Self-Deicing Electrolyte Hydrogel Surfaces with Pa-level Ice Adhesion and Durable Antifreezing/Antifrost Performance

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ABSTRACT: Despite the remarkable advances in mitigating ice formation and accretion, however, no engineered anti-icing surfaces today can durably prevent frost formation, droplet freezing, and ice accretion in an economical and ecofriendly way. Herein, sustainable and low-cost electrolyte hydrogel (EH) surfaces are developed by infusing salted water into a hydrogel matrix for avoiding icing. The EH surfaces can both prevent ice/frost formation for an extremely long time and reduce ice adhesion strength to ultralow value (Pa-level) at a tunable temperature window down to −48.4 °C. Furthermore, ice can self-remove from the tilted EH surface within 10 s at −10 °C by self-gravity. As demonstrated by both molecular dynamic simulations and experiments, these extreme performances are attributed to the diffusion of ions to the interface between EH and ice. The sustainable anti-icing properties of EH can be maintained by replenishing in real-time with available ion sources, indicating the promising applications in offshore platforms and ships.

KEYWORDS: anti-icing, ice adhesion strength, antifreezing, antifrost, durability

1. INTRODUCTION

Ice formation and accretion, via frost, snow, and freezing rain, results in severe challenges for infrastructures and transportation, including the collapse of grid infrastructures, failure of turbine blades, and traffic accidents.1–3 These hazards can be divided into two aspects. First, ice formation on a surface (e.g., frost, rime) can cause change in the intrinsic surface properties, such as optical property and wettability.4,5 Second, ice accretion on a surface can result in a loss of equipment efficiency and even the destruction of the infrastructure by the gravity of ice.6,7 A general anti-icing strategy requires the surfaces to possess effective functionalities, namely, to truly prevent ice formation and/or to have low ice adhesion strength for easy removal of eventually formed ice.

The current industrial practices, including mechanical, chemical, and thermal deicing methods, despite their effectiveness, are energy-intensive and/or environmentally unfriendly.8 To solve the high cost of deicing-associated environmental challenges, various anti-icing or icephobic surfaces have been developed, including superhydrophobic surfaces (SHS),9,10 interfacial slippage surfaces (ISS),2,7,11 cracks/modulus-controlled low ice adhesion surfaces8,12,13 and lubricating surfaces.14–21 They mitigate the hazards by delaying ice formation time and/or reducing ice adhesion strength on surfaces. However, the delayed ice formation time of these surfaces is limited and/or the reduction of ice adhesion is not significant enough, which has not reached for practical requirements of durable antifreezing/frost and self-removal of ice.7,22 For example, SHS delays ice formation within a limited time by repelling water droplets,9 and they increase ice adhesion because of an interlocking effect.23,24 The durable low ice adhesion is obtained from extremely soft ISS surfaces, however, their poor mechanical properties restricted the practical applications.2,7 Macrocrack initiators (MACI), stress localization, and modulus reduction strategies have been found to reduce the ice adhesion of surfaces by prompting crack initiation and propagation via stiffness inhomogeneity and deformation incompatibility.8,12,15,25,26 However, the ice adhesion strength of these methods are still in need of reduction for the self-removal of ice. The lubricating surfaces (surfaces with lubricating layer), including slippery lubricant-infused surfaces (SLIS) and hydrated surfaces, mitigate ice formation and reduce ice adhesion.15,16,18,27–29 However, ice forms on the SLIS, as the depletion of an organic lubricant from SLIS results in the reduction of icerobic ability.27,30 In contrast, the hydrated surfaces self-generate an aqueous lubricating layer at the interface between ice and materials, opening a sustainable avenue to mitigate ice formation and lower ice adhesion. So far, antifreeze proteins, ethanol/water, and polyelectrolytes with counterions have been used to develop the hydrated surfaces to mitigate the ice chal-
For example, the ice formation time has been delayed to ca. 4500 s and the ice adhesion has been reduced to ca. 20 kPa on the polyelectrolyte hydrogel surface simultaneously. Nonetheless, the ice could form on the hydrated surfaces after the delay time and the ice adhesion needs further improvement. It remains a daunting challenge to avoid ice formation and accretion.

Herein, we report an easy and low-cost method to truly prevent the ice hazards by employing transparent electrolyte hydrogels (EHs), which are fabricated by infusing salted water into a polymer network. The EH surfaces can durably prevent ice formation and have ultralow ice adhesion simultaneously within a tunable anti-icing temperature range (down to −48.4 °C). The molecular dynamic (MD) simulations and in situ experiments show that the diffusion of ions to the interface lead to the melting of ice crystals. In addition, the EH can be quickly replenished with the salted water (e.g., straight forward seawater), resulting in sustainable icephobicity. Our strategy could potentially be used as a sustainable ice protection system in the offshore platforms and ships.

2. RESULTS AND DISCUSSION

2.1. Morphology, Composition, and Properties of EH. To demonstrate the anti-icing/icephobic properties of EHs, they were fabricated by infusing sodium chloride (NaCl) as a typical ion source into the poly(vinyl alcohol) (PVA) hydrogel, resulting in a solid compound with liquid inside. The EH sample prepared from a 20 wt % aqueous NaCl solution is used for investigation unless stated otherwise. The fabrication procedure of EH is shown in Figure 1a,b as well as the experimental part. As shown in Figure 1b, the PVA hydrogel shrinks after soaking in comparison with the EH due to the loss of water from the hydrogel caused by the concentration difference. Finally, the as-prepared EH was obtained with a thickness of 1.2 mm after further soaking without obvious volume and weight change, indicating that the concentration of ions in a PVA hydrogel and a NaCl solution reached an equilibrium. As shown in Figure 1b (3), the obtained EH shows a transparent character with an average transmittance of ca. 90.0% at a wavelength range of 400–800 nm (Figure S1). Two and three-dimensional atomic force microscopy (AFM) images (Figure 1c,d) reveal that the EH surface is smooth, with a root-mean-square roughness of 5 nm from an area of 500 × 500 nm².

Furthermore, the EDS test and weight measurement reveal that the EH consists of 40.7 wt % water, 30.0 wt % ions, and 29.3 wt % PVA matrix, demonstrating that the ions have been infused into the hydrogel. (Supporting Information, Figures S2 and S3). Furthermore, Raman spectroscopy was performed to reveal the existing configuration of water inside the EH. The peak (symmetric −OH stretch) shifts from about 3410 cm⁻¹ to about 3457 cm⁻¹ (blue arrow in Figure 1e), and the shoulder peak (3220 cm⁻¹, asymmetric −OH stretch) decreases significantly in comparison to Raman spectra of PVA hydrogel and water, demonstrating that the configuration of water is strongly influenced by ions. This is consistent with previous studies of the influence on the configuration of water by ions (NaCl), in which the ions and water molecules in EH interact with each other and form hydrated ions in comparison with random water molecules (Figure S4b). Furthermore, the crystallization/melting points of EHs with variable concentration of ions (0, 5, 10, 15, 20, and 23 wt %) are revealed by the differential scanning calorimetry (DSC) tests (Figure S5a) shows an example about how to identify the points), with obtained values summarized in Figure 1f. The crystallization/melting points of EHs decrease from −17.3/1.7 to −47.1/−21.8 °C with the concentration of ions increasing from 0 to 23 wt % due to the hydration. The crystallization/melting points of EH are also compared with those of water, 20 wt % NaCl solution, and PVA hydrogel (Figure S5b), revealing that the reduction of crystallization/melting points of EH is due to the addition of NaCl solution. Notably, it is due to the lack of nucleation sites during the cooling process, resulting in much lower crystallization points than the melting points. Hence, the melting point is used to characterize the antifreezing point of EH.

2.2. Anti-icing Mechanism of the EH Surface. According to our hypothesis, the ions inside the EH have mobility. They may diffuse to the interface due to the osmotic pressure between EH and ice/supercooled water, and then interact with the ice or supercooled water (Figure 2a−c). Consequently, ice crystals can be inhibited from formation or broken up, resulting in the generation of a liquid lubricating layer at the interface (Figure 2c). The mechanism of the destruction of ice crystals by ions at the interface is first demonstrated by MD simulations (Supporting Information, Figure S6). The results show that the ice crystal is destroyed by ions due to electrostatic and van der Waals interactions between the ions and ice crystals. Moreover, the detailed information is shown in the Supporting Information to demonstrate the process. Figure 2d shows an image of the pilot process via the MD simulations of ice and cooled water, in which a part of the ice is destroyed and forms a liquid state. Figure 2e further reveals that the melting rate of ice decreases with the decrease of temperature (from −5 to −15 °C), indicating that the generation of the lubricating layer is positively correlated to temperature.

Furthermore, the self-melting of ice at an EH surface is demonstrated by in situ observation of a 5 μL ice on the EH surface at −10 °C. As shown in Figure 2f, the morphology of the ice changes from a dark opaque triangular-like appearance
In detail, the slight transparent part of the droplet, close to the surface prepared from 20 wt % NaCl solution at temperatures by MD simulation. (f) Melting process of ice on the EH surface prepared from 20 wt % NaCl solution at −10 °C.

Figure 3. Antifreezing properties of the EH surface prepared from 20 wt % NaCl solution. (a–c) Schematic of droplets freezing process on different surfaces at −10 °C. (d) Freezing time of water droplets on the EH surface in comparison with PVA hydrogel and glass surfaces at different temperatures. The asterisk (*) in (d) indicates that the water droplets have not been frozen at −5 and −10 °C after 43 200 s.

Figure 4. Antifrost property of the EH surface prepared from 20 wt % NaCl solution. (a) Area ratio of the formed ice on different surfaces as a function of time at −10 °C and 50% RH. The insets in (a) are micrographs of surfaces at different elapsed times. (b) Micrographs of EH surfaces with decreased temperature after being stored at RH of 50% for 30 min. (c) Micrographs of EH surfaces with increased humidity after storage at −15 °C for 30 min. The white arrows in (c) indicate the condensed liquid water droplets on the EH surface.
glass surface, where the area ratio of ice on the surface increases to 95% within 60 s. The PVA hydrogel freezes at −10 °C with an opaque property, covered with a 100% area ratio of ice since 0 s (Figure 4a (2)). The EH shows a transparent property (insets in Figure 4a (3) and Video S1), and the surface does not change from 0 to 1800 s (0% area of ice), demonstrating antifrost property within minute-scale. Furthermore, how the variable temperatures and humidities influence the EH surface is revealed in Figure 4b,c, respectively. The micrographs are recorded after the samples are stored for 30 min at different temperatures (−25 to −10 °C) with the RH of 50% or different RH (0−80%) at −15 °C, respectively. As shown in Figure 4b, the EH surface has no ice after moisture flux (RH of 50%) is injected for 30 min above −15 °C; whereas the surface freezes when the temperature further decreases to −25 °C. In Figure 4c, when the RH is less than 50%, no water droplets condensate on the EH surface after 30 min; when the RH increases to 80%, water gradually condensates and droplets form on the EH surface. Hence, the antifrost function of the current EH works at temperatures down to −15 °C, despite water condensation at RH of 80%. Moreover, despite the limited time (30 min) for the antifrost test, it is predictable that the EH could durably prevent frost formation due to the destruction of ice via ions (Figure 2).

2.5. Ice Adhesion Strength and Deicing Time of the EH Surface. The ice adhesion strength is a key factor in determining how easily the ice can be removed from a surface. The normal and shear ice adhesion strengths of EH prepared from 20 wt % NaCl solution are measured at variable temperatures, respectively (Figure S9). Notably, when we tested the ice adhesion at −20 °C, the EH separated from the cooling stage with the ice still attached to the EH surface due to the larger interfacial adhesion between ice and EH than that between the stage and EH. Hence, the practical normal and shear ice adhesions of the EH surface at −20 °C are larger than 110 and 138 kPa, respectively (dotted ellipse in Figure 5a). At −15 to −5 °C, the normal and shear ice adhesions are all negligible due to the instrument capacity, where the normal and shear ice adhesions are all recorded as 0 (Figure 5a, the details are shown in the experimental part), which is ascribed to the self-generation of a sufficient lubricating layer between the interface of ice and EH (Figure 2). It is worth noting that the ice cylinder self-moves from the EH surface quickly at −15 to −5 °C during the shear ice adhesion test, showing the self-deicing property. In fact, during the ice adhesion test, the interface changes from EH–ice contact to that the lubricating layer insulates ice and EH, where the ice adhesion is influenced by the lubricating layer. With ion diffusion at the interface, the thickness of the lubricating layer increases, and the ice adhesion is determined by the properties of the lubricating layer finally. Furthermore, the thickness of the lubricating layer is estimated. The moving ice is hydroplaning on the lubricating surface.\(^{40}\) The system may change to hydrodynamic friction when ice moves. The thickness of the lubricating layer may be in the micrometer scale when the ice starts moving if the lubricating layer is roughly considered as pure water.\(^{41}\) Furthermore, the concept of anti-icing temperature window is introduced to describe the effective anti-icing temperature range of EH. The critical temperature of anti-icing temperature window is defined by the EH melting point obtained from the DSC test, as the EH freezes, and the ion diffusion process is inhibited below the melting point. Hence, the melting point is used as the critical temperature. Hence, the anti-icing temperature window of EH prepared from 20 wt % NaCl solution is above ca. −17.6 °C. Moreover, the anti-icing temperature window of the EH can be controlled by infusing different types of ions with different concentrations, as shown in Figure 5b. The EHs are prepared from different concentrations and types of salted water (typical deicing agent, 20 wt % KCl, 20 wt % NaCl, 23.0 wt %, and 30% CaCl\(_2\) solutions). The DSC tests show that the critical temperature of EHs can be tuned down to −48.4 °C. The speed for self-removal of ice is quantified by the deicing time, which is defined as the interval between the time when the stage reaches the target temperature and the time when the ice starts moving. As shown in Figure S10, the ice is first frozen on an EH surface at −25 °C (below the anti-icing temperature window), and then the EH with ice is rotated to target angle θ. Finally, the temperature of the EH surface is regulated to a target temperature (within the anti-icing temperature window). Once enough lubricating layers are generated with time, the component of gravity (\(mg\sin\theta\), in which \(m\) is the mass of the ice and \(g\) is the acceleration of gravity) drives the ice to slide off the EH surface. Figure 5c shows that the deicing time first decreases and then holds steady with the increase of \(\theta\) at a temperature range of −15 to −8 °C using the ice (5 μg). The reduction of deicing time is due to the increase of \(mg\sin\theta\) along the ice sliding direction and decrease of the normal pressure by \(mg\cos\theta\). After the tilted angle of the EH surface increases sufficiently, the speed for the formation of the lubricating layer dominates the deicing time, resulting in a steady deicing time.
The deicing times at \(-5 \, ^\circ\mathrm{C}\) and at \(-8 \, ^\circ\mathrm{C}\) (at a tilted angle beyond \(20^\circ\)) are recorded as 0 s, as the ice starts moving before the surface reaches the target temperature. In addition, the deicing time decreases with the increase of temperature from \(-15\) to \(-5 \, ^\circ\mathrm{C}\) at the same tilted angle due to the faster generation of the lubricating layer at higher temperatures. Furthermore, Figure 5d reveals how the mass of ice influences the deicing time, in which the ice cylinder with the same basal area (15 mm diameter) but with variable masses (0.5–3.0 g) are used for the experiments. The deicing time first increases with the increase of the mass of the ice, followed by a decrease. This may result from the increase of the component of gravity (via ice) and the decrease of normal pressure by \(mg \cos \theta\).

Figure 5e further shows the side-view optical images of the ice cylinder (1.0 g) moving on the EH surface at different elapsed times as a typical example, indicating self-removal of ice (self-deicing property), which is also shown in Video S2. The ice moving from the surface can be considered as the solid–solid friction. The thickness of an interfacial lubricant continuously increases as the deicing time increases, resulting in the lateral threshold force at interface decreasing with time. When the thickness of the lubricating layer reaches the critical value, ice starts moving as \(mg \sin \theta\) exceeds the lateral threshold force. Herein, the adhesion strength to ice can be roughly estimated as \(mg \sin \theta/S\), where \(S\) is the contacting area between the ice and EH and \(\theta\) is the tilted angle of the surface. For example, the adhesion strength is ca. 7.1 Pa for the system in Figure 5e, in which the difference in adhesion strength is due to the transition from static to kinetic friction. In short, the order of magnitude of the deicing time is within ca. 100 s above \(-15 \, ^\circ\mathrm{C}\) with a tilted angle of more than \(15^\circ\) and the ice adhesion is ultralow.

2.6. Sustainability. The durability of the material is an important aspect of practical anti-icing applications. The lubricating layer with ions could be depleted from the EH during the deicing process, resulting in reduction of ions in the EH and thus a decrease in the anti-icing ability. Herein, as shown in the side-view schematic of the EH (Figure 6a), we propose the methods (1) and (2) for the replenishment of salted water to solve the depletion problem based on self-diffusion of ions and water into the EH. In detail, method (1) represents the replenishment of salted water on the surface via a coating method, and (2) represents the real-time replenishment of ions solution by directly connecting the EH with salted water. As the ions and water can self-diffuse in the EH, the side edge of EH (1.5 \(\times\) 0.2 cm\(^2\)) directly contacts the salted water to demonstrate the sustainable diffusion of ions to EH. The normalized weight of the EH samples (size: 4 \(\times\) 1.5 \(\times\) 0.2 cm\(^2\)) with and without the replenishment as a function of time is shown in Figure 6c. In contrast to about 50% weight loss of the EH without replenishment due to the evaporation, the weight of EH with replenishment (2) almost does not change, demonstrating the self-diffusion of salted water and the replenishment of ions from any part of EH.

Furthermore, the recovery ability of the EH is demonstrated in Figure 6d. We imitate the depletion of ions and water inside the EH by infusing the EH in water for 2 min and thereafter removing water by heat treatment. Then, the EH was replenished by soaking it in the 20 wt % NaCl solution for 2 min. After the first cycle, the normalized weight of the EH after replenishment decreases by about 30%, which may be due to the collapse of EH during the first dry treatment. Notably, the recovery of the EH remains stable with similar normalized weight from 2th to 10th cycle, showing the sustainable recovery property of EH. To further investigate the recovery ability, the EH is soaked in the solution for 12 h, and the normalized weight shows no obvious change. This indicates that the salted water has high mobility in EH, showing a fast recoverable property. In practical application, such fast recoverable property could potentially be used as an ice protection system for offshore platforms and marine ships, where the replenishment via seawater (typical salted water) after concentration is low cost and straightforward.

3. CONCLUSIONS

In this work, we have developed the sustainable and transparent anti-icing EH surfaces based on the salted water-
infused polymer network. According to the MD simulations and experimental results, when water droplets/ice contact the cooled EH surface, the ions inside the EH diffuse to the interface, preventing ice nucleation and destroying the ice crystals into liquid water at the interface between EH and ice/water. It has been found that the EHs have durable antifreezing/frost properties and ultralow ice adhesion within a tunable anti-icing temperature window (down to −48.4 °C). Moreover, the ice can self-remove from the EH surface within a short time as 10 s at −10 °C, showing fast self-removal of ice. In addition, the EH can be quickly replenished with the salted water (e.g., seawater) by coating or directly contacting methods, resulting in sustainable anti-icing property. Our current anti-icing strategy opens up a new and low-cost strategy to prevent ice formation and accretion for multiple icing scenarios.

**ASSOCIATED CONTENT**

Supporting Information
The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsami.0c06912.

Materials and methods, molecular dynamic simulations details, transmittance spectrum of EH, SEM, and EDS results, the composition of EH, schematic of the configuration of water and a NaCl aqueous solution, a side-view snapshot of deposited water droplets on different surfaces, digital images of PVA hydrogel and EH, schematic of normal and shear ice adhesion tests, schematic of the deicing process, the displacement of ice as a function of time squared (PDF)
The antifrost test on an EH surface (AVI)
An ice cylinder moving with time on an EH surface (AVI)

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