

Controllable synthesis of Ni/SiO₂ hollow spheres and excellent catalytic performance in 4-introphenol reduction

Zhongyi Niu, Shenghuan Zhang, Yanbo Sun*, Shili Gai, Fei He, Yunlu Dai, Lei Li,
and Piaoping Yang*

Table S1 Experimental conditions for the synthesis of SiO₂ particles with different sizes ^a

SiO ₂ (nm)	V(TEOS)	V(H ₂ O)	V(NH ₃)	V(C ₂ H ₅ OH)
140	14	49.8	27.9	277.2
210	14	31.4	183	119.8
350	14	28.2	164.8	106.4
580	14	4	117.8	88.2

a V: volume (mL)

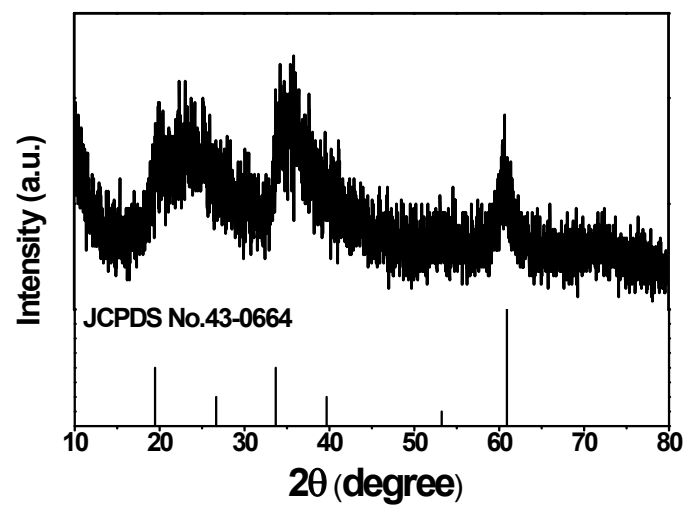


Fig. S1 XRD pattern of nickel silicate.

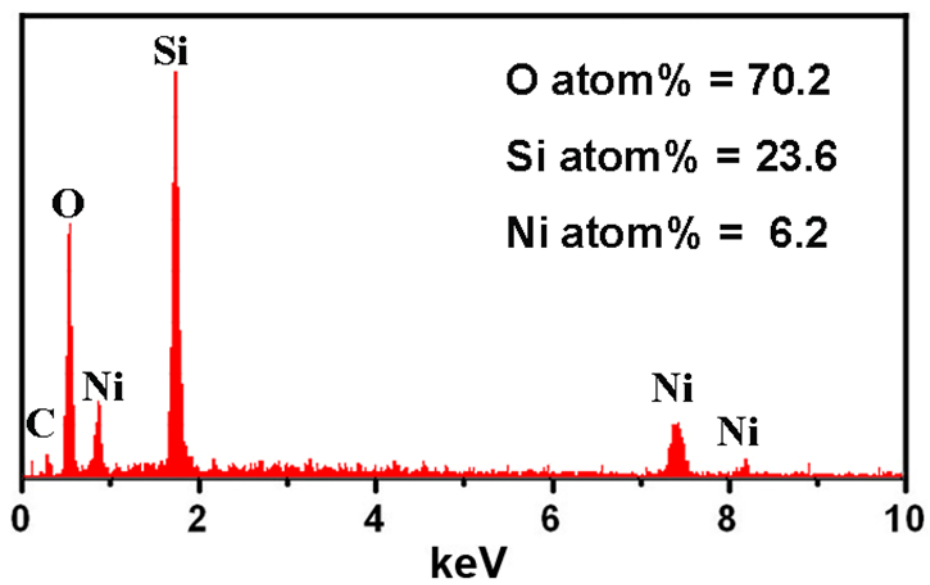


Fig. S2 EDS spectrum of nickel silicate.

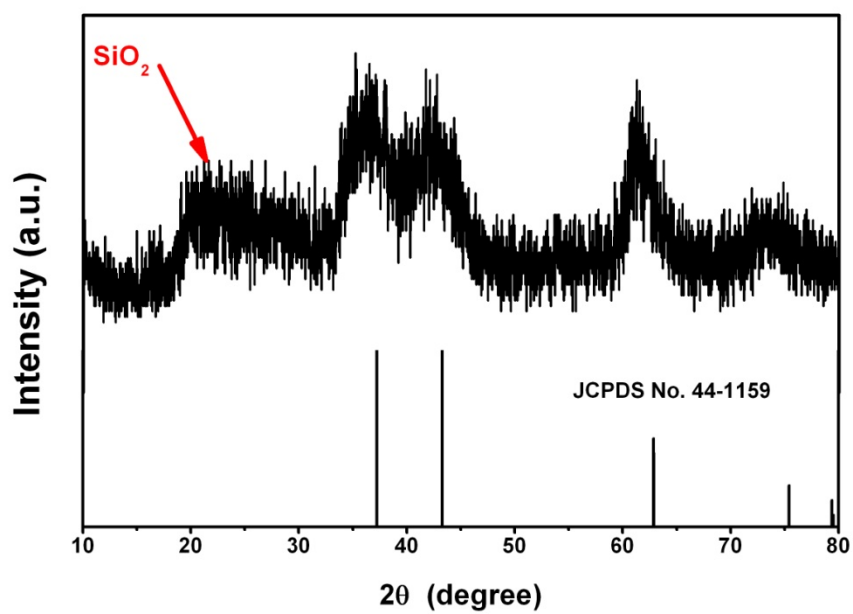


Fig. S3 XRD pattern of NiO/SiO₂ and the standard data of rhombohedral phased NiO.

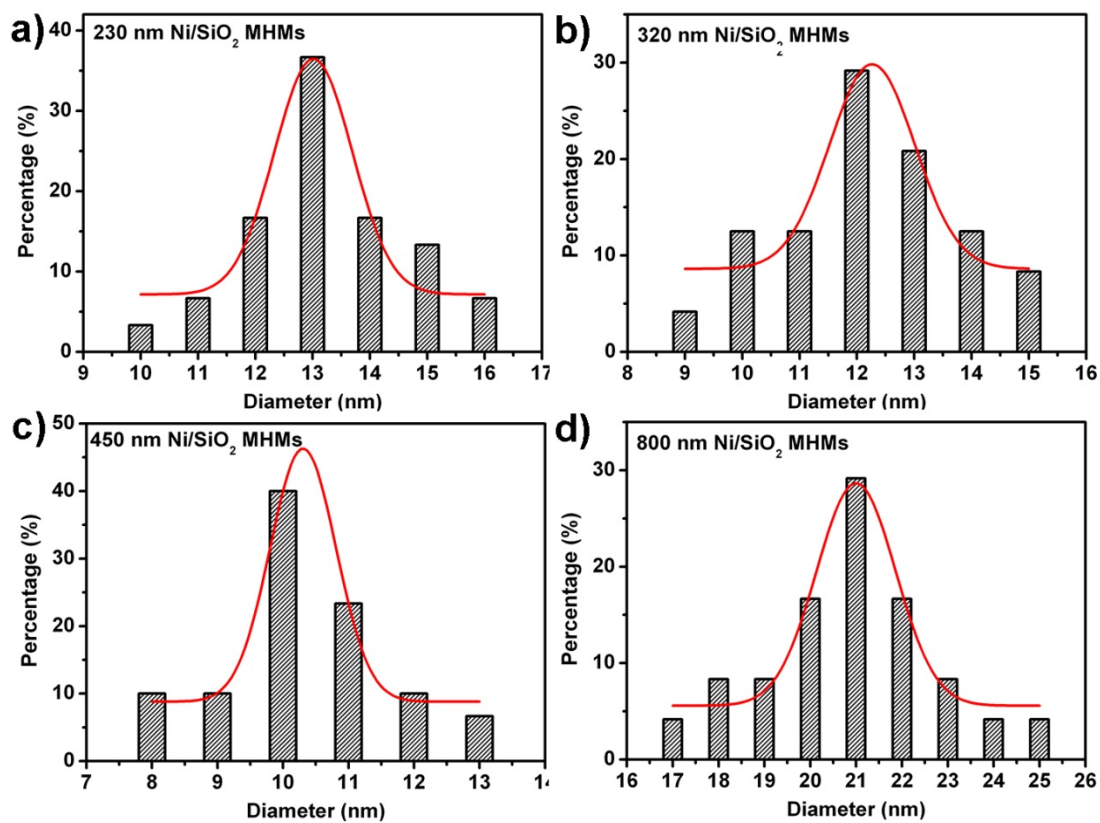


Fig. S4 Size distribution histogram of the Ni NPs calculated from a single Ni/SiO₂ MHMs with the diameter of a) 230 nm, b) 320 nm, c) 450 nm and d) 800 nm.



Fig. S5 photograph for the magnetic separation of Ni/SiO₂ MHMs.

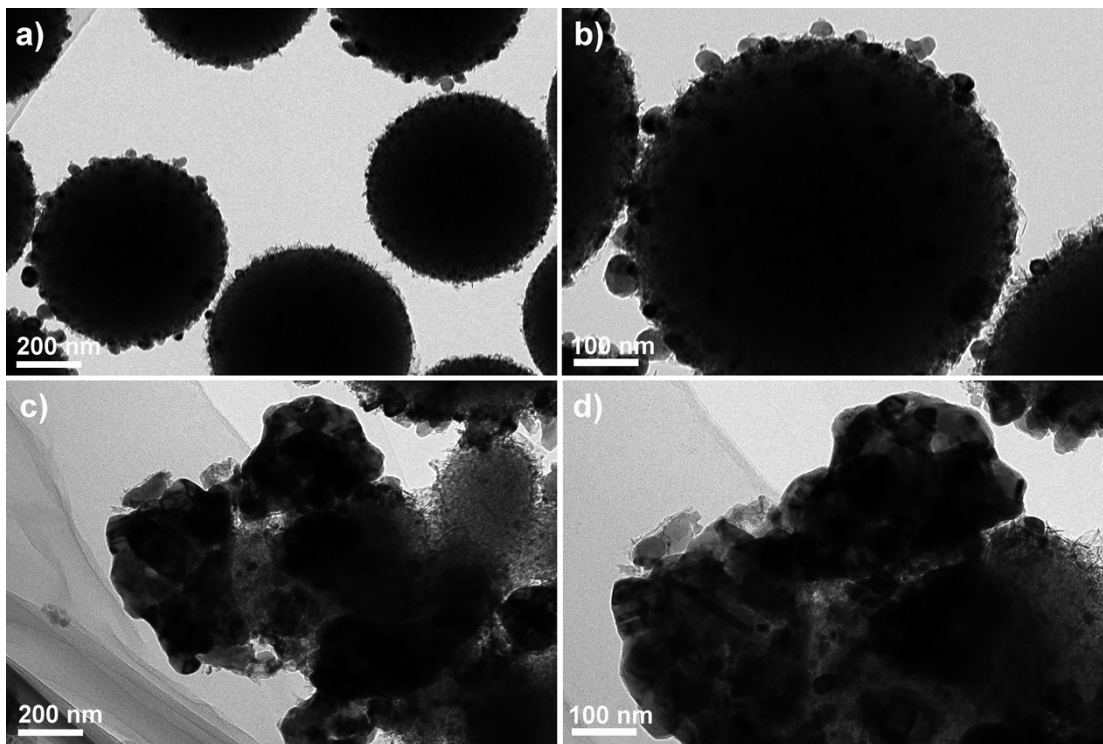


Fig. S6 TEM images of Ni/SiO₂ synthesized by wet impregnation (a, b) and bare Ni NPs synthesized by calcination and reduction of Ni(NO₃)₃ (c, d).

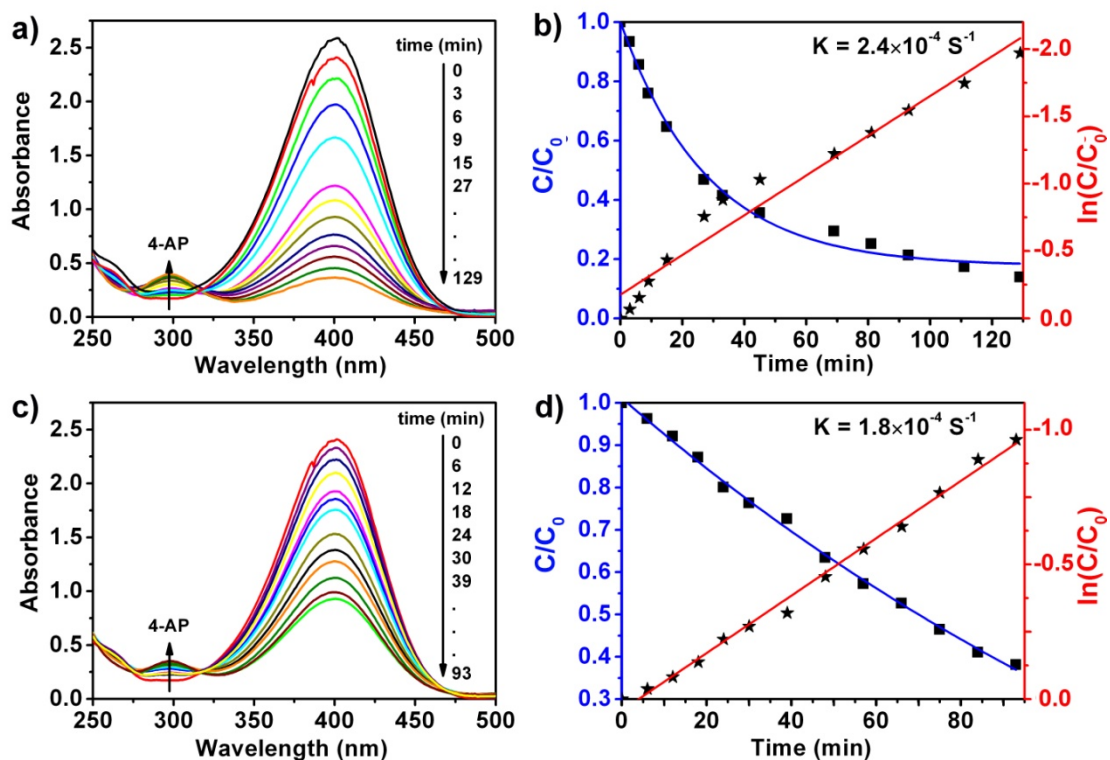


Fig. S7 UV-vis spectra of the catalytic reduction of 4-NP to 4-AP developed at different reaction times over Ni/SiO₂ synthesized by wet impregnation (a) and bare Ni NPs synthesized by calcination and reduction of Ni(NO₃)₃ (b); C/C_0 and $\ln(C/C_0)$ versus time for the reduction of 4-NP over Ni/SiO₂ synthesized by wet impregnation (c) and bare Ni NPs synthesized by calcination and reduction of Ni(NO₃)₃ (d), the ratio of 4-NP concentration (C_t at time t) to its initial value C_0 is directly represented by the relative intensity of the respective absorption peak at 400 nm.

Table S2 The ICP data of Ni/SiO₂ MHMs with different size before and after catalytic reaction.

	Ni (μg/mg)	Si (μg/mg)
230 nm Ni/SiO ₂ MHMs	14.6	24.3
320 nm Ni/SiO ₂ MHMs	16.4	29.5
450 nm Ni/SiO ₂ MHMs	13.2	27.6
800 nm Ni/SiO ₂ MHMs	14.4	25.2
230 nm Ni/SiO ₂ MHMs after recycling	12.4	25.6

For calculating the dispersion of Ni/SiO₂ HMHs, the equation can be formulated as follows (see Ref. S1 and S2)

$$\text{The number of nickel particles } N_1 = \frac{d_{Ni}}{\frac{2}{3}\pi\left(\frac{d_{Ni}}{2}\right)^2\rho_{Ni}}$$

$$\text{The overall surface area of Ni particles } S = 2\pi\left(\frac{d_{Ni}}{2}\right)^2N_1$$

$$\text{Dispersion} = \frac{N_S}{N_T} = \frac{Sk}{n_{Ni}N_A}$$

$$\text{Where } \rho_{Ni} = 8.90 \times 10^3 \text{ kg m}^{-3}$$

N_S = total number of surface nickel atoms

N_T = total number of nickel atoms

The nickel atom density (k) is $1.54 \times 10^{19} \text{ m}^{-2}$

$$N_A = 6.02 \times 10^{23} \text{ mol}^{-1}$$

$$n_{Au} = m_{Au}/M_{Au}$$

Therefore, the equation can be written as:

$$\text{Dispersion} = \frac{10.06}{d_{Ni}} = \frac{5.03}{r_{Ni}}$$

References

- 1 J. R. Anderson, *Structure of Metallic Catalysts*, Academic Press, 1975
- 2 Q. Bi, X. Du, Y. Liu, Y. Cao, H. He and K. Fan, *J. Am. Chem. Soc.*, 2012, **134**, 8926.