Supporting Information

Hollow Mesoporous Ceria Nanoreactors with Enhanced Activity and Stability for Catalytic Application

Baocang Liu,[a,b] Shengli Yun,[a] Qin Wang,[a] Wenting Hu,[a] Peng Jing,[a] Yang Liu,[a] Wenjing Jia,[a] Yongxin Liu,[a] Lixia Liu,[a] and Jun Zhang[a,b]*

[a] College of Chemistry and Chemical Engineering, Inner Mongolia University, Hohhot 010021, P. R. China
[b] College of Life Science, Inner Mongolia University, Hohhot 010021, P. R. China

1. Experimental section

Synthesis of amino-modified SiO_2 spheres: The monodispersed SiO_2 spheres were synthesized by a Stöber sol-gel method. Typically, 280 mL of absolute ethanol was mixed with 8 mL of tetraethylorthosilicate (TEOS). A mixture of water (56 mL) and ammonia solution (8.4 mL) was injected into the above solution at room temperature under magnetic stirring in a 500 mL beaker. After reacting for 24 h, the colloidal spheres were collected by centrifugation. The spheres were washed three times with deionized water and absolute ethanol, respectively, and then finally dried at 65 ^oC for 2 h. The SiO_2 spheres (1 g) were dispersed into absolute ethanol (30 mL), and APTMS (1 mL) was added and stirred at room temperature for 12 h to ensure the amino groups were decorated on the surface of SiO_2 spheres. The white precipitates were collected by centrifugation and washed with ethanol. The particles were then dried at 65 ^oC for 6 h.

*Synthesis of Au/SiO_2, Pd/SiO_2, and Au-Pd/SiO_2 spheres:*The Au/SiO_2, Pd/SiO_2, and Au-Pd/SiO_2 spheres were prepared via the colloidal deposition. In a typical procedure, $HAuCl_4$ solution (1 g/L, 12.6 mL) and polyvinyl alcohol (PVA) solution (1 wt%, 0.75 mL) were diluted to 100 mL with deionized water under vigorous stirring. After 30 min, $NaBH_4$ solution (0.1 mol/L, 1.5 mL) was injected into the above solution, and a dark orange-brown solution was obtained, indicating that the Au colloids were formed. After another 30 min, SiO_2-NH_2 (0.2 g) was added immediately. After stirring for 12 h, the Au colloids were completely absorbed, as indicated by the discoloration of the solution. The Au/SiO_2 was collected by centrifugation, and washed more than five times with deionized water to completely remove the chloridion. For the synthesis of Pd/SiO_2, all the experimental conditions were kept except replacing the $HAuCl_4$ solution (1 g/L, 12.6 mL) with $PdCl_2$ solution (0.5 g/L, 20 mL). For Au-Pd/SiO_2 spheres, the volumes of $HAuCl_4$ solution and $PdCl_2$ solution were 6.3 and 10 mL, respectively.

Synthesis of @M/CeO_2 (M = Au, Pd, and Au-Pd) spheres: Sol-type ceria coating precursor was prepared by a peptization method. Typically, 1 g of $Ce(NO_3)_3{\cdot}6H_2O$ and 1 g of NaOH were separately dissolved in 20 mL of ethanol under vigorous stirring for 24 h at 50°C. H_2O_2 (30%, 0.05 mL) was added to above solution as the oxidizing agent. After centrifugation and washing with distilled water, 1 g of the precipitate was redispersed in 20 mL distilled water under continuous stirring. The pH of the suspension solution was adjusted to 0.1 by adding concentrated HNO_3 to obtain a peptized ceria sol. The solution was heated to 40°C and kept for 2 h. The transparent light yellow solution obtained was cooled to room temperature. Then, 0.4 g of M/SiO_2 was dispersed in 30 mL of distilled water. Subsequently, 3 mL of ceria precursor was added. After stirring for 30 min, the pH of solution was adjusted to 6.8 by adding ammonium hydroxide solution, followed by heating to 60°C, for 4 h. When cooling to room temperature, the mixture solution was centrifuged to obtain the SiO_2/M@CeO_2

spheres, then washed with water and ethanol, respectively. After drying at 60°C, the prepared SiO_2/M@CeO_2 was calcined at 500°C for 2 h. Next, 0.4 g of SiO_2/M@CeO_2 spheres was dissolved in 40 mL of NaOH aqueous solution (5 mol/L). The @M/CeO_2 was obtained after etching for 4 h, and washed five times with distilled water and dried at 60°C for 6 h.

For comparing the catalytic activity, the Au and Pd nanoparticles sols, @CeO_2, @CeO_2/Au, Au/CeO_2, and Pd/CeO_2 catalysts were also prepared. For the preparation of Au NPs, $HAuCl_4$ solution (1 g/L, 5 mL) and PVA solution (1 wt%, 0.3 mL) were diluted to 25 mL with deionized water under vigorous stirring. After 30 min, $NaBH_4$ solution (0.1 mol/L, 0.6 mL) was injected into the above solution, and a dark orange-brown solution was obtained, which indicated that the Au colloids were formed. For the synthesis of Pd NPs, similar synthetic procedures were followed, replacing the $HAuCl_4$ solution with $PdCl_2$ solution (0.5 g/L, 8 mL). For the preparation of @CeO_2/Au, 1 g/L of $HAuCl_4$ solution (12.6 mL) and, 0.75 mL of PVA solution (1 wt%) were diluted to 100 mL with deionized water under vigorous stirring. After 30 min, $NaBH_4$ solution (0.1 mol/L, 1.5 mL) was injected into the above solution, and a dark orange-brown solution was obtained, which indicated that the Au colloids were formed. After another 30 min, the @CeO_2 support (0.2 g, acidified by 0.1 M of sulfuric acid) was added immediately. After stirring for 12 h, the Au colloids were completely absorbed, as indicated by the discoloration of the solution. The @CeO_2/Au was collected by centrifugation and washed more than five times with deionized water to completely remove the chloridion. Finally, the @CeO_2/Au was dried at 65 °C for 6 h. For the synthesis Au/CeO_2 and Pd/CeO_2 catalysts, the CeO_2 support was prepared using co-precipitation route according to the previous report with slightly modified.[1] Typically, to a 250 mL baker containing $Ce(NO_3)_3$

solution (0.05 M, 100 mL), Na_2CO_3 solution (0.5 M) was added dropwise under vigorously stirring to adjust the pH of the suspension to 9 until the complete precipitation of cerium ions. Then, the precipitates were recovered by centrifugation, and washed with hot deionized water for five times. Next, the precipitates were dried in oven at 80 ^{o}C for 12 h. Before the noble nanoparticle loading, the precipitates were acidified using 0.1 M of sulfuric acid. Following the synthetic procedures for @CeO_2/Au catalyst, the Au/CeO_2 and Pd/CeO_2 catalysts were obtained. Finally, the catalysts were calcined in furnace at 500 ^{o}C for 2 h.

2. Characterization

Powder X-ray diffraction (XRD) was used to characterize the phase structures of the samples. Measurements were performed using a PuXi XD3 diffractometer (China) operated at 36 kV and 20 mA with a slit of 1/2 at a scanning rate of 4° min^{-1} in a scanning range of 2θ = 10–80°, using Cu Ka radiation (λ = 0.15406 nm). Transmission electron microscopy (TEM) characterization was performed on a JEM-2010 system and a HITACHI 800 system operated at acceleration voltages of 120 kV and 200 kV, respectively. The samples for the TEM measurements were suspended in ethanol and supported onto a holey carbon film on a Cu grid. Scanning electron micrographs were recorded with a JEOL-JSM 6700-F field emission scanning electron microscope (FE-SEM). Samples for SEM measurements were deposited on silicon substrates and coated with 5 nm of Pt for characterization. Actual Au and Pd contents in the catalysts were estimated with a VARIAN VISTA-MPX inductively coupled plasma-mass spectroscope (ICP-MS, USA). The absorption spectra were recorded by a SPECORD.50 ultraviolet-visible (UV-vis) spectrophotometer (Analytikjena).

3. Catalytic tests of the reduction of 4-Nitrophenol over @Au/CeO_2, @Pd/CeO_2, @Au-Pd/CeO_2, @CeO_2/Au, @CeO_2/Pd, @CeO_2, Au/CeO_2, Pd/CeO_2 catalysts, and Au and Pd sols

In order to activate the performance of the catalysts, all the catalysts were treated under 500°C for 2 h, and reduced by $NaBH_4$ (0.5 M) before the catalyst test. The catalytic performance of the catalysts was checked using the reduction of 4-nitrophenol (4-NP) to 4-aminophenol (4-AP) with $NaBH_4$ aqueous solution at room temperature as a model reaction. The @Au/CeO_2, @Pd/CeO_2, @Au-Pd/CeO_2, @CeO_2/Au, @CeO_2/Pd, @CeO_2, Au/CeO_2, Pd/CeO_2, Au and Pd sol catalysts were utilized for the catalytic tests. Typically, the catalyst (1 mg) for @Au/CeO_2, @Pd/CeO_2, @Au-Pd/CeO_2, @CeO_2/Au, @CeO_2/Pd, @CeO_2, Au/CeO_2, and Pd/CeO_2 and 0.5 mL of Au and Pd sols were added into millipore water (40 mL) to form a homogeneous suspension under ultrasonication. Then, $NaBH_4$ aqueous solution (0.5 M, 0.5 mL) was added into the above suspension, and the suspension was stirred at room temperature for 10 min. Then, 4-NP (0.012 M, 0.25 mL) was infused into the above suspension. After stirring for several seconds, the mixture was rapidly transferred to the quartz cell to monitor the reaction progress. The UV-vis absorption spectra of the mixture was measured to evaluate the catalytic activity and stability of the catalysts, as the reactant of 4-NP has a strong absorption peak at 400 nm, while the product of 4-AP has a median absorption peak at 295 nm.

Table S1. Actual Au and Pd contents loaded in different catalysts and their corresponding values of turnover frequency (TOF).

Catalysts	Actual content of Au (wt%)	Actual content of Pd (wt%)	Turnover frequency TOF (h^{-1})[a]
Au/SiO_2	2.73	—	108
Pd/SiO_2	—	0.85	161
Au-Pd/SiO_2	0.93	0.84	178
SiO_2/Au@CeO_2	1.63	—	73
SiO_2/Pd@CeO_2	—	0.07	547
SiO_2/Au-Pd@CeO_2	0.65	0.15	96
@Au/CeO_2	2.97	—	240
@Pd/CeO_2	—	0.18	1068
@Au-Pd/CeO_2	1.10	0.38	281
@CeO_2/Au	3.12	—	101
@CeO_2/Pd	—	1.12	171
Au/CeO_2	2.26	—	98
Pd/CeO_2	—	1.08	253
CeO_2	—	—	0
Au sol	—	—	126
Pd sol	—	—	187

[a] The TOF is defined as the moles of reduced 4-NP molecules per mole of noble metal atoms in the oxide microspheres per hour. When M = Au-Pd, the TOF is calculated by the moles of product divided by the sum moles of Au and Pd.

Table S2. Recent studies on the reduction of 4-NP over Au nanocatalytsts.

Catalyst support/template*	Molar ratio			size of Au (nm)	K_{app} (s^{-1})	k per Au content (s^{-1} μmol Au^{-1})	TOF (h^{-1})	Ref.
	NaBH$_4$	4-NP	Au					
GO	874	38	1	20	1.9×10^{-1}	1.0	126	2
4,4-bpy	2,000	20	1	50.9	7.2×10^{-4}	9.6×10^{-2}	19	3
PDDA/NCC	36,585	37	1	2.95	5.1×10^{-3}	1.2×10^{-1}	212	4
Boehmite	36	0.36	1	15-40	1.7×10^{-3}	2.1×10^{-6}	0.69	5
PANI	250	57	1	2	1.2×10^{-2}	2.0×10^{-1}	570	6
GO/SiO$_2$	12,000	60	1	2-5	1.7×10^{-2}	8.3	1,028	7
SNTs	150	3.6	1	3-5	1.1×10^{-2}	1.1×10^{-2}	46	8
PNIPAP-b-P4VP	167	5	1	3.3	1.5×10^{-3}	2.5×10^{-2}	16	9
PDMAEMA-PS	28	0.14	1	4.2	3.2×10^{-3}	2.3×10^{-3}	1	10
Poly(DVP-co-AA)	9,800	267	1	10	6.0×10^{-3}	4.1×10^{-2}	222	11
Chitosan	20	6	1	3.1	1.2×10^{-2}	1.0	50	12
CSNF	15,000	150	1	< 5	5.9×10^{-3}	5.9×10^{-1}	563	13
PMMA	22,500	15	1	6.9	7.2×10^{-3}	7.5×10^{-2}	89	14
DMF	200,000	100	1	~20	3×10^{-3}	3×10^{-3}	83	15
SiO$_2$	2,755	9.4	1	3.3 ± 0.7	1×10^{-3}	8×10^{-2}	14	16
PAMAM	1,667	98	1	2.3 ± 0.8	2×10^{-3}	1.6×10^{-2}	196	17
EGCG-CF	1,320	1	1	10.7 ± 3.8	2.4×10^{-3}	2.4×10^{-3}	2	18
Biomass	1,316	20	1	25.3 ± 0.37	4.6×10^{-4}	8.9×10^{-5}	20	19
TWEEN/GO	37	1.6	1	6 - 15	4.2×10^{-3}	9.8×10^{-4}	7	20
HPEI-IBAm	1,055	10.5	1	14	-	-	120	21
graphene hydrogel	164	2.3	1	14.6	3.2×10^{-3}	2.6×10^{-2}	12	22
ZnO	300	0.3	1	4.3 ± 2.1	2.4×10^{-3}	4.8×10^{-4}	3	23
αCD	250	6	1	11	4.7×10^{-3}	7.8×10^{-2}	34	24
Peptide	133	0.54	1	172 ± 2	1.3×10^{-2}	5.5×10^{-2}	7	25
PC/PEI/PAA	6,100	3.8	1	16 ± 2	7×10^{-3}	2.0×10^{-2}	33	26
MPFs	8,000	20	1	4.0	3×10^{-3}	1.2	80	27
P(AN-co-VA)-g-PEDOT	108	0.27	1	-	6.1×10^{-4}	2.2×10^{-5}	0.32	28
Lysozyme	83	1.7	1	17	6.5×10^{-4}	5.4×10^{-5}	2	29
RF	250	2.5	1	8.0	7.4×10^{-3}	1.2×10^{-1}	15	30
P2VP	200	10	1	4.2 ± 0.7	8.0×10^{-3}	7.9×10^{-2}	200	31
SiO$_2$	200	1	1	7.9 ± 2.6	9.1×10^{-4}	3.6×10^{-4}	1.2	32
CSPQ	1,000	10	1	2 - 5	-	-	10	33
PEGDMA	3,750	0.15	1	23	-	-	0.7	34
β-d-Glucose	75	1	1	8.2 ± 2.3	6.5×10^{-3}	1.1×10^{-1}	10	32
P(AA–DVB)–Fe$_3$O$_4$	0.81	1.63	1	5	1.1×10^{-2}	9×10^{-2}	2	33
Hollow SiO$_2$	750	2.1	1	43 ± 7	3.9×10^{-3}	2.4×10^{-3}	6	34
SiO$_2$	1,000	10	1	21			38	35
@Au/CeO$_2$	1,667	20	1	3-5	1.3×10^{-2}	9.6×10^{-2}	240	This work

* GO: graphene oxide, 4,4-bpy: 4,4-bipyridine, PAMAM: poly(methylacrylate)-b-poly(ethylenediamine), SNTs: silica nanotubes, NCC: nanocrystalline cellulose, Boehmite: AlOOH, PANI: Polyaniline, PNIPAP-b-P4VP: poly(N-isopropylacrylamide)-b-poly(4-vinylpyridine), Poly(DVP-co-AA): Poly(divinylbenzene-co-acrylic acid), PDMAEMA-PS:Poly(2-(dimethylamino)ethyl methacrylate) grafted onto solid polystyrene core, EGCG-CF: Epigallocatechin-3-gallate- collagen fiber, TWEEN:polyoxyethylene sorbitol anhydride monolaurate, HPEI-IBAm: Polysciences-b-benzoylated cellulose membrane, PC/PEI/PAA: polycarbonate/ poly(ethylenimine)/ poly(acrylic acid) sodium salt, MPFs: multilayer

polyelectrolyte films, P(AN-co-VA)-g-PEDOT: poly(acrylonitrile-co-vinyl acetate) -graft-poly(3,4-ethylenedioxythiophene), RF: phloroglucinolcarboxylic acid-formaldehyde, CA: Calcium-Alginate, P2VP: Poly(2-vinylpyridine), CSPQ: cyanopropyl polysilsesquioxane, PEGDMA: poly(ethyleneglycol dimethacrylate), P(AA–DVB): poly(acrylic acid–divinylbenzene)

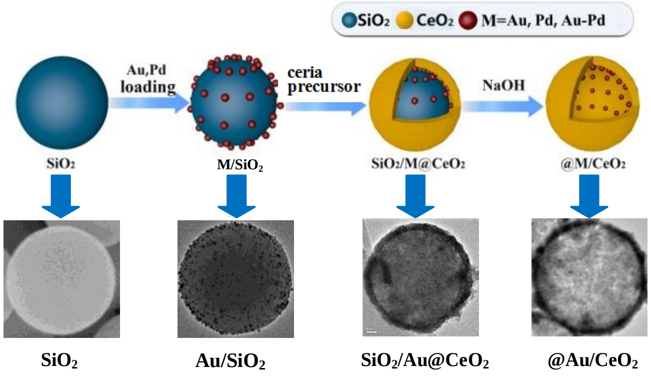
Table S3. Recent studies on the reduction of 4-NP over Pd nanocatalysts.

Catalyst support/template*	**Molar ratio**			**Size of Pd (nm)**	**K_{app} (s^{-1})**	**k per Au content (s^{-1} µmol Pd^{-1})**	**TOF (h^{-1})**	**Ref.**
	$NaBH_4$	**4-NP**	**Pd**					
CNT/PiHP/Pd	2,000	25	1	2.7, 51	5×10^{-3}	5×10^{-2}	300	36
Fe_3O_4	1,390	10	1	6.9 ± 1.3	3.3×10^{-2}	1.0×10^{-1}	300	37
PEDOT-PSS-	excess	1.3	1	<9	6.58×10^{-2}	8.75×10^{-2}	13	38
SPB	27,322	273	1	2.4 ± 0.5	4.41×10^{-3}	4	819	39
Microgels	4,651	46.5	1	3.8 ± 0.6	1.5×10^{-3}	2.34×10^{-1}	139	40
PPy/TiO_2	419	38	1	2.0	1.22×10^{-2}	2.08	326	41
SBA-15	1,000	1	1	~8	1.2×10^{-2}	4×10^{-1}	6	42
@Pd/CeO_2	14,836	178	1	3-5	8×10^{-3}	4.7×10^{-1}	1,068	This work

***** CNT: carbon nanotubes, PiHP: hyperbranched polymers, SPB: Spherical Polyelectrolyte Brushes, PEDOT: poly-(3,4)ethylenedioxythio-phene

Table S4. The leaching content of Au and Pd for @Au/CeO_2, @Pd/CeO_2, and @CeO_2/Pd catalyst before and after eight cycles of tests determined by ICP-MS.

The contents of Au or Pd	Before tests	After eight cycles of tests
The content of Au in @Au/CeO_2 catalysts/wt%	2.97	2.75
The content of Au in mother liquor/ppb	0	40
The content of Pd in @Pd/CeO_2 catalysts/wt%	0.18	0.16
The content of Pd in mother liquor/ppb	0	4
The content of Pd in @ CeO_2/Pd catalysts/wt%	1.12	0.96
The content of Pd in mother liquor/ppb	0	68



Scheme S1. (Upper) Schematic illustration of the synthetic procedures for @M/CeO_2 (M = Au, Pd and Au-Pd) hollow nanospheres. (Lower) The corresponding SEM image of a SiO_2 sphere and TEM images of Au/SiO_2, SiO_2/Au@CeO_2, and @Au/CeO_2 spheres.

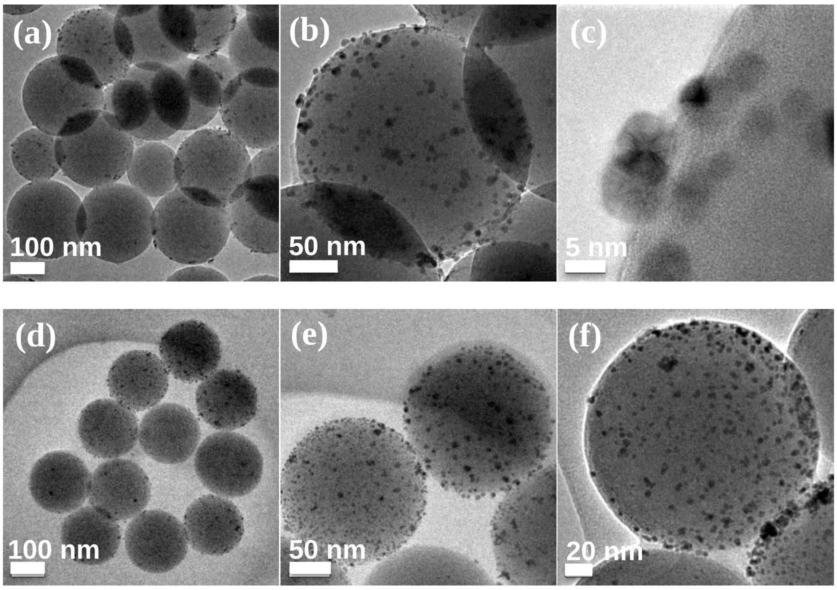


Figure S1. (a-c) TEM images of Pd/SiO_2 nanospheres, and (d-f) TEM images of Au-Pd/SiO_2 nanospheres in different magnification.

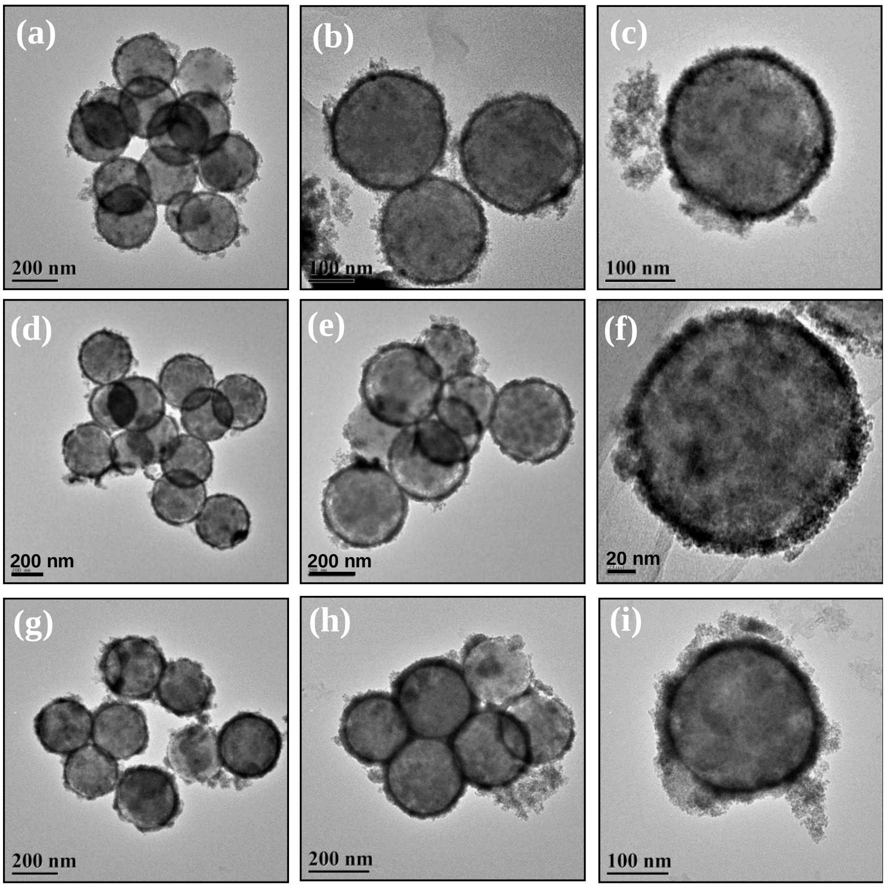


Figure S2. TEM image of (a-c) $SiO_2/Pd@CeO_2$, (d-f) $SiO_2/Au@CeO_2$, and (g-i) $SiO_2/Au\text{-}Pd@CeO_2$ nanospheres under different magnifications.

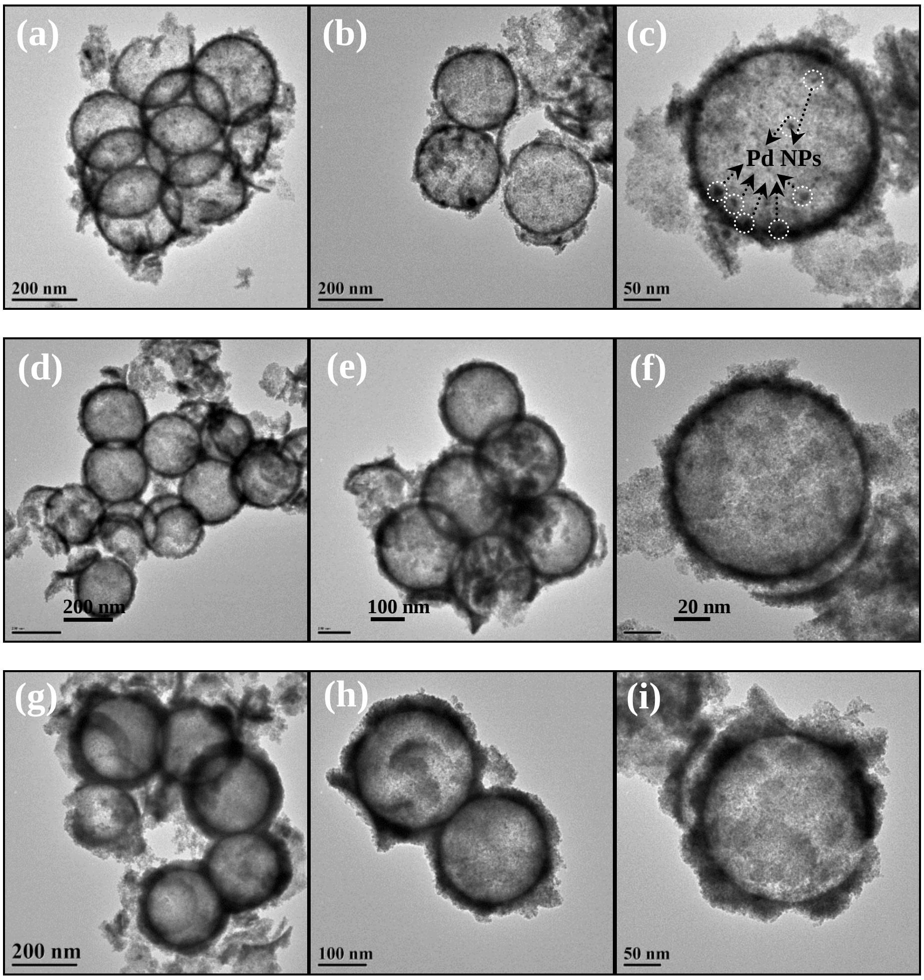


Figure S3. TEM image of (a-c) @Pd/CeO_2, (d-f) @Au/CeO_2, and (g-i) @Au-Pd/CeO_2 nanospheres under different magnification. The white cricles marked in Figure S3c indicate the existence of Pd nanoparticles closely contact with inner wall of CeO_2 shells.

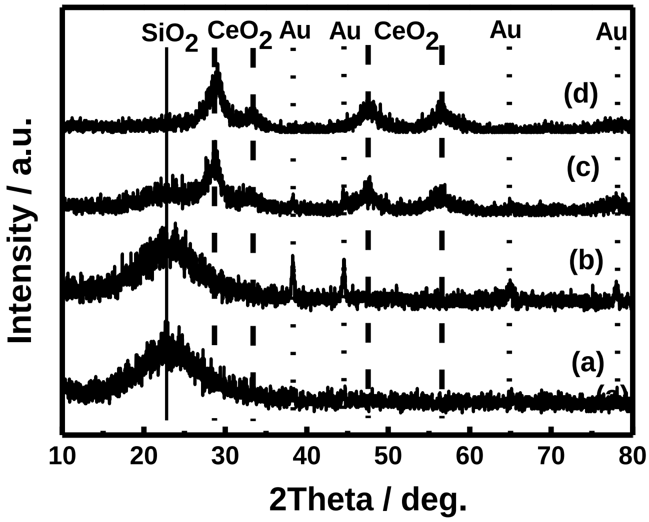


Figure S4. XRD patterns of (a) SiO_2, (b) Au/SiO_2, (c) SiO_2/Au@CeO_2, and (d) @Au/CeO_2 nanospheres

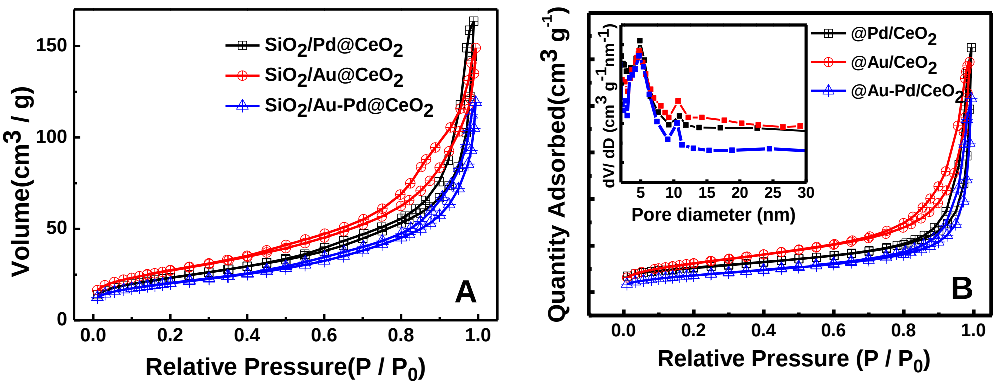


Figure S5. Nitrogen adsorption–desorption isotherms of (A) SiO_2@Pd/CeO_2, SiO_2@Au/CeO_2, and SiO_2@Au-Pd/CeO_2 catalysts, and (B) @Pd/CeO_2, @Au/CeO_2, and @Au-Pd/CeO_2 catalysts. Inset is the BJH pore size distribution.

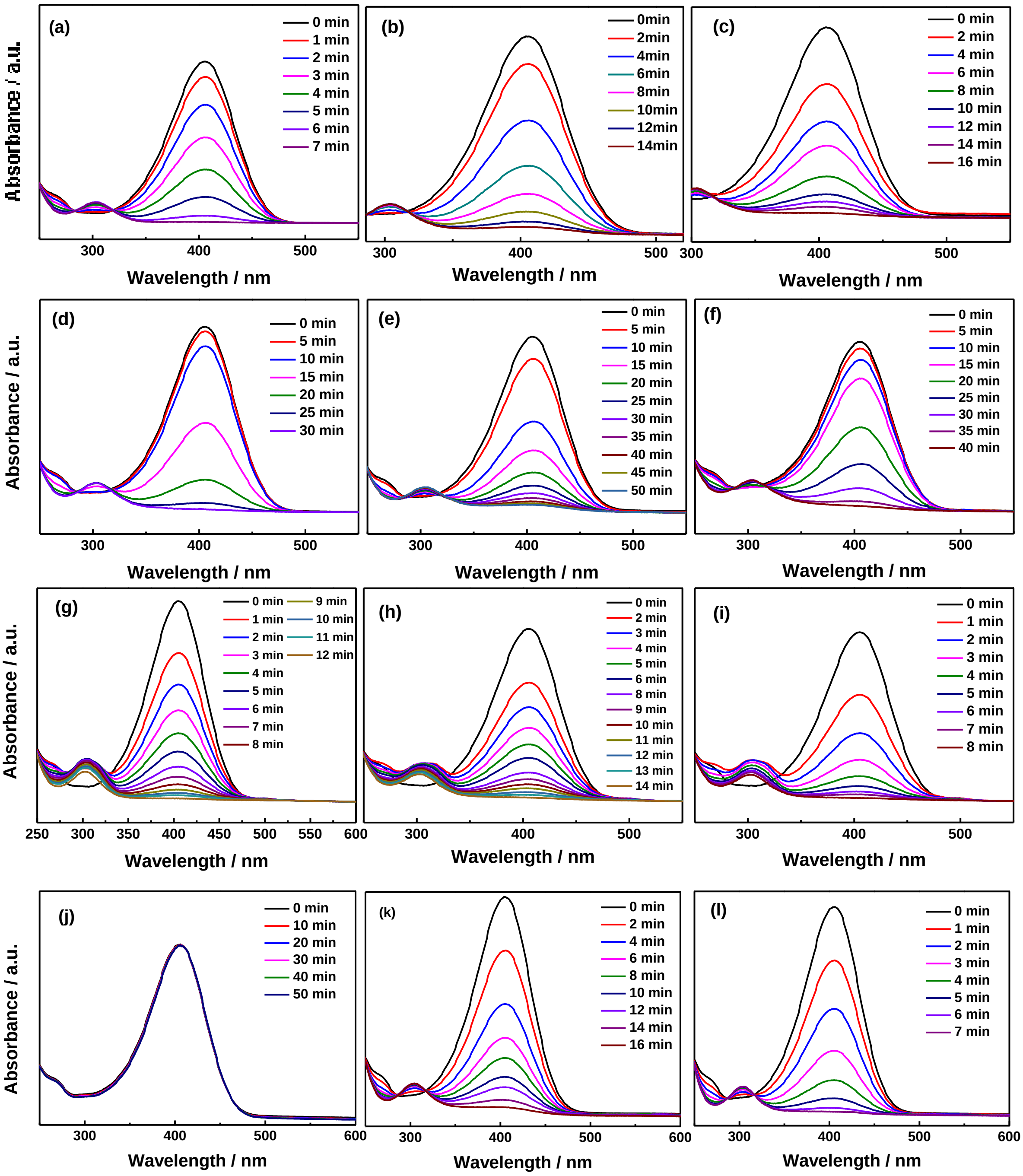


Figure S6. UV-vis spectra of catalytic reduction of 4-NP to 4-AP over various catalysts: (a) @Au-Pd/CeO_2, (b) @CeO_2/Au-Pd, (c) @CeO_2/Au, (d) SiO_2/Au@CeO_2, (e) SiO_2/Pd@CeO_2, (f) SiO_2/Au-Pd@CeO_2, (g) Au/SiO_2, (h) Pd/SiO_2, (i) Au-Pd/SiO_2, (j) @CeO_2, and (k) Au/CeO_2, and (l) Pd/CeO_2.

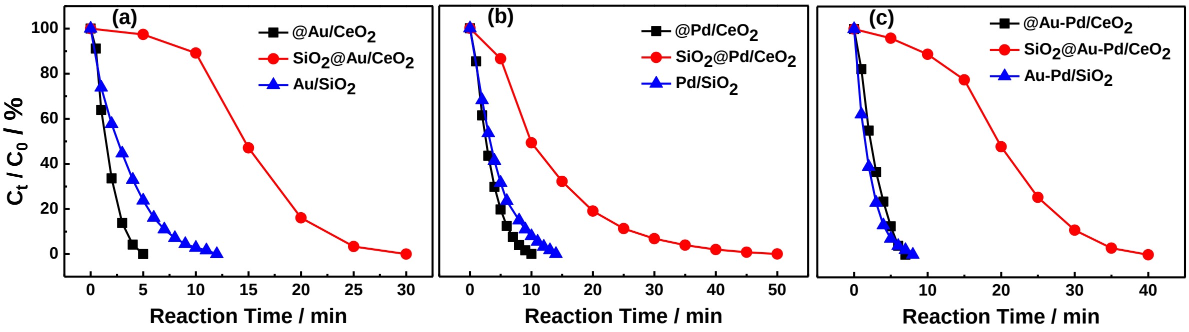


Figure S7. Catalytic tests of the reduction rate for the reduction of 4-NP over (a) @Au/CeO_2, SiO_2@Au/CeO_2, and Au/SiO_2; (b) @Pd/CeO_2, SiO_2@Pd/CeO_2, and Pd/SiO_2; and (c) @Au-Pd/CeO_2, SiO_2 @Au- Pd/CeO_2, and Au-Pd/SiO_2 catalysts.

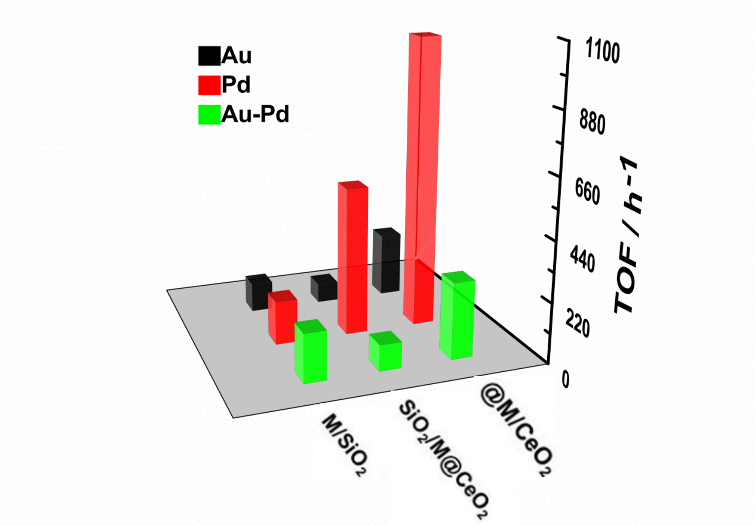


Figure S8. TOFs of M/SiO_2, SiO_2/M@ CeO_2, and @M/CeO_2 catalysts.

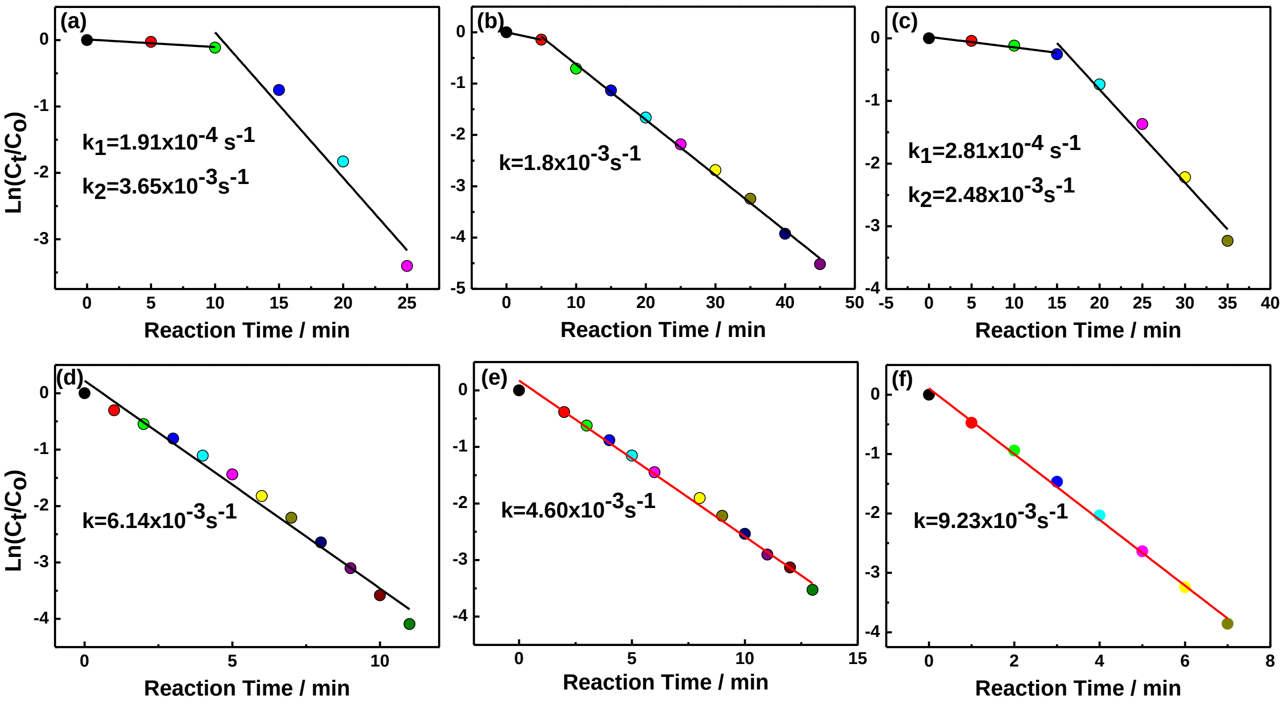


Figure S9. Plot of Ln(C_t/C_0) against the reaction time of (a) SiO_2/Au@CeO_2, (b) SiO_2/Pd@CeO_2, (c) SiO_2/Au-Pd@CeO_2, (d) Au/SiO_2, (e) Pd/SiO_2, and (f) Au-Pd/SiO_2 catalysts.

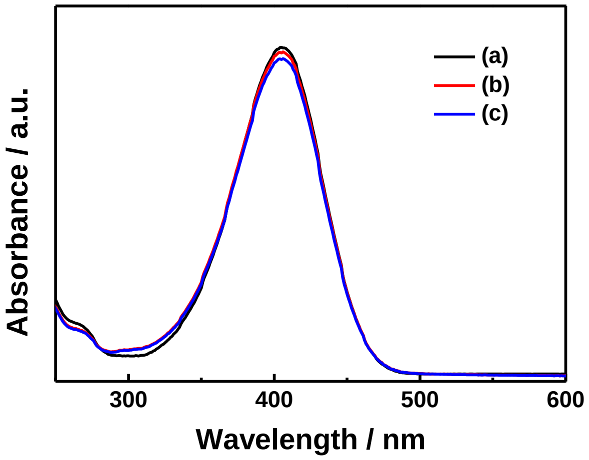


Figure S10. The absorbance change of solution for different catalysts in 10 min: (a) the original solution, (b) the solution with SiO_2/Pd@CeO_2 catalyst, and (c) the solution with @Pd/CeO_2 catalyst.

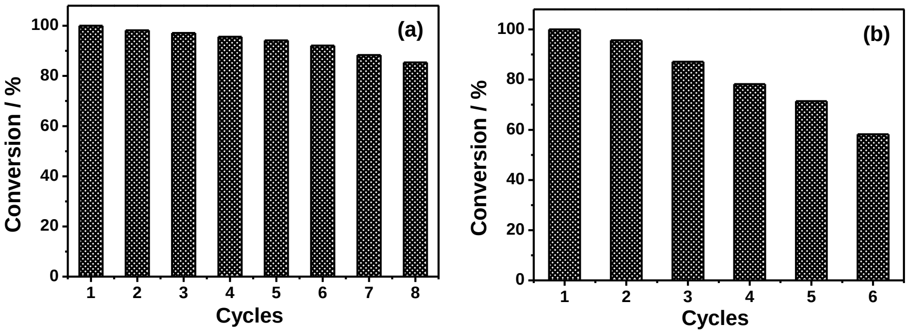


Figure S11. The catalytic stability tests of (a) @Pd/CeO_2 and (b) @CeO_2/Pd catalysts.

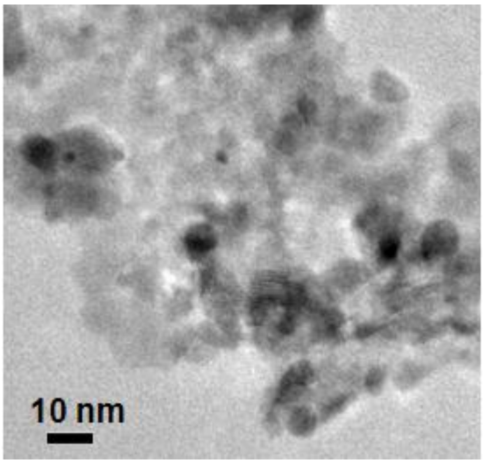


Figure S12. The TEM image of the traditional noble metal supported Au/CeO_2 catalyst.

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