Renewable Water Harvesting by Amyloid Aerogels and Sun

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Solar distillation is an appealing and facile technology for seawater desalination and water purification, but constructing stable, environmentally-friendly, and cost-effective solar evaporators often remain unfeasible. Here, amyloid hybrid aerogels are introduced for universal and renewable freshwater production by relying on the combination of sustainable materials and solar energy, thus making the process highly ecological and virtually costless. Amyloid fibrils obtained from whey, a byproduct of the dairy industry, are used as the main scaffold for the aerogels, whose solar energy harvesting properties of the top surface are enhanced by bioinspired dark polymers, such as polydopamine. Due to the fast water transport throughout the porous aerogels, the high-yielding photothermal conversion ability of the top surface, and efficient heat insulation at the bottom, the hybrid amyloid fibrils aerogel exhibits a water evaporation rate of 1.61 kg m\(^{-2}\) h\(^{-1}\) with a solar-thermal conversion efficiency as high as 91.3\% under 1 sun illumination. The process allows desalinating various seawater sources with different salinities to levels below the drinking water threshold recommended by the World Health Organization. Moreover, the same hybrid amyloid evaporator is highly efficient in removing heavy metals, organic pollutants, bacteria, and viruses from water, introducing a general, sustainable and energy-efficient solution for desalination and water purification.

1. Introduction

One of today’s most severe global challenges that will have an even greater impact in the future, is the ongoing water crisis and the inaccessibility to clean water.[1] With climate change,[2] growing world population, and increasing water pollution,[3,4] there is a urgent demand for sustainable and economical solutions that can deliver safe drinking water worldwide. A solution that could address this challenge is desalinating largely abundant seawater turning them into potable water.[4] The main desalination technologies can be categorized into thermal desalination and reverse osmosis membrane separation.[5] Both methods demand centralized and technically-advanced water treatment plants which are tied to high energy consumption and great costs, restricting their broad application.[6] Thus, sustainable on-site water purification technologies with affordable cost and low energy input are still highly sought-after.[7]

Recently, distillation powered by solar energy (solar/solar-driven evaporation) has been proposed as a possible alternative to conventional desalination techniques[8] if decentralized water purification could be enabled by this method, safe drinking water could become accessible to many. Yet, solar-driven evaporation systems still face significant challenges that prevent them from being implemented into real-life applications.[9] While novel materials often lead to improved efficiency and evaporation rates,[10] these are neither cost-efficient for real-life production nor are they sustainable against a full life cycle assessment, since they may cause, among other drawbacks, secondary pollution.[11] Furthermore, many solar evaporators do not have a consistent long-term performance and evaporation rates decrease gradually, which is further accelerated by fouling and salt precipitation.[12] Among these materials, hydrogels,[13] aerogels,[14] foams,[15] and porous materials,[16] have also been considered as cost-effective precursors for solar evaporators, yet, with all the drawbacks quoted above. These shortcomings hinder the transition from lab-scale to large-scale evaporation systems.

To address this open challenge, we report a two-layered biodegradable solar evaporator aerogel with whey amyloid fibrils (WAFs) as the main component. WAFs are highly sustainable and environmentally-friendly as whey is a protein-rich byproduct from the dairy industry,[7] the stability of WAF-derived aerogels can be further enhanced by using biodegradable polymers as gelators—such as polyvinyl alcohol (PVA) in the present case—instead of more commonly used salts or other harmful crosslinking agents such as glutaraldehyde.[13,17]
2. Aerogel Fabrication and Characterization

The first step of the fabrication process is preparing WAFs spanning both layers of the aerogel (Figure 1b). Self-assembly of the whey protein under proper conditions yielded WAF (see Supporting Information) whose characteristic birefringence was confirmed by cross-polarized light (Figure S1, Supporting Information). The bottom part of the hydrogel (precursor of the aerogel) was prepared through inducing gelation of WAF by adding PVA; the top layer was prepared in a similar way, but prior to gelation by PVA mixing, PDA was added to render the hydrogen black-colored top layer of the aerogel can be realized by adding a dye or any radiation-absorbing material, such as black carbon; to maintain a bio-inspired approach, we realize here the dark top layer by adding the WAFs polydopamine (PDA), a polymerized form of dopamine, a catechol-based amino acid involved in mussel byssus adhesion.[18] The hydrophilic bottom layer made of WAF and PVA allows water transport via capillary interactions, whereas the top dark layer of the aerogel constituted by WAF, PVA, and PDA has the energy-harvesting role (Figure 1a). In such a way, all the components of the aerogel are cost-effective, biodegradable, and cause no secondary pollution that may threaten humans or the environment.

Figure 1. Solar evaporation with hybrid amyloid fibrils aerogel and its fabrication process. a) Schematic illustration of solar water purification by WAF aerogel. b) Schematic of the WAF aerogel’s preparation process.

The aerogel’s spectrum, the characteristic peak around 1077 cm$^{-1}$ is ascribed to the C–O vibration[13] visible also in the PVA spectrum. Stretching vibrations at 1229, 1523, and 1633 cm$^{-1}$ were attributed to WAF’s amide III, amide II, and amide I, respectively.[20] The –OH vibration peak within WAF at approximately 3250 cm$^{-1}$ decreases significantly after PVA mixing, reflecting strong hydrogen bond interactions in the hybrid aerogel matrix.[13]

For efficient evaporation, long-term aerogel’s mechanical stability is essential. For this reason, the aerogel’s properties were characterized under compression and shear strain, (Figure S5, Supporting Information). The material resists against deformation up to over 50% of its initial height: the compression curves show limited local drops of compressive stress, confirming the mechanical resistance under high deformation. The extracted Young’s modulus of the hydrated aerogels is in the order of tens of kPa ($E \approx 30$ kPa); oscillatory sweep rheological experiments additionally confirmed the material’s elasticity (Figure 2d, Supporting Information), with the shear elastic modulus of the material ($G’$) one order of magnitude above the shear viscous modulus ($G''$), and both nearly frequency-independent.

Low density enables the hybrid aerogels to float on water (Figure S6, Supporting Information), while being exposed to the solar energy source. The upper black layer absorbs sunlight and confines the heat at the surface, enhancing the evaporation rate. The WAF aerogels’ absorption efficiency was determined by studying the optical properties of the PDA-coated fibrils within the solar spectrum range (0.25–2.5 μm) with a UV–Vis/near infrared spectrometer (Jasco V770) and an ILN-925 integrating sphere at near-normal incidence (see Supporting Information for further information). Figure 2a, Supporting Information shows the absorption spectrum and the global AM1.5 solar spectrum, with a mean absorption of 93.36%. As shown in Figure 2b, Supporting Information, most of the absorption losses stem from the impedance mismatch between the fibrils and air, resulting in an unavoidable, yet low reflectance of $R = 4.45\%$; the sample is fully opaque, as demonstrated in Figure 2c, Supporting Information (the visible ripples are associated with noise at higher wavelengths).
Low thermal conductivity of the amyloid aerogel minimizes conductive heat losses to the water bulk and enhances heat localization, allowing the efficient harvesting of solar heat for evaporation. Figure 2e shows the thermal conductivity (measured with a laser flash analyzer, \( N = 3 \)) for our aerogels in dry and wet states and compared to wood and water. The thermal conductivity increases in the wet and saturated aerogels as water acts as a filler, increasing conduction pathways.\[21\]

In the presence of the aerogel, the bulk water remains at constant room temperature while there is an evident temperature increase at the surface caused by heat localization, reaching \( \approx 32 \) °C; this is visualized as a graph in Figure 2f and as a map gradient in the infrared images of Figure 2g.

The heat localization effect was further investigated by visualizing the temperature distribution in a COMSOL model (for additional information, refer to Supporting Information). Figure 2h shows the results for the cross-section of the wet aerogel on the water surface and its steady-state temperature distribution, confirming a surface temperature of \( \approx 32 \) °C and negligible heat losses to the bulk water.

3. Evaporation and Desalination Performance

Next, the interfacial evaporation performance of the WAF aerogel was assessed. Figure 3a illustrates the comparison of time-dependent mass change of water under different conditions. The mass change of water was measured as 0.38 kg·m\(^{-2}\) for 1 h irradiation under 1 sun. After placing the aerogel on its surface, the evaporation rate quadrupled under the same conditions and reached 1.61 kg·m\(^{-2}\)·h\(^{-1}\). By applying two suns, the
evaporation rate doubled and increased to 3.32 kg·m⁻²·h⁻¹. The enhanced solar evaporation rate with amyloid hybrid aerogel is ascribed to the excellent heat confinement, well-integrated structure, in-time upward water convection, as well as lowered energy demand for water evaporation: part of the water molecules that enter the aerogel interact and bind with the abundant -OH groups of the water channels, increasing wetting ability; the resulting hydration layer leads to an energy reduction for water vapor formation in the aerogel network. The vaporization enthalpy of the water in the aerogel was estimated under dark conditions (for further information, consult Experimental Section) and compared with the bulk water's latent heat. As demonstrated in Figure 3b, the theoretical water vaporization enthalpy (2450 J g⁻¹) decreased by 20% (2042 J g⁻¹) after using the WAF aerogel, lowering the energy demand for evaporation. The energy efficiency (η) for solar-vapor conversion was calculated (for further information see Supporting Information) and shown in Figure 3c, amounting to a 91.3% evaporation efficiency for WAF aerogel under 1 sun, whereas without the aerogel evaporation efficiency drops down to 26.1%, confirming the enhanced evaporation rate enabled by the aerogel. The water harvesting performance of the hybrid amyloid evaporator is good and in the range of some other reported green evaporators (for further information see Table S1, Supporting Information). To evaluate the performance under real, daily conditions, a continuous 12 h illumination/12 h dark condition cycle was applied for 3 continuous days. As observed in Figure 3d, the evaporation rate was essentially constant within the 3 days, indicating excellent stability and durability.

The effect of salinity on evaporation rates was evaluated by distilling synthetic seawaters with salinities ranging from 0 to 450 g·kg⁻¹. As observed in Figure 3e, the evaporation rate remained above 1.5 kg·m⁻²·h⁻¹ until the salt saturation point at room temperature (350 g·kg⁻¹) was reached, demonstrating the generality of this technique for water desalination over a broad range of salinity.

A well-known complication that solar evaporators face is the surface accumulation of salt. However, during the long-time operation of the WAF aerogel, no salt was visible on the evaporation surface. This can be attributed to the evaporator’s well-forged water channels that allow the salt to efficiently diffuse back toward the bulk water from its surface. Considering that salt ions have been previously used to produce amyloid fibril hydrogels, their presence during desalination actually strengthens the structure of the wet WAF aerogel. Thus, the aerogel’s antifouling properties were further evaluated by adding salt crystals on the surface of the aerogel: its self-cleaning properties are observable in Figure 3f, where the salt crystals are transferred back to the bulk water within a time span of 1 h.

The desalination performance of the evaporator was assessed by comparing the concentrations of salt in bulk water and condensed vapor (Figure S9, Supporting Information). The solar-driven evaporator distilled synthetic seawater samples containing the four most abundant cations (Na⁺, Mg²⁺, K⁺, and Mg²⁺) found in seawater. The concentration for all four ions was successfully decreased below the threshold levels set by the World Health Organization (WHO) (Figure 4a). The concept was further validated by desalinating samples from Lake of Constance.
(low salinity), Red Sea (high salinity), and Siwa Salt Lakes (saturated). As observed in Figure 4b–d, regardless of salinity and the coexistence of other minor ions, the hybrid evaporators were able to make the water sources potable in each case.

In contrast to previous solar evaporation systems, the proposed aerogel machinery is affordable and scalable for large-scale manufacturing and applications. The scalability of the approach was demonstrated by producing an aerogel with a diameter of 20 cm (see Supporting Information and Figure S10a,b, Supporting Information). After hydration (Figure S10c, Supporting Information), the aerogel was applied for outdoor solar evaporation with actual solar illumination (Figure S10d, Supporting Information). Condensed droplets formed within 1 h evaporation time are shown in Figure S10e,f, Supporting Information.

The as-described WAF aerogel is equally efficient for purifying water from heavy metals, organic contaminants, bacteria, and even viruses. To demonstrate this, we first tested the evaporator’s ability for simultaneous removal of six heavy metals (mercury, zinc, platinum, cobalt, copper, and gold). As shown in Figure 4e, the concentration of heavy metals in the condensed vapor dropped by five orders of magnitudes, exhibiting removal efficiencies above 99.99%.

At lower or without energy input, WAF aerogels can remove heavy metals from the water via adsorption mechanisms (strong chelation binding affinity). These adsorption mechanisms were further studied by placing the aerogels in contaminated water samples with each of the six metals under dark conditions. After 24 h the metal adsorption capacity of the aerogel was calculated and summarized in Figure S11, Supporting Information.

Figure 4. Desalination and water purification performance with hybrid amyloid solar evaporators. Desalination performance of hybrid aerogel for a) synthetic seawater, b) Lake Constance water, c) Red Sea water and d) Siwa Salt Lakes water. e) Heavy metal removal performance. f) Dye removal performance (CV: crystal violet, AF: acid fuchsin, MB: methylene blue, MG: malachite green, RB: rhodamine B, CR: Congo red), g) E. coli bacteria removal performance. h) MS2 virus removal performance. LOD: limit of detection; * indicates measurements below the LOD.
Information; the aerogels exhibited excellent removal capacities particularly for gold, mercury, and platinum with adsorption capacities amounting to 535, 216, and 191 mg g\(^{-1}\), respectively. However, the major advantage of solar distillation is that under illumination the process does not rely on adsorption and can be used in a renewable cyclic way without adsorption-driven decrease in performance, making this concept virtually limitless in the amount of water that can be treated.

Similarly to the removal of heavy metals, evaporation experiments were also performed on water contaminated by organic molecules to evaluate organic pollutants removal efficiency. Cationic and anionic dyes, namely crystal violet, acid fuchsin, methylene blue, malachite green, rhodamine B, and Congo red were used. Figure 4f shows that no dye was detected in the vapor within the detection limits; here again, when illumination power decreases, the adsorption mechanism takes over,\(^{[20]}\) as shown by the aerogel’s change in color after contact with the dye solution (Figure S12, Supporting Information).

A universal water purifying system should be able to remove inorganic and organic contaminants from water, but ideally also biological threats.\(^{[30]}\) To this end, we also tested the evaporator’s capability in removing waterborne bacteria and viruses. Again, the performance of the aerogel evaporator was found outstanding: in Figure 4g the concentration of *Escherichia coli* (*E. coli*) decreased from \(\approx 10^9\) CFU mL\(^{-1}\) to below the detection limit; similarly, the concentration of the infectious MS2 virus decreased by at least six orders of magnitude (Figure 4h), also in this case below the detection limit. These results show convincingly that the water evaporation performed by WAF aerogels can be extended to microbiological contaminants, making it a universal concept in water purification.

4. Conclusions

In summary, we have shown that it is possible to produce clean drinking water from virtually any water source by using suitably designed amyloid aerogels to harvest solar energy and use this same energy to distill water. We have selected a protein byproduct from the dairy industry as a source for amyloid proteins and freeze-thawing cycles and freeze-drying as a process to endow the aerogel with the necessary porosity for efficient water transport. The aerogel maintains its stability over time and possesses self-cleaning properties, avoiding potential fouling from salt. The outstanding performance in water evaporation of the aerogels at moderate solar illumination from a multitude of salinity ranges and pollution contexts, combined with the inexpensive nature of the precursor materials makes this approach a valid candidate for scalable water production at virtually no cost and with no energy requirements, besides the freely available, renewable and unlimited solar energy.

5. Experimental Section

A full detailed description of the materials and methods used in this work is provided in Supporting Information. A summary is given below.

Materials: Whey protein isolate was provided from Fonterra and directly used for amyloid fibril preparation. Hydrochloric acid (36%) and PVA (fully hydrolyzed, \(M_w = 200,000\)) were obtained from Merck. Dopamine hydrochloride, Trizma base, all the dyes and the salts of all metals studied were purchased from Sigma Aldrich. MS2 bacteriophage (DSM no. 13767) and *E. coli* (DSM no. 5695) were acquired from DSMZ-German Collection of Microorganisms and Cell Cultures GmbH (Braunschweig, Germany). Natural waters with various salinities were collected from Lake Constance, Red Sea, and Siwa Salt Lake.

**Hybrid Amyloid Aerogel Preparation:** The schematic steps for the preparation of hybrid amyloid aerogel is shown in Figure 1b. Amyloid fibrils were prepared by denaturation and self-assembly of whey protein isolate (4 wt% in aqueous solution) at pH 2 and a temperature of 90 °C over a duration of 5 h.\(^{[29]}\) The amyloid fibril solution was dyed black by using a PDA (2 wt%) aqueous solution, as documented in previous protocols.\(^{[24]}\) To form hybrid hydrogels, the 4 wt% amyloid fibril solution was mixed with 10 wt% PVA solution with a fibrils to PVA mass ratio of 1:5. The double-layered hydrogels were made by casting the transparent hydrogel (WAF-PVA) in a disk-shaped mold, followed by casting the black hydrogel (WAF-PDA-PVA) on its top. In the next step, the hydrogels were unidirectionally frozen and thawed three times, to form the internal water channels within the hydrogel. Finally, the aerogels were prepared by lyophilization of the frozen hydrogels.

**Solar Desalination and Water Purification Experiments:** In order to execute the desalination experiments, the wet aerogels were placed on the water’s surface under different sun illuminations. For evaluating the performance of the evaporator for desalination under 1 sun, the chloride salts of sodium, potassium, magnesium, and calcium were dissolved in water, according to their actual concentrations in real seawater. Desalination of real water samples from Lake Constance, Red Sea, and Siwa Salt Lakes were performed by directly running the desalination experiments without any additional pretreatment. The evaporator’s heavy metals, organic pollutants, bacteria, and virus removal efficiency was evaluated by using a separated solution for each compound. The distillation removal performance was then calculated by comparing the concentrations of each individual contaminant in the condensed water vapor with respect to the bulk contaminated water. To demonstrate the synergistic aerogel adsorption capacity for every single metal and dye without applying a solar energy source, the aerogels were directly placed in pure solutions of each contaminant under dark conditions.

**Additional Methods Used:** Experimental details on hybrid amyloid aerogel preparation, solar desalination and water purification experiments, thermal conductivity measurements, and heat localization modeling are given in full in Supporting Information. Details on characterization, including microscopy and molecular (atomic force microscope, SEM, and FTIR), mechanical and optical, are also given in full in Supporting Information.

**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.

Data Availability Statement
Research data are not shared.

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