

Electronic Supporting Information (ESI) for

Ni, FeO Nanocrystal-Integrated Hollow (Solid) N-Doped Carbon Nanospheres: Preparation, Characterization and Electrochemical Properties

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Table S1. Pore parameters of samples Ni(0)-HNCNSs-n (n =1, 2, 3), Ni(0)-HNCMSs-4, Ni(0)-SNCMSs-5 and FeO-HNCNSs-6 from N₂ physisorption analysis.

sample	$a_{\text{BET}}^{\text{a}}$ / m ² g ⁻¹	$V_{\text{micropore}}^{\text{b}}$ / cm ³ g ⁻¹	$V_{\text{single point}}^{\text{c}}$ / cm ³ g ⁻¹	$d_{\text{p,ads}}^{\text{d}}$ / nm	$d_{\text{p,des}}^{\text{e}}$ / nm
Ni(0)-HNCNSs-1	494	0.16	0.78	-	3.93
Ni(0)-HNCNSs-2	505	0.16	0.93	-	3.75
Ni(0)-HNCNSs-3	411	0.13	0.68	-	3.62
Ni(0)-HNCMSs-4	387	0.16	0.25	-	3.81
Ni(0)-SNCMSs-5	422	0.17	0.29	-	3.92
FeO-HNCNSs-6	404	0.08	0.85	-	3.54

^a Specific BET surface area.

^b Micropore volume.

^c Single point adsorption total pore volume at $P/P_0 = 0.99$; Samples were degassed at 250 °C under vacuum until the pressure was < 10⁻³ mbar for 4 h.

^d Pore diameter according to the maximum of the BJH pore size distribution calculated from the adsorption branch of the isotherm. For all samples, the maximum of the BJH mesopore size distribution cannot be determined due to very broad pore size, no uniform pore size can be determined.

^e Pore diameter according to the maximum of the BJH pore size distribution calculated from the desorption branch of the isotherm.

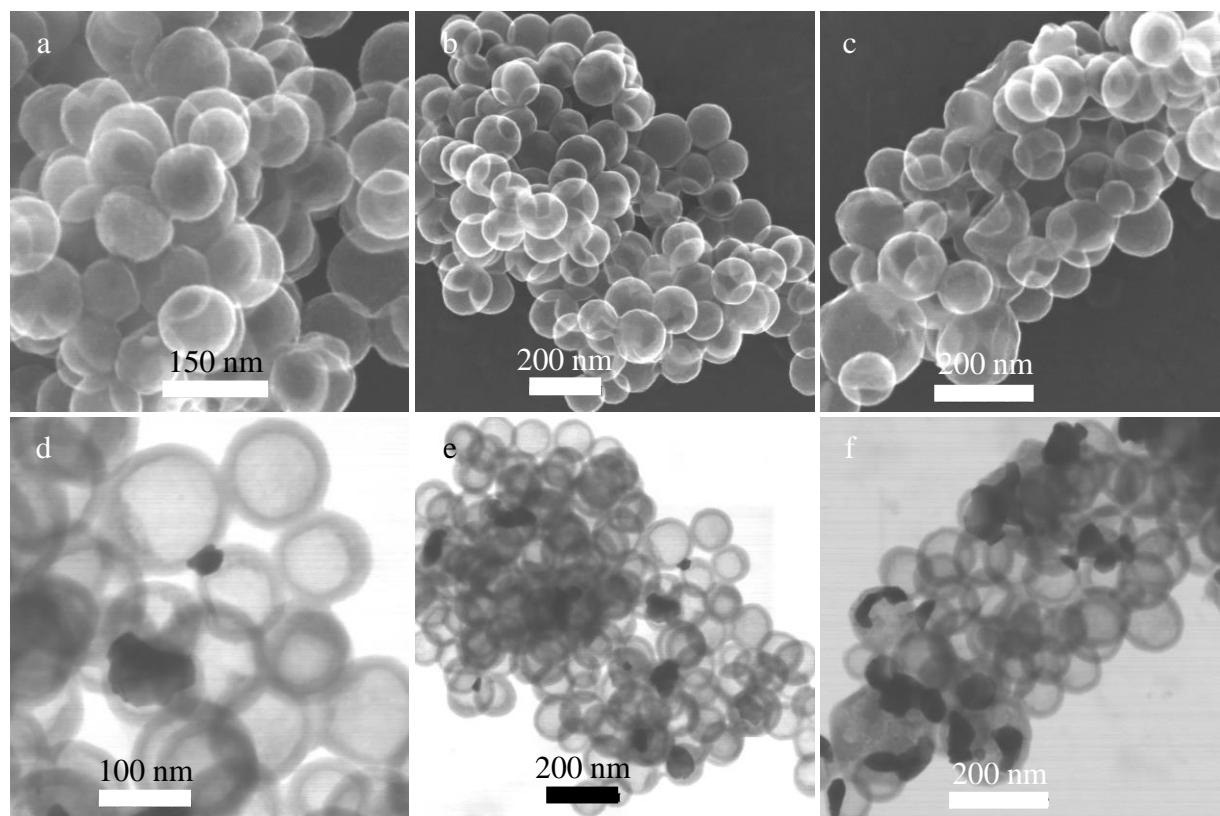


Fig. S1 (a-c) SEM and (d-f) TEM images of Ni(0)-HNCNSs-3.

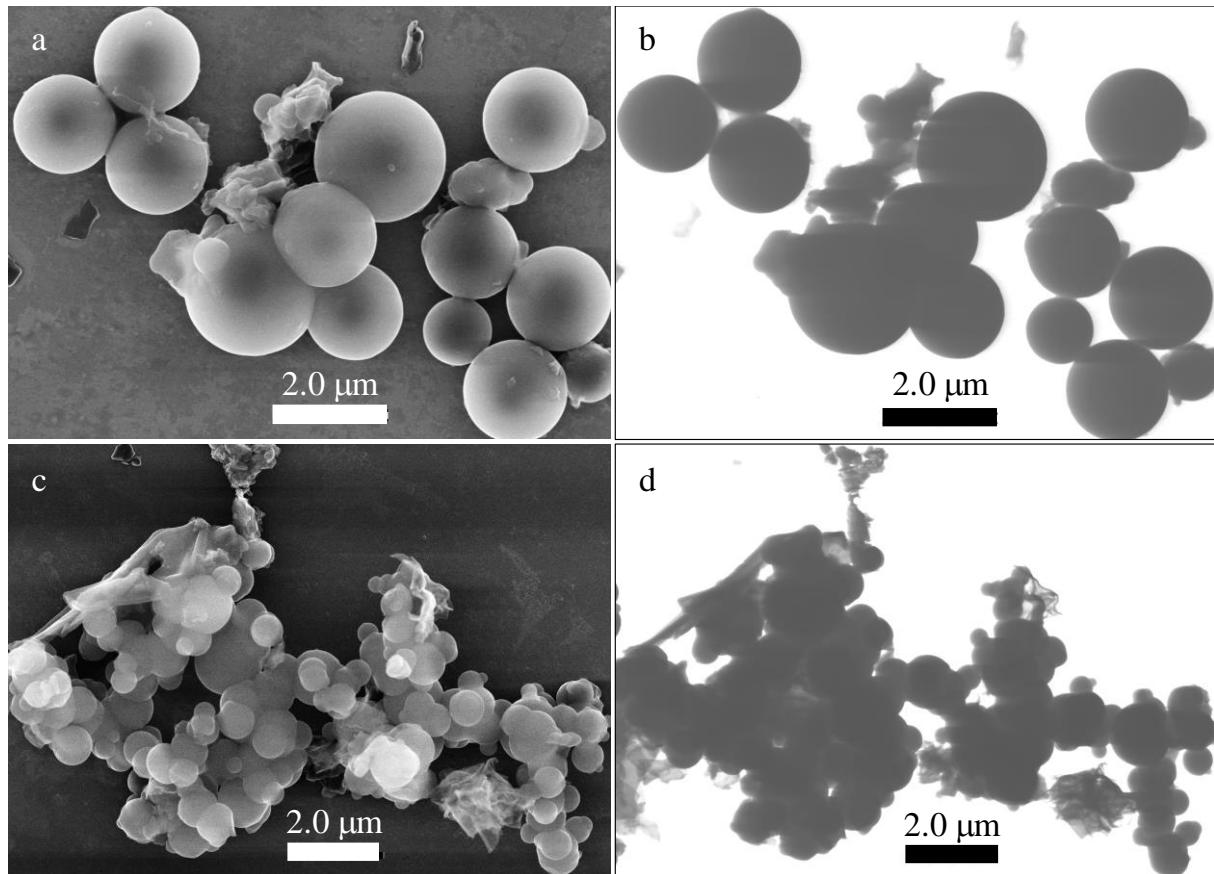


Fig. S2 SEM and TEM images of (a,b) Ni(II)-PRMSs-4 and (c,d) Ni(II)-PRMSs-5.

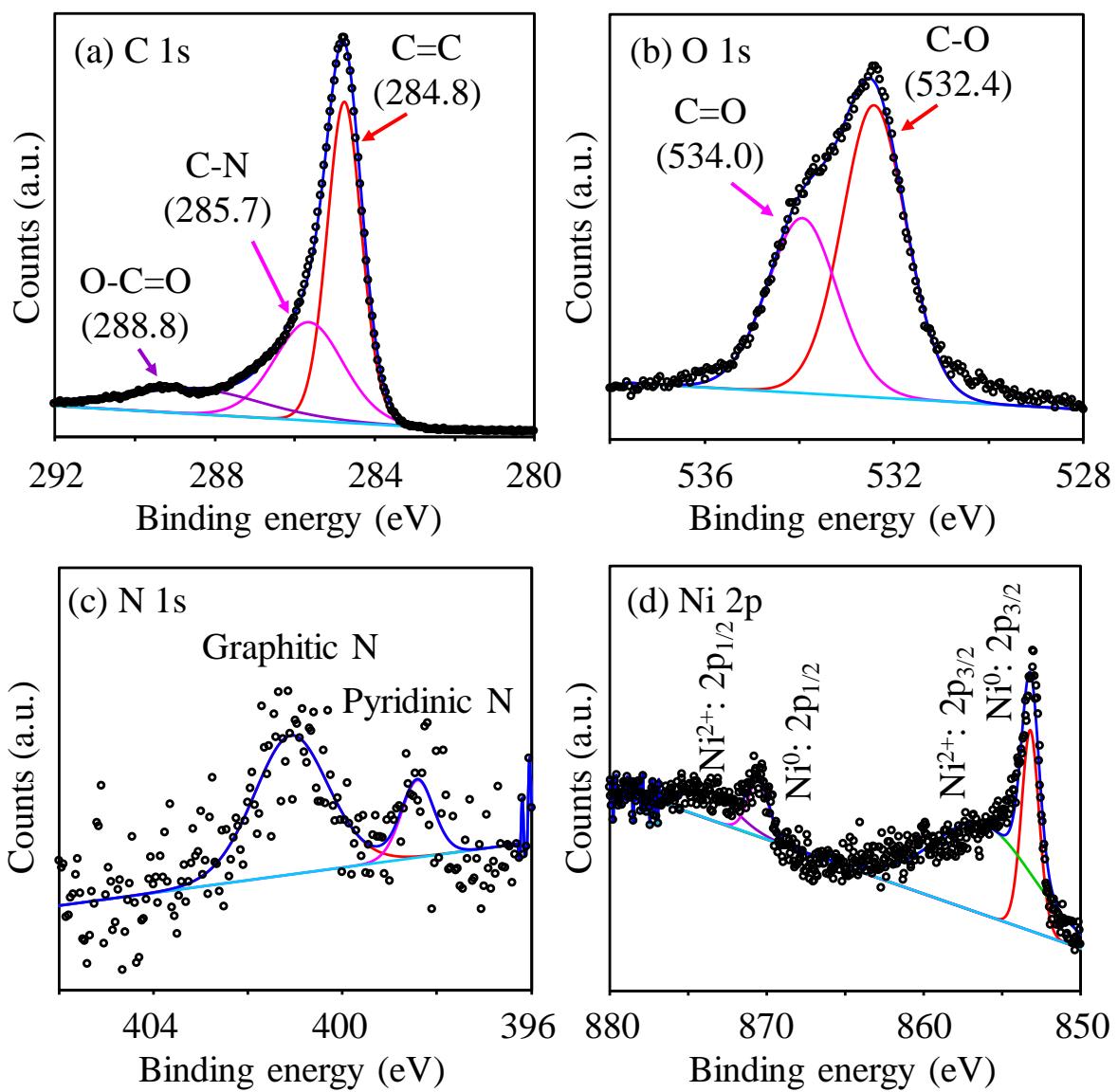


Fig. S3 High-resolution XPS spectra of (a) C 1s, (b) O 1s, (c) N 1s and (d) Ni 2p for Ni(0)-SNCMSs-5.

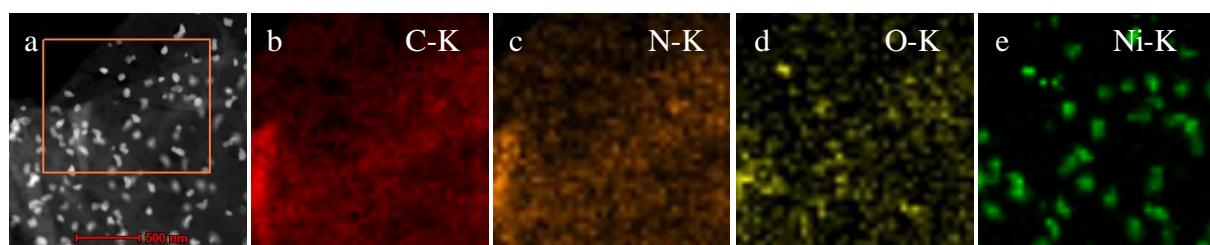


Fig. S4 (a) SEM image and EDX spectroscopic elemental mappings of (b) C, (c) N, (d) O and (e) Ni in Ni(0)-SNCMSs-5.

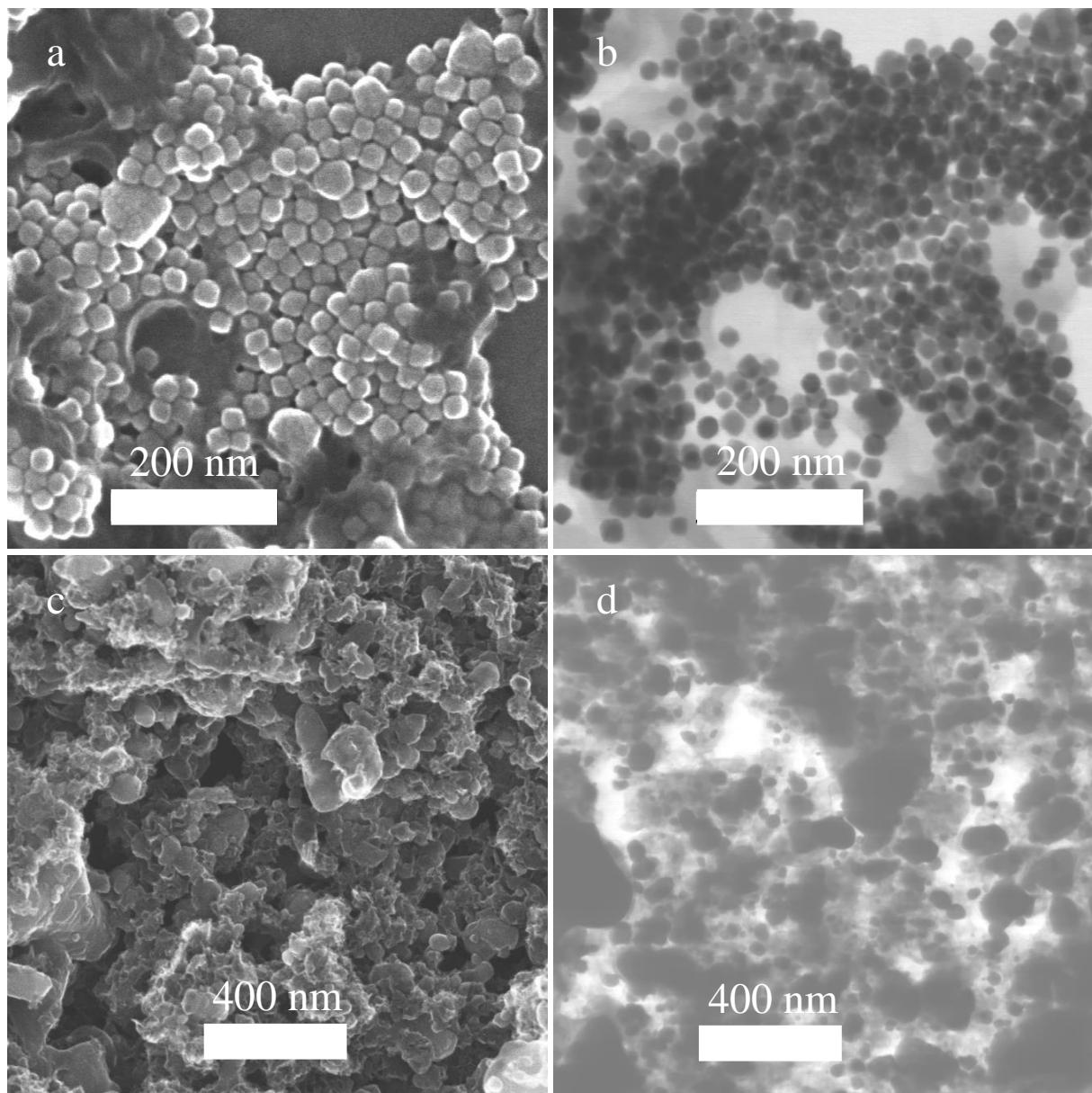


Fig. S5 SEM and TEM images of (a,b) FeO-PR and (c,d) FeO-NCC.

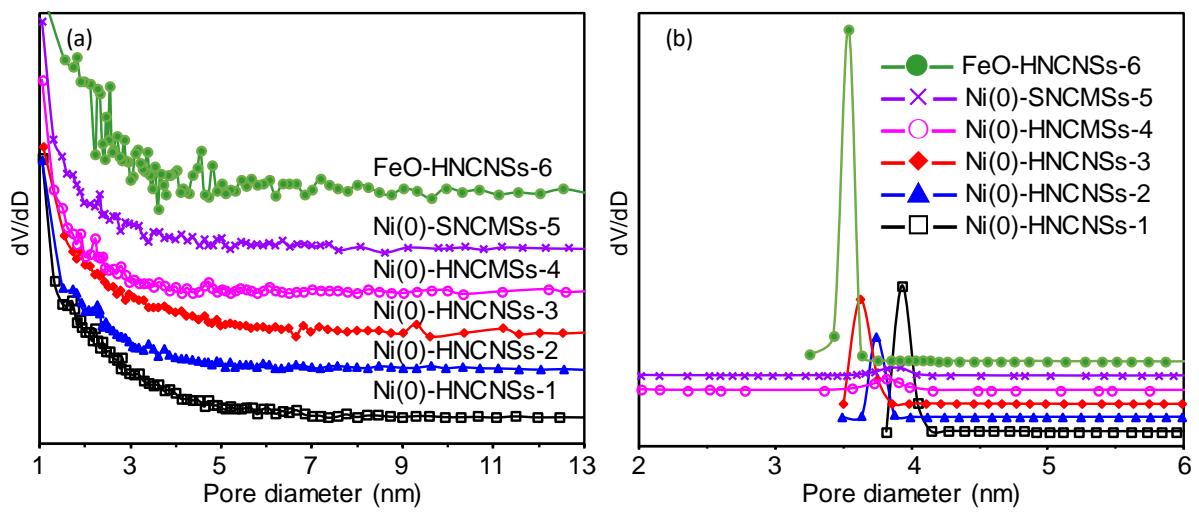


Fig. S6. The BJH pore size distributions of the samples Ni(0)-HNCNSs-n ($n = 1, 2, 3$), Ni(0)-HNCMSs-4, Ni(0)-SNCMSs-5 and FeO-HNCNSs-6 from (a) the adsorption branch of the isotherm, and (b) the desorption branch of the isotherm.