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Electronic Supplementary Information

Size-controlled nitrogen-containing mesoporous carbon nanospheres

by one-step aqueous self-assembly strategy⁺

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Sample	Ammonia	F127	H_2O	HMT	MAP	TMB	Particle Size
	(M)	(mM)	(ml)	(mmol)	(mmol)	(mmol)	(nm)
APS-1	0.37	0	52	5	10	-	-
APS-2	0.37	1.5	52	5	10	-	-
APS-3	0.37	3.8	52	5	10	-	-
APS-4	0.37	4.6	52	5	10	-	324 ± 45
APS-5	0.37	5.3	52	5	10	-	136 ± 14
APS-6	0.37	6.1	52	5	10	-	-
APS-7	0	4.6	52	5	10	-	-
APS-8	0.14	4.6	52	5	10	-	-
APS-9	0.57	4.6	52	5	10	-	302 ± 31
APS-10	0.71	4.6	52	5	10	-	221 ± 28
APS-11	0.86	4.6	52	5	10	-	202 ± 16
APS-12	0.37	4.6	52	5	10	3.3	-

Table S1Synthesis parameters, particle size of spherical nitrogen-containing resols-F127composites (APS)

Sample	Particle size (nm)	Pore Size (nm)	V _{micro} (cm ³ g ⁻¹)	S_{BET} $(m^2 g^{-1})$	$\frac{V_t}{(cm^3 g^{-1})}$
NMCS-1-600	-	-	0.15	403	0.19
NMCS-7-600	-	4.7	0.14	497	0.32
NMCS-8-600	-	4.7	0.14	517	0.33
NMCS-4-600	225 ± 23	4.2	0.14	497	0.36
NMCS-9-600	163 ± 23	4.0	0.14	469	0.38
NMCS-10-600	143 ± 17	4.0	0.15	532	0.41
NMCS-11-600	119 ±14	4.0	0.17	595	0.50
NMCS-5-600	97±13	4.0	0.18	629	0.57
NMCS-4-800	164 ± 23	4.2	0.24	793	0.54
NMCS-12-600	~100	5.1	0.13	492	0.52

Table S2 Texture properties of NMCS with different particle sizes.

Table S3 Elemental composition of the typical samples from Elemental analysis and XPS spectra.

Consulta	XPS				Elemental analysis				
Sample	С	Ν	0	N/C	С	Ν	0	Н	C/N/H/O
	(at%)	(at%)	(at%)	N/C	(wt%)	(wt%)	(wt%)	(wt%)	
APS-4	74.3	5.1	20.6	0.069	65.9	3.8	23	7.3	100/5.8/11.1/24.9
NMCS-1-600	87.2	5.3	7.4	0.061	82.4	7.4	8.5	1.7	100/9.0/2.1/10.3
NMCS-4-600	88.0	4.5	7.4	0.051	81.5	7.3	9.3	1.9	100/8.9/2.3/11.4
NMCS-4-800	91.2	3.1	5.7	0.033	86.0	4.4	8.4	1.2	100/5.11/9.8/1.4
CMK-3	95.4	-	4.2	0	-	-	-	-	-
N-CMK-3 ^ª	93.9	1.3	4.8	0.014	-	-	-	-	-

a: N-CMK-3 represents the nitrogen-containing CMK-3, which is synthesized through two-step method: CMK-3

is firstly oxidized by concentrated HNO3 at 60 $^{\circ}$ C for 3 h and then treated by ammonia at 800 $^{\circ}$ C for 3 h.

	Comple	Description	Position	Area	FWHM	1 /I
	Sample	Description	(cm⁻¹)	(a.u.)	(cm⁻¹)	I _D /I _G
		D4	1195.1	70793	191	
		D	1348.8	186100	183	2.40
	NIVICS-1-600	D3	1507.2	102760	64	2.18
		G	1591.8	85169	94	
		D4	1173.5	55857	2216	
Before		D	1341.5	189190	200	2.00
Reaction	NIVICS-4-600	D3	1503.5	76090	60	2.08
		G	1586.7	90929	97	
		D4	1180.3	147440	243	
	NMCS-4-800	D	1334.5	408810	183	4.00
		D3	1493.7	184850	58	1.98
		G	2582.6	206130	95	
	NMCS-1-600	D4	1198.6	98243	183	
		D	1348.6	215070	173	2.26
		D3	1509.6	155950	70	2.36
		G	1593.1	91093.9	84	
		D4	1183.4	110840	210	
After		D	1340.9	191380	189	2.45
Reaction	NIVICS-4-000	D3	1520.2	142730	79	2.45
		G	1593.7	78079	80	
	NMCS-4-800	D4	1169.3	121450	90	
		D	1330.6	259930	181	2 12
		D3	1509.2	164220	76	2.13
		G	1589.6	121640	76	

Table S4 Sample denotations and comparison of the fit parameters from the peaks fitted in theRaman spectra of NMCS before and after reaction.

Table S5 C, N, O content of typical samples from XPS spectra after reaction.

Sample	C (at %)	N (at %)	O (at %)	N/C
NMCS-1-600	89.1	4.6	6.4	0.052
NMCS-4-600	90.1	4.2	5.7	0.047
NMCS-4-800	93.4	2.4	4.2	0.026



Fig. S1 Yield variation of **APS** and **NMCS-600** with the change of F127 concentration (A) and ammonia concentration (B); Yield variation of **APS** with the change of HMT amount (C).



Fig. S2 The upper solution after centrifugation. The synthesis condition: F127: 3.8 mM. 3-Aminophenol, 10 mmol; HMT, 5 mmol; ammonia concentrations: 0.37 M.



Fig. S3 Typical SEM image (A) and TEM image (B) of APS-4.



Fig. S4 Typical SEM image (A) and TEM image (B) of NMCS-1-600.



Fig. S5 Small-angle XRD patterns of the prepared **NMCS** with different synthesis conditions: A) different F127 concentration; B) different ammonia concentration; C) different carbonization temperature; D) adding TMB.



Fig. S6 Typical SEM images (A1-F1), TEM images (A2-F2) and N₂ adsorption-desorption isotherms (A3-F3) of NMCS-7-600 (A1-A3), NMCS-8-600 (B1-B3), NMCS-9-600 (C1-C3), NMCS-10-600 (D1-D3), NMCS-11-600 (E1-E3) and NMCS-5-600 (F1-F3). The insects show the corresponding pore size distributions.



Fig. S7 TEM image (A) and N_2 adsorption-desorption isotherms (B) of NMCS-12-600. The insect shows the corresponding pore size distributions.



Fig. S8 XPS spectra of C1s (A), N1s (B), and O1s (C) in NMCS-1-600 and NMCS-4-600.



Fig. S9 Catalytic performance of **N-CMK-3** for direct dehydrogenation of ethylbenzene to styrene. Reaction conditions: 550 °C, 2.8 % of Ethylbenzene in He, 10 mL min⁻¹, 100 mg catalyst. For comparison, we also plot the results of CMK-3 and NMCS-4-800.



Fig. S10 N_2 adsorption-desorption isotherms (A) and corresponding pore size distributions (B) of NMCS-1-600, NMCS-4-600, and NMCS-4-800 after reaction.



Fig. S11 TEM images of **NMCS-4-600** (A), **NMCS-4-800** (B), and **NMCS-1-600** (C) after reaction and small-angle XRD patterns of the catalysts after reaction (D).



Fig. S12 Raman spectra of NMCS samples after reaction.