Bioinspired elastomeric fibrillar surfaces have significant potential as reversible dry adhesives, but their adhesion performance is sensitive to the presence of liquids at the contact interface. Like their models in nature, many artificial mimics can effectively repel water, but fail when low-surface-tension liquids are introduced at the contact interface. A bioinspired fibrillar adhesive surface that is liquid-superrepellent even toward ultralow-surface-tension liquids while retaining its adhesive properties is proposed herein. This surface combines the effective adhesion principle of mushroom-shaped fibrillar arrays with liquid repellency based on double re-entrant fibril tip geometry. The adhesion performance of the proposed microfibril structures is retained even when low-surface-tension liquids are added to the contact interface. The extreme liquid repellency enables real-world applications of fibrillar adhesives for surfaces covered with water, oil, and other liquids. Moreover, fully elastomeric liquid-superrepellent surfaces are mechanically not brittle, highly robust against physical contact, and highly deformable and stretchable, which can increase the real-world uses of such antiwetting surfaces.

Certain species of insects, arachnids, and reptiles, most notably the gecko lizards, have micro- or nanoscale fibril arrays on their footpads that function as high-performance dry adhesives, allowing them to effortlessly climb on diverse surfaces. Such remarkable attachment ability is based on van der Waals and capillary forces, and the principles of contact splitting and crack trapping. Synthetic mimics of these fibrillar adhesive systems have been actively developed in the past two decades, with performance reaching and even surpassing that of geckos on smooth surfaces by using mushroom-shaped fibril designs. The fibrillar adhesive pads not only provide strong attachment to diverse surfaces, but they are also superhydrophobic. Water repellency has been suggested to contribute to self-cleaning of the gecko footpads and their synthetic mimics through the lotus leaf effect, and it likely helps in keeping the dry adhesion mechanism functional. Indeed, wetting by water dramatically decreases the adhesion of gecko footpads to substrates. Similar reduction in performance has been reported for artificial bioinspired adhesives under water.

In the case of low-surface-tension liquids, such as oils, wetting becomes a major concern if the liquid spreads easily at the interface and between the fibrils. Even though carefully controlled, thin layers of viscous oil (0.1–2.1 µm thick) applied on the fibril tips of artificial dry adhesives can enhance adhesion on both smooth and rough surfaces. Larger volumes of liquids (0.1–0.4 µL) at the solid–solid interface have been shown to drop adhesion to a fraction compared to dry conditions. For overall adhesion performance in various wetting conditions, it would be advantageous to displace (push away) liquid from the contact interface and make a dry contact. Preferably all liquids, regardless of their surface tension, should remain in the Cassie state (i.e., the liquid droplet staying suspended on top of the fibrils), even during contact with the target surface. The transition barrier to the Wenzel state (i.e., the droplet fully wetting the substrate and fibrils) should also be sufficiently high to provide robust liquid repellency, since the adhesion would drop drastically in the Wenzel state.

Combining high adhesion and low-surface-tension liquid repellency on the same fibrillar surface has not been possible yet as the two properties have fundamentally opposing requirements for solid fraction (the fraction of the solid surface in contact with the liquid or the solid surface to adhere)—it should be large for adhesion and small for liquid repellency. Furthermore, high liquid repellency has been traditionally achieved by a combination of surface chemistry and roughness modification, an approach which is typically incompatible with the goal of high adhesion. For example, sprayable coatings can be extremely effective at turning a surface superrepellent to all liquids, but they rely on ultralow surface energy and hierarchical micro- and nanoscale roughness, both of which are detrimental to adhesion.

Another prominent avenue for achieving repellency toward low-surface-tension liquids is based on arrays of microscale features with re-entrant geometry, inspired by the skin of springtails. In recent years, this approach has been taken further by the introduction of double re-entrant structures, which can repel all liquids regardless of surface chemistry. However, the fabrication techniques have mostly focused on rigid materials, which are not suitable for dry adhesives involving elastomeric compliant fibrils. Although rigid double re-entrant structures for liquid repellency have been fabricated on flexible substrates, and compliant double re-entrant structures have been demonstrated by shape-altering metal
deposition on poly(dimethylsiloxane) (PDMS) fibril tips,\[28\] fabrication of entirely compliant elastomeric liquid repellent surfaces without hindering their adhesion has not been accomplished yet.

Here, we report an elastomeric, stretchable mushroom-like fibrillar surface that is at the same time highly adhesive and superrepellent even toward ultralow-surface-tension liquids. We achieve these two properties simultaneously by adding a double re-entrant geometry to the bioinspired mushroom-like design of fibrillar dry adhesives. This approach allows the fibril tip surfaces to remain smooth for high dry adhesion, and involves no surface chemistry modification. Pull-off forces up to five times compared to flat smooth surface control are demonstrated, even for small preload forces on the order of 1 mN. Our scalable fabrication technique is based on 3D microprinting of a rigid master, followed by double molding to produce a soft replica.

We use PDMS as a representative elastomer, but the molding method is suitable for a wide range of elastomeric materials.

For the elastomeric fibrillar surface fabrication (Figure 1a), we first used two-photon polymerization method to 3D-print the master fibril array on a glass substrate. The master was fluorosilanized to allow subsequent demolding, and PDMS was then cast and cured on the fibril array. The negative replica was peeled off and fluorosilanized, and the subsequent molding yielded a PDMS replica of the original master. A softer PDMS composition (20:1 monomer to crosslinker ratio) for the negative replica was employed to facilitate demolding of the final replica, for which standard PDMS was used (10:1 monomer to crosslinker ratio). The final replicas were characterized as prepared without any further processing. The fibril dimensions (Figure 1b) were fixed as base diameter $B = 30 \, \mu m$, height $H = 40 \, \mu m$, neck diameter $d = 10 \, \mu m$, overhang height $h = 5 \, \mu m$,
and overhang thickness $t = 3 \mu m$. The cap diameter $D$ was varied for the adhesion studies (Figure S1, Supporting Information). The overhang dimensions could easily be reproduced in 3D-printing (Figure 1c), and were set large enough to facilitate replication in PDMS (Figure 1d). Fibril pitch for the replicated arrays (Figure 1e) was chosen as 60 $\mu m$ to maximize their liquid repellency (Figure S2, Supporting Information).

To achieve reliable liquid repellency, particularly for ultralow-surface-tension liquids, the fibril cap diameter and center-to-center distance in the array were chosen in a way that favors robustness of the Cassie state rather than simply maximizing advancing and receding contact angles by decreasing the solid fraction. In practice this meant inclining toward a larger fibril cap diameter and smaller center-to-center distance. This tradeoff is reasonable, because the increased contact area also leads to higher dry adhesion. We found that, for fibrils with a cap diameter of 28 $\mu m$, reducing the center-to-center distance to 60 $\mu m$ was required for keeping all the tested liquids in the Cassie state, suspended by the vertical components of surface tension (Figure 2a). Liquid repellency of the double re-entrant PDMS fibrils was characterized using contact angle measurements.

Advancing and receding contact angles were determined for a variety of liquids covering surface tensions in the range of 11.91–72.80 mN m$^{-1}$ (Figure 2b). Perfluorohexane was included in the test liquids as a representative fluorinated organic solvent, which typically wets most surfaces completely due to its ultralow surface tension (11.91 mN m$^{-1}$). Even perfluorohexane (Figure 2b, inset), and an excessive amount of decafluoropentane (Movie S1, Supporting Information) remained in the Cassie state on the double re-entrant soft fibril array. Due to the high robustness of the Cassie state, the elastic surface could be bent to a curve and could still repel low-surface-tension liquids, such as methanol (Figure 2c), even allowing droplet pick-and-place manipulation of various liquids including methanol (Figure 2d; Movies S2 and S3, Supporting Information).

Adhesion of the fabricated fibrillar surfaces was characterized with pull-off force measurements (Figure 3a) using a smooth hemispherical glass probe (radius of curvature: 4 mm) as the contacting surface at a speed of 25 $\mu m$ s$^{-1}$ (Figure S3, Supporting Information). Pull-off forces increased with increasing cap diameter, when all other fibril dimensions were fixed (Figure 3b), reaching values up to five times higher than...
a flat control surface for the largest diameter (32 µm) with a preload force of 2 mN. Considering the projected contact area at the moment of maximum pull-off force, adhesion stress surpassed 100 kPa for the cap diameter 28 µm and larger. Overall work of adhesion of these fibrillar structures\[29,30\] was calculated from the representative measured force–displacement curves with a preload of 2 mN, loading/unloading speed of 25 µm s\(^{-1}\), and projected maximum contact areas extracted from corresponding video recordings. Measured overall work of adhesion values are 6.3, 7.3, and 8.4 J m\(^{-2}\) for cap diameters 28, 30, and 32 µm, respectively. The well-known dependence of adhesion on the preload force\[31\] was also observed (Figure S4, Supporting Information). Finite element (FE) simulations showed that the stress in the fibrils is concentrated around the fibril tip center and neck (Figure 3c), while the double re-entrant overhang remains almost free of stress (Figure 3d). The FE simulations (Figure S5, Supporting Information) and our experiments confirm that adding a double re-entrant overhang to the fibrils does not interfere with their adhesion performance with the selected fibril material and geometries, but may actually help preventing cracks initiated near the fibril edges from propagating and leading to adhesion failure, which is consistent with recent modeling of the T-shaped fibrils.\[32\]

We also simulated adhesion for a flat punch and a fibril with single re-entrant tip shape, and compared to the double re-entrant fibrils (Figure S6, Supporting Information). Cap diameter was the same (28 µm) for all three cases. The results show that single re-entrant and double re-entrant geometries lead to similar stress distribution, which is more equal along the contact interface compared to the flat punch. Their adhesion is thus expected to be similar and performance significantly higher than the flat punch, assuming the same cap diameter. On the other hand, shear adhesion simulations (Figure S7, Supporting Information) indicated that the increased bending compliance of the double re-entrant tip shape allows more stress to propagate through the tip compared to the single re-entrant case,\[33\] which may lead to lower adhesive shear strength. While there have been some studies for optimization of adhesion (pull-off force) for bio-inspired fibrillar structures,\[34,35\] optimization of the single and double re-entrant fibrils for maximum shear strength/adhesion is a future work, as a computationally intensive and complex problem to solve.

To investigate the advantages of liquid superrepellency for bioinspired dry adhesives in realistic wetting conditions, we compared adhesion of the double re-entrant fibrils with 5 µL droplets of different test liquids (water, ethylene glycol, and methanol) at the contact interface (Figure 4a) to the adhesion in a dry condition. The results show that keeping liquid in the Cassie state enables direct dry contact between the fibril and the contact surface, so that short-range van der Waals forces dominate, and dry adhesion performance is almost completely

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**Figure 3.** Adhesion of double re-entrant PDMS fibrils. a) A representative force curve of a dry adhesion measurement using a smooth hemispherical glass probe (radius of curvature: 4 mm) indenter at a speed of 25 µm s\(^{-1}\). b) Pull-off forces for the double re-entrant PDMS fibril arrays as a function of fibril cap diameter for a 2 mN preload force (dashed line indicates adhesion on flat PDMS). c) Finite element simulation of the normal stress distribution of a single fibril when stretched 25%—normalized to stress at the base—and d) the corresponding stress profile at the contact interface. Such relatively equal stress distribution is required for maximizing the dry adhesion.\[32,35\]
retained. On the other hand, collapse to the Wenzel state greatly reduced short-range forces, and the remaining weak long-range capillary forces resulted in significantly reduced adhesion (Figure 4b). Notably, if liquid is fully pushed away from the contact interface (e.g., in the case for water), adhesion is similar to the dry case; if the liquid is partially pushed away (e.g., in the case for ethylene glycol), adhesion is slightly reduced; and if the liquid is not pushed away (e.g., in the case for methanol) and collapses to the Wenzel state, adhesion is significantly reduced. Our adhesives therefore do not have strong adhesion under fully submerged wet conditions. For low-surface-tension liquids, such as methanol, a hydrophobic contacting surface (static water contact angle 103.7°) enables partially displacing it, thus preventing collapse to the Wenzel state and therefore retaining the adhesion performance (Figure 4c,d; Movie S4, Supporting Information). With a hydrophilic contact surface...
(static water contact angle 45.6°), the representative force curves for water, ethylene glycol, and methanol illustrate the transition from strong short-range forces facilitated by the Cassie state to weak capillary forces due to the Wenzel state (Figure 4e–g). Liquid repulsion/displacement ability of the proposed fibrillar adhesive during contact depends on wettability (e.g., hydrophobicity/hydrophobicity) of the contacting surface (Figure 4a,h), which therefore significantly affects the adhesion performance.

Our approach of integrating recent advances in bioinspired adhesion and wetting research effectively provides a new perspective for robust soft fibrillar adhesive surfaces that can function in real-world applications with possible liquids on surfaces. The fibrils retain adhesion after repeated cycles (Figure S8, Supporting Information). Since liquids collapsing to the Wenzel state do not induce any changes to the fibrils, adhesion remains the same after liquid has fully evaporated. They also retain their adhesive and liquid-superrepellent properties even after being exposed to high mechanical contact forces and harsh physical handling (Movie S5, Supporting Information), and can be used, for example, to pick up wet objects as shown in Movie S6, Supporting Information. The fully elastomeric liquid-superrepellent surfaces are also deformable and stretchable, which could advance the real-world uses of such antiwetting surfaces. We also studied the effect of stretching the fibril arrays on apparent contact angles of water (γ = 72.8 mN m⁻¹), diethylene glycol (γ = 44.8 mN m⁻¹), and methanol (γ = 22.7 mN m⁻¹). The samples were clamped from both ends and a uniaxial stretch of 16% was applied using a motorized stage. Apparent contact angles dropped for all three liquids (Figure S9, Supporting Information). Mechanical robustness and compliance have been shown to be a key feature in achieving robust liquid repellent surfaces with high impalement resistance.²² The scalable molding-based fabrication process allows future scaled-up manufacturing of such liquid-superrepellent fibrillar adherives for a wide range of industrial applications in apparel closures, robotics, automotive industry, medical devices, portable electronics, and manufacturing, where there could be oil or other liquids at the contact interfaces.

Experimental Section

**Master Mold Design and Fabrication:** The fibrils were designed using CAD software (Inventor Professional 2016, Autodesk Inc., San Rafael, CA, USA), converted to laser writing files (DeScribe, Nanoscribe GmbH, Karlsruhe, Germany), and printed using a laser 3D printer based on two-photon polymerization (Photonic Professional GT, Nanoscribe GmbH, Karlsruhe, Germany). The masters were printed on 25 mm x 25 mm x 0.7 mm (thickness) indium tin oxide (ITO)-coated glass substrates using the commercial IP-S photoresist (Nanoscribe GmbH, Karlsruhe, Germany). In order to enhance the adhesion of the photoresist to the substrate for molding purposes, the ITO-coated glass was placed in a closed desiccator next to a droplet of 1,1,1,3,3,3-hexamethyldisilazan (HMDS, Carl Roth GmbH, Karlsruhe, Germany) and left overnight. After exposure in the 3D laser printer, the master fibril array was developed in propylene glycol monomethyl ether acetate (PCMEA, Sigma-Aldrich Inc., St. Louis, MO, USA) for 30 min, followed by a short rinse in isopropl alcohol (IPA).

**Master Negative Replication in PDMS:** The fibril master mold was first activated in an oxygen plasma chamber (model: Zepto, Diener electronic GmbH, Ebhausen, Germany) for 3 min at 90 W, and then fluorosilanized by placing it in a vacuum desiccator together with a glass vial containing 0.1 mL trichloro(1H,1H,2H,2H-perfluoroctyl)silane (Sigma-Aldrich Inc., St. Louis, MO, USA) for 1 h, followed by baking in oven at 90 °C for 1.5 h. The fluorosilanized master substrate was fixed to a glass vial containing a 0.1 mL mixture (20:1 base monomer to crosslinker ratio) was prepared and poured around the fluorosilanized master substrate, and after 15 min of degassing in a vacuum desiccator the PDMS was allowed to flow over the master by tilting the desiccator. The negative replica was cured at room temperature for 48 h, then in oven at 65 °C for 24 h, and finally peeled off carefully.

**Master Positive Replication in PDMS:** The positive PDMS replica was placed on a glass plate with the cavities facing up and activated in a UV-ozone chamber (model: PS8D-UVT, Novascan Technologies Inc., Boone, IA, USA) for 30 min and then fluorosilanized by placing it in a vacuum desiccator together with a glass vial containing 0.1 mL Trichloro(1H,1H,2H,2H-perfluoroctyl)silane (Sigma-Aldrich Inc., St. Louis, MO, USA) for 5 h, followed by baking in oven at 90 °C for 1.5 h. Standard PDMS (10:1 base monomer to crosslinker ratio) was prepared and poured on the fluorosilanized negative replica around the cavity and by 15 min of degassing in a vacuum desiccator the PDMS was allowed to flow over the negative replica and into the cavities. The positive replica was cured at room temperature for 48 h, then in oven at 65 °C for 24 h, and finally peeled off carefully.

**Wettability Characterization:** Surface wettability was characterized using the sessile drop method on a commercial contact angle measurement device (model: DSA100, Krüss GmbH, Hamburg, Germany). Advancing and receding contact angles of ~2 μL droplet were measured three times at three different locations on each sample, for a total of nine measurements per liquid. Slow liquid dosing and aspiration speeds of 0.1–0.2 μL s⁻¹ were used.

**Adhesion Characterization Setup:** The custom adhesion setup was built around an inverted optical microscope (model: Axio Observer A1, Carl Zeiss AG, Oberkochen, Germany) connected to a video camera (model: Grasshopper 3, FLIR Systems Inc., Wilsonville, OR, USA) for visualization of the contact interface. Adhesion forces were recorded with a sensitive load cell (model: GSO-25, Transducer Techniques LLC, Temecula, CA, USA) mounted on a computer controlled high-precision motorized piezo stage (model: LPS-65 2”, Physik Instrumente GmbH & Co. KG, Karlsruhe, Germany) moving in the z-direction with 5 nm positioning resolution. Another high-precision motorized piezo stage was used for y-direction. Fine adjustments in x- and y-direction are enabled by a manual xy-stage and tilt correction by two goniometers. Motion control of the piezo stages and data acquisition were done using custom software running on Ubuntu Linux. The load cell was connected to the computer through a signal conditioner (model: BNC-2110, National Instruments, Austin, TX, USA) and a data acquisition board (model: PCIe-6259, National Instruments, Austin, TX, USA).
employed to create a wall enclosing the micropatterns to limit the flow of 50 µL fluid. The glass probe was cleaned after each series of experiments with a particle-free tissue and isopropanol alcohol. A minimum of ten measurements were carried out for each data point. The experiments were conducted at room temperature (23 °C) and a humidity of 30%.

Droplet Pick-and-Place Experiments: Droplets of water and methanol were picked up from liquid super-repellent microfibrillar adhesive patches and placed on flat surfaces using the custom setup described above. Both the surface initially having the droplet (bottom surface) and the picking surface (top surface) had identical topography consisting of the same double re-entrant microfibril arrays. To facilitate the process, the bottom surface was fluorosiliconized with trichloro(1H,1H,2H,2H-perfluorooctyl)silane while the top surface remained untreated. The resulting surface energy contrast between the bottom and top surfaces was enough to make droplets stick slightly more to the untreated top surface, allowing them to be picked up from the fluorosiliconized bottom surface. Since both surfaces were liquid super-repellent, the droplets maintained a highly spherical shape until being placed down on a flat PDMS surface. Picking droplets were also demonstrated with the bottom surface attached to a highly curved, concave piece (half pipe) of flat PDMS surface. Picking droplets were also demonstrated with the flat PDMS surface. Since both surfaces were liquid super-repellent, the droplets maintained a highly spherical shape until being placed down on a flat PDMS surface. Picking droplets were also demonstrated with the bottom surface attached to a highly curved, concave piece (half pipe) of plexiglass and the top surface accordingly to a convex half cylinder.

Fibril Stress Simulations: Simulation studies were conducted to analyze stress distributions for contact interfaces and fibril bodies when tensile or shear load is applied. Using finite element analysis (FEA) in a commercial simulation software (COMSOL Multiphysics 5.4, COMSOL Inc., Burlington, MA, USA), the stress distribution at the contact interface under tensile load was studied following the numerical approach of Balijepalli et al.[32] In addition to interfacial stress, stress distribution on the fibril body was further studied to identify where the maximum stress appears due to the unconventional shape of the fibril. For the simulation, the fibril was assumed fixed at the base and fibril top initially attached to a flat rigid surface (probe), while tensile load was applied to the far end of the probe. The hyperelastic behavior of the stretched fibril was represented by the Mooney–Rivlin hyperelastic model.[30] Stresses were analyzed at infinitesimal stretch and 25% stretch of the fibril body, and normalized to the maximum stress occurring at the fibril base. A total of 11 278 free quadrilateral elements were used for the double re-entrant fibrils, with extremely fine meshes for the contact interface and edge of the fibril top. Material properties in the simulation were chosen for PDMS cured in room temperature (25 °C), where the Young’s modulus is 1.32 MPa and the Poisson’s ratio 0.499.[31] The Mooney–Rivlin model parameters were 75.5 kPa (C10) and 5.7 kPa (C00).[32]

For the stress simulations under shear load, the fibril was assumed fixed at the base and fibril top initially attached to a flat rigid surface (probe). Compression (preload) was then applied to the far end of the probe, followed by lateral displacement. Since the axisymmetric modeling approach used for normal adhesion cannot be applied for shear adhesion, a full 3D model was adopted. To make the simulation computationally feasible, the hyperelastic fibril model was replaced with a simple linear elastic model, and solutions were sought with infinitesimal compression and a very small lateral shear distance of 1 µm. Within this small displacement regime, the linear elastic model for the fibril should give qualitatively similar results as the hyperelastic model. Material properties and other simulation parameters were the same as in the normal adhesion simulations.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

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