Supplementary Information (SI) for Soft Matter.

A facile method to fabricate syndiotactic polystyrene aerogels by freeze-drying

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Characterization

High-temperature gel permeation chromatography (HT-GPC) analysis of the weight-average molecular weight (M_w) and molecular weight distribution (PDI) of sPS at 150°C were performed on a PL-GPC 220 high-temperature chromatograph equipped with two GPC columns (USA PLgel 10 μm MIXED-B) and a differential refractive-index detector. 1,2,4-Trichlorobenzene (TCB) was used as the eluent at a flow rate of 1.0 mL/min. The calibration curves were made by narrow polystyrene standards (Polymer Laboratories).

Carbon-13 nuclear magnetic resonance (^{13}C NMR) spectra of the samples were obtained with a Bruker Avance III 500 MHz NMR instrument at 130°C. The samples were dissolved in a 5 wt % solution of a mixed solvent of deuterated o-dichlorobenzene/o-dichlorobenzene with a volume ratio of 1:1 in the 10 mm high temperature NMR tubes.

Differential scanning calorimetry (DSC) curves of sPS were collected using a Perkin-Elmer DSC 4000 instrument (USA) under nitorgen atmosphere. The follow programs were set: first, weighed about 3 mg samples and heated it from 40 to 300 °C at a rate of 10 °C/min, kept at 300 °C for 5 min to remove the thermal history. Then cooled down to 40 °C at a rate of 10 °C/min, kept at 40 °C for 1 min followed by heating to 300 °C at a rate of 10 °C/min again, finally recorded the second heating curves. The protective gas was nitrogen at a flow rate of 20 mL/min.

The thermograms of solidification and melting of pure *p*-xylene and *p*-xylene in sPS gels were recorded on a Netzsch DSC204F1 DSC instrument (Germany). Samples of about 5.0 mg were sealed in aluminum DSC pans to undergo the following temperature program: holding the samples at 30°C for 5 min and then cooling to -50°C at 10°C/min; holding the samples at -50°C for 5 min and then heating to 30°C at 10°C/min; the purge gas and protective gas were nitrogen, with 20 mL/min flow rate; finally recorded the cooling and heating curves.

Thermogravimetric analyses (TGA) were performed on a Netzsch TGA 209 F1 instrument (Germany) from 30 to 800°C at a heating rate of 10°C/min, under a nitrogen atmosphere with a flow rate of 20 mL/min.

Addition Results

Stereoregularity of sPS. The ^{13}C NMR solution spectra of our homemade five different molecular weights sPS samples recorded in o-dichlorobenzene at 130°C are presented in Figure S1b. That region of the spectra containing the protonated phenyl ring carbon resonances ($C_{4\text{-}6}$ in Figure S1b) is not shown because of extensive overlap with the more abundant solvent o-dichlorobenzene resonances (127.7, 130.5, 132.6 ppm). However, it is apparent from the remaining spectral regions, particularly the methylene carbon region known to be highly sensitive to the PS stereosequence, that only single resonances are observed for the CH_2 (C_1, 45.0 ppm), CH (C_2, 41.6 ppm), and C_3 (145.7 ppm) carbons, which correspond to the syndiotactic stereosequence [1].

Table S1. Summary of HT-GPC, ^{13}C NMR, and DSC analysis of different molecular weights sPS.

samples	M_w (g/mol)	PDI	sPS tacticity [rrrr]	$T_{m,1}$ (°C)	$T_{m,2}$ (°C)
sPS-1	600,000	2.38	> 96%	263.9	268.4
sPS-2	350,000	2.65	> 96%	260.1	268.2
sPS-3	150,000	2.90	> 96%	261.0	269.5
sPS-4	100,000	2.71	> 96%	254.0	268.7
sPS-5	50,000	2.42	> 96%	254.1	264.5

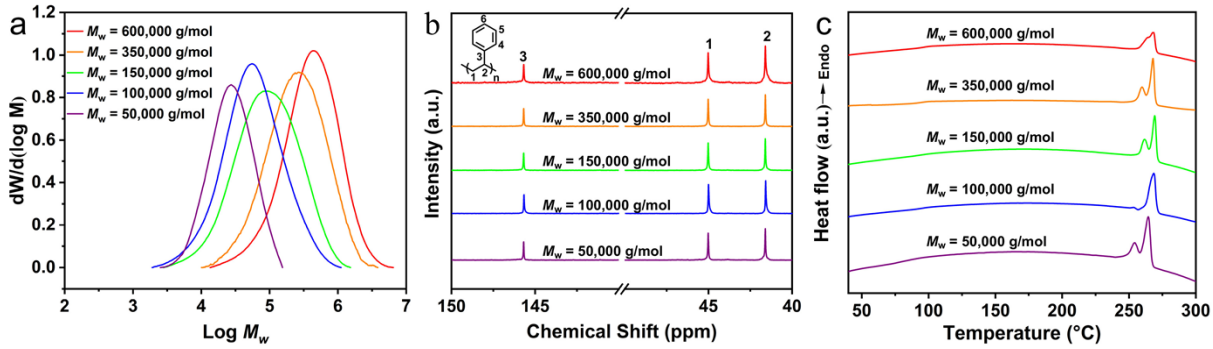

Figure S1. (a) HT-GPC profiles, (b) ^{13}C NMR spectra, and (c) DSC traces of different molecular weights sPS.

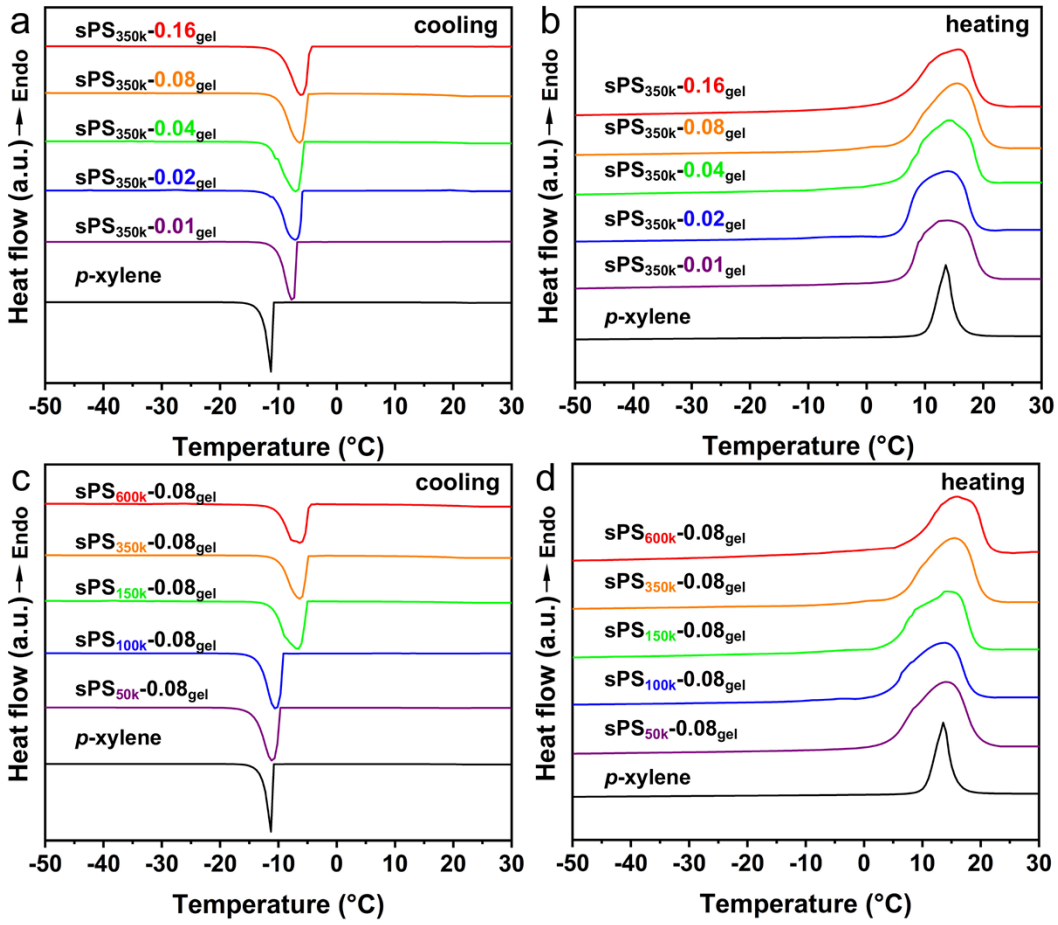

Figure S2. DSC traces of pure *p*-xylene and *p*-xylene-based sPS gels with different sPS contents (a) cooling and (b) heating, and different sPS molecular weights (c) cooling and (d) heating.

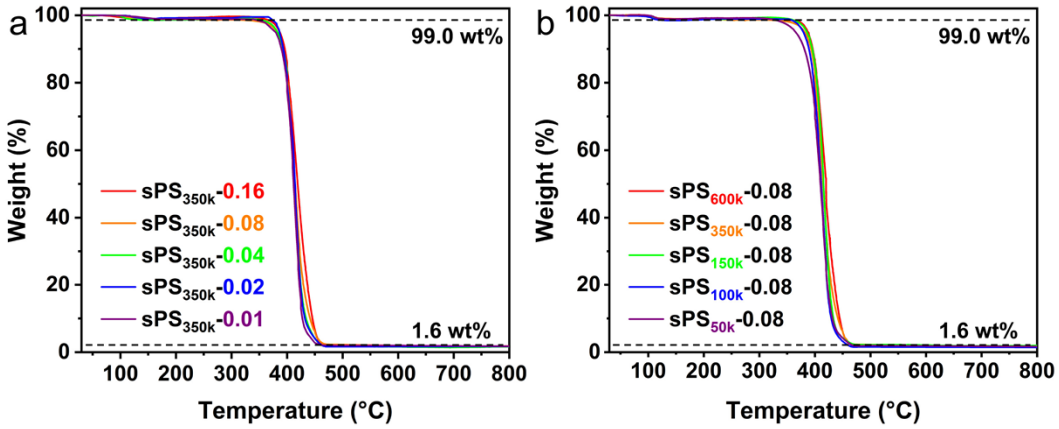

Figure S3. TGA curves of the sPS aerogels with (a) different sPS contents and (b) different sPS molecular weights.

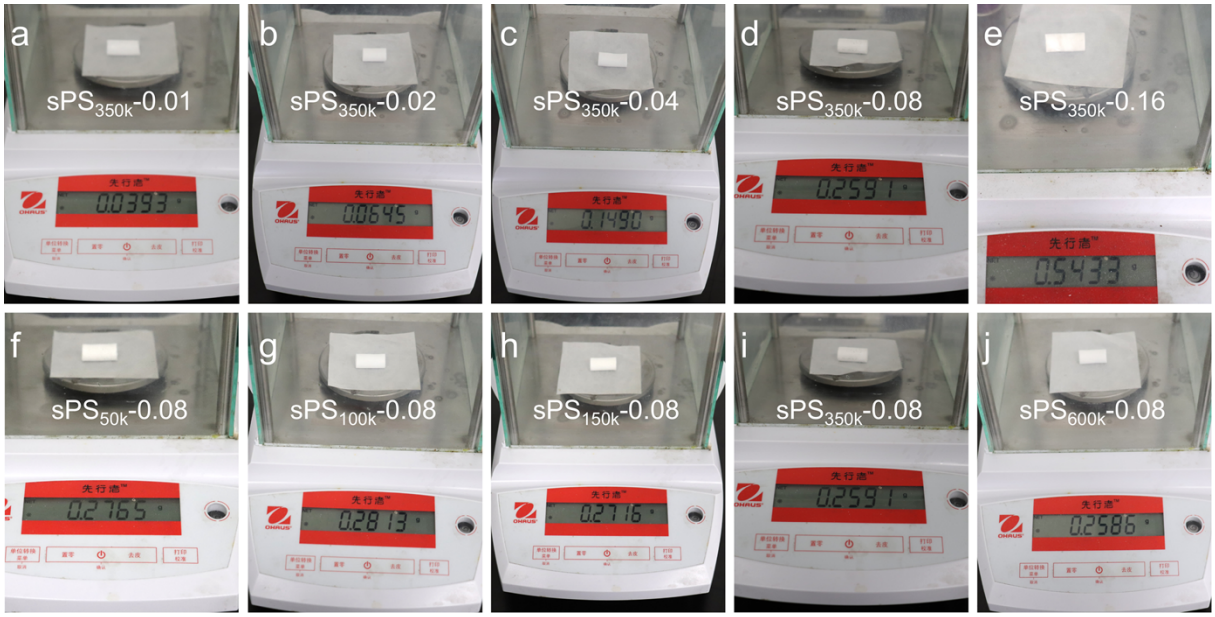

Figure S4. Weight of the sPS aerogels with (a-e) different sPS content and (f-j) different sPS molecular weights.

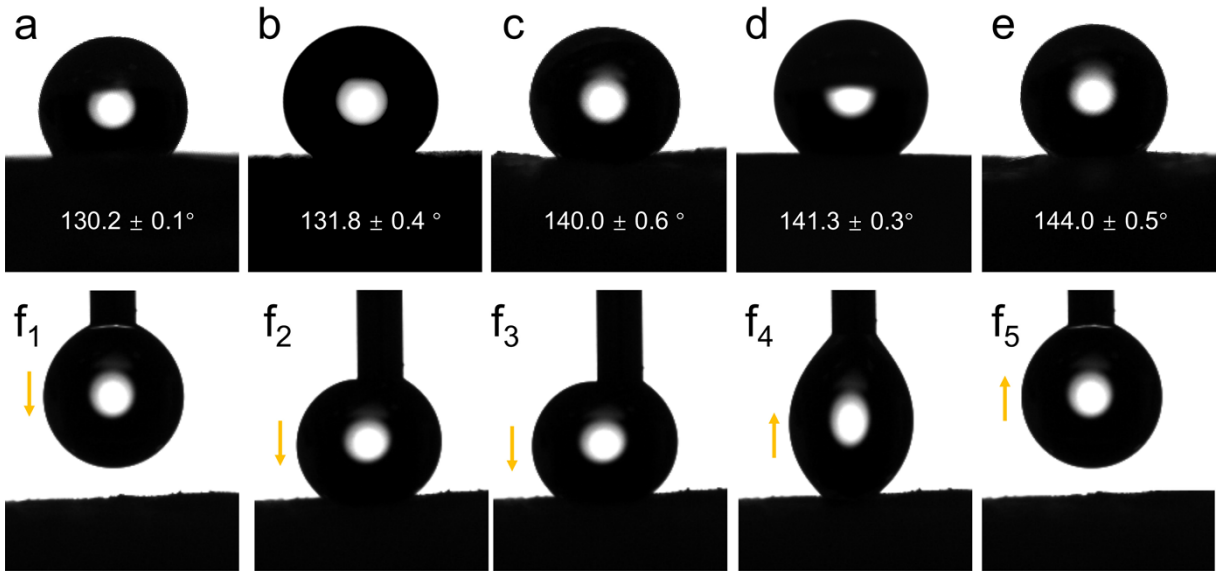

Figure S5. Water contact angles of the sPS aerogels with different sPS contents. (a) sPS_{350k}-0.16, (b) sPS_{350k}-0.08, (c) sPS_{350k} -0.04, (d) sPS_{350k}-0.02, and (e) sPS_{350k}-0.01. ($f_{1\text{-}4}$) Dynamic water repelling images on sPS_{350k}-0.01 surface.

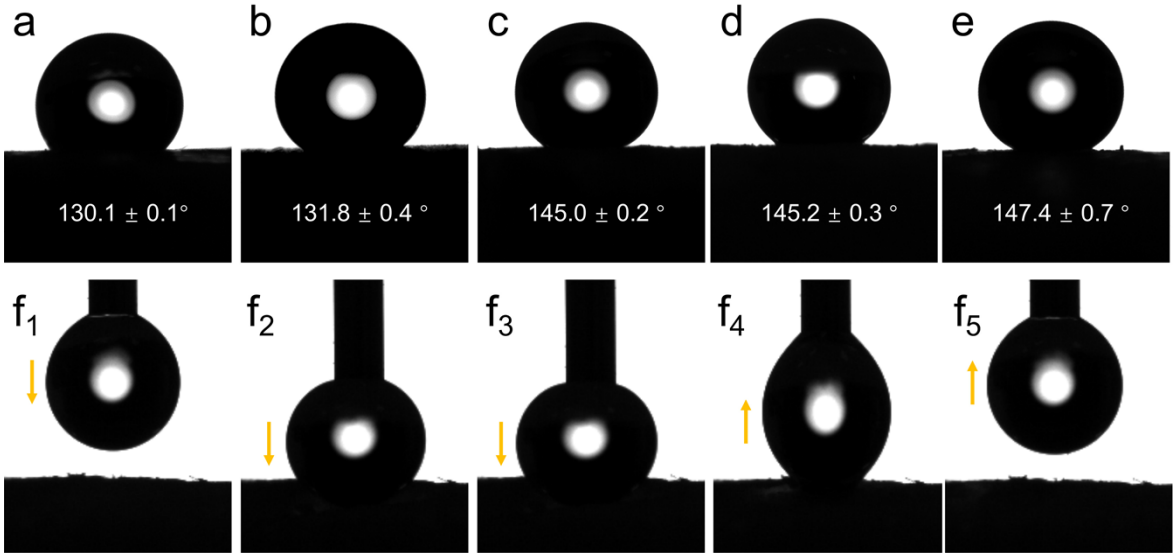

Figure S6. Water contact angles of the sPS aerogels with different molecular weights. (a) sPS_{600k}-0.08, (b) sPS_{350k}-0.08, (c) sPS_{150k} -0.08, (d) sPS_{100k}-0.08, and (e) sPS_{50k}-0.08. ($f_{1\text{-}4}$) Dynamic water repelling images on sPS_{50k}-0.08 surface.

Table S2. Comparison of the absorption capacity of various polymeric absorbents.

			absorption capacity (g/g)			
oil sorbents	ρ_a (g/cm^3)	P (%)	hexane	chloroform	pump oli	references
PVDF	0.200	90.0	3.1	-	4.0	[2]
PDMS	0.180	81.0	18.0	34.0	8.5	[3]
modified PU	0.086	-	20.4	22.4	-	[4]
PI/MXene	0.023	-	20.0	45.0	-	[5]
sPS-emulsion-1	0.015	98.5	33.9	81.3	-	[6]
sPS_{350k}-0.01	0.012	98.9	38.1	60.0	40.3	this work

References

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