Supporting Information

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Ultralight and Flexible Monolithic Polymer Aerogel with Extraordinary Thermal Insulation by A Facile Ambient Process

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Synthesis of Polyvinyl Chloride (PVC) Aerogel

First, the reaction sources, i.e. PVC powder (389293, Sigma-Aldrich, USA) is mixed with dimethylformamide (DMF) (DX1727, EMD Millipore, Germany) with ratio from 0.2 g/10 mL to 1 g/10 mL at temperature of 60 °C. The different mass to volume ratio of PVC to DMF is used to control the porosity of aerogel. Then, the PVC solution is sonicated for an hour and exposed to air for 12 hours. Consequently, with the water vapor is absorbed into DMF/PVC solution, the solubility of PVC decreases gradually resulting in the precipitation of polymer particles. Finally, a white and jelly-like PVC solid was formed. To remove the liquid from the PVC gel while avoiding shrinkage of the porous structure, the solvent is exchanged with ethanol, which has a small surface tension. In the solvent exchange process, the volume percentage of ethanol in the solvent is gradually increased. In the solvent exchange process, the volume percentage of ethanol in the solvent is gradually increased. After the gelation is finished, ethanol is added into the solution to increase the volume percentage of ethanol to 25%. After 6 hours we extract some liquids from the solution and add more ethanol into the solution to improve the volume percentage of ethanol to 50%. This process is repeated 5 times with a time interval of 6 hours, and the eventual ethanol concentration increases from 0 to 93.8%. Following aged for twelve hours, the gel is exposed to ambient environment. After the solvent is completely evaporated, the solid PVC aerogel is formed.
Thermal Stability of PVC Aerogel

The thermal stability of PVC aerogel is investigated using Thermogravimetric analysis (TGA) up to 700 °C (Figure S1). The measurement shows a two-stage weight loss process, including a major weight loss step (around 280 ~ 335 °C) and a secondary weight loss step (around 450 ~ 475 °C). The first step is attributed to the emission of hydrogen chloride, and the second step is attributed to the break of conjugated double bonds due to the dehydrochlorination of the polymer.

Figure S1. TGA thermograms of the PVC aerogel.
SEM Images of PVC Aerogels

SEM images were taken for PVC aerogels prepared with different initial mass-volume ratio of PVC/DMF solution (Figure S2). In general, a lower initial mass-volume ratio of PVC/DMF solution leads to a higher porosity in the as-synthesized PVC aerogel.

![SEM images of PVC aerogels prepared from different initial PVC/DMF mass-to-volume ratio of (a) 0.02 g/mL, (b) 0.025 g/mL, (c) 0.038 g/mL and (d) 0.10 g/mL. The scale bar is 20 μm.](image)

**Figure S2.** SEM images of PVC aerogels prepared from different initial PVC/DMF mass-to-volume ratio of (a) 0.02 g/mL, (b) 0.025 g/mL, (c) 0.038 g/mL and (d) 0.10 g/mL. The scale bar is 20 μm.