Supporting Information

Ultrafine Silver Nanoparticles Supported on a Covalent Carbazole Framework as High-Efficiency

Nanocatalysts for Nitrophenol Reduction

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1. Materials and Characterization

3,6-Dibromo-9H-carbazole, ethynyltrimethylsilane, 1-bromo-4-fluorobenzene, 1,3,5-tribromobenzene, dipalladium(0) tris(dibenzylideneacetone) $(Pd_2(dba)_3),$ tetrakis(triphenylphosphine)palladium(0) $(Pd(PPh_3)_4),$ bis(triphenylphosphine) palladium(II) dichloride (Pd(PPh₃)₄Cl₂), cuprous iodide (CuI), triphenylphosphine (PPh₃), N,Ndimethylformamide (DMF), dimethyl sulfoxide (DMSO), toluene (Tol), cesium carbonate (Cs₂CO₃), tetrahydrofuran (THF), diisopropylamine (DIPA), triethylamine (Et₃N) were purchased from Energy Chemical Co. and used as recevied. Other reagents of analytical grade were commercially available and utilized without further purification. Fouriertransformed infrared (FT-IR) spectra were collected on KBr disks in transmission mode using a Bruker Tensor 27 FTIR spectrometer. Ultraviolet-visible spectra were measured on a UV-VISNIR spectrophotometer (S-3100, Scinco China). ¹³C cross polarization magic angle spinning nuclear magnetic resonance (¹³C CP/MAS NMR) spectra were recorded on a WB 400 MHz Bruker Avance II spectrometer with the contact time of 2 ms (ramp 100) and pulse delay of 3 s. Specific surface area, N₂ adsorption isotherm (77 K) and pore size distribution were measured using Micromeritics ASAP 2460 surface area and porosity analyzer. Before analysis, the samples were degassed at 120 °C for 12 h under vacuum (10-5 bar). The specific surface area was calculated based on nitrogen adsorption isotherms by Brunauer-Emmett-Teller (BET) or Langmuir analysis. The pore size distribution was calculated by non-local density functional theory (NLDFT) methods via the adsorption branch. The N₂ gas sorption isotherm was measured at 77 K. Elemental analysis (EA) was performed on a Vario Microcube Elemental Analyser (Elementar, Germany). Thermogravimetric analysis (TGA) was performed using a TA Q500 (TA, United States). The samples were heated at the rate of 10 °C/min under a nitrogen atmosphere up to 700 °C. Powder X-ray diffraction (PXRD) measurement was carried out at room temperature on PAN alytical X'Pert Pro diffractometer using Cu K α radiation (λ = 1.5418 Å). Scanning electron microscopy (SEM) was conducted with Nova NanoSEM 430 (FEI, Holland). Transmission electron microscopy (TEM) was performed with Tecnai G2 F30 (FEI Holland) transmission electron microscope. X-ray photoelectron spectroscopy (XPS) analysis was conducted on an K-Alpha⁺ (Thermo Fisher Scientific, United States) with an Al Kα X-ray line (1486.6 eV), Ag content was determined by Jobin Yvon Ultima 2 inductively coupled plasma atomic emission spectrometer (ICP-AES).

2. Synthesis of CZ-TEB

Synthesis of 3,6-dibromo-9-(4-bromophenyl)-9H-carbazole (2)



The compound (2) was synthesized according to a literature procedure with slight modifications.¹ A mixture of 3,6-dibromo-9H-carbazole (1) 2.5 g (7.7 mmol), Cs₂CO₃ 7.5 g (23 mmol), and 1-bromo-4-fluorobenzene 4 g (22.9 mmol) in DMSO (25 mL) was allowed to react under argon atmosphere. The reaction mixture was stired and heated under 120 °C for 24 h. After the reaction was completed, the mixture was poured into 200 mL petroleum ether (PE)/H₂O (v/v = 1:1). The pale solid was collected by filtration, followed by washing with 30 mL H₂O for 3 times. The product was purified by flash chromegraphy (PE) on silica gel (200- 300 mesh) column, giving a white solid (1.41 g. 45.1% yield). ¹H NMR (400 MHz,DMSO- *d6*): $\delta = 8.59$ (d, 2 H, J = 2.0 Hz), 7.88 (m, 1 H), 7.86 (m, 1 H), 7.61 (m, 2H), 7.59 (m, 2 H), 7.36 (s, 1 H), 7.33 (s 1 H). ¹³C NMR (100 MHz, DMSO- *d6*): $\delta = 139.59$, 135.82, 133.76, 130.04, 129.39,124.23, 124.18, 121.62, 113.16, 112.39.

Synthesis of 1,3,5-triethynylbenzene (5)



The compound 1,3,5-tri((trimethylsilyl)ethynyl)benzene (**4**) was synthesized according to a reported procedure.² 1,3,5-Tribromobenzene (1.259 g, 4.0 mmol), Pd(PPh₃)₂Cl₂ (0.421 g, 0.6 mmol), PPh₃(0.158 g, 0.6 mmol) and Cul (0.114 g, 0.6 mmol) were dissolved in a mixed solvent of tetrahydrofuran (THF) and diisopropylamine (DIPA) (10 mL/10 mL). Then (trimethylsily)acetylene (4.4 mL, 30 mmol) was added to the stirred solution. The mixture was stirred at 50 °C for 24 h. The solvent was then evaporated, and dichloromethane (DCM) was added to the residue and extracted with brine. The organic phase was dried with MgSO₄. The crude product was purified by flash chromatography (PE) on silica gel (200-300 mesh) column, giving a colorless solid (1.061 g, 84.3% yield). ¹H NMR (400 MHz, DMSO- *d*6) δ : 7.49 (s, 3H), 0.23 (s, 27H). ¹³C NMR (100 MHz, CDCl₃): δ 134.9, 123.9, 103.1, 96.9, 88.5, 87.1, 0.2.

Methanol (MeOH), 1,3,5-tri((trimethylsilyl)ethynyl)benzene (1.061 g, 2.7 mmol) in DCM were added to a 100 mL round-bottomed flask, and then KOH (1.68 g, 30 mmol) was added. The mixture was allowed to stir at room temperature overnight under argon atmosphere. MeOH and DCM were removed under reduced pressure. Residual white solid was dissolved in water and washed twice with DCM. The organic layer was collected and solvent was removed under reduced pressure, giving a colorless solid (0.400 g, 89% yield), which was used immediately without further purification. ¹H NMR (400 MHz, CDCl₃) δ : 7.57 (s, 3H), 4.34 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 135.3, 123.5, 83.1, 81.8.

General procedure for the synthesis of CZ-TEB



1,3,5-Triethynylbenzene (**5**) (57.2 mg, 0.381 mmol), 3,6-dibromo-9-(4-bromophenyl)-9H-carbazole (**2**) (122 mg, 0.254 mmol), Pd catalyst (0.038 mmol), PPh₃ (10 mg, 0.038 mmol) and Cul (7.3 mg, 0.038 mmol) were added to a Schlenk tube (o.d. = 26, length = 125 mm) charged with 2 mL mixed solvent (v/v = 1/1). After 10 min of sonication, the tube was frozen with a liquid nitrogen bath, degassed through three freeze-pump-thaw cycles, recharged with argon and sealed with the screw cap, then heated at 80 °C for 3 days. The formed brown product was collected *via* filtration and rinsed with MeOH. The product was added to a round-bottom flask charged with 40 mL of a mixed solvent of MeOH, acetone, dichloromethane and H₂O at a volume ratio of 1:1:1:1. The mixture was heated to reflux and stirred overnight. The product was collected *via* centrifugation and then extracted with MeOH in Soxhlet extractor for 12 h, following by drying at 60 °C under vacuum for 24 h, so obtaining the product **CZ-TEB** (isolated yield 88% for Pd₂(dba)₃). Anal. Calcd for (C₅H₁₃N)n: C, 70.18; H, 4.09; N, 16.37. Found: C, 70.74; H, 3.96; N, 2.38.

Table S1The screening of reaction conditions.									
Entry ^a	Solvent	Palladium catalyst	S _{BET} (m ² g ⁻¹) ^b	S _L (m ² g ⁻¹) ^c	PV (cm ⁻³ g ⁻¹) ^d				
1	DMF/DIPA	Pd(PPh ₃) ₄	371	402	0.36				
2	Tol/DIPA	Pd(PPh ₃) ₄	384	421	0.37				
3	DMF/Et₃N	Pd(PPh ₃) ₄	381	420	0.37				
4	DMF/DIPA	$Pd(PPh_3)_4Cl_2$	1232	1233	1.49				
5	DMF/DIPA	$Pd_2(dba)_3$	1600	1537	2.09				

^a Without further statement, the reaction performed as the general procedure.

^b Specific surface area calculated from nitrogen adsorption isotherms at 77.3 K using BET equation.

^c Specific surface area calculated from nitrogen adsorption isotherms at 77.3 K using Langmuir equation.

^d Pore volume calculated from nitrogen isotherm at $P/P_0 = 0.99$, 77.3 K.

3. NMR Spectra







Figure S2 ¹³C NMR spectrum of compound 2.







Figure S4 ¹³C NMR spectrum of compound 4.







Figure S6 ¹³C NMR spectrum of compound 5.

4. FT-IR Spectra



Figure S7 FT-IR spectra of compound 2 (black), 5 (red), and CZ-TEB (blue).



Figure S8 FT-IR spectra of Ag⁰@**CZ-TEB** (black) and Ag⁰@**CZ-TEB** after 5 cycles (red). No significant variation could be observed which indicated the structure of Ag⁰@**CZ-TEB** was robust after 5 consecutive recycle experiment.

5. PXRD Spectra of CZ-TEB



Figure S9 PXRD spectra of **CZ-TEB** with different Pd catalyst. (a) Pd(PPh₃)₄; (b) Pd(PPh₃)₄Cl₂; (c) Pd₂(dba)₃. The dispersed peak indicated that the **CZ-TEB** synthesized with different Pd⁰ catalysts was in amorphism.



6. SEM and TEM Images

Figure S10 SEM images of (a, b) **CZ-TEB**, (c) Ag⁰@**CZ-TEB**, (d) Ag⁰@**CZ-TEB** after 5 catalytic cycles, (e, f) HR-TEM images of Ag⁰@**CZ-TEB**.

N Ka1_2

Ag La1

Figure S11 EDX mapping of Ag⁰@**CZ-TEB.** C: red; N: green; Ag: blue. The background interference in Ag La1 is relatively severe due to the low loading of Ag nanoparticles on **CZ-TEB** which make the amount of Ag nanoparticles in restrict area is trivial.

Figure S12 TEM EDX mapping of fresh Ag⁰@**CZ-TEB.** C: red; N: orange; Ag: green.

Figure S13 TEM EDX mapping of Ag⁰@**CZ-TEB** for 5 cycles. C: red; N: orange; Ag: green.

7. TGA Curve

Figure S14 TGA curve of CZ-TEB. TGA analysis indicates that CZ-TEB are thermally stable up to about 700 °C.

8. N₂ Adsorption-Desorption Analysis

Figure S15 N₂ adsorption (filled symbols) and desorption (empty symbols) isotherms of CZ-TEB. The adsorption isotherms show a typical type-IV shape at low relative presure, indicating that CZ-TEB possesses a mesoporous structure.

Figure S16 BET surface area plot for **CZ-TEB** calculated from N₂ isotherms (left) and pore-sizedistribution of **CZ-TEB** (right). The right figure showed that the pore-size-distribution of **CZ-TEB** was dispersed, and the pore size was mainly centred in the range of 1.2-12 nm.

9. XPS Spectra of Ag⁰@CZ-TEB

Figure S17 XPS spectra of Ag⁰@CZ-TEB.

10. Preparation of Ag⁰@CZ-TEB Composite Materials

The **CZ-TEB** (10 mg) and AgNO₃ solution (0.1 mL, 1 mol L⁻¹) were added to 10 mL H₂O, 0.5 mL freshly prepared NaBH₄ solution (1 mol L⁻¹) was added in one portion under 273 K and then the mixture was stirred overnight at room temperature in the dark. The solid was collected by filtration, washed three times with distilled water and dried under vacuum in 60 °C for overnight. The Ag content in the composite materials was determined by ICP-AES with a mean loading of ca. 5.1 *wt*%.

11. Catalytic Activity of Ag⁰@CZ-TEB, unsupported Ag nanoparticles and CZ-TEB for Nitrophenols Reduction

The catalytic activity of $Ag^0@CZ$ -TEB was tested by the reduction reaction of n-nitrophenol (n-NP, n = 2, 3, 4) into aminophenol (n-AP, n = 2, 3, 4) in the presence of large excess of sodium borohydride (100 equiv to n-NP). In a typical procedure, 2 mg of $Ag^0@CZ$ -TEB nanocatalyst was added to the mixture of n-NP (1 mL, 0.05 mol L⁻¹) and freshly prepared NaBH₄ (1.0 mL, 5.0 mol L⁻¹) solution under contineously stirring by magnetic bar at room temperature. After adding the $Ag^0@CZ$ -TEB, the reaction started. Next, the mixture was taken from the bottom of the flask every 10 seconds by quickly inserting a 1 mL syringe pre-charged with 0.96 mL H₂O, and then filtrated by a membrane fillter to remove the catalyst. Because the low density and hydrophobic property of catalyst, most of the catalyst was floating on the surface of the reaction solution, so the catalyst loss is trivial. The reduction of n-NP to n-AP was monitored by UV-vis spectra, and the control experiment was performed without catalyst. After the reaction was completed, the mixture was centrifuged to recover the $Ag^0@CZ$ -TEB nanocatalyst. Consecutive recycling reactions were carried out to examine the reusability of nanocatalyst. Between every two consecutive

reactions, Ag⁰@**CZ-TEB** nanocatalyst was recovered by repeating operation of centrifugation and washing with ethanol three times very carefully to avoid the catalyst loss, and the recovered nanocatalyst was dried and used for the next run.

As control experiments, the reduction reaction of 4-NP to 4-AP catalyzed by unsupported Ag nanoparicles and **CZ-TEB**, respectively, was also conducted under the same reaction condition. Similarly, the corresponding recycling reactions were carried out to examine the reusability of unsupported Ag nanoparicles. The unsupported Ag nanoparicles were prepared under the same condition as $Ag^0@CZ$ -**TEB**. The apparent rate constants (k_{app}) of unsupported Ag nanoparicles and **CZ-TEB** were much lower than $Ag^0@CZ$ -**TEB** (2.41 × 10⁻³ s⁻¹ for Ag NPs, 1.67 × 10⁻⁴ s⁻¹ for **CZ-TEB**), but the catalytic ability of Ag NPs keep robust after 5 consecutive runs (Fig. S18). Apperent rate constant (k) was calculated using Eq. (1) as follows:

$$\ln A = -kt + C \tag{1}$$

where A, t and C are molar absorption coefficient, reaction time and constant, respectively.

Figure S18 Apperent rate constant k_{app} for reduction reactions of 4-NP catalyzed by unsupported Ag nanoparticles in five consecutive runs. The rate constant is 2.41×10^{-4} s⁻¹ for the first use which is much lower than that of Ag⁰@**CZ-TEB**.

12. Reported Works about Ag Nanoparticles Catalyzed Reduction Reaction of 4-NP to 4-AP

Table S2 Some representative papers related to the reduction reaction of 4-NP to 4-AP catalyzed byunsupported/supported Ag nanoparticles.

Catalyst	Catalyst amount	4-NP amount	Reaction condition	Rate constant k	Reference
AgNPs/SugPOP-1	1.0 mg/mL, 0.10 mL	1.44 mM, 0.10 mL	R.T. in water	5.14 × 10 ⁻³ s ⁻¹	[3]
AgNPs(x)/ZHL	10 mg	25 mL, 1.50 mmol L ⁻¹	R.T. in water	0.875 min ⁻¹	[4]
Ag-DHSS	0.002 mg/mL, 10mL	1.8 mg/mL, 10 mL	R.T. in water	no detailed data	[5]
Au@NiAg	0.06 mL, 0.250 mL	2.0 mL, 0.125 mM	R.T. in water	0.0266 s ⁻¹	[6]
Ag@Gd-MOFs	1.0 mg	1.8 × 10 ⁻⁴ mol·L ⁻¹ , 3.0 mL	R.T. in water	$21.33 \times 10^{-3} \text{ s}^{-1}$	[7]
Ag-SiO ₂ NWs	2 mg mL ⁻¹ , 100 μL	0.25 mmol L ⁻¹ , 1.5 mL	R.T. in water	2.535 × 10 ⁻³ s ⁻¹	[8]
	1 mg	3 mL of 1 × 10 ⁻⁴ M	R.T. in water	0.38 min ⁻¹ mg ⁻¹	[9]
				(<i>k'</i> = <i>k</i> /m; <i>k</i> is a	
				rate constant, and	
				m is the amount of	

				catalyst)	
PSMAA/Ag	2 mg	0.1 mM, 2.0 mL	R.T. in water	3.19 ±0.22 × 10 ⁻³ s ⁻ 1	[10]
BDP@Ag	Not mentioned	1 mM, 100 μL	R.T. in water	3.23 × 10 ⁻³ s ⁻¹	[11]
Ag@Vesicle	15.0 μg/mL	10 μL, 0.01 M	R.T. in water	Not mentioned	[12]
SBA-15/PDA _{0.6} /Ag	40 μL, 0.5 mg/mL	200 μL, 20 mM	R.T. in water	0.4404 min ⁻¹	[13]
Ag-coated PVDF nanofiber mat	Not mentioned	25.0, 37.5,or 50.0 μg/mL,	R.T. in water	5.5 × 10 ⁻⁴ s ⁻¹	[14]
PVA/PAA/Fe ₃ O ₄ /Mxene@AgNP composite nanofibers	Not mentioned	2 mL, 5 mmol/L	R.T. in water	0.152 min ⁻¹	[15]
Ag NPs@CMG nanohybrids	25 mL, 0.5 mM, containing 1.35 mg Ag	5 mL, 1.2 mM	R.T. in water	$3.4 \times 10^{-3} \text{ s}^{-1}$	[16]
cl-AP/exf.LT-AgNPs Ternary Nanocomposite Hydrogels	5 mg	25μL, 10mM	R.T. in water	Not mentioned	[17]
Ag-NP-SPBs	5.0× 10 ⁻⁴ mol dm ³	1.0 × 10 ³ mol dm ³	R.T. in water	0.1772 min ⁻¹	[18]
p(AA)-Ag composites		0.01 M	R.T. in water	0.134(±0.0042) min ⁻¹	[19]
A-EDTA-PEG600/OA-POSS/Ag NPs	30 mg	2.5 mL, 1.438 mmol L ⁻¹	R.T. in water	$9.4 \times 10^{-3} \mathrm{s}^{-1}$	[20]
Fe ₃ O ₄ -Ag/rGO-1	3.5 mg	5 mL 0.4 mM	R.T. in water	1.9 × 10 ⁻³ mg ⁻¹ s ⁻¹	[21]
Ag@PS	1 mg	2 ml, 2 mM	R.T. in water	0.54 min ⁻¹	[22]
Nano Ag End Capped PCL-L- Glutathione-PTHF Diblock Copolymer	0.02 g	50 mL, 1 × 10 ⁻³ M	R.T. in water	3.22 × 10 ⁻³ s ⁻¹	[23]
Ag-MBTHs	Not mentioned	Not mentioned	Visible-NIR irradiation, in water	0.058 s ⁻¹	[24]
Ag/H2CaTa2O7	3 mg	$1.0 \times 10^{-4} \text{mol L}^{-1}$	R.T. in water	0.74 min ⁻¹	[25]
AuAgNC/GO	5 μL, 0.25 mg mL ⁻¹	1 mL, 0.1 mM	R.T. in water	0.394 min ⁻¹	[26]
Fe3O4@P(MBAAm-co- MAA)@Ag	2 mg	5.8 mg	R.T. in water	1.5285 min ⁻¹	[27]

13. Molecular Model

Figure S19 3D-view of the simulated structure of CZ-TEB.

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