Facile One-Pot Synthesis of Mesoporous Hierarchically Structured Silica/Carbon Nanomaterials

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Supporting Information

Experimental Section

Preparation of polymer-silica-surfactant (PSS) composites:

PSS composites were synthesized in basic solution at room temperature using ethanol and water as cosolvents, cetyltrimethylammonium bromide (CTAB) as surfactant, tetraethylorthosilicate (TEOS) and resorcinol/formaldehyde (RF) as precursors, and ammonia as catalyst. Typically, ammonia aqueous solution (NH_4OH, 0.2 mL, 25 wt%) and resorcinol (0.2 g) were first mixed with a solution containing absolute ethanol (EtOH) and deionized water (H_2O) with a total volumes of 28 ml (for different samples in this work, the volume ratios of ethanol/water were 4/24, 8/20 and 12/16). After the mixture was stirred for 0.5 h at room temperature, CTAB (0.2 g) was added and continually stirred for 0.5 h. Then the formaldehyde solution (0.28 mL) was added and stirred for 1~5 min, and subsequently TEOS (1 mL) were added to the reaction solution and stirred for 24 h at room temperature. Then the product was heated for 24 h at 80 ℃ under a static condition in a Teflon-lined autoclave. The solid product was recovered by centrifugation and air-dried at 80 ℃ for 24 h.

Preparation of mesoporous hierarchically structured silica/carbon mesoporous nanomaterials:

The obtained PSS composites were pyrolysis in a tubular furnace at 600 ˚C for 3 h under a N_2 flow with a heating rate of 3ºC/min. The silica was removed by immersing the obtained carbon-silica composites in 10 wt% HF aqueous solution at room temperature for 24 h, followed by washing with water. Mesoporous hierarchical structured carbon nanomaterials were obtained after drying for overnight. Mesoporous hierarchical structured silica nanomaterials were obtained by calcination the PSS composites at 550ºC for 5 h in air.

Characterization:

Scanning electron microscopy (SEM) images of samples were recorded on a Hitachi S-4800 field emission scanning electron microscope operated at 10kV. Transmission electron microscopy (TEM) experiments were conducted on a JEOL JEM-1011CX (Japan) operated at 100 kV. The S-4800 scanning electron microscope was used to acquire the EDX analysis results. Nitrogen sorption isotherms were measured at 77 K with an ASAP 2020 Nitrogen System. Before measurements, the samples were degassed in a vacuum for at least 6 h at 300ºC. The specific surface areas were calculated by using the Brunauer-Emmett-Teller (BET) method. The pore size distributions were derived from the adsorption branches of isotherms by using the Barrett-Joyner-Halenda (BJH) model. The total pore volumes were estimated from the adsorbed amount at a relative pressure P/P_0 of 0.971.

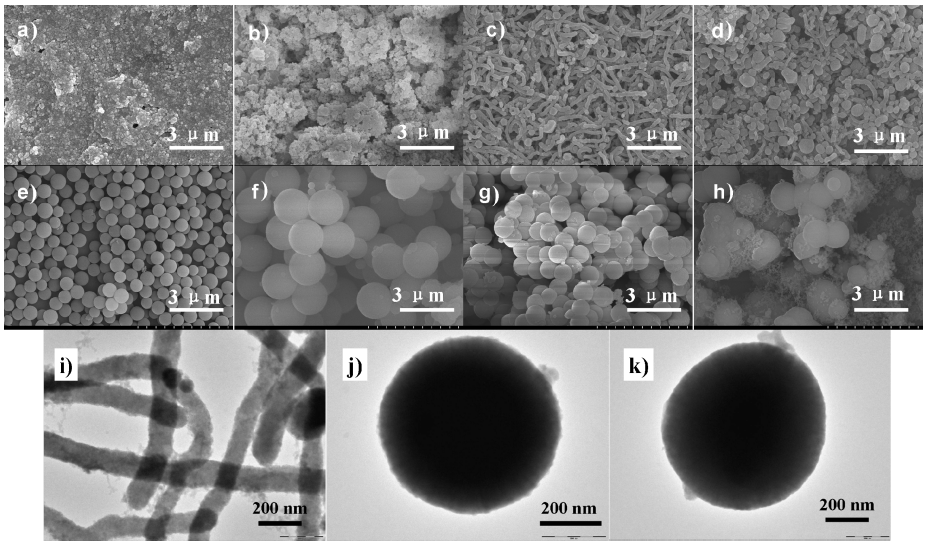
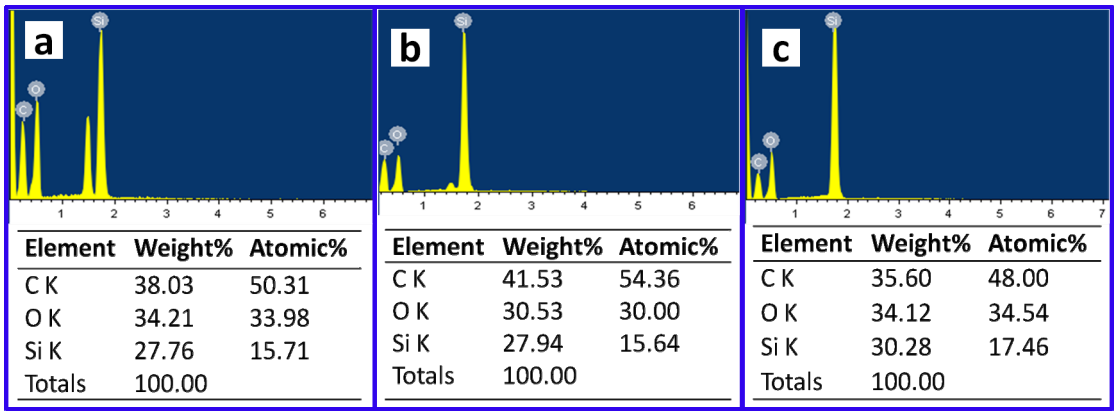


Fig. S1. SEM images (a-g) and TEM images (i-k) of the PSS composites prepared with different ethanol/water volume rations: ethanol/water volume ratio = 0:28 (a); 2/26 (b); 4/24 (c, i); 6/22 (d); 8/20 (e, j); 10/18 (f); 12/16 (g, k); 14/10 (h).



Element	Weight%	Atomic%
C K	38.03	50.31
O K	34.21	33.98
Si K	27.76	15.71
Totals	100.00	

Element	Weight%	Atomic%
C K	41.53	54.36
O K	30.53	30.00
Si K	27.94	15.64
Totals	100.00	

Element	Weight%	Atomic%
C K	35.60	48.00
O K	34.12	34.54
Si K	30.28	17.46
Totals	100.00	

Figure S2. EDX spectra and the element composition of CS composites (a: E4-CS, b: E8-CS, c: E12-CS).

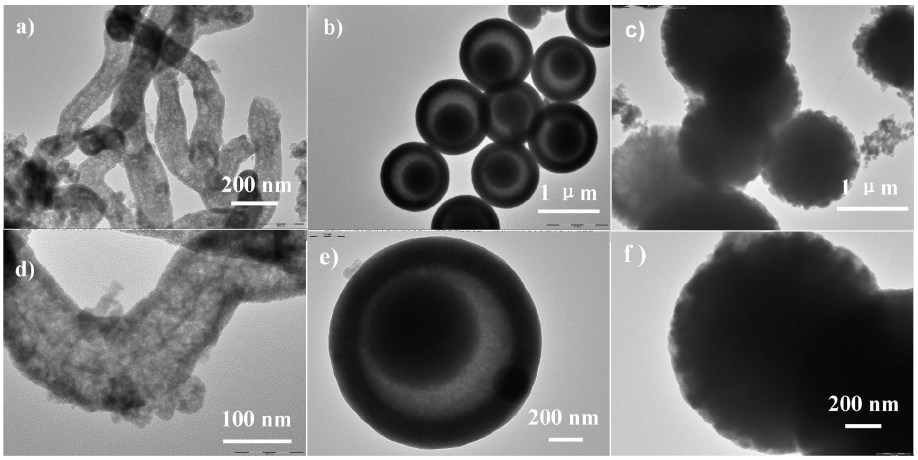
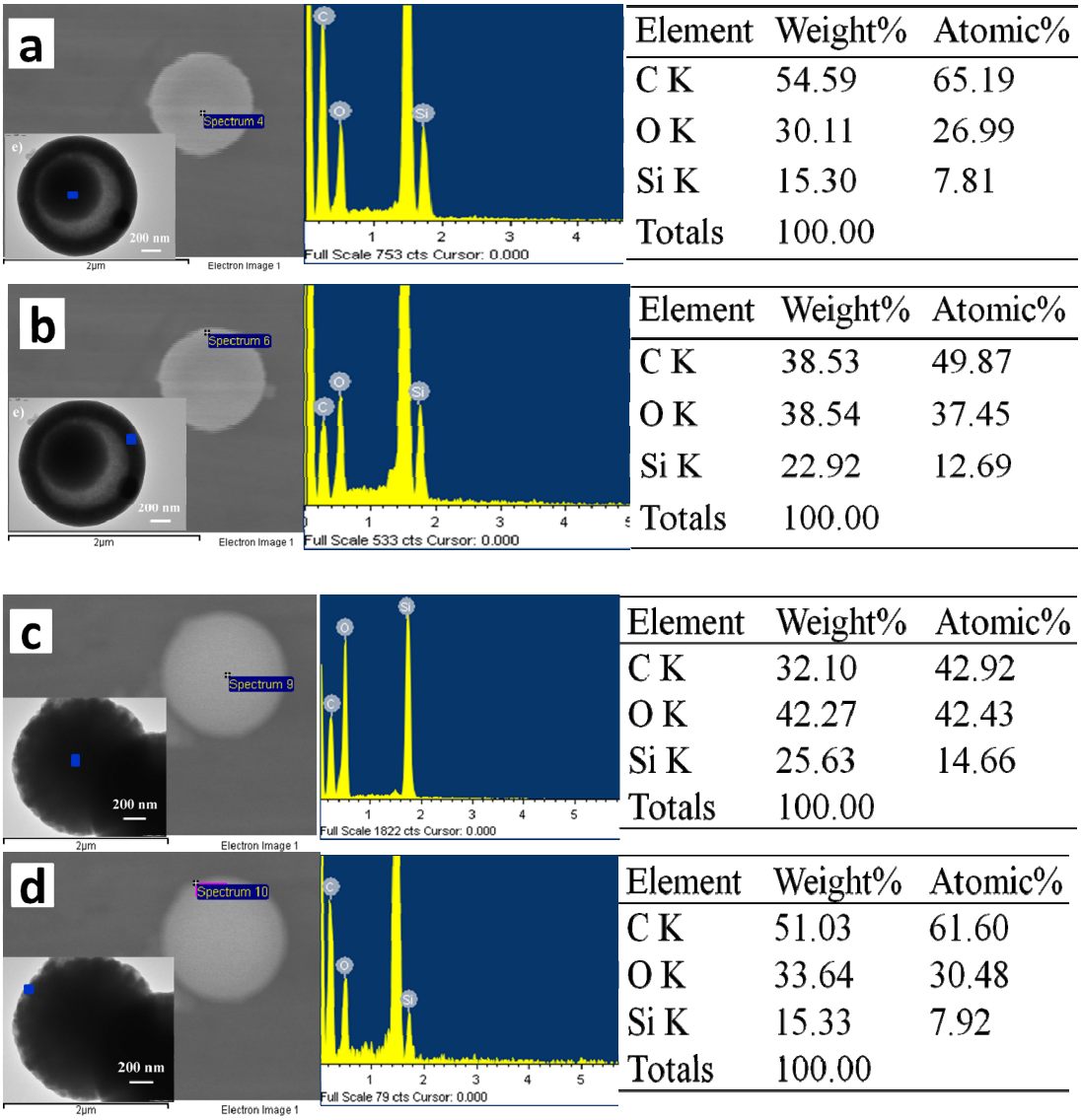


Fig. S3. TEM images of the carbon-silica (CS) samples E4-CS (a,d), E8-CS (b,e), and E12-CS (c,f).



Element	Weight%	Atomic%
C K	54.59	65.19
O K	30.11	26.99
Si K	15.30	7.81
Totals	100.00	

Element	Weight%	Atomic%
C K	38.53	49.87
O K	38.54	37.45
Si K	22.92	12.69
Totals	100.00	

Element	Weight%	Atomic%
C K	32.10	42.92
O K	42.27	42.43
Si K	25.63	14.66
Totals	100.00	

Element	Weight%	Atomic%
C K	51.03	61.60
O K	33.64	30.48
Si K	15.33	7.92
Totals	100.00	

Fig. S4. EDX spectra and the element composition of the core and shell of the CS composites (a,b: E8-CS, b,c: E12-CS).

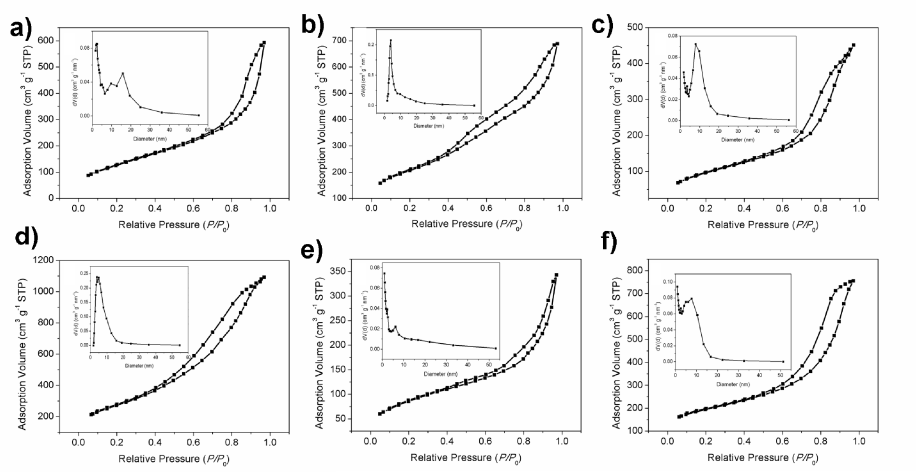


Fig. S5. N_2 sorption isotherms and pore size distributions (inset) of the samples prepared with different ethanol/water volume rations: (a) E4-SiO_2; (b) E4-C; (c) E8-SiO_2; (d) E8-C; (e) E12-SiO_2; (f) E12-C.

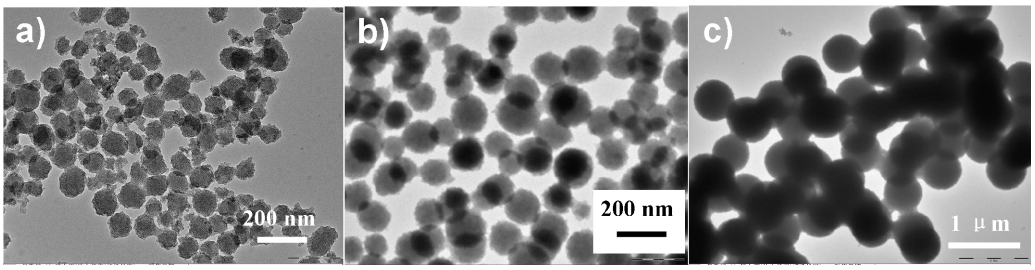


Fig. S6. TEM images of the mesoporous silica spheres prepared without the addition of RF precursor. The ethanol/water volume ratios were 4/24 (a), 8/20 (b), and 12/16 (c).

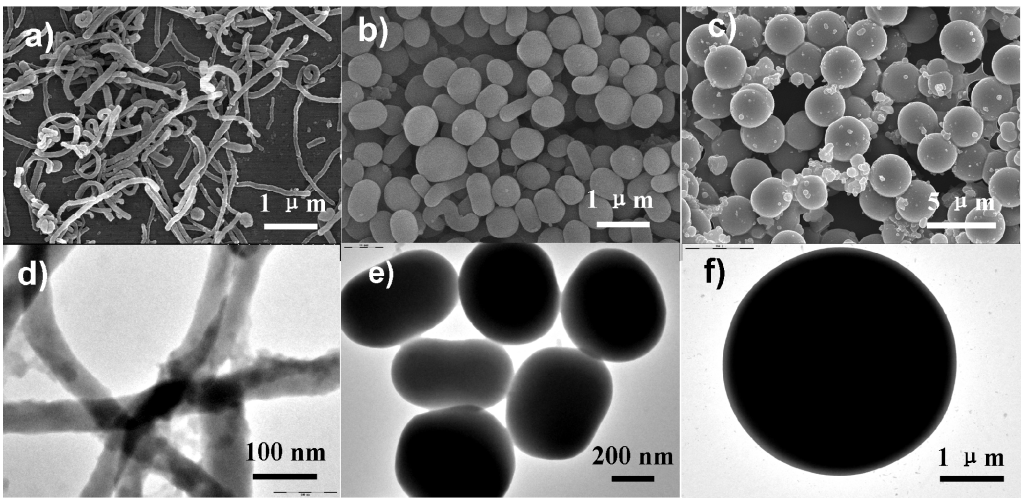


Fig. S7. SEM images (a-c) and TEM images (d-f) of the RF polymer products without the addition of TEOS. The ethanol/water volume ratios were 4/24 (a,d), 8/20 (b,e), and 12/16 (c,f).

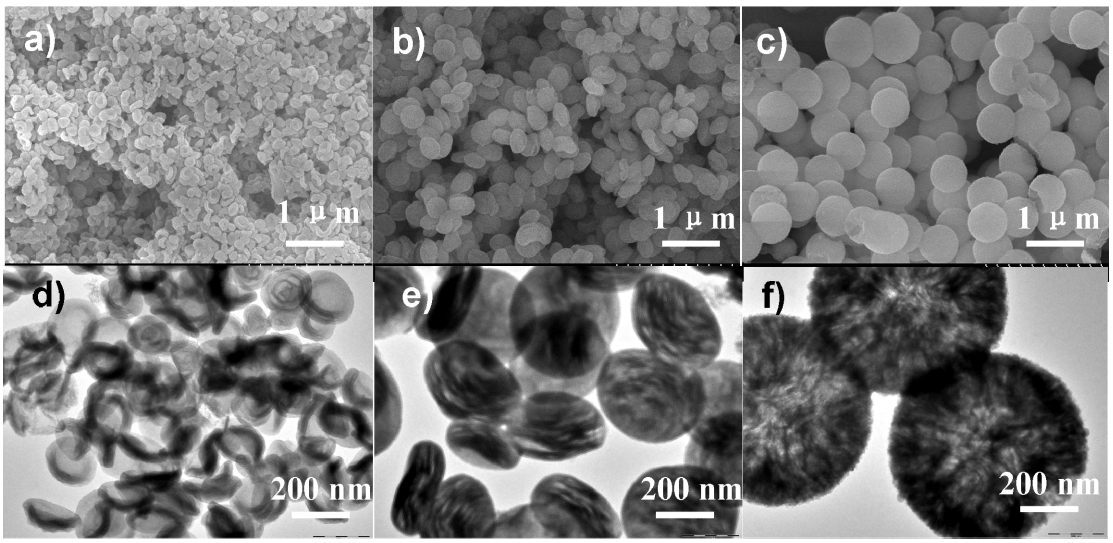


Fig. S8. SEM images (a-c) and TEM images (d-f) of the silica products without the addition of formaldehyde. The ethanol/water volume ratios were 4/24 (a,d), 8/20 (b,e), and 12/16 (c,f).

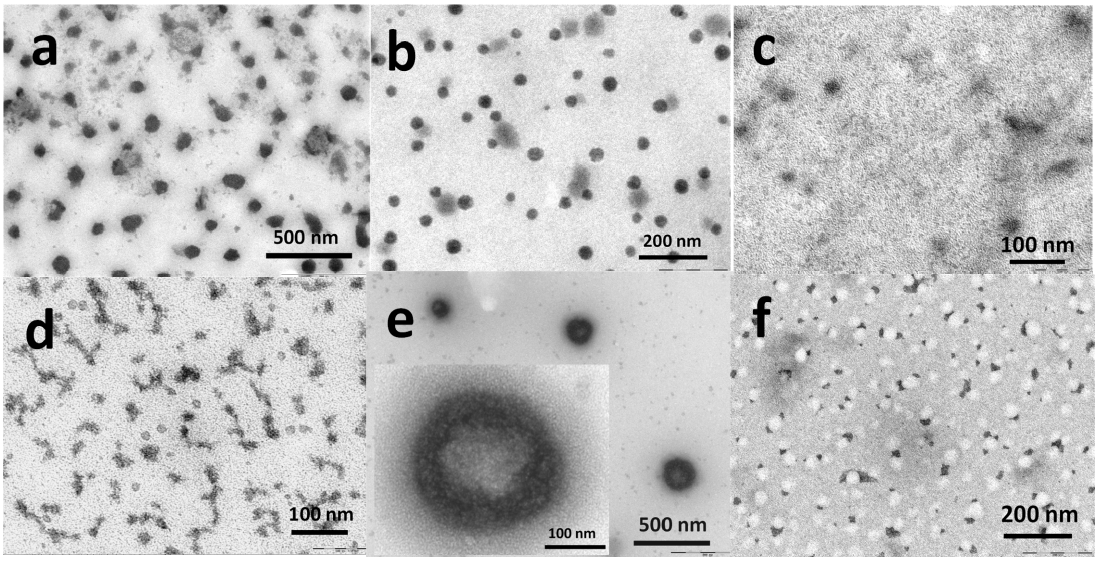


Fig. S9. TEM images (a, b, c) of the formed aggregates and micelles before the addition of formaldehyde, and TEM images (d, e, f) of the formed aggregates and micelles after 1 min of the formaldehyde was added, with different ethanol/water volume ratios: (a, d) 4/24, (b, e) 8/20, and (c, f) 12/16.