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Abstract: Sorption-based atmospheric water harvesting (SAWH) is a promising strategy to alleviate global water scarcity. However, the practical application of SAWH is hindered by the low water production efficiency of sorbent, especially in arid climate. This limitation results from sluggish sorption-desorption kinetics, a consequence of the long transport path required for vapor and liquid water due to sorbent stacking. Herein, we report a thin hygroscopic polyampholyte hydrogel layer on a solid porous substrate, which greatly mitigate stacking. The development dramatically improves the sorption-desorption kinetics, which allows 61 sorption-desorption cycles per day in an arid climate and results in 33.3 L kg⁻¹ theoretical freshwater collection within 24 h (30% RH). Besides, the strategy demonstrates remarkable scalability—even at a hundred-gram scale, the hydrogel layer can stably produce 1.16 L of water per day (10.5 L kg⁻¹ day⁻¹) in the dry, cold winters of central China (12.4 °C, 70.4% RH). Our work provides a promising solution to mitigate the severe water crisis in arid climate.

Only about 2.5% of the Earth's water is freshwater¹, and this limited resource is under increasing strain due to population growth, urbanization, and climate change, leading to severe water scarcity for approximately 4 billion people worldwide.^{2,3} There are great research interests in developing atmospheric water harvesting (AWH) technologies to alleviate global water scarcity, especially in arid climates⁴⁻⁶. Compared with traditional AWH technologies, such as fog collection and dew harvesting, sorption-based atmospheric water harvesting (SAWH) effectively captures water vapor in a broad RH range, which shows great applications potential⁷⁻⁹.

In SAWH, to address the disadvantages of conventional sorbents (e.g., low water vapor uptake, high energy for desorption, and deliquescence, etc.¹⁰), various functional sorbents were developed, such as metal-organic frameworks (MOFs)¹¹⁻¹³, covalent-organic frameworks (COFs)¹⁴⁻¹⁶, hygroscopic polymeric hydrogels (HPH)¹⁷⁻¹⁹, and their salt-based composites (e.g., LiCl)²⁰⁻²², etc. However, the daily water production efficiency of current sorbents remains insufficient, largely because sorbent stacking is unavoidable in prevalent designs that rely on porous structures or reduced particle size^{6,7}. Sorbent stacking significantly increases the internal transport distances for both vapor and liquid water. This, in turn, slows the sorption-desorption kinetics of sorbent and ultimately reduces the daily water production efficiency. To mitigate stacking, most state-of-the-art sorbents in SAWH system are therefore limited to a few grams or tens of grams²³⁻²⁸, which consequently caps daily water production at millilitres to tens of millilitres. While increasing the sorbent loading in a SAWH system is a straightforward way to boost water yield, it also exacerbates sorbent stacking. Consequently, the water collection efficiency drops to below 4 L kg⁻¹ day⁻¹²⁹⁻³⁴, detrimentally affecting the system's capital cost, weight, and footprint.

In recent years, hygroscopic salt-based hydrogels (HSBH) have emerged as promising AWH materials due to their high moisture uptake (e.g., 1.7 g g⁻¹) in arid climate, along with relatively high sorption-desorption kinetic, low leakage risk, as well as material cost, and excellent processability^{29, 35-39}. The daily water production of HSBH can be improved by morphological optimization (e.g., forming porous bulk gels³⁰, films²³, or microgels²⁵), which works through an enlarged vapor-sorbent contact area, reduced stacking, or a combination thereof. However, the relatively long vapor and liquid transport distance still greatly slows the sorption and desorption performance of porous bulk gels, as the thickness of porous sorbents is usually more than 100 μ m to maintain their mechanical structure²³. Although hydrogel microparticles with larger vapor-sorbent contact area and smaller sorbent size (e.g. 500 nm to 25 μ m) showed better kinetics, they are prone to stacking due to the electrostatic and hydrogen bonding interactions, which in turn increase the vapor and liquid transport distance^{24, 25}. These limitations reduce the sorption-desorption kinetics and thus reduce the daily water production. Furthermore, most current strategies for preparing high-performance HSBH rely on delicate porous structures, which makes the fabrication process time-consuming, equipment-intensive and capital-intensive. In summary, a key challenge lies in the concurrent reduction of sorbent size and suppression of stacking, as achieving both without compromising performance remains difficult. It is thus imperative to mitigate these disadvantages in order to develop SAWH system capable of achieving synergistic kinetic enhancement and meeting daily water collection requirements—advances that would pave the way for large-scale production.

Herein, we report a new HSBH and an associated protocol that addresses the dual challenge of reducing sorbent size and suppressing stacking. The implementation of this protocol led to significant enhancements in sorption-desorption kinetics and overall water production efficiency. We employed frontal polymerization (FP)⁴⁰ as a means to rapidly synthesize porous (poly (acrylic acid-*co*-2-acryloyloxyethyl trimethylammonium chloride) (P(AA-*co*-AETAC)) hydrogel on a kilogram scale with very low energy input, allowing for the large-scale and low-cost fabrication of HSBH. Hygroscopic polyampholyte hydrogel microparticles loaded with LiCl (denoted as

HPHMs) were prepared by swelling and fragmenting P(AA-co-AETAC) hydrogel in aqueous LiCl solution, yielding particles approximately 1 μm in size. Leveraging the strong electrostatic and hydrogen-bonding interactions of the functional group of P(AA-co-AETAC), the HPHMs exhibited self-healing, which facilitated their reassembly into a thin ($\sim 10\ \mu\text{m}$), adherent hydrogel layer on single-layer porous beds (e.g., nylon mesh) that did not block the pores. (Fig. 1a). This design effectively mitigated sorbent stacking, shortened the liquid transport distance, and expanded the water vapor contact area. Thus, the sorption kinetics were significantly increased compared with reported strategies (Fig. 1b). Besides, the ionic functional groups in polymers strongly interacted with LiCl to form a coordinate salt, which further improved the sorption kinetics and reduced the LiCl leakage risk. As a result, the material allowed 61 sorption-desorption cycles within 24 h. Typical HPHMs achieved 8.46, 16.65, and 33.3 L kg^{-1} theoretical freshwater collection at 15%, 20%, and 30% RH within 24 h, respectively. In practical applications, HPHMs were adhered to a multilayer sorption bed (Fig. 1c). The air and heat flow could move freely between sorption layers, which enables HPHMs to fast capture and release water vapor (Fig. 1d). In an actual indoor arid climate (21.8 $^{\circ}\text{C}$, 23% RH), in grams scale, HPHMs collected 44.60 mL (20.18 L $\text{kg}^{-1}\ \text{day}^{-1}$) clean water. Besides, a continuous 24-hour automatic SAWH device was developed for the evaluation of water production performance of hundred-gram-scale HPHMs under real outdoor arid climate (12.4 $^{\circ}\text{C}$, 70.4% RH), demonstrating a daily yield of 1162 mL of clean water (10.5 L $\text{kg}^{-1}\ \text{day}^{-1}$) (Fig. 1e). This means that only approximately 293 g of HPHMs are needed to meet the recommended daily water intake for an adult (3.07 L), as established by the U.S. National Academy of Medicine. To sum up, both in gram and hundred-gram levels, HPHMs outperformed state-of-the-art sorbents in water collection efficiency. (Fig. 1f and Supplementary Table 1).

Results

Synthesis of HPHMs

HPHMs were prepared through following steps: synthesis of copolymers, salt loading, mechanical fragmentation, and drying process (Fig. 2a). Typically, copolymers of poly (acrylic acid (AA)-co-2-acryloyloxyethyl trimethylammonium chloride (AETAC)) (P(AA_x-co-AETAC_y)) were synthesized via FP, with ammonium persulfate (APS) and methylene-bis-acrylamide (MBA) as initiator and crosslinker respectively (Supplementary Fig. 1). P(AA₁-co-AETAC₃) was applied for further studies due to the fast sorption kinetics, rapid FP synthesis (FP velocity was 11.2 cm min^{-1}), and high salt loading capacity (no leakage with 70 wt% LiCl under 70% RH) (Supplementary Fig. 3, 4, Supplementary Table 2, and Supplementary Note 1, respectively). Results of the self-perpetuating and exothermic property of P(AA₁-co-AETAC₃) during the FP process were shown in Fig. 2b, Fig. 2c, Supplementary Video 1 and 2, respectively, where the FP temperature was 144 $^{\circ}\text{C}$ (Supplementary Table 2). The heat released during the front propagation enabled the evaporation of water in AETAC, which resulted in porous polymers directly. Due to the large swelling ratio in DI water (54 g g^{-1}) and porous structure, P(AA₁-co-AETAC₃) could be easily broken into porous hydrogel pieces after drying (Fig. 2d). The SEM image suggested the porous and loose surface morphology (Fig. 2e).

In following steps, P(AA₁-co-AETAC₃) hydrogel pieces were broken into microparticles by mechanical fragmentation in LiCl aqueous solution (Fig. 2f). This mixed solution was then dried to produce HPHMs (Fig. 2g). To investigate the large-scale production ability, $\sim 1\ \text{kg}$ of P(AA₁-co-AETAC₃) was synthesized, which was used in preparation of around 31 kg of P(AA₁-co-AETAC₃)/LiCl mixed solution (Supplementary Fig. 5, and Supplementary Video 3 and 4). After drying, around 2.2 kg of HPHMs could be obtained.

Atmospheric water harvesting performance of HPHMs

We first measured the water vapor sorption capacity of HPHMs (LiCl mass fractions from 10 to 80 wt%). The water vapor sorption isothermal results indicated that pure P(AA₁-co-AETAC₃) only

captured 0.15 g g⁻¹ water vapor at 30% RH, while HPHMs captured more water vapor (0.24 g g⁻¹ to 1.43 g g⁻¹) with the increasing of LiCl amount (10-80 wt%) (Fig. 3a). To choose suitable LiCl content, the concentration of LiCl aqueous solution was optimized to be 4.2, 8.4, and 12.6 wt%, respectively. The mass fraction of P(AA₁-co-AETAC₃) in LiCl aqueous solution was 1: 20, 1: 30, 1: 40, and 1: 50 (w: w), respectively. In this step, nylon mesh was applied as a porous sorption bed to hold HPHMs (Fig. 3b). Samples of P(AA₁-co-AETAC₃) prepared in 4.2, 8.4, and 12.6 wt% LiCl aqueous solutions at polymer-to-solution mass ratios of 1: 30, 1: 50, and 1: 50 were labeled as HPHMs-1, HPHMs-2, and HPHMs-3, respectively. All samples exhibited high sorption rates. This result is attributed to the reduced stacking of the sorbent (Supplementary Fig. 6 and Supplementary Note 2), aided by the mesh substrate. The average water capacity of HPHMs-1, HPHMs-2, and HPHMs-3 was 0.91, 1.27, and 1.42 g g⁻¹ at 30% RH (Fig. 3c and Supplementary Fig. 7), corresponding to 13.6, 21.5, and 33.6 min to reach 80% saturated water uptake, respectively. So, the related calculated average sorption rate was 3.22, 2.84, and 2.03 L kg⁻¹ h⁻¹ for HPHMs-1, HPHMs-2, and HPHMs-3, respectively (Supplementary Fig. 7).

Next, we investigated the desorption performance of HPHMs. Fig. 3d indicated that the time to release over 75% of captured water from HPHMs-1, HPHMs-2, and HPHMs-3 was 4, 7, and 8 min, respectively. Besides, the time to release over 80% of captured water of HPHMs-1 and HPHMs-2 was 10 and 18 min, respectively, while HPHMs-3 failed to release 80% water within 20 min. The desorption performance of HPHMs-1, HPHMs-2, and HPHMs-3 at different temperatures (60, 80, and 90 °C) also showed the same trends (Supplementary Fig. 8). Related results indicated that all HPHMs samples failed to release 80% captured water when the desorption temperature was as low as 60 °C. These results highly agreed with the evaporation and melting behavior from differential scanning calorimetry (DSC) measurements (Supplementary Fig. 9 and Supplementary Note 3). As such, HPHMs-1 was selected in the following studies due to its fast sorption-desorption kinetics and promising water vapor capacity, and 70 °C was kept as the appropriate desorption temperature.

As mentioned previously, the stacking of hydrogel materials strongly hampers the performance of sorbents. To address this, nylon mesh was applied as a solid substrate to help distribute microparticles. HPHMs-1 could be firmly adhered to nylon mesh due to the charge stoichiometry between the polyions of P(AA₁-co-AETAC₃) towards cationic groups. In detailed investigations, nylon mesh (6.5 cm × 6.5 cm) was loaded with different amount of HPHMs-1, which were prepared through optimizing the number of dip coating-drying cycles (Supplementary Fig. 10a). In applications, for samples after 2 or 3 dip coating-drying cycles (refer to as HPHMs-1-2 and HPHMs-1-3), the mass of the HPHMs-1-2 and HPHMs-1-3 on nylon mesh was 25.94 g m⁻² and 52.18 g m⁻². Results indicated that the time to reach 80% saturation increased from 22.1 min (HPHMs-1-2 saturate water uptake of 0.93 g g⁻¹) to 43.7 min (HPHMs-1-3 saturate water uptake of 0.95 g g⁻¹) (Supplementary Fig. 10b), and the corresponding calculated sorption rate decreased from 2.0 to 1.0 L kg⁻¹ h⁻¹ (Supplementary Fig. 10a). In addition, the desorption time of HPHMs-1-2 to evaporate 80% of captured water was 16 min, while HPHMs-1-3 released 77% of captured water after 20 min at 70 °C (Supplementary Fig. 10b). Results from both SEM and optical microscope images suggested that the meshes of nylon mesh were gradually blocked with increasing mass of HPHMs (Supplementary Fig. 11). These results strongly indicated that the stacking inevitably increased the liquid transport distance and reduced the water vapor contact area, which decreased the sorption-desorption kinetics. As such, HPHMs-1 on nylon mesh (1 dip coating-drying cycle) exhibited promising effects and was applied in the following experiments.

Results from dynamic vapor sorption-desorption measurements suggested that HPHMs-1 captured 0.57, 0.75, 0.91, and 1.57 g g⁻¹ vapor water under 15%, 20%, 30%, and 60% RH, and took 36, 30, 13, and 16 min to reach 80% of its saturation, respectively (Fig. 3e). Then, 75%, 80%,

80%, and 80% of captured water were released within 16, 8, 10, and 14 min at 70 °C, respectively. The fast sorption-desorption kinetics enabled HPHMs-1 to achieve 27, 37, 61, and 48 cycles under 15%, 20%, 30%, and 60% RH, respectively. Therefore, the daily water collection of HPHMs-1 approached 8.46, 16.65, 33.3, and 45.21 L kg⁻¹ at 15%, 20%, 30%, and 60% RH, respectively. The efficiency is among the best state-of-the-art sorbents (Fig. 3f, Fig. 3g, and Supplementary Table 3). In addition, stability measurements indicated that HPHMs-1 maintained stable water capacity and sorption performance after 61 sorption-desorption cycles (Fig. 3h). Both P(AA₁-co-AETAC₃) and HPHMs-1 were stable when the temperature approached 200 °C (Supplementary Fig. 12).

In further investigations, results from scanning electron microscopy (SEM) (Fig. 4a) studies indicated that HPHMs-1 could self-heal after being dried and adhered to the surface of nylon mesh. The self-healing property could be attributed to the abundant electrostatic interactions and hydrogen bonding between quaternary amine and carboxyl of the HPHMs-1 (Supplementary Fig. 13). The quaternary amine and carboxyl could form strong electrostatic interactions and hydrogen bonding with the polar group (-OH, -COOH, -NH₂ and -CO-NH-) on the surface of nylon mesh, which resulted in the strong adhesion. Due to the firm adhesion of the hydrogel to the nylon mesh and the absence of blockages, moisture was able to flow freely. This promoted an effective expansion of the water vapor contact area. In this design, the ultrathin layer of HPHMs-1 (around 10 μm) adhered to a thin nylon mesh (around 170 μm) greatly reduced the liquid transport distance (Fig. 4b), thus strongly promoting the sorption kinetics. So, the dry HPHMs-1 could rapidly capture moisture within 5 min and reached saturation after 10 min. Meanwhile, no blocking happened when the HPHMs-1 was saturated (Fig. 4c). In addition, we need to point out that the nylon mesh only served as a sorption bed instead of a LiCl holder. This was evidenced by related energy-dispersive X-ray spectroscopy (EDX) results, which indicated that most of the LiCl was loaded into HPHMs-1 (Fig. 4d), because the affinity of LiCl for P(AA₁-co-AETAC₃) was much stronger than that of the nylon mesh.

In the AWH process, there is no crystal and agglomeration of LiCl salt in P(AA₁-co-AETAC₃) (Fig. 4a and Fig. 4d), which not only avoids the reduction of water vapor contact area, but also shortens the liquid transport distance in HPHMs-1. Besides, X-ray diffraction (XRD) pattern also indicated the absence of characteristic peaks of LiCl in HPHMs-1, suggesting that P(AA₁-co-AETAC₃) could form strong coordinate salt with LiCl (Fig. 4e). This resulted in the rapid sorption-desorption kinetics as mentioned (Fig. 3e). We hypothesize that this was attributed to the cationic and anionic functional groups in P(AA₁-co-AETAC₃), which interacted with LiCl salt to form coordinate salt instead of free salt⁴¹. FTIR spectra showed that the antisymmetric and symmetric stretching vibration of N-(CH₃)₃ shifted from 3021 and 2961 cm⁻¹ to 3028 and 2966 cm⁻¹, respectively, which was due to the interaction between the quaternary amine with LiCl (Fig. 4f). Furthermore, the antisymmetric stretching vibration absorption peak of COO⁻ was significantly enhanced, widened and shifted from 1645 to 1634 cm⁻¹, which suggested the transition of carboxyl ionizes to carboxylate after interacted with LiCl. Theoretically, Li⁺ is more prone to bind to COO⁻ instead of Cl⁻ due to the lower binding energy of COO⁻ Li⁺ (-30.12 Kcal mol⁻¹, compared with Li⁺ Cl⁻ (-27.11 Kcal mol⁻¹)⁴². The binding energy of N⁺(CH₃)₃Cl⁻ (-5.08 Kcal mol⁻¹) is higher than COO⁻ N⁺(CH₃)₃ (-4.58 Kcal mol⁻¹) in H₂O systems, which help to form coordinate salt⁴². As reported, free salt moves faster than coordinate salt in hydrogels because of the absence of electrostatic interaction^{43 44}. Therefore, free salt should be more prone to agglomerate compared with coordinate salt. In the HPHMs-1 system, the abundant coordinate salt stretches the polymer chains to rule out salt agglomeration. As such, P(AA₁-co-AETAC₃) could strongly interact with LiCl salt to form plenty of coordinate salt rather than free salt. The high AWH performance of HPHMs-1 could be attributed to the synergistic contribution from enlarged water-vapor contact area, free of LiCl agglomeration in P(AA₁-co-AETAC₃), and reduced vapor and liquid transport

distance.

Demonstration of the water collection performance of HPHMs

To investigate the water collection performance of HPHMs, we developed a test device consisting of a heating chamber, sorption bed, vacuum pump, condenser tube, and water collecting bottle (more details see Supplementary Note 4). The water collection efficiency of the test device was 84% and 73% at temperatures of 20 °C and 25 °C, respectively (Supplementary Fig. 15a and Methods), which is high enough to test the water collection performance of HPHMs. Results from dynamic vapor sorption measurements indicated that the HPHMs-1 sorption bed (containing ~ 2.21 g HPHMs-1) captured 0.59, 0.79, and 0.97 g of water vapor under 15%, 20%, and 30% RH, and took 20, 10, and 8 min to reach 80% saturation (Fig. 5a), respectively. The calculated sorption rates of HPHMs-1 for 80% water uptake at 15%, 20%, and 30% RH were 1.41, 3.79, and 5.82 L kg⁻¹ h⁻¹, respectively. Meanwhile, the temperature of the PTC heater and desorption time were set at 120 °C and 20 min to efficiently release the captured water (Fig. 5b, Supplementary Fig. 15b, and 15c). Owing to the enthalpy of evaporation of water, the temperature of HPHMs-1 sorption bed gradually increased from 25 to 70 °C within the first 8 min and finally reached 90 °C after 20 min (Supplementary Fig. 15d). As a result, 0.24, 0.41, and 0.65 g g⁻¹ water was collected at 15, 20, and 30% RH, respectively, after 20 min (Fig. 5b). This indicated that 50.00%, 64.87%, and 83.76% of captured water were collected at 15, 20, and 30% RH, respectively. Therefore, the corresponding daily number of sorption-desorption cycles was 36, 48, and 51, respectively. Results of 24 h continuous cycling experiments suggested that the daily water collection was 19.84 g (8.97 L kg⁻¹), 40.61 g (18.37 L kg⁻¹), and 60.89 g (27.55 L kg⁻¹) at 15, 20, and 30% RH, respectively (Fig. 5c).

We further measured the 24 h water collection performance of ~2.21 g HPHMs-1 under indoor arid environments. The sorption and desorption times were set to 10 and 20 min, respectively. Results (Fig. 5d and Supplementary Fig. 16) indicated that HPHMs-1 consistently excelled in an actual arid scenario, which collected 44.60 g (20.18 L kg⁻¹) water at 18.7% - 27% RH, 18.3 - 30.2 °C (Fig. 5e). The collected water under the low RH scenario (RH ≤ 30%) was shown in Fig. 5f. Quality measurements suggested that concentrations of most elements and ions in the collected water were far below the standard drinking water quality (Supplementary Table 4 and Supplementary Table 5). In particular, the concentration of Li⁺ ion was only 0.016 ppm, which is far below the Li⁺ safety standard (0.06 ppm) of drinking water from the USGS and US EPA.

Outdoor water collection of HPHMs

To demonstrate that the HPHMs could meet the practical applications, we designed a 24-hour automated continuous operating SAWH device, which loaded ~110 g of HPHMs. The device is composed of a heating module, a vacuum pump, two 3D sorption bed modules, a transmission module, a condenser, a water collector, cooling fans, and an electrical control module (Fig. 6a, more details see Supplementary Note 5). Fig. 6b presents the water collection principle of the device. Driven by the vacuum pump, the water vapor released by HPHMs in the heating module flows into the condenser, which rapidly cools into liquid water within the condenser (spiral aluminum tube) and flows into the water collector (more details see Supplementary Note 5). Moreover, the outlet of the water collector is connected to the inlet of the pump, and the outlet of the pump is connected to the heating chamber to return the not fully condensed water vapor for condensing during the next cycle. The sorption-desorption time was set to 30 min (Supplementary Fig. 18, Supplementary Note 5), Fig. 6c indicated that the daily water collection of the device was 1162 mL within 24 h under 9 - 20 °C, 45 - 85.9% RH (the average temperature and relative humidity was 12.4 °C and 70.4% RH respectively, which is equivalent to 25 °C, 32% RH) (Fig. 6d and Supplementary Video 7). These results suggested promising practical water collection applications.

Summary

We demonstrated the synthesis and application of functional materials that concurrently reduces sorbent size, mitigates sorbent stacking, and enables large-scale scalability in sorbent design. The thin hydrogel layer demonstrated exceptional sorption-desorption kinetics and water production efficiency at both the gram and hundred-gram scales under practical arid climate.

Methods

Chemicals and Materials

Acrylic acid (AA, 95%), acryloyloxyethyl trimethyl ammonium chloride (AETAC-80, 80 wt% in water), ammonium persulfate (APS, $\geq 98\%$), N, N'-Methylenebisacrylamide (MBA, 99%), ethanol (ET, $> 99.5\%$), and lithium chloride (LiCl, 99.0%) were purchased from Aladdin shanghai Co. Ltd. Deionized water was obtained by a Milli-Q Gradient water system (Millipore). All other chemicals were used without further purification.

Frontal polymerization of poly (acrylic acid-co-2-acryloyloxyethyl trimethylammonium chloride) (P(AA_x-co-AETAC_y))

Frontal polymerization was used in the synthesis of P(AA_x-co-AETAC_y). Typically, MBA (0.019 g, 0.3 wt%) was dissolved in AA (0.7 g, 1 mol) at room temperature and mixed with AETAC-80 (7.0546 g, 3 mol), followed by adding APS aqueous solution (containing 0.0634 g APS, 1 wt%, 130 μ L). The as-prepared mixed solution was then transferred into a glass tube (diameter = 15 mm, length = 150 mm) and initiated with a solder iron (300 $^{\circ}$ C), which was against the glass tube wall of the liquid surface of the mixed solution until the polymerization started. The obtained P(AA₁-co-AETAC₃) was soaked in DI water for 48 h to swell, followed by washing with ethanol five times to remove the unreacted residual. The P(AA₁-co-AETAC₃) was dried in a vacuum drying chamber at 90 $^{\circ}$ C overnight to offer light-yellow porous particles.

Preparation of the mixed solution of P(AA₁-co-AETAC₃) and LiCl aqueous solution

P(AA₁-co-AETAC₃) was immersed in 4.2, 8.4, and 12.6 wt% LiCl aqueous solution (LiCl (H₂O)) at room temperature for 24 h, respectively. The mass fraction of P(AA₁-co-AETAC₃) and LiCl (H₂O) was 1: 20, 1: 30, 1: 40, and 1: 50 (w: w), respectively. Next, the swollen P(AA₁-co-AETAC₃) in LiCl (H₂O) was broken by mechanical fragmentation, which offered the mixed solution of P(AA₁-co-AETAC₃) microparticles and LiCl (H₂O). Finally, the as-prepared mixed solution was placed for another 24 h to enable P(AA₁-co-AETAC₃) microparticles to fully swell. For example, the mixed solution of P(AA₁-co-AETAC₃) and 4.2 wt% LiCl (H₂O) at mass fraction of 1: 30 (w: w) was prepared as follows: 1 g P(AA₁-co-AETAC₃) was immersed into 30 g 4.2 wt% LiCl (H₂O) for 24 h, then the P(AA₁-co-AETAC₃) was broken into microparticles by mechanical crusher at rpm of 8000 for 3 times, each time kept at least 1 minute.

Frontal polymerization of P(AA₁-co-AETAC₃) at kg scale

3.23 g MBA was first dissolved in 119.13 g AA at room temperature and mixed with 1200 g AETAC-80, followed by adding 20 g APS aqueous solution (containing 10.79 g APS). The well-mixed precursor solution was poured into a 50 $\times$ 35 $\times$ 10 cm³ stainless steel basin and initiated with a solder iron (500 $^{\circ}$ C), which was against the basin wall of the liquid surface of the mixed solution until the polymerization started. The obtained P(AA₁-co-AETAC₃) bulk was soaked in 90 wt% ethanol aqueous solution for 48 h, and then washed with ethanol five times to remove the unreacted residual. The dried $\sim$ 1 kg P(AA₁-co-AETAC₃) was immersed in 30 kg 4.2 wt% LiCl aqueous solution for 48 h, then broken into microparticles by mechanical fragmentation.

Preparation of HPHMs

In a typical procedure, the mixed solution of P(AA₁-co-AETAC₃) and LiCl (H₂O) was stabilized by a homogenizer. Next, a 6.5 cm $\times$ 6.5 cm 200 mesh nylon mesh was immersed in a 200 g P(AA₁-co-AETAC₃) and LiCl (H₂O) mixed solution and carefully pulled out. Finally, nylon

mesh was dried in a drying oven at 110 °C for 30 min to remove all residual water, which offered white HPHMs adhering to nylon mesh.

Characterization of P(AA_x-co-AETAC_y) and HPHMs

A scanning electron microscope (SEM) was used for microstructural investigation and conducted on (ZEISS, Gemini 500). Constituent elements of the samples were analyzed by energy dispersive spectroscopy (EDS, Oxford, X-Max). The samples were vacuum-dried overnight before taking SEM images. The FTIR spectra were obtained by the FTIR Spectrometer (ThermoFisher Scientific, Thermo Nicolet 6700). The TG analysis was conducted by the thermogravimetric analyzer (NETZSCH, TG209F1) with an airflow rate of 50 mL min⁻¹, and a heating rate of 10 K min⁻¹. The XRD pattern was recorded by an X-ray diffractometer (Rigaku, SmartLab). The evaporation and melting behaviors of HPHMs were observed by a differential scanning calorimeter (TA instrument, DSC214). The test atmosphere was nitrogen with a flow rate of 50 mL min⁻¹, and the ramping rates were 2 K min⁻¹ and 5 K min⁻¹ for evaporation and melting, respectively. The optical microscopic images of HPHMs on nylon mesh were captured by a metallurgical microscope (MV3000, NOVEL).

Measurements of frontal polymerization

Frontal temperature was measured by a K-type thermocouple that was inserted into the center of a reaction tube. A digital camera and thermal infrared camera (CEM, DT9887) were used to track the front propagation; besides, frontal velocity was recorded as a function of frontal position and time.

Calculation of the swelling ratio

The swelling ratio of P(AA_x-co-AETAC_y) hydrogel in LiCl aqueous solution is calculated by the following Equation:

$$\text{Swelling ratio} = \frac{m_1 - m_0}{m_0}$$

where, m_0 (g) is the original weight of the dried P(AA_x-co-AETAC_y) hydrogel, m_1 (g) is the weight of the hydrate P(AA_x-co-AETAC_y) hydrogel after immersing in DI water for 48 h. Typical procedure is as follows: 0.3 g P(AA_x-co-AETAC_y) is immersed in 50 mL DI water and is named as P(AA_x-co-AETAC_y)-0 wt% LiCl. Air-laid paper is used to carefully adsorb the residual solution on the surface of P(AA_x-co-AETAC_y) hydrogel, and then it is weighed.

Leakage measurements of P(AA₁-co-AETAC₃)

In a typical procedure, 0.7 g LiCl was first dissolved in 3 g H₂O to prepare a LiCl aqueous solution, followed by immersing 0.3 g P(AA₁-co-AETAC₃) into the LiCl aqueous solution for 48 h to ensure full swelling. Then dried in a 110°C drying oven for 12 h, which offered dehydrated P(AA₁-co-AETAC₃)-70% LiCl. Next, P(AA₁-co-AETAC₃)-70% LiCl was placed in an electric constant temperature and humidity chamber (25 °C and 70% RH) for 48 h to reach sorption equilibrium. The hydrated P(AA₁-co-AETAC₃) was first gently wiped with air-laid paper to remove the free water on the hydrogel surface, and was placed on another new air-laid paper to observe the leakage.

Measurements of sorption kinetics

P(AA₁-co-AETAC₃)-50% LiCl is taken as an example. The typical procedure is as follows: P(AA₁-co-AETAC₃) was ground into powder to reduce the influence of porosity on the sorption kinetics. 0.15 g P(AA₁-co-AETAC₃) was mixed with 4 g LiCl aqueous solution (containing 0.15

g LiCl) in a glass petri dish (diameter is 3.5 cm) for 48 h. After that, the petri dish was heated in a 90 °C drying oven for 12 h to remove most of the water, which offered P(AA₁-co-AETAC₃)-50% LiCl. P(AA₁-co-AETAC₃)-50% LiCl was placed in an electric constant temperature and humidity chamber (25 °C and 70% RH) for 48 h to ensure LiCl in P(AA₁-co-AETAC₃) was uniformly distributed. Then, P(AA₁-co-AETAC₃)-50% LiCl was totally dried in 110 °C drying oven for 12 h, following by test its sorption kinetics under 25 °C and 30% RH in homemade RH and temperature-controlled system with temperature and RH accuracy of ± 0.2 °C and ± 1% RH, which composed of acrylic chamber, weather meter (Kestrel Meters, kestrel 5500), and analytical balance (Mettler Toledo, ME104, 0.1 mg). The RH in the chamber was stabilized by a specific supersaturated salt solution (Supplementary Fig. 2). A supersaturated solution of LiCl was used to stabilize the RH at ~15% and ~20%, and CH₃CO₂K was used to stabilize the ~30% RH²³. The environmental temperature was controlled by an air conditioning system. A weather meter was used to monitor the RH and temperature of the chamber. The mass change of sorbent was measured by an analytical balance and recorded on a laptop.

Measurements of sorption and desorption performance of HPHMs

The water vapor sorption isotherm performance was measured by a dynamic vapor sorption (DVS) system (BSD-DVS, DSD Instrument). The sorption-desorption performance was conducted in a homemade RH and temperature-controlled system. Before the sorption test, samples were pre-dried on a hot plate at 110 °C for 30 min, and then quickly placed on an analytical balance. In addition, to diminish the influence of moisture uptake of nylon mesh, the sorption performance of pristine nylon mesh was recoded as background data before coating and subtracted. For the desorption test, the sample was put on a hot plate at 60, 70, 80, and 90 °C, respectively, and the weight changes were recorded.

Measurements of the water collection efficiency of the AWH device

A Teflon disk containing 1 g of deionized water was put into the heating chamber, whose PTC heaters' temperatures were 110 °C, until it was completely dried. Then it was collected and weighed. The water collection efficiency is calculated by dividing 1 g of water by the weight of the collected water.

Measurements of the AWH performance of the HPHMs system

For the SAWH system, the standard sorption performance (in specific RH and temperature) of the sorption bed was conducted in a homemade RH and temperature-controlled system. Forced convection (wind speed at 4.47 m s⁻¹) was applied by a 3 W electric fan to provide sufficient moisture. The weight change of the sorption bed was recorded using an analytical balance. For practical experiments, the surrounding RH and temperature were recorded by a weather meter (Kestrel Meters, kestrel 5500). To precisely weigh the collected water of each cycle during the cycling collection experiment, the silicon tube was squeezed, which enabled the desorbed water vapor condensed in the silicon tube to flow into a glass bottle (60 mL) with the air flow driven by a vacuum pump. The collected water was weighed by an analytical balance (Mettler Toledo, ME104, 0.1 mg). The concentrations of ions and elements in collected water were tracked by ion chromatography (Dionex ICS-5000, Thermo Fisher Scientific) and inductively coupled plasma mass spectrometry (Agilent, 7850 ICP-MS). For a 24 h automated SAWH system, the mass change of the collected water was tracked by an analytical balance (0.01 g) connected to a laptop. The ambient RH and temperature were recorded by the weather meter. The 24 h water collection process was recorded by a digital camera (DJI, Osmo Action 4). The power of the SAWH device was recorded by a power analyzer (AWS2103, Aitek) (Supplementary Fig. 19).

The grams scale 24 h continuous practical indoor water collection experiment and hundreds-gram scale automated SAWH device were conducted in the south campus of Hefei University of Technology, Hefei, China (117.296° N, 31.843° E) in January 2025 and December 2025 respectively.

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Author contributions:

T.H. and H.H.H. supervised the project. T.H. and W.C.X. conceived and designed the experiments. T.H., W.C.X. and R.J. wrote the original draft. W.C.X., W.W.F., L.X.F and B.X. prepared and characterized the materials. R.J. designed the AWH device, R.J., L.L., S.Y.J. and Y.Y.W. manufactured the AWH device. Z.H.F., Y.F.L., Y.J.K. and Z.H.X. assisted in experiments. L.X., L.X.T., L.J.C., S.F., L.P., Y.J.R., Z.X.Y. and L.J.P. commented on the paper. T.H., H.H.H., W.C.X., R.J., Y.L. and J.J. reviewed and edited the draft.

Competing interests:

Authors declare that they have no competing interests.

Data and materials availability:

All data are available in the main text or the supplementary materials.

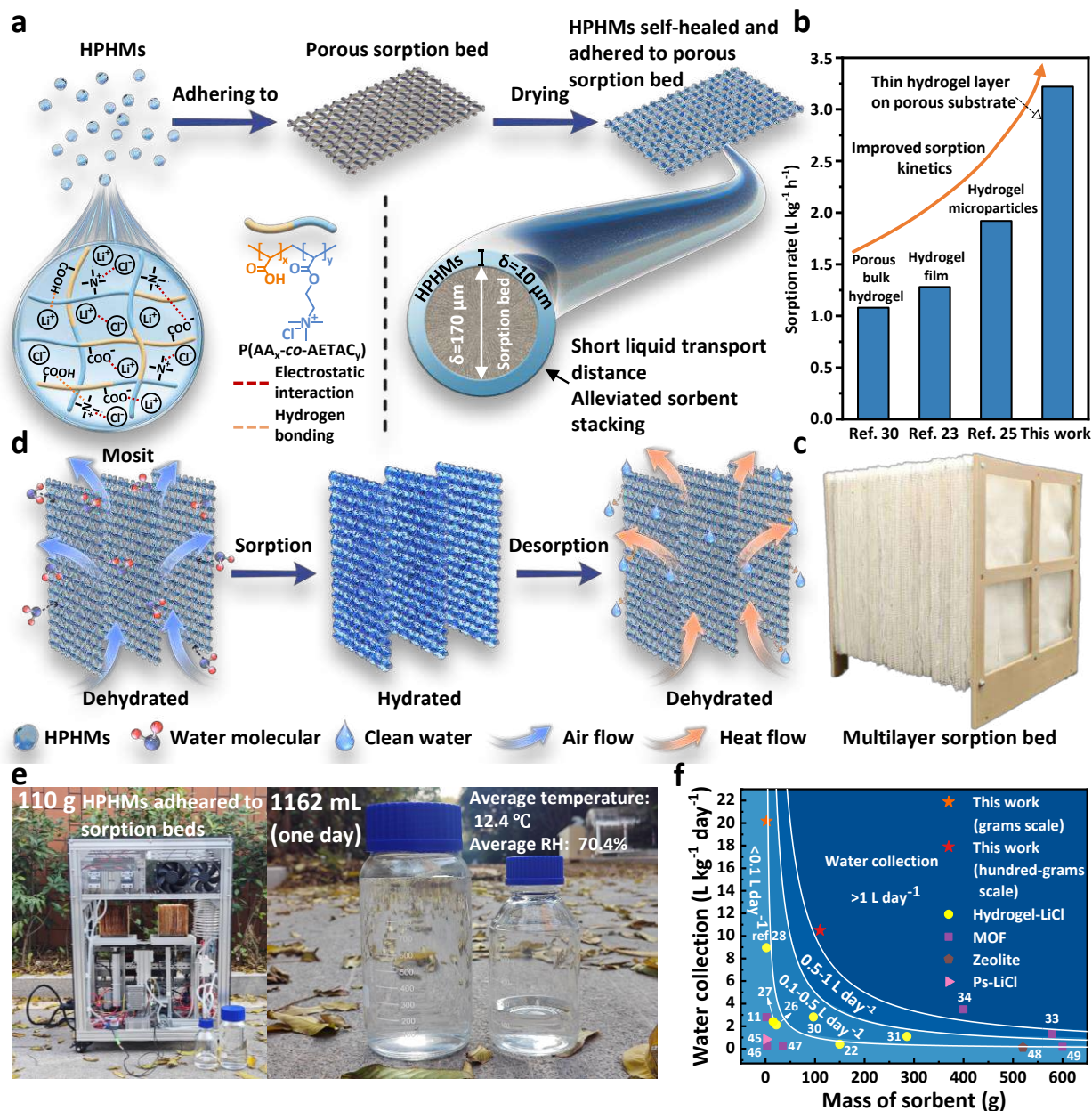


Fig. 1 Development of air water harvesting of HPHMs system. (a) Schematic of the fabrication process of thin HPHMs hydrogel layer adhered to porous sorption bed. (b) comparison of sorption kinetics of previous strategies with this strategy. (c) photo of multilayer sorption bed adhered with HPHMs. (d) the sorption-desorption process of HPHMs adhered to multilayer sorption bed. (e) photos of automated SAWH device loaded with ~110 g HPHMs and the collected water within 24 h. (f) comparison of practical AWH efficiency among HPHMs and state-of-the-art sorbents.

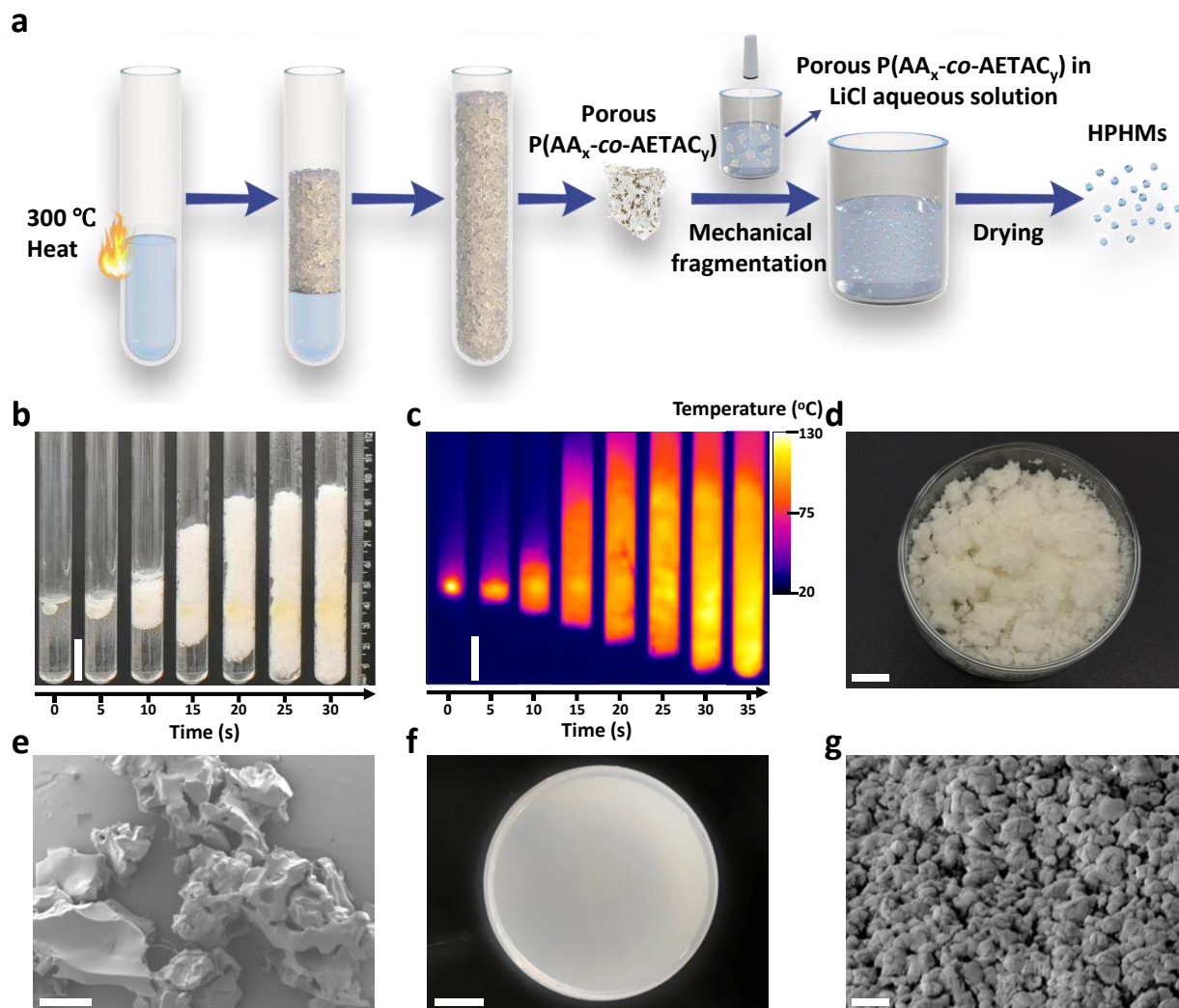


Fig. 2 Preparation of HPHMs. (a) Schematic diagram of the fabrication process of HPHMs. (b) Photo and (c) thermal infrared image of the FP process of P(AA₁-co-AETAC₃) (scale bar: 2 cm). (d) Photo of P(AA₁-co-AETAC₃) (scale bar: 1 cm). (e) SEM image of P(AA₁-co-AETAC₃) (scale bar: 500 μm). (f) Photo of the mixed solution of P(AA₁-co-AETAC₃) microparticles and LiCl aqueous solution (scale bar: 2 cm). (g) SEM image of HPHMs (scale bar: 2 μm).

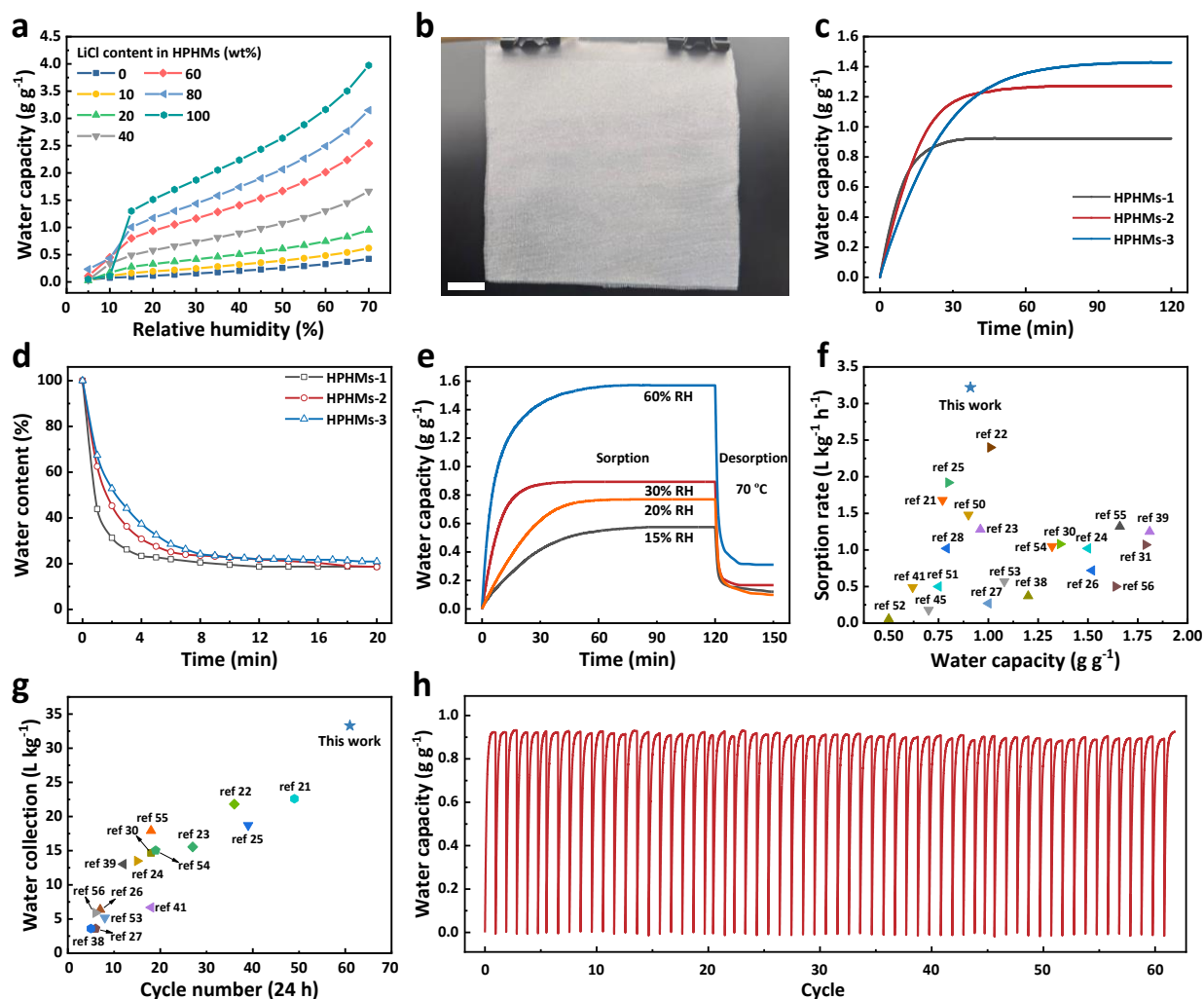


Fig. 3 Water vapor sorption-desorption performance of HPHMs. (a) Water vapor sorption isotherm of HPHMs at 25 °C. (b) Photo of HPHMs-1 being adhered to nylon mesh (scale bar: 1 cm). (c) Water vapor sorption curves of HPHMs-1 to 3. (d) Desorption curves of HPHMs-1 to 3 at 60 °C to 90 °C respectively. (e) Dynamic water vapor sorption-desorption performances of HPHMs at 15%, 20%, 30% and 60% RH, 25 °C respectively. (f) Comparison of water vapor sorption rate and capacity with the state-of-the-art LiCl salt-based sorbents at 30% RH. (g) Comparison of sorption-desorption cycle number and water collection in 24 h with the state-of-the-art LiCl salt-based sorbents at 30% RH. (h) Cycle stability performance of HPHMs-1.

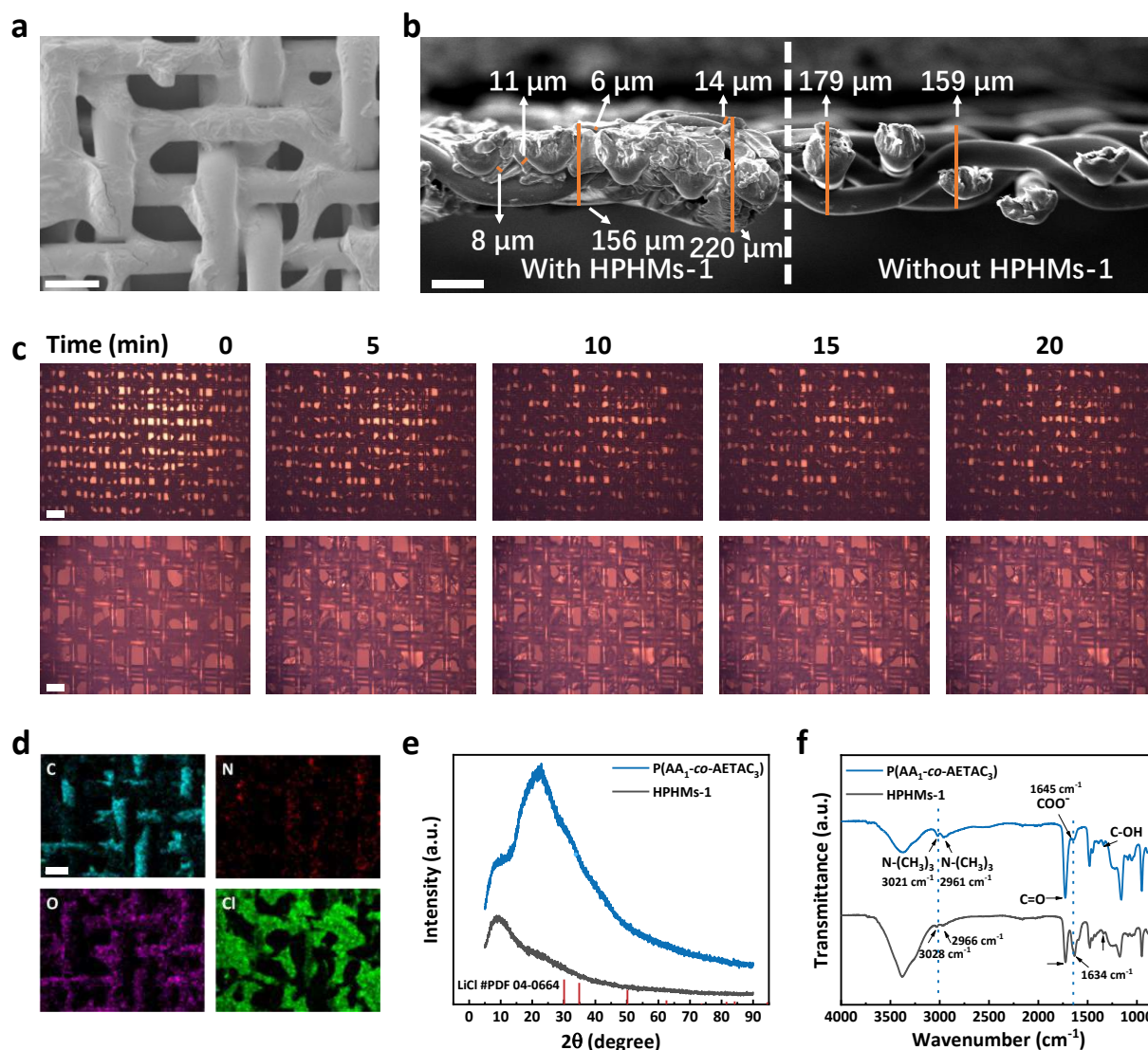


Fig. 4 AWH performance of HPHMs-1. (a) SEM image of HPHMs-1 being adhered to nylon mesh (scale bar: 100 μm). (b) SEM image of the thickness of pristine nylon mesh and nylon mesh adhered with HPHMs-1 (scale bar: 100 μm). (c) Optical microscope images of the water sorption process of HPHMs-1 (scale bar: 300 μm (up row), 100 μm (down row), test environment: 27 $^{\circ}\text{C}$, 40% RH). (d) EDX mapping of HPHMs-1 being adhered to nylon mesh (scale bar: 100 μm). (e) XRD pattern and (f) FTIR spectrum of P(AA₁-co-AETAC₃) and HPHMs-1.

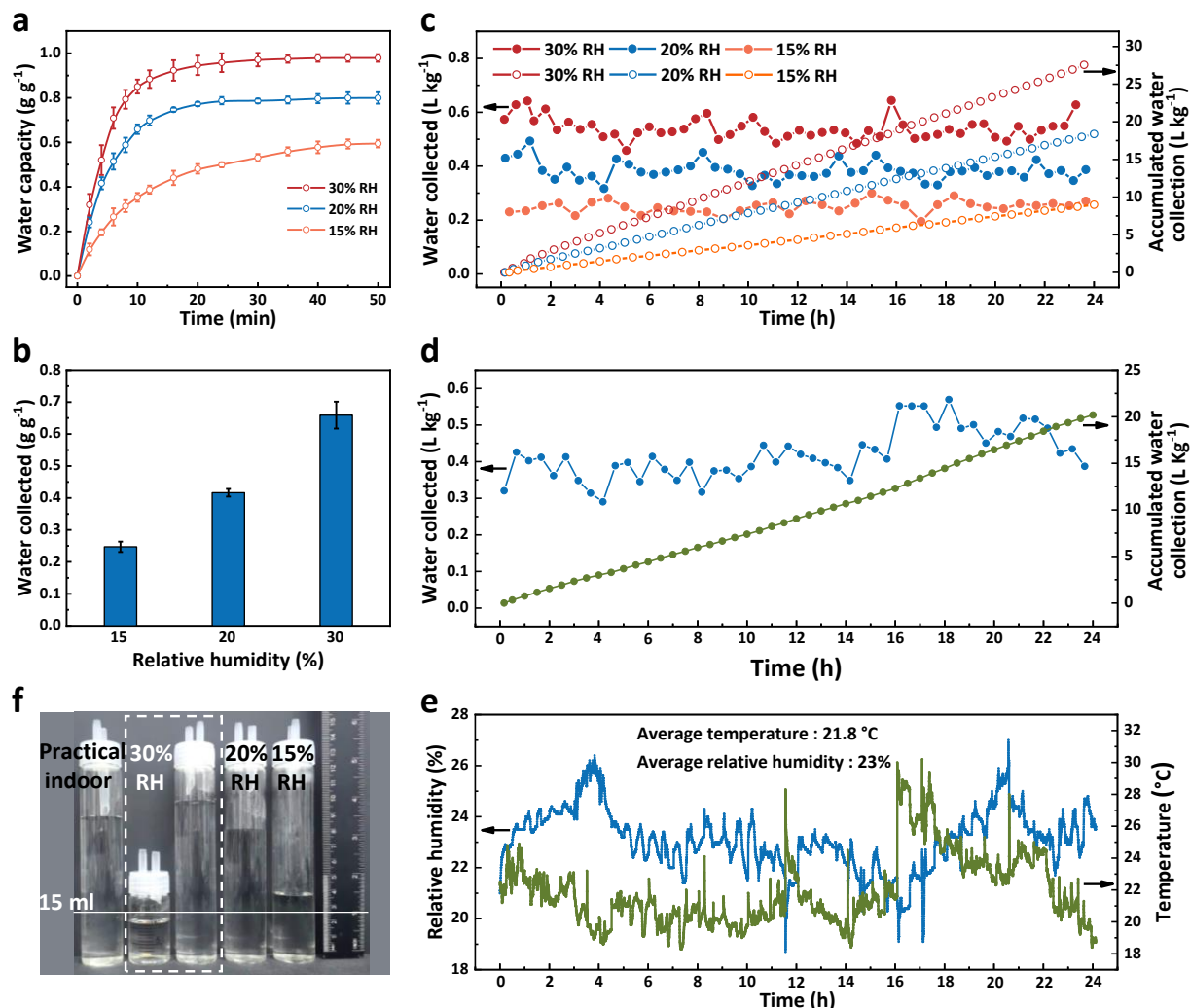


Fig. 5 Water collection of the grams scale HPHMs-1. Schematic diagram of the structure of (a) sorption kinetics curves of HPHMs-1 adhered to sorption bed. (b) water collection and (c) 24 h cycling water collection of HPHMs-1 adhered to sorption bed at different relative humidity. (d) practical water collection of HPHMs-1 adhered to sorption bed in arid environment. (e) temperature and relative humidity of practical environment. (f) photo of collected water at different relative humidity.

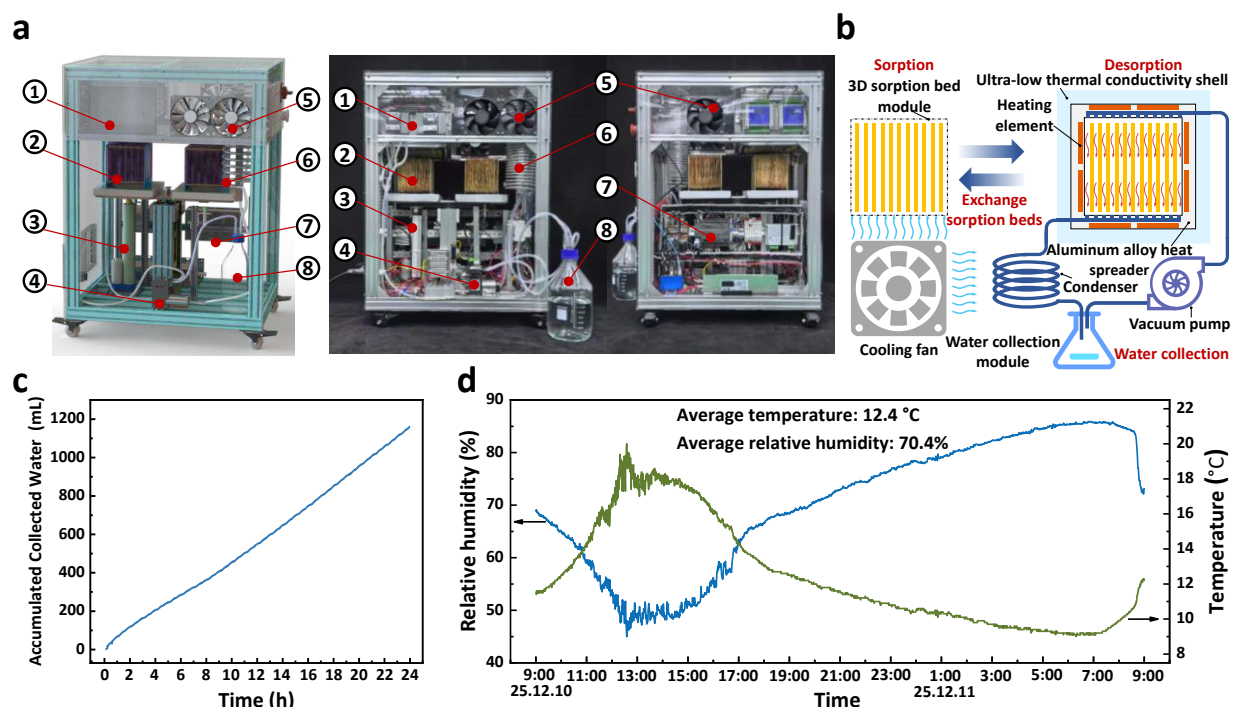


Fig. 6 Water collection of the hundred-grams scale HPHMs. (a) 3D model and photos of the automated air-efficient water harvesting device; ①: Heating module, ②: Three-dimensional moisture-absorbing module, ③: Transmission module, ④: Vacuum pump, ⑤: Cooling fan, ⑥: Condenser, ⑦: Electrical control module, ⑧: Water collector. (b) schematic illustration of the process of automated water collection. (c) 24 h continuous cycling practical water collection of HPHMs. (d) temperature and relative humidity of practical environment.

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