Oxygen-Free Water-Promoted Selective Photocatalytic Oxidative Coupling of Amines

Peng Bai, a Xinli Tong, *a Yiqi Gao a and Pengfei Guoa

^a Tianjin Key Laboratory of Organic Solar Cells and Photochemical Conversion, School of Chemistry and

Chemical Engineering, Tianjin University of Technology, Tianjin 300384, P. R. China; Tel:+86-22-60214259; E-

mail: tongxinli@tju.edu.cn or tongxli@sohu.com

Supporting Information



Figure S1. XRD patterns of 1%Pt@TiO₂-500 catalysts during the recycling experiment



Figure S2. XPS spectrum of O 1s core of the $1\%Pt@TiO_2$ catalyst

Catalyst	Surface area	Pore volume	
	(m^2/g)	(cm^3/g)	
1%Pt@TiO ₂ -300	195.7	0.25	
1%Pt@TiO ₂ -400	122.1	0.21	
1%Pt@TiO ₂ -500	66.5	0.17	
1%Pt@TiO ₂ -600	35.1	0.11	
1%Pt@TiO ₂ -700	21.8	0.08	
0.5%Pt@TiO2-500	53.4	0.16	
1.5%Pt@TiO ₂ -500	63.3	0.17	
2%Pt@TiO ₂ -500	59.5	0.17	

Table S1. BET data for different catalysts



Figure S3. Nitrogen adsorption and desorption isotherms curve of 1%Pt@TiO₂-500

Entry	Oxidant	Conversion (%) ^b	Product distribution (%) ^b	
			2	Others
1	water	76.1	97.7	2.3
2	water + oxygen	98.5	> 99	< 1
3	oxygen	61.6	98.3	1.7
4 ^{<i>c</i>}	water	64.1	85.7	14.3

Table S2. The experimental results for the oxidative coupling of 1 using different oxidant

^{*a*} Reaction condition: 0.1 g of **1**, 50 mg of 1%Pt@TiO₂-500 catalyst, 0.1 g H₂O, in 10 mL acetonitrile solvent, under 0.3 MPa of N₂ atmosphere or under 0.3 MPa mixed O₂ and N₂ (1 : 2) atmosphere, Xe lamp with a light intensity of 250 mW/cm², 6 h, 25 °C. ^{*b*} The data are obtained by GC with internal standard technique. ^{*c*} During the oxidation reaction, 0.2 mmol of TEMPO is added as the scavenger of active oxygen species.



Figure S4. The GC analysis of the reaction product (conditions: 0.1 g of benzylamine, 50 mg catalyst, 0.1 g H_2O , in a 10 mL acetonitrile solvent, 0.3 MPa of nitrogen, Xe lamp with a light intensity of 250 mW/cm², 16h, 25 °C)



Figure S5. The GC analysis of product of control experiment (reaction conditions: 0.1 g benzylamine and β -phenylethylamine, 50 mg catalyst, 0.1 g H₂O, in 10 mL acetonitrile solvent, 0.3 MPa of nitrogen, Xe lamp with a light intensity of 250 mW/cm², 6h, 25 °C).



Figure S6. GC detection of H_2



Figure S7. The testing result of basic gas product (NH₂OH or NH₃) with wet red litmus paper [The left is before detection, and the right is after detection]