

Patterned Nanobrush Nature Mimics with Unprecedented Water-Harvesting Efficiency

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Water scarcity is one of the most alarming problems of the planet. An ambient ion based method is developed to make hydrophilic-hydrophobic patterned silver nanowires (NWs) as humidity harvesters of unprecedented efficiency. Such water harvesters are developed by two-step surface modification of the as-synthesized NWs (known from a report earlier) using electrospray. These patterned NWs of $\approx 20 \mu\text{m}$ length and $\approx 200 \text{ nm}$ width grown over a relatively large area ($2 \times 2 \text{ cm}^2$) exhibit atmospheric water capture (AWC) efficiency of $56.6 \text{ L m}^{-2} \text{ d}^{-1}$, the highest reported so far. The whole fabrication process of the surface is performed under ambient conditions with a home-built nanoelectrospray ion source, without the help of any sophisticated instrumentation. The synthesized material combines and mimics two exciting examples of AWC in nature, which are cactae and Namib Desert beetles, which utilize AWC for their living. It is believed that the combination of the special features of the above two natural species helps to achieve the highest water capture efficiency reported till date. A working prototype using this surface for AWC is also fabricated.

Availability of clean fresh water is one of the measures of development of a society. However, water resources have become severely depleted or highly polluted due to the demands of rapid economic growth as well as poor management in most parts of the planet. This has resulted in water scarcity to be one of the gravest social risks of the modern world while it was considered to be an environmental risk a few years ago.^[1] Around 1.2 billion people, or almost one-fifth of the world's population already live in areas with water shortage, and another 500 million more people are being pushed to this situation. By the year 2025, 1.8 billion people will be living in regions with sheer water paucity, and two-thirds of the world's population could be living in water-stressed circumstances. Atmospheric water capture is being considered as a possible solution for providing clean water to people living in those areas, for sometime now.

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This is due to the fact that at any given moment, the earth's atmosphere contains an astounding 37.5 million billion gallons of water as vapor,^[2] and an efficient device to capture a fraction of this water vapor, in a cost-effective way would help solve the water crisis.

Over the period of human existence, it has become apparent that biomimicking is the most efficient way to tackle such problems. When we look into nature, there are organisms, which in the course of evolution have acquired physical traits that enabled them to capture atmospheric water, even in the most arid corners of our planet. One such example is the *Stenocara* beetle of Namib Desert which capture water on its hardened forewings.^[3] Electron microscopic images of these have revealed unique array of hydrophilic regions distributed on a superhydrophobic background,^[4] creating a surface

energy gradient, which facilitates efficient condensation and transportation of atmospheric water. Another such example is spider silk, which shows unique periodic spindle-knot structure when wetted and efficiently collects water from atmosphere through the combination of surface energy gradient and Laplace pressure difference. Other inspirations from nature are some cactaceae species which live in arid environments and are extremely drought-tolerant.^[5] These species are shown to have structures with spines and trichomes which enable them to condense humidity efficiently from the atmosphere. Grasslands are also examples of natural atmospheric water harvesters. Hence, micro/nanostructuring of the water-collecting surface plays a critical role in determining the efficiency of water capture. With the advancement of nanotechnology, significant efforts have been directed toward fabricating surfaces with similar morphological features and chemical patterning to enable efficient water capture.^[6,7] However, these natural and nature-mimicked surfaces collect atmospheric water in the form of dew/fog and require the temperature to drop below the dew point to cause condensation. For building a practical and round-the-clock operating atmospheric water generator, it is important to cool the condensing surfaces and the surrounding air efficiently, with minimum energy input. Heat transfer efficiency of the condensing surface material is as important as the water transfer efficiency for creating a viable radiative condenser. Although many biomimetic, patterned surfaces have been made for fog collection, inspired by active condensing

surfaces found in nature,^[7,8] most of these make use of polymers or polymer-hybrids, which might not be best suited for radiative condensers due to their poor heat-transfer efficiencies.

Metallic nanostructures are of great interest in recent research due to their excellent electrical and thermal conductivity.^[9–11] The enhanced electric field around these structures makes them potential substrates for surface-enhanced Raman spectroscopy (SERS)^[10,12] enabling sensing of various biological and nonbiological compounds present in water.^[13] Apart from these, metallic nanostructures exhibit interesting mechanical properties,^[14] while maintaining high degree of porosity,^[15] ordered arrays,^[16] and many more. Even though such nanostructures have potential applications in different areas, it has been difficult to synthesize them over large areas for commercial applications. Several methods have been developed to create patterned nanostructures which include chemical^[17] and physical^[18] vapor deposition, plasma chemical method,^[19] spray pyrolysis,^[20] etc. They can also be synthesized using photolithography^[21] or electron beam lithography^[9,22] as top-down approaches. Template mediation^[23] and self-assembly are also used for making desired nanostructures. However, all these methods need special conditions like vacuum, high temperature, templates, etc. Ambient, solution state techniques to create well-defined metallic nanostructures over large areas are still limited. In an earlier report from our group, long (80 μm) nanowire (NW) arrays were made through electrospray ionization-induced creation and deposition of Ag nanoparticles (NPs), under ambient conditions.^[24] These wires could be grown over several cm^2 areas to make a metallic grassland like morphology. In this report, we have shown growth of these structures over 4 cm^2 area; however, they can also be grown over several tens of cm^2 area using a programmable moving stage.

Here, we report the creation of a hydrophilic-hydrophobic patterned humidity-collecting surface as an efficient radiative atmospheric water capture device, by chemical modification of the metallic grasslands through electrospray deposition (ESD). While the use of NW arrays maximize the available surface area for water condensation, chemical modification of the NW surface helps with the quick transfer of the condensed water and regeneration of active sites for further condensation to happen. The surface modification strategy also improved the mechanical strength and stability of the NWs in water. In the design of the surface, we have successfully fabricated surfaces which mimic the hairy structures of cactae which in turn

have hydrophilic-hydrophobic patterns resembling the wings of *Stenocara* beetles along with superior heat transfer capabilities of metallic nanostructures. A combination of all these effects led to high water capture efficiency ($56.6 \text{ L d}^{-1} \text{ m}^{-2}$) at a prototype-scale. Although we have used Ag NWs reported previously^[24] as the initial building block of our patterned NWs, the present work focuses on a method of creating hydrophilic-hydrophobic patterned NWs using two simple steps, which mimic the above mentioned examples from nature and combine their special features to achieve highest water capture efficiency reported till date.

Hydrophilic-hydrophobic patterned Ag NWs were fabricated using ESD at room temperature in air. A schematic representation of the ESD process along with the dimensional descriptions is shown in Figure S1 (Supporting Information). As the growth of unpatterned NWs was described previously,^[24] we present only the essential aspects here. A detailed description of the synthesis and characterization (electron microscopy, elemental analysis) of the Ag NWs on an empty transmission electron microscopy (TEM) grid, which form the core of the patterned NWs, is given in the Supporting Information (Figures S2–S8, Supporting Information). For all our AWC experiments, we have selected stainless steel (SS) wire mesh as the substrate to create a cost-effective substrate for the application. Figure S9a,b (Supporting Information) shows scanning electron microscopy (SEM) images of the Ag NWs grown on an empty TEM grid and SS wire mesh, respectively, proving identical morphology of the NWs on both the surfaces. Hence, for all our water capture experiments, we used the hydrophilic-hydrophobic patterned Ag NWs grown on SS wire meshes (50 μm). Use of a larger mesh size would reduce the active area for water condensation because of the reduction of number of wires (on which the Ag NWs are grown) in a specified area. On the other hand, use of a lower mesh size would increase the number of wires (on which the Ag NWs are grown) but it would also reduce the active surface area by affecting the size of the Ag NWs. The Ag NWs grown on a reduced mesh size SS wire mesh were smaller in size because of the coulombic repulsion between the growing NWs. We have also compared the growth of these NWs on top of meshes of different shapes.

Figure 1a shows a SEM image of the as-synthesized Ag NWs on a SS wire mesh using ESD. It shows an arrangement of 1D NWs, which upon extension will resemble a grassland-like structure. This can be created over a larger area of the order of

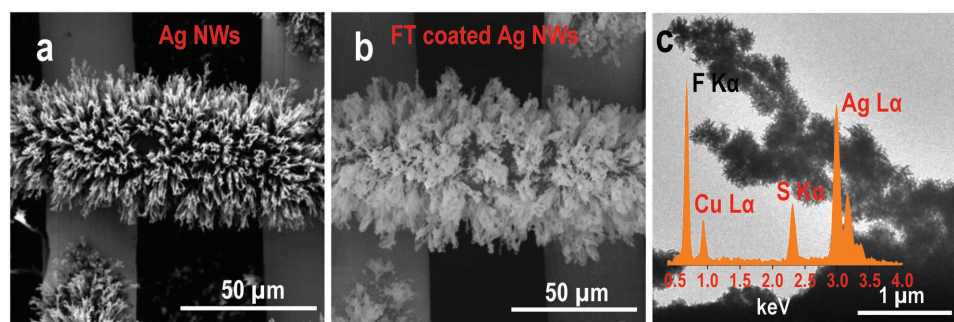


Figure 1. Electron microscopic characterization of the hydrophilic-hydrophobic patterned Ag NWs. a) SEM image of the as-synthesized Ag NWs on a 50 μm SS wire mesh, b) SEM image of similar NWs after fluorothiol coating. Difference in contrast is due to the organic content. c) TEM image of the FT-coated Ag NWs, showing the intact morphology of the material, EDS spectrum (inset) showing presence of F, S, and Ag. Cu is due to the grid.

4 cm². Figure S10 (Supporting Information) shows a large-area SEM image (300 μm²) of the Ag NWs on a TEM grid. These metallic NWs were the core of the patterned NWs with specific hydrophilic regions on a hydrophobic background. Fabrication of the patterned NWs was designed by successive ESD processes, as explained below.

At first, as-synthesized Ag NWs were subjected to an electrospray coating of a fluorothiol (FT), namely, 3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heptadecafluoro-1-decanethiol. A solution in dichloromethane (DCM) and acetonitrile (AcN) mixture was used for ESD of FT. This made the Ag NWs superhydrophobic in nature by the formation of a chemical bond between Ag and the thiol group, of the FT. Metal-sulfur bond is strong and is the basis of one class of self-assembled monolayers.^[25] Figure 1b shows a SEM image of FT-coated Ag NWs. The change in contrast (with the image in Figure 1a) is due to the coating of a nonconducting layer of FT on the metal. To prove the superhydrophobic nature of the surface, the contact angle of a water droplet on it was measured. Surface energy of the FT-coated surface was so low that a water droplet did not stand static on the surface. Figure S11 (Supporting Information) shows images of a water droplet rolling on the FT-coated Ag NWs. No pinning of the droplet was observed, proving the superhydrophobicity of the surface. Figure S12a–c (Supporting Information) shows optical images of a water droplet bouncing off from the FT-coated Ag brush substrate while another water droplet is stranded on the bare stainless steel wire mesh. This proves that the FT coating on the Ag brushes made them superhydrophobic in nature. Figure S12d (Supporting Information) shows contact angle measurement of a water droplet on stainless steel wire mesh before (normal wire mesh) and after modification (FT-coated Ag NWs). Contact angle of the water droplet was measured to be ≥125° in the case of normal mesh indicating that the normal stainless steel surface is hydrophobic (90° < θ_c < 150°). Measurement of resting contact angle was not possible for the superhydrophobic surface as it was too slippery for the droplet to sit on it. Hence, the contact angle was measured when the water droplet was touching the surface. A measurement of advancing and receding contact angles (173° and 166°, respectively) was also performed (Figure S13, Supporting Information). The contact angle hysteresis (173°–166° = 7°), is within the desired 10° for good superhydrophobicity. These measurements prove the superhydrophobic nature of the Ag NWs after FT coating. Roughness due to the presence of NWs also plays a significant role in acquiring superhydrophobicity. Control experiments were performed with normal stainless steel wire mesh and using the same sprayed with FT. It was seen that water droplets did not bounce off from the normal stainless steel mesh but wetted it instead, shown in Figure S14a (Supporting Information), clearly indicating that it was not superhydrophobic. Then FT was sprayed on the stainless steel mesh and the same experiment was repeated, images of which are shown in Figure S14b (Supporting Information). Here also the droplets did not bounce off and got stuck to the surface. However, when sprayed over by FT, the stainless steel mesh surface becomes hydrophobic to some extent. Hence, the presence of Ag NWs, providing surface roughness in nm scale, was essential to make it superhydrophobic. ESD is a gentle process; hence, it does not harm the morphology of the as-synthesized

nanobrushes. This was the prime reason for us to choose ESD of FT over Ag NWs. Other procedures of FT coating, such as spray coating with a mechanical sprayer were not useful. Although the spray-coated surface became superhydrophobic, it destroyed the morphology of the NWs (Figure S15, Supporting Information). To optimize the ESD time for FT coating, a time-dependent ESD experiment was performed (spray time of 2, 4, 6, 8, and 10 min) on five different spots containing identical Ag NWs. It was observed that each spot became superhydrophobic proving that ESD of FT for 2 min was enough to make the surface superhydrophobic. However, these FT-coated NWs lost their morphology when subjected to water wash. In view of that we have enhanced the ESD time of FT to 20 min at a rate of 20 nA deposition current. In this case, the FT-coated NWs retained their morphology after water wash. This indicates that ESD of FT also has a role in imparting stability to the Ag nanobrushes by Ag–S bond formation (there is another aspect how ESD of FT provides stability to the Ag NWs which will be discussed at the end of this paragraph). Hence, for all other experiments, the above mentioned time and rate of ESD was taken as optimum. Figure 1c shows a TEM image of the FT-coated Ag NWs. Inset in Figure 1c shows an energy-dispersive spectrum (EDS) taken from these NWs showing the presence of all the expected elements (Ag, S, and F).

From our data we know that our Ag NWs are made by an organization of NPs. During the ESD of FT on Ag NWs, the NPs constituting the NWs coalesce with each other, making a solid metallic core inside. Figure S16 (Supporting Information) shows the TEM images of the Ag NWs before and after FT coating. From these images, we can easily compare and come to a conclusion that the NPs in the FT-coated Ag NWs are fused, providing a solid core on which the superhydrophobic coating of FT is formed. This solid metallic core gives strength to the NWs, making them more resistant toward mechanical strain.

In the second step, we have performed ESD of AgOAc (aqueous solution, 10 × 10^{−3} M) over the superhydrophobic NWs to create Ag NP decorated NWs. These bare Ag NPs act as hydrophilic protrusions on the superhydrophobic NWs. In the following paragraph, we describe the structure of our material using electron microscopic images. The contact angle of a water droplet was measured after creating the hydrophilic zones. Figure S17 (Supporting Information) shows that the contact angle of a water droplet was 152°. Reduction of the contact angle, with respect to the superhydrophobic NWs, proves the presence of hydrophilic protrusions.

In Figure 2, we represent our material and compare it with the examples available in nature. Figure 2a shows a cartoon of a cactus with water droplets on the trichomes. Figure 2b shows a schematic of the cactus showing that its sharp spines and trichomes are responsible for water capture. Figure 2c shows a TEM image of our material which resembles the hairy structure of a cactus although the scales shown are different. Figure 2d shows a cartoon of a *Stenocara* beetle and the hydrophobic-hydrophilic patterning of the wings (Figure 2e). Figures 2f,g show a schematic of the synthesized hydrophilic-hydrophobic patterned Ag NW and a TEM image of the same with three distinct regions: (i) the Ag-core, (ii) hydrophobic coating on the Ag-core, and (iii) tiny hydrophilic regions (Ag NPs, see below for its synthesis) on the hydrophobic background. Ag core gives

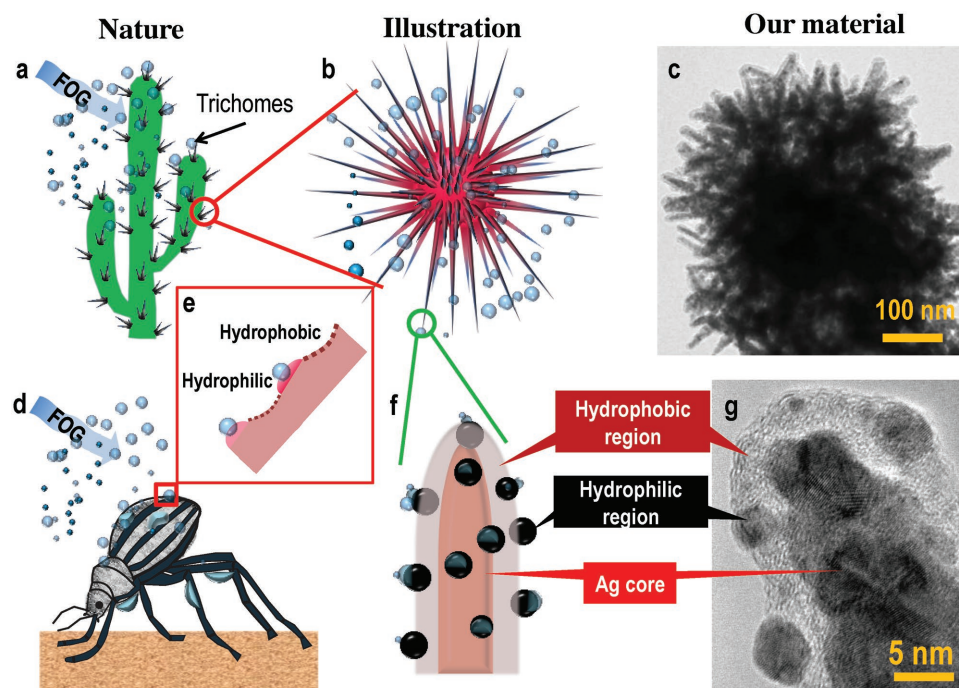


Figure 2. Graphical demonstration of the natural species with atmospheric water-harvesting capabilities, illustration of their special properties and comparison with our material. a) Graphical representation of a cactus, b) illustration of hairy structure of cactus, c) TEM image of a single Ag NW showing similar hairy structures resembling the cactus, d) graphical representation of a *Stenocara* beetle collecting water from atmosphere, e) schematic representation of hydrophobic-hydrophilic patterning of their wings, f) illustration of our superhydrophobic-hydrophilic patterned Ag NW showing a solid Ag core, a superhydrophobic coating on the core, and hydrophilic regions (Ag NPs) on the superhydrophobic background, and g) TEM image of a single Ag NW showing all three regions.

the strength to the brushes, while the FT coating creates the hydrophobic background. Subsequent ESD of Ag on this FT-coated surface resulted in Ag NPs decoration all over the NWs. These Ag NPs, embedded in the FT layer, act as hydrophilic zones, known to be responsible for the condensation of atmospheric water. A schematic illustration of atmospheric water capture by these hydrophilic-hydrophobic patterned NWs is shown in Figure 2f.

A 2 cm × 2 cm SS wire mesh covered by these patterned NWs was created and used for water capture experiments described in the following sections. Evaluation of humidity condensation was performed using this surface mounted on a Peltier cooler, which was then videographed (Video S1, Supporting Information) using a microscope. Two experiments, one control (with normal stainless steel mesh) and another with the prepared surface, were performed to test the efficiency of the nanostructured material for atmospheric water capture. In both the experiments, the surface being examined was carefully mounted on a Peltier cooler, using silver paste as glue, so that it remained in adequate contact with the cooling stage. The entire arrangement was placed atop the viewing stage of an inverted fluorescence microscope (Leica), in a controlled room with 40% relative humidity and 28 °C temperature (the dew point at this condition ≈ 13 °C). An arrangement was made for illumination of the surface being examined, for the purpose of microscopy. Time-lapse optical microscopy of the stainless steel mesh as well as the prepared surface upon exposure to cool humid air was performed to monitor droplet

nucleation and condensation. The temperature of the surface under examination was maintained at 12 °C, measured with a thermocouple. Figure S18a,b (Supporting Information) shows the optical images of a normal stainless steel wire mesh after 60 and 120 s of collection, respectively. Condensation of water droplets was seen on the wire mesh. Figure S18c,d (Supporting Information) shows the optical images of condensation of water on the stainless steel wire mesh containing superhydrophobic Ag NWs with hydrophilic protrusions. It is seen that the amount of water captured in this case is much larger in comparison to a normal wire mesh. In a time span of 2 min, a volume of 2.6×10^{-6} L water was captured (approximately calculated from the number of droplets considering them to be spheres, $\approx 1.3 \times 10^{-8}$ L of water was captured over the normal SS wire mesh) on the brushes, even though the humidity was less and no water vapor was forcefully passed over the surface during the experiment. The video clearly shows nucleation of tiny water droplets on the patterned NWs followed by fusion of them to bigger droplets. Once the droplet is larger in size, it rolls off the surface because of its superhydrophobic nature.

Transport of the condensed water is a crucial criterion of any atmospheric water capture material. Hence, we calculated the water collection rate of the surface on a 2 × 2 cm² area considering all the parameters like, condensation rate, transport of the collected water, etc. Figure 3a shows an optical photograph of the total setup where the hydrophilic-hydrophobic patterned NWs containing SS wire mesh was firmly mounted (the black

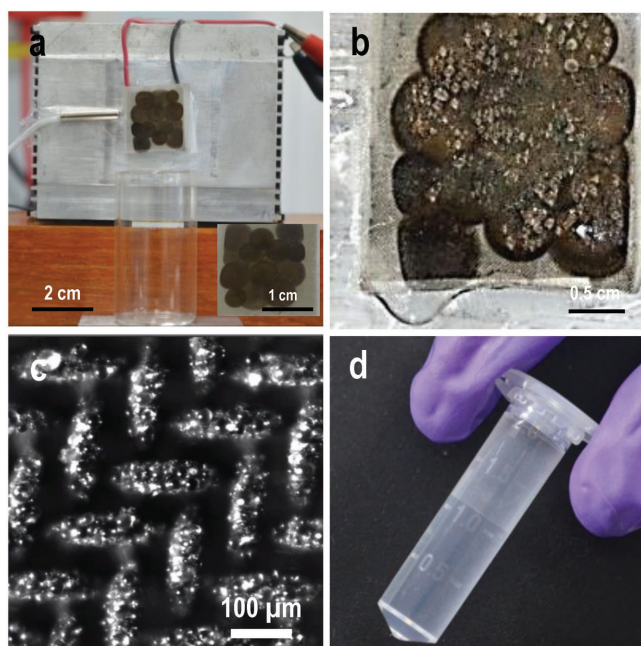


Figure 3. Optical and microscopic photographs of the condensed water droplets on a $2 \times 2 \text{ cm}^2$ area. a) Optical photograph of the water collection setup. Inset shows a photograph of the hydrophilic-hydrophobic patterned surface over a $2 \times 2 \text{ cm}^2$ area. Each ESD results in a 5 mm dia dark colored disk indicating the growth of NWs. This process was repeated multiple times to cover the $2 \times 2 \text{ cm}^2$ area. b) Zoomed optical photograph of the surface after switching on the Peltier cooler showing condensed water droplets on it and dripping of water at the bottom of the surface. c) Optical photograph (at a $100\times$ magnification) of the Ag NWs with condensed water droplets. d) Optical photograph of the collected water in a 2 mL vial.

square surface) on a Peltier cooler, using a carbon tape. Carbon tape was used in this case, instead of silver paste, for better transport of the collected water. Inset of Figure 3a shows a photograph of the surface. A 12 V DC fan was used for cooling the hot side of the Peltier cooler. Temperature of the surface was measured and kept constant (7.5°C) during the experiment. The above experiment was performed inside an air-conditioned room where the relative humidity was 48% and the ambient temperature was 26°C . There was no forced airflow. Figure 3b shows the formation of water droplets on the hydrophilic-hydrophobic patterned nanobrushes, just after switching on the Peltier cooler. Figure 3c shows an optical microscopic image of the surface at $100\times$ magnification, clearly showing the accumulated droplets on it. In the above experiment, 3.75 mL of water was collected in 13 h. If we convert the water collection to $\text{L d}^{-1} \text{ m}^{-2}$ area, it comes to $7.9 \text{ L d}^{-1} \text{ m}^{-2}$. Figure 3d shows an optical photograph of the collected water in a 2 mL vial. Control experiments were performed to compare the water capture efficiency of the hydrophilic-hydrophobic patterned NWs with as-synthesized Ag NWs and hydrophilic Ag NWs. Although the parent surfaces without chemical functionalization were unstable during water capture experiments, we made the structures stable by exposing them to 4-mercaptobenzoic acid. In addition to enhanced stability during water capture, this functionalization made the surface hydrophilic. Such surfaces showed a

water capture efficiency of 4.3 L d^{-1} under the same conditions as before.

For the next set of experiments, we designed a prototype to evaluate the water capture efficiency of the surface in presence of air flow. Figure 4a shows a schematic of the prototype. In these experiments, a 12V DC (air flow speed of 2.55 m s^{-1} volume 103–105 cfm) fan was used to blow atmospheric air toward the cold hydrophilic-hydrophobic patterned surface. Two sets of experiments were performed with this prototype: (i) inside an air-conditioned room (at lower humidity) (Figure 4b shows an optical photograph of the setup), and (ii) outside the lab, under ambient conditions (Figure 4c shows a photograph). Conditions and data collected from these experiments are described here. In the first set of experiments, surface temperature and average relative humidity were 8°C and 58%, respectively. The water collection efficiency was $26.8 \text{ L d}^{-1} \text{ m}^{-2}$. In the second set of experiments, the temperature of the surface was kept the same and average relative humidity was $\approx 87\%$. In this case, the water collection efficiency was $56.5 \text{ L d}^{-1} \text{ m}^{-2}$. This is the highest water collection reported till date by any surface. The high efficiency of water collection can be attributed to the combination of cactus effect as well as hydrophilic-hydrophobic patterning. Water droplets start nucleating on the hydrophilic regions and as they grow larger in size, they start to roll off from the surface, generating fresh surface for collection.

For long-term use of the substrate, stability of the brushes is an important aspect. Reusability and the morphology of the surface were checked after the water capture experiment. Figures S19a,b (Supporting Information) show SEM images of the hydrophilic-hydrophobic patterned Ag nanobrushes after five cycles of water capture. The images show that the morphology of the brushes is almost intact after the experiment. Figures S19c,d (Supporting Information), respectively, show contact angle measurements of a water droplet on the substrate before and after the water capture experiment. The contact angle was found to be the same in both the cases, proving that the nature of the surface is the same after dew collection.

An ambient solution-state method of creating hydrophilic-hydrophobic patterned 1D Ag NWs, using electrospray deposition in three simple steps, is presented. The whole synthesis including the Ag NWs (the core of the patterned NWs) was performed at room temperature without the help of any sophisticated instrumentation. This method of synthesis enabled us to create a material that could mimic two efficient natural atmospheric water harvesters. Combination of hydrophilic patterns on a superhydrophobic background along with hairy structures of the Ag NWs led to highest water capture efficiency, reported till date. We also built a prototype and tested its water capture efficiency under various conditions. At $\approx 87\%$ relative humidity, our material showed a water capture efficiency of $56.5 \text{ L d}^{-1} \text{ m}^{-2}$.

Experimental Section

For all experiments, nanospray emitters were made using a micropipette puller (P-97) purchased from Sutter Instrument, USA. To confirm the

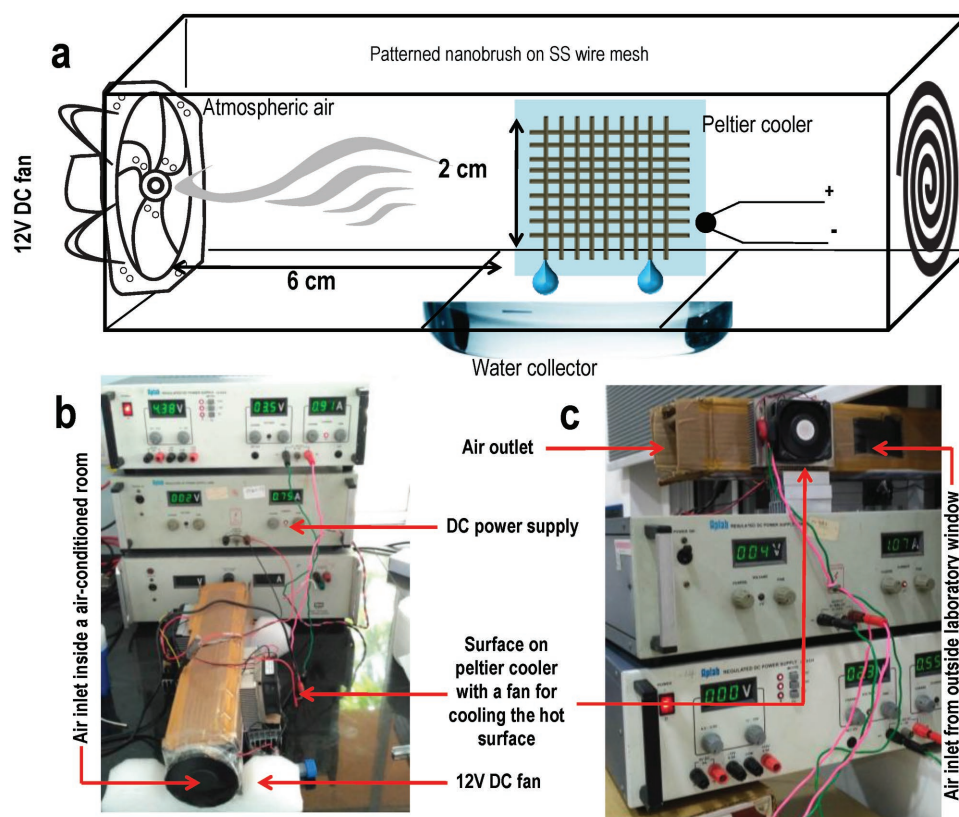


Figure 4. Prototype of the atmospheric water capture experiment. a) Schematic and b,c) photographs of water capture experiments inside and outside the laboratory. The photograph in (c) shows that the air inlet of the prototype is kept outside the laboratory window.

presence of solvated Ag^+ ions, mass spectra were collected using an ion trap LTQ XL (Thermo Scientific, San Jose, CA) mass spectrometer. Indium tin oxide-coated glass slides (Toshniwal brothers (SR) Pvt. Ltd., India) was the usual deposition substrate. Locally available stainless steel wire mesh was used as a surface to grow the Ag NWs. All TEM measurements were performed using a JEOL 3010 (JEOL Japan) transmission electron microscope. Copper TEM grids were purchased from SPI Supplies. A FEI Quanta 100 instrument with tungsten filament source was used for SEM imaging. EDS analyses were performed with an attachment on the SEM instrument. Contact angle and contact angle hysteresis (CAH) of water droplet on the different coated substrates were measured using a Holmarc contact angle meter. Optical photographs of water droplets condensed over the hydrophilic-hydrophobic patterned Ag NWs and normal SS mesh was performed using a 100 $\times$ objective fitted to a Leica DFC365 FX microscope. Optical photographs of the surface, prototype, and water droplets bouncing off the surface were captured using a Nikon D5100 camera using an 18–55 mm lens. Videos were also captured using the same camera.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

nature mimics, prototype, superhydrophobic-hydrophilic patterned NWs, water harvesting

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Supporting Information

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

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