

Facile Preparation of Monodisperse Carbon Spheres: Template-free Construction and Further Their Hydrogen Storage Properties

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1. Materials

Analytically pure styrene (St), which was supplied by Kemiou Chemical Reagents Researching Center in Tianjin, was purified by distillation under reduced pressure in nitrogen atmosphere. Divinylbenzene (DVB-55), containing 55% of the para- and meta-divinyl isomers and 45% of para- and meta-ethylvinyl isomers, was purchased from Aldrich Chemical Co. Potassium persulfate (KPS) was purchased from Aijian Reactant Factory in Shanghai, and was used after recrystallization. Analytically pure tetrachloromethane (CCl_4) was obtained from Chemical Reagent Factory in Luoyang. Analytically pure acetone was provided by Chemical Reagent Factory in Tianjin. Analytically pure anhydrous aluminum trichloride (AlCl_3) was supplied by Damao Chemical Reagent Factory in Tianjin.

2. Recipes for the preparation of core-shell P(St-co-DVB) spheres with different DVB dosage

Table S1. Recipes for the preparation of core-shell P(St-co-DVB) spheres with different DVB dosage

Samples	KPS(g)	St(g)	DVB	
			(g)	(wt%)
SPSs-35	0.12	1.95	1.05	35
SPSs-40	0.12	1.80	1.20	40

3. SEM photos of the SPSs and HPCs samples

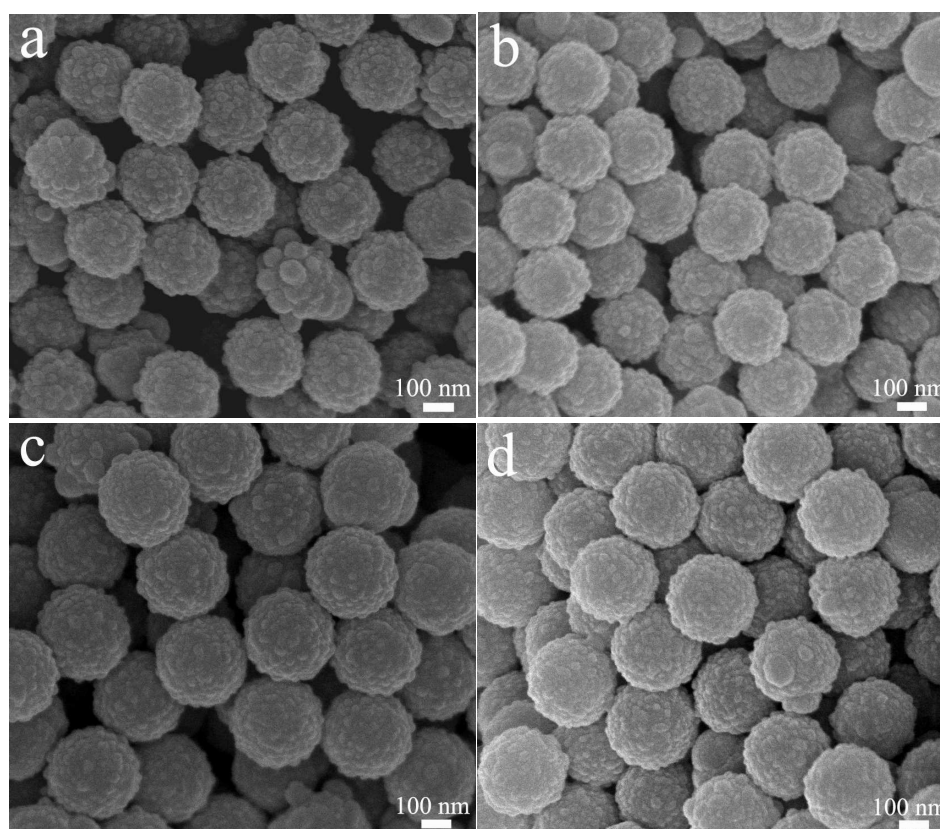


Fig. S1. SEM photos of (a) SPSs-35, (b) HPCs-35, (c) SPSs-40, and (d) HPCs-40.

SEM photos of the SPSs and HPCs samples are shown in Fig. S1. The diameter of the SPSs-35 and HPCs-35 was about 270 nm, and the diameter of the SPSs-40 and HPCs-40 was about 310 nm. According to Figure 4, the diameter of the CS35, CH35, CS40, and CH40 prepared from SPSs-35,

HPCs-35, SPSs-40 and HPCs-40 was respectively about 200 nm, 160 nm, 240 nm, and 220 nm. It is obvious that the size of carbon spheres is much smaller than that of the SPSs and HPCs.

4. TEM photos of the s-HCPSs and h-HCPSs samples

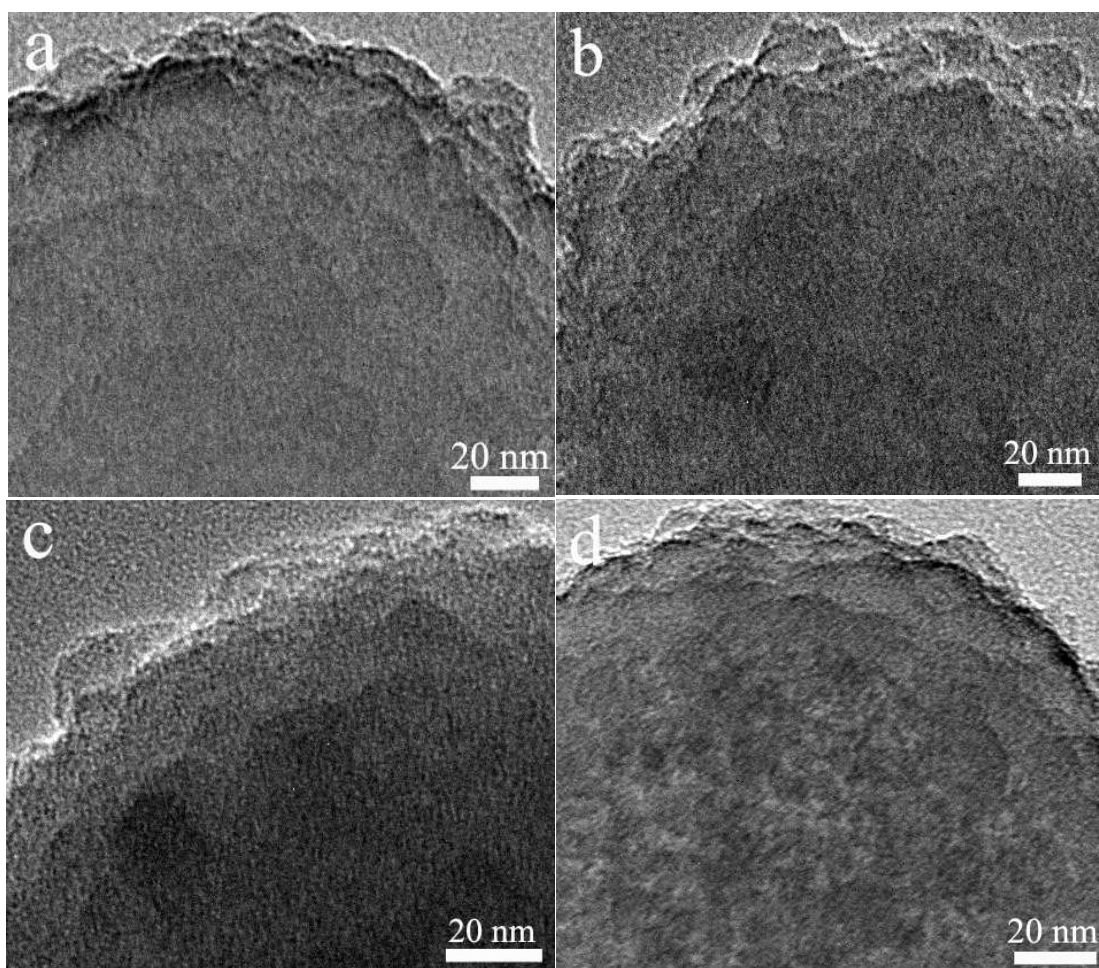


Fig. S2. TEM photos of (a) s-HCPSs-35, (b) h-HCPSs-35, (c) s-HCPSs-40, and (d) h-HCPSs-40

5. XRD patterns of the carbon spheres powder samples

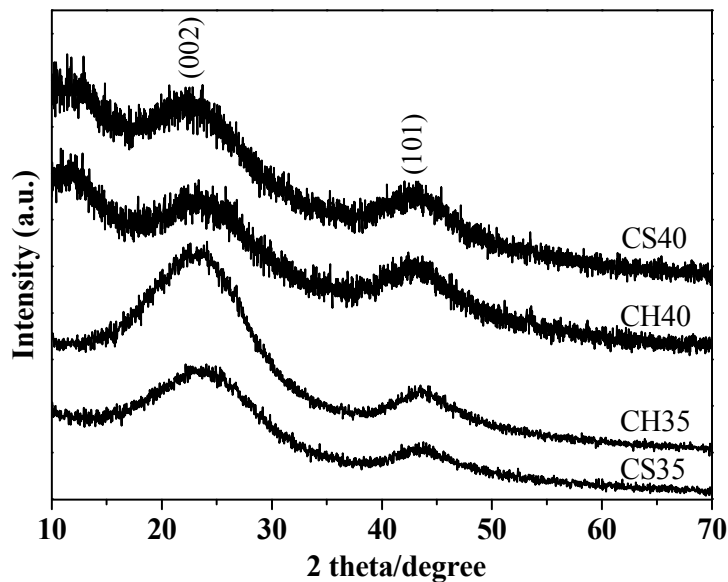


Fig. S3 XRD patterns of the carbon spheres powder samples.

XRD patterns of the carbon spheres powder samples are shown in Fig. S2. The wide reflection peaks indicated that these carbon spheres were amorphous graphite phase. As a measure of the number of carbon sheets arranged as single layers, the empirical parameter (R), which was defined as the ratio of height of the (002) Bragg peak to the background, was used.¹ Usually a larger R value indicates a higher degree of the graphitization.^{2,3} The R values of CS35, CH35, CS40, and CH40 are respectively 1.9, 2.3, 1.4, and 1.4, showing different degree of the graphitization. Besides, the broad reflection peak at 12° might be related with the microporous structure in the matrix of carbon spheres (CS40 and CH40).

6. Raman spectra of the carbon spheres

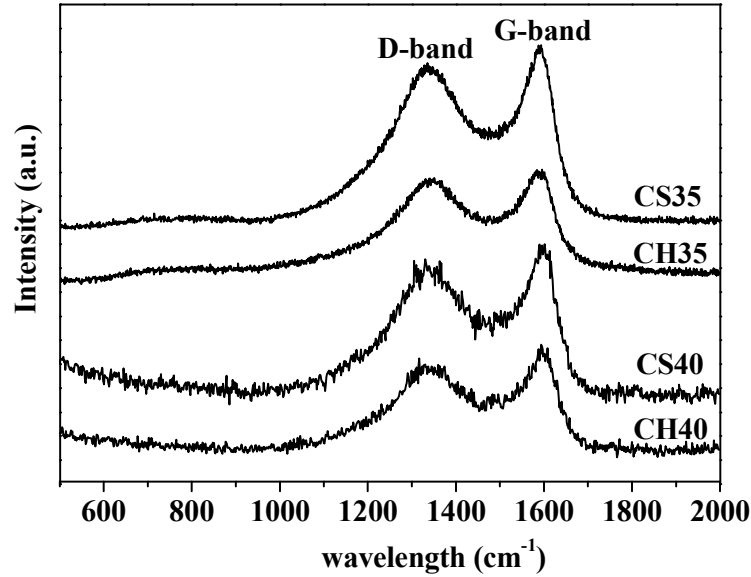


Fig. S4. Raman spectra of the carbon spheres.

Raman spectra for the carbon spheres samples are shown in Fig. S2. The D-band around 1350 cm⁻¹ is associated with the vibrations of carbon atoms with dangling bonds for the in-plane terminations of disordered graphite. The G-band around 1580 cm⁻¹ is attributed to an E_{2g} mode of graphite and is related to the vibration of sp²-bonded carbon atoms in a two-dimensional hexagonal lattice, such as in a graphite layer. The low ratio of I_D/I_G is characteristic of a graphite lattice with perfect two-dimensional order in the graphitic layer.⁴ The intensity ratio of I_D/I_G corresponding to CS35, CH35, CS40, and CH40 was respectively calculated to be 0.91, 0.95, 0.92, and 0.86.

7. Nitrogen adsorption/desorption isotherms of the carbon spheres

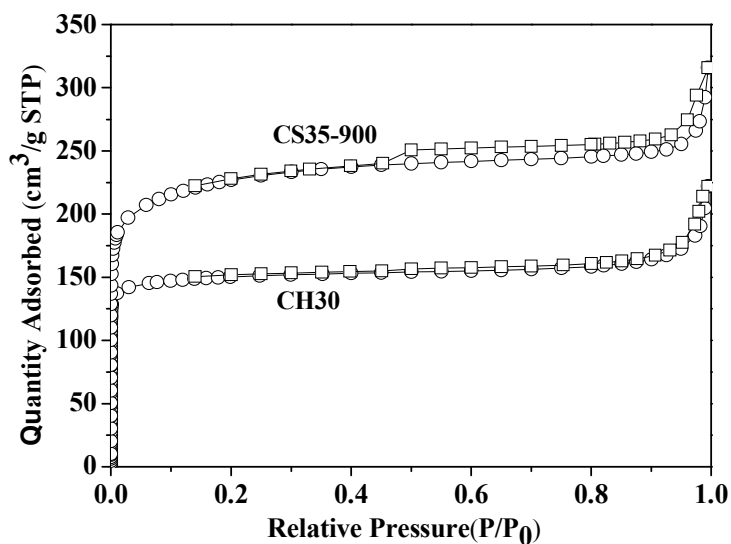


Fig. S5. Nitrogen adsorption/desorption isotherms of the carbon spheres

8. Textural parameters of the carbon spheres

Table S2. Textural parameters of the carbon spheres

Samples	$S_{\text{BET}}^{\text{a}}$ (m^2/g)	V_{t}^{b} (m^3/g)	$V_{\text{micro}}^{\text{c}}$ (cm^3/g)	$V_{\text{micro}}/V_{\text{t}}$ (%)	D_{HK}^{d} (nm)
CS35-900	776	0.41	0.32	78	0.77
CH30	454	0.34	0.23	68	0.49

^a Specific surface area (SSA) calculated by the Brunauer-Emmett-Teller (BET) method.

^b Total pore volume.

^c Microporous volume determined by the DR analysis.

^d Median micropore size calculated by the HK method.

According to our previous report,⁵ the shell thickness of hollow nanospherical precursor increases with increasing of DVB dosage. The textural parameters of the CH30, CH35 and CH40 samples respectively prepared from HPCs-30, HPCs-35 and HPCs-40 show that the shell thickness

influences the porosity of the carbon spheres (Tables 1 and S2). Besides, the carbon spheres labeled as CS35-900 were obtained by carbonizing s-HCPSs-35 at 900 °C. The N₂ adsorption-desorption results showed that the pore volume of CS35-900 was much larger than that of CS35 obtained by carbonizing s-HCPSs-35 at 700 °C (Tables 1 and S2). It was suggested that the pore volume was further increased after the carbonization temperature was increased to 900 °C.

References

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