

Electronic Supplementary Information (ESI) for

**Intrinsic microstructure of supramolecular hydrogels derived from
 α -cyclodextrin and Pluronic F127: nanosheet building blocks and
hierarchically self-assembled structures**

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Materials. α -CDs were purchased from Aladdin Industrial Corporation and used as received. Pluronic F127 were purchased from Sigma, USA. Distilled water was used, and all the other reagents are of analytical purity and used as received.

Synthesis and drying of SMHs. SMHs could be formed with a varying feed volume ratio of α -CD saturated aqueous solution to Pluronic F127 aqueous solution (0.1 g/ml). Take the SMH1-1 for example, 2 ml α -CD solution and 2 ml Pluronic F127 solution were mixed and stirred for 5 min, then the mixture were presented at 20 °C and a white and opaque SMH was formed in 1h. The SMHs were dried by different method. The ambient pressure drying was carried out by presenting the SMH in an oven at 80 °C. The freeze-drying method was carried by freezing the SMHs at -20 °C and dried in vacuum at room temperature. SCL drying was performed with CO₂ under the condition of 9 MPa and 42 °C, the SMHs were solvent-exchange with acetone before drying.

Characterization. XRD was performed on a Bruker D8 Advance spectrometer. The radiation source used was Ni-filtered Cu KR radiation with a wavelength of 0.154 nm. Samples were mounted in a sample holder and scanned from 4.5 to 50° at a speed of 5° min⁻¹. Field-emission scanning electron microscopy (Quanta 400 FEG) was performed to determine the microstructure of the SMHs. The samples were coated with Au nanopowder under current of 20 mA for 2 min. The specific surface area was determined by the Brunauer-Emmett-Teller (BET) method (ASAP 2020, Micromeritics, USA), based on the amount of N₂ adsorbed at pressures 0.05 < P/P₀ < 0.3.

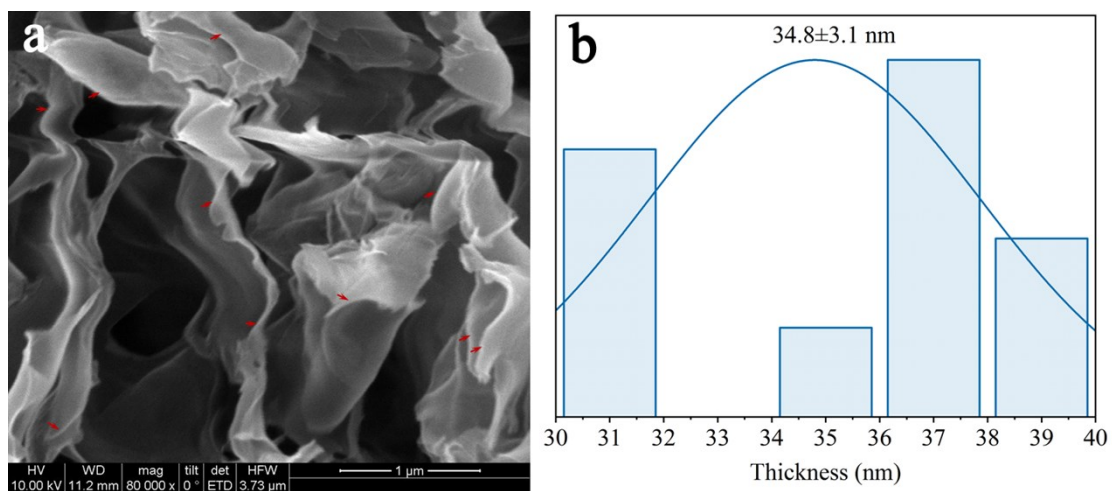


Fig. S1 (a) SEM image of SMH1-1 dried by SCLD; (b) Thickness of the nanosheets obtained by ImageJ, the measured sites are indicated by red arrows, and the thickness ranged from 30 to 40 nm (the columns corresponding to specific nanosheets) with an average value of 34.8 nm and a coefficient of variation of 3.1 nm (the fitted curve).

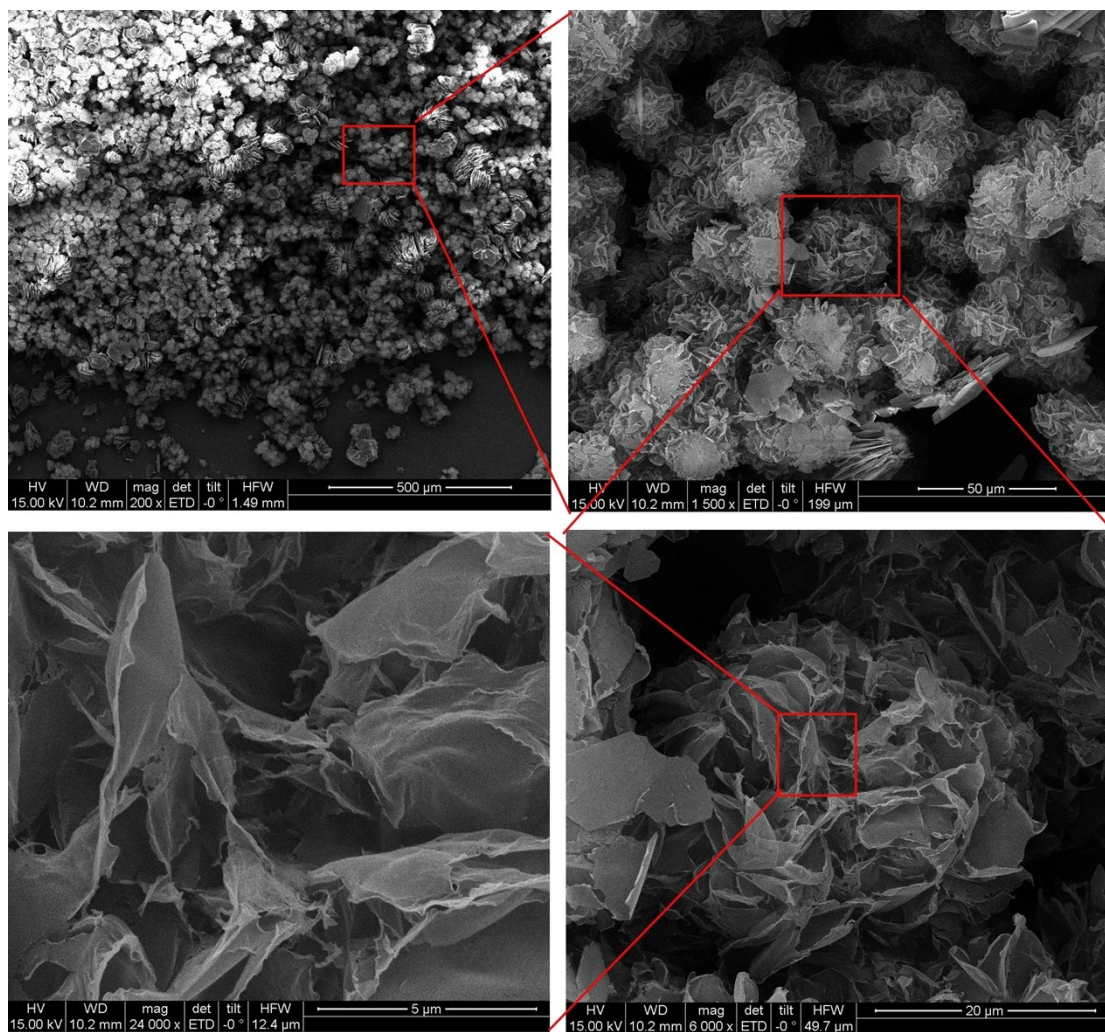


Fig. S2. SEM image of SMH15-1 dried by SCLD.