Electronic Supplementary Material (ESI) for RSC Advances.

Preparation of mesoporous silica nanosheets through electrospinning: a novel scroll mechanism

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Electronic Supplementary Information (ESI)

Reagents and materials

Polyvinylpyrrolidone (PVP, M_w 1,300,000), polyamidoamine dendrimers $C_{22}H_{48}N_{10}O_4$ (PAMAM-0G, 20% solution in methanol) and $C_{62}H_{128}N_{26}O_{12}$ (PAMAM-1G, 20% solution in methanol) were purchased from Sigma-Aldrich. Tetraethyl orthosilicate (TEOS, SiO_2% = 28.4) and ethanol (99.7%) were provided by Sinopharm Chemical Reagent Co., China. PAMAM-0G and 1G were treated by removing methanol solvent using vacuum distillation before use, other chemicals were used without further purification. Deionized water was used throughout this work. Molecular structures of PAMAM-0G and 1G are illustrated with Fig. S1.

Preparation of silica precursor sols

Magnetic stirring was applied throughout this part. In a typical process in this work, 0.034 g PAMAM-1G was dropped into a mixed solvent containing 22 mL ethanol and 2 mL water. After PAMAM was fully dissolved, 1.6 g PVP was added to the mixture. The viscosity of solution became higher and higher during dissolution. A total content of 3.0 g TEOS was added dropwise to the system after the polymer was completely dissolved. Stirring was continued for another 30 min, thus the silica precursor sol was obtained. Various contents of TEOS ranging from 0.3 g to 4.0 g, and the zero-generation PAMAM were employed to form different sols. For a comparison, no structuring agent was utilized in the blank experiment. Moreover, polymer was not used in another comparison experiment in order to obtain a normal silica sol.

Preparation of PVP/SiO_2 nanofibers

The as-prepared precursor sol was moved to a plastic syringe with a metal nozzle, of which the inner diameter was 1.2 mm. A high voltage of 18 kV was applied to the nozzle to start the electrospinning process. The distance between the nozzle and the counter electrode was 20 cm. During electrospinning, the sol was pushed out the syringe by a pump at a constant rate of 0.8 mL h^{-1}. The deformed drops converted into ultrathin fibers when they touched the high voltage. The fibers were collected on the counter electrode covered with aluminum foil. Then, they were dried at 60 ℃ for 24 h under vacuum, as the PVP/SiO_2 nanofibers (see Fig. S2). The normal silica sol did not experience this procedure. The electrospinning process is illustrated with Fig. S3.

Preparation of SiO_2 nanosheets

The fibers were calcined in air in order to remove the coexisted polymer and template. The procedures of calcination were as follows. First, the chamber temperature was increased from 30 °C to 100 °C with the rate of 2 °C min^{-1}. Then, the nanofibers were heat-treated at the rate of 5 °C min^{-1} to 500 °C and held at this temperature for 6 h. After a spontaneous cooling procedure to room temperature, the mesoporous silica nanosheets were obtained (see Fig. S4). On the other hand, the normal silica sol was kept static until it formed a gel. The gel was dried at 80 °C under vacuum for 24 h. A xerogel was thus obtained and calcined later through the identical procedures mentioned above.

Characterization

Scanning electron microscopy (SEM) measurements were taken on a Philips XL-30 scanning electron microscope. Transmission electron microscopy (TEM) experiments were performed on a FEI Tecnai G2 F20 S-TWIN electron microscope. Wide-angle X-ray diffraction (WAXD) pattern of the adsorbent sample was recorded using an X-ray diffractometer (Bruker D8 Advance) at 40 kV and at a current of 40 mA with Cu Kα radiation. Low-angle X-ray diffraction pattern of the adsorbent sample was recorded using a Rigaku RINT2000 vertical goniometer at 40 kV and at a current of 50 mA with Cu Kα radiation. The nitrogen adsorption and desorption isotherms were measured using a Micromeritics ASAP 2020 analyzer at 77 K. The Brunauer-Emmett-Teller (BET) method was utilized to calculate the specific surface area. The pore volume and pore size distribution were derived by using the Barrett-Joyner-Halenda (BJH) model.

Physicochemical parameters of the as-prepared materials are defined as follows: mesopore surface area (S_{meso}, $m^2\ g^{-1}$) was calculated using BJH method from the desorption branches; total pore volume (V_{total}, $cm^3\ g^{-1}$) was measured at $P/P_0 = 0.995$; mesopore volume (V_{meso}, $cm^3\ g^{-1}$) was derived from the difference between the total pore volume (V_{total}) and the micropore volume determined from *t*-Plot method; average pore diameter (D_{pore}, nm) was calculated using BJH method from the desorption branches. Nitrogen adsorption-desorption isotherm data and BJH desorption pore distribution data of the typical sample as-prepared (TEOS 3.0 g as silica source, PAMAM-1G as template) are shown in Table S1 and S2, respectively.

Figures and experimental data

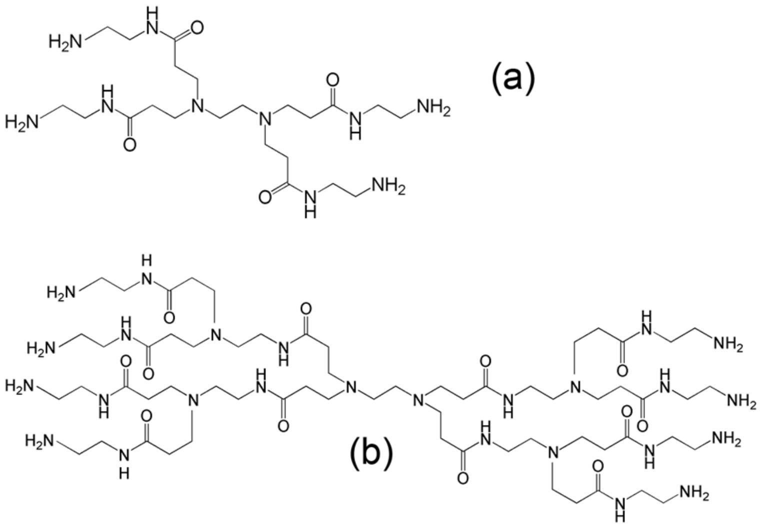

Fig. S1 Molecular structures of dendrimers: (a) PAMAM-0G and (b) PAMAM-1G.

Fig. S2 Photograph of the product before calcination (TEOS 3.0 g, PAMAM-1G).

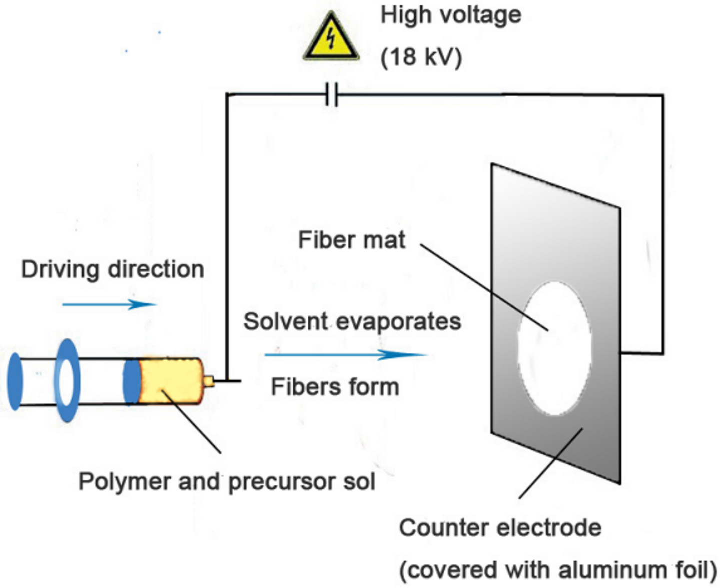

Fig. S3 The electrospinning process.

Fig. S4 Photograph of the product after calcination (TEOS 3.0 g, PAMAM-1G). The product was stored in a plastic tube.

Table S1 Nitrogen adsorption-desorption isotherm data.

Relative pressure (P/P_0)	Quantity adsorbed (cm^3 g^{-1})
0.00184	104.0474
0.00357	117.3640
0.00720	132.5151
0.01604	151.1314
0.05136	181.6048
0.08183	195.4255
0.12025	207.8763
0.15567	217.1075
0.19120	225.1766
0.22665	232.5915
0.26196	239.6444
0.29701	246.4652
0.33177	253.2763
0.36466	259.8348
0.39955	267.0447
0.43459	274.6163
0.47003	282.7705
0.50497	291.3524
0.53985	300.8055
0.57504	311.1356
0.60946	322.3540
0.64479	335.2661
0.67974	349.7670
0.71463	366.5300
0.74953	386.1236
0.78434	409.4046
0.81940	437.3050
0.85430	469.8634
0.88986	506.1713
0.92641	541.8219
0.96126	569.6638
0.99432	591.8157
0.94530	582.8736
0.90922	576.5541
0.87506	568.3525
0.85579	559.4136
0.82410	534.9546
0.78629	495.1345
0.74734	450.5942
0.71230	414.5089
0.67941	385.8878

Relative pressure (P/P_0)	Quantity adsorbed (cm^3 g^{-1})
0.64417	361.5264
0.61001	342.4128
0.57493	325.8793
0.53975	311.5714
0.50502	299.2753
0.47074	286.6263
0.43448	275.4054
0.39992	266.8101
0.36532	259.1901
0.33016	251.9109
0.29508	244.7614
0.26000	237.6358
0.22508	230.3871
0.19008	222.8137
0.15517	214.6214
0.12026	205.2772
0.08573	193.9597
0.05092	178.1970
0.01438	145.0730
0.00736	129.9723
0.00381	116.4579
0.00194	104.1164

Table S2 BJH desorption pore distribution data.

Average diameter (nm)	Incremental pore volume (cm^3 g^{-1})
1.7	0.01112
1.9	0.00991
2.1	0.00942
2.2	0.00924
2.4	0.00942
2.6	0.00975
2.8	0.01086
3.1	0.01364
3.3	0.02061
3.6	0.02505
4.0	0.02331
4.3	0.02809
4.8	0.03336
5.3	0.03953
5.9	0.05162
6.6	0.06166
7.4	0.07791
8.6	0.09540
10.3	0.08345
12.6	0.04976
15.0	0.01760
18.3	0.01544
26.1	0.01129
39.8	0.01600