-coating for different durations of immersion as shown in Supplementary Fig. 19. Before dip-coating process, the PS4 membrane was conducted with plasma surface modification to obtain the hydroxy function group. Also, the PFPE lubricant was mixed with acetic acid (5 wt%) and stirred for 12 h under room temperature. In the dip-coating process, the PS4 membrane $(5 \text{ cm} \times 5 \text{ cm})$ was immersed in the lubricant solution by a dip-coater. The time from the bottom edge of the membrane touching the lubricant solution until the membrane was completely removed was defined as the immersion time. The coating layer became cross-linked with PS4 membrane through condensation reaction as shown in Supplementary Fig. 20. After dip-coating, the unreacted excess lubricant was guickly removed by adsorption with blotting paper. Finally, the lubricant-coated membrane was then dried in oven at 150 degrees overnight for further solidification to prevent any flow movements of the lubricant and to enhance the surface stability.

Membrane characterizations

Liquid contact angle (CA) and surface energy. The CAs of DI water, 0.5 mM SDS solution, castor oil, and 75 wt% ethanol on the employed membranes were measured using a CA measuring system (KRÜSS, Germany). The CA value took the average of three repeated measurements. To determine the surface energy, the CA of diiodomethane was evaluated for all membranes. The membranes' surface energies were investigated by double sessile drop measurement coupled with KRÜSS software.

Surface morphology, roughness, and composition. The PS bead morphology on membrane surfaces was detected using scanning electron microscopy (SEM; EVO MA10, ZEISS, Germany). The membranes' surface roughness was explored by an optical surface profiler (Wyko NT9300, Vecco, U.S.A.). To further demonstrate the membranes' composition, Fourier transform infrared spectroscopy (FTIR) analysis was conducted using an IRAffinity-1 FTIR spectrometer (Shimadzu, Japan).

Porosity, liquid entry pressure, and pore size. Membrane porosity was measured by a gravimetric method based on the dry and wetted weight of the employed membranes $(3 \text{ cm} \times 3 \text{ cm})$. 1-butanol was used as the wetting agent. The porosity was calculated based on the volume of 1-butanol remaining in the membrane samples, the weight

difference, and the densities of 1-butanol and the PS polymer. Liquid entry pressure (LEP) and pore size distribution were measured using a porometer (PoroLux[™], Germany).

Water bouncing test. 0.5 mL water droplets were dropped from a needle 20 mm above the membrane surface, and the bouncing phenomena of the water droplets on the membranes were recorded using a high-speed camera (Photron, frame rate of 3600 fps). The contact time and the number and height of water bouncing were measured as indicators of membrane hydrophobicity that indirectly reveal the contact area between the droplets and the membrane surface.

Surface energy measurement. The membrane surface energy was measured using the Owens–Wendt method (Eq. 1).

$$\frac{0.5\sigma_1 \cdot (\cos\theta + 1)}{\sqrt{\sigma_L^{\rm D}}} = \sqrt{\sigma_{\rm s}^{\rm p}} \frac{\sqrt{\sigma_L^{\rm p}}}{\sqrt{\sigma_L^{\rm D}}} + \sigma_{\rm s}^{\rm D} \tag{1}$$

Wherein, σS is the surface energy of the solid membrane surface, σSD is the dispersive component of the solid membrane surface, σSP is the polar component of the solid membrane surface, and θ is the contact angle. Surface tension (σL , mN m–1), the dispersive component (σLD), and the polar component (σLP) of the aforementioned solvents (water and di-iodomethane) at room temperature are presented in Supplementary Table 2.

Direct contact membrane distillation (DCMD)

The overall DCMD experimental set-up consisted of a MD module in the center to separate the hot feed side and the cold permeate side. The effective area of membrane in the MD module was 9.8 cm². Real seawater mixed with 10 mg L⁻¹ Bull Serum Albumin (BSA) and different concentrations of SDS was circulated in the feed side using a gear pump at a flow rate of 500 mL min⁻¹, and DI water was circulated in the permeate side at 500 mL min⁻¹. The surface tension of 1.0 mM SDS mixed with real seawater was measured to be around 22.36 mN m⁻¹. The temperature in the feed side was maintained at 60 °C by a hot plate, and the permeate temperature was maintained at 20 °C using a chiller cooling unit. The initial volume of feed solution was 2.2 L, and the permeate side was filled with 500 mL of DI water. The tube's surface was covered with a rubber protector to reduce heat loss. The conductivity in the permeate side, which is an indicator of water quality, was continuously measured by a conductivity meter. Water flux was also calculated by monitoring the changes in the weight of the permeate side, which was recorded every 10 min with a digital balance.

Permeate flux and salt rejection efficiency

The water flux J in an MD process can be expressed as follows:

$$J = \frac{\Delta W}{\Delta t \times A}$$
(2)

where ΔW is the weight of water collected in permeate side, Δt is the MD operation time, and A is the effective area of the employed membrane, which was 9.8 cm².

The salt rejection rate can be calculated using the following equation:

$$\mathbf{R} = \left(1 - \frac{Cp}{Cf}\right) \times 100\% \tag{3}$$

where C_f (mg L⁻¹) is the initial conductivity in the feed solution, and C_p (mg L⁻¹) is the conductivity in the permeate water after the MD process.

Data availability

The data supporting the findings of the study are included in the main text and supplementary information files. Raw data can be obtained from the corresponding author upon request.

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Author contributions

J.G., M.J., and A.K.A. conceived the research. A.K.A. supervised the research. J.G. designed and carried out the experiments and analyzed data. X.L., M.U.F., B.J.D, B.Z., J.S., Z.W., C.Y., P.W.W., and S.J., contributed to the characterization and discussion of data. B. G. contributed to CFD simulation. J.G. wrote the manuscript. M.J., M.U.F., and A.K.A. discussed and revised the manuscript.

Competing interests

The authors declare no competing interests.

Additional information

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