

Supporting Information

Selective hydrogenation of phenylacetylene over TiO₂ supported Ni₂P nanoparticles under visible light irradiation

Xincheng Li,^{a,b} Ruiyi Wang,^{a*} Jin Zhang,^c Jing Wang,^{a,b} Yunwei Wang^{a*} and Zhanfeng Zheng^{a,b*}

^a *State Key Laboratory of Coal Conversion, Institute of Coal Chemistry, Chinese Academy of Sciences, Taiyuan 030001, PR China*

^b *Center of Materials Science and Optoelectronics Engineering, University of Chinese Academy of Sciences, Beijing 100049, China.*

^c *.Shanxi Institute of Science and Technology, Jincheng 048000, China*

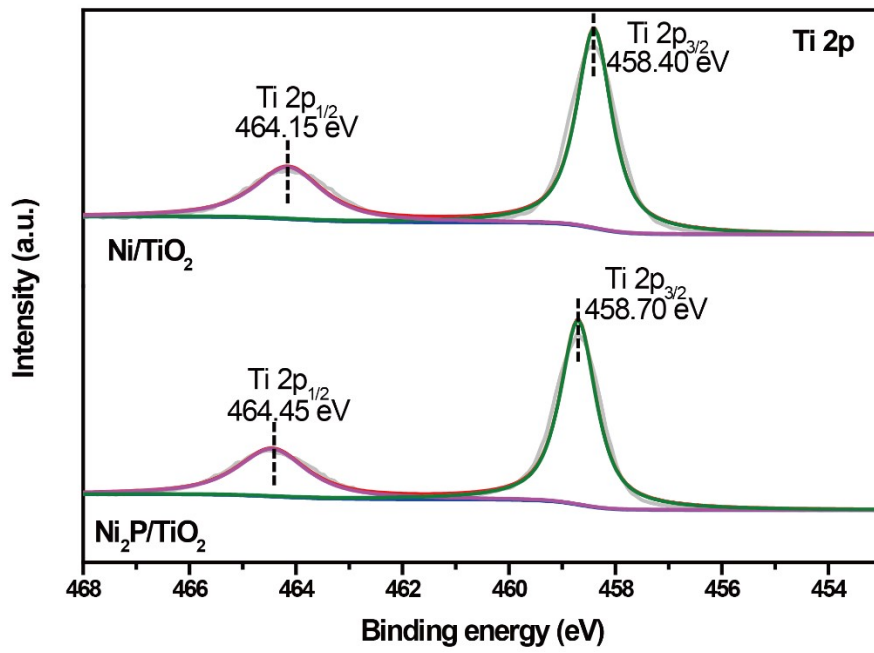


Fig. S1 High-resolution XPS Ni 2p of Ni₂P/TiO₂ and Ni/TiO₂

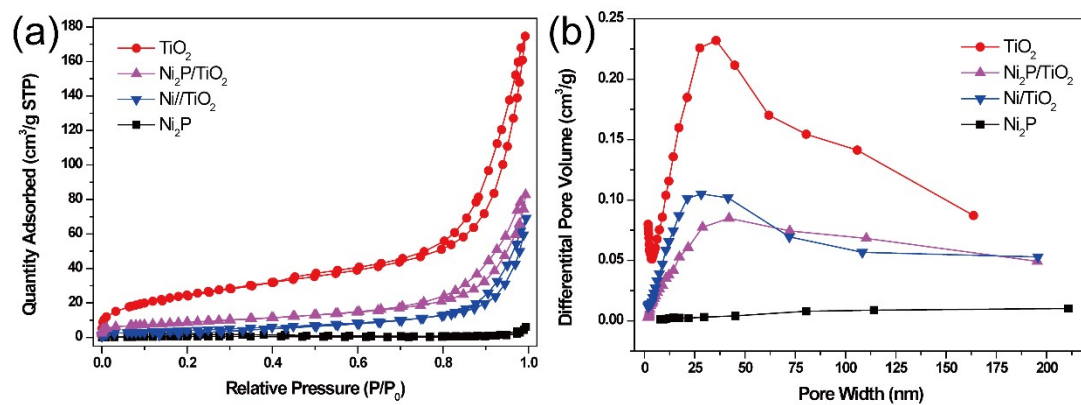


Fig. S2 (a) N_2 sorption isotherms and the (b) corresponding pore size distribution curves of the prepared catalysts.

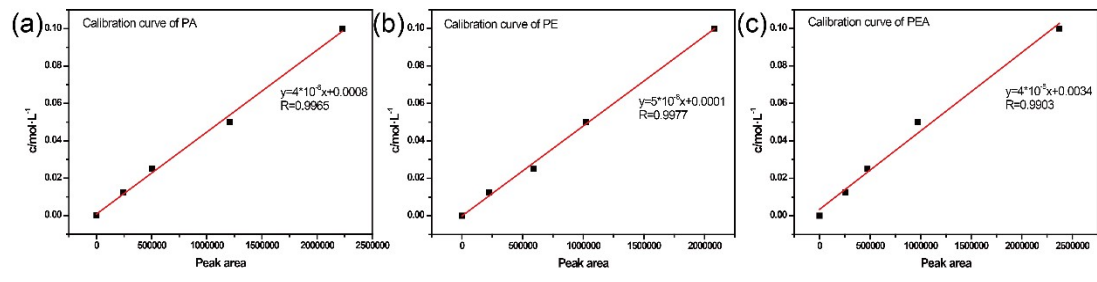


Fig. S3 Standard curves of PA, PE and PEA were plotted using the external standard method.

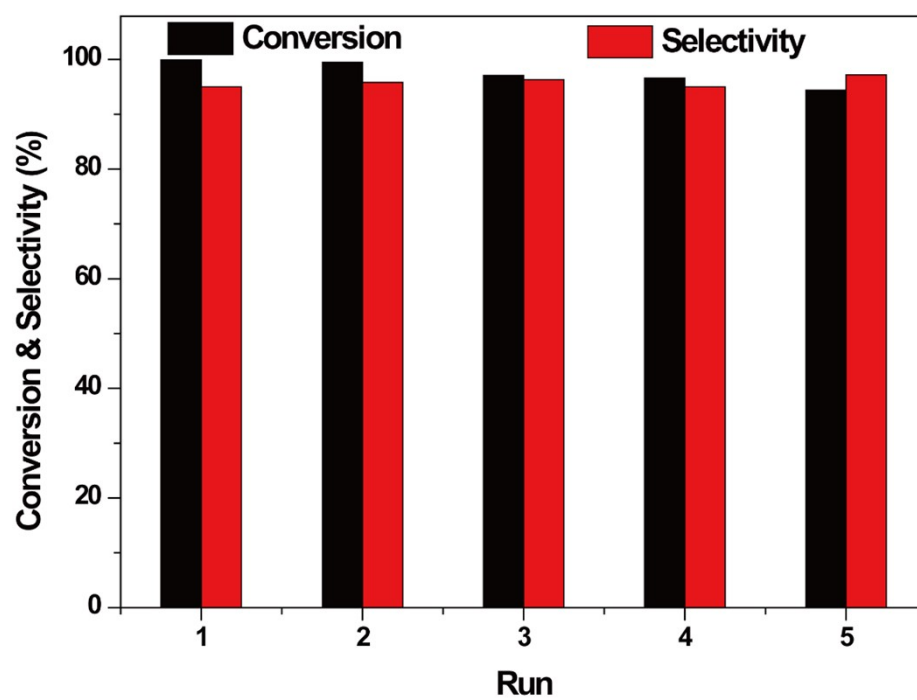


Fig. S4 Reuses of the Ni₂P/TiO₂(R) photocatalyst in selective hydrogenation of PA.
Reaction conditions: 20 mg catalyst, 0.1 mmol PA, 2 mL i-PrOH, 12 h, 80 °C, 1 atm H₂, 0.5W/cm²LED white.

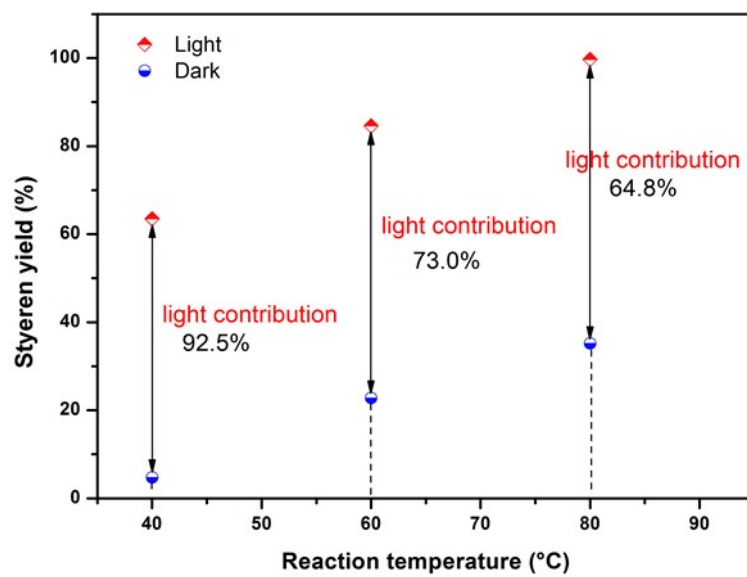


Fig. S5 Photocatalytic performance at different temperatures. Reaction conditions: 20 mg Ni₂P/TiO₂ catalyst, 0.1 mmol phenylacetylene, 2 mL, i-PrOH, 12 h, 80 °C, 1 atm H₂, LED white light (0.5 W cm⁻²) or dark.

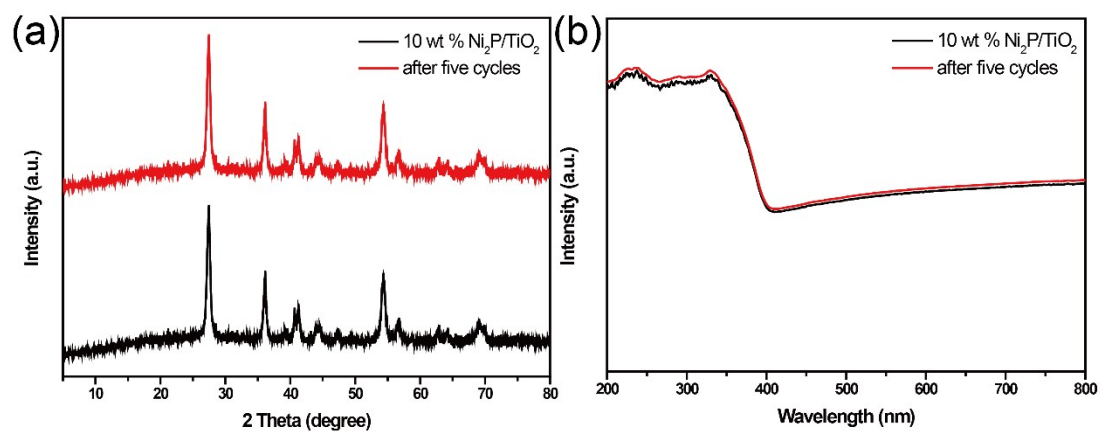


Fig. S6 The XRD (a) and UV-Vis DRS of fresh and recycled Ni₂P/TiO₂ catalyst.

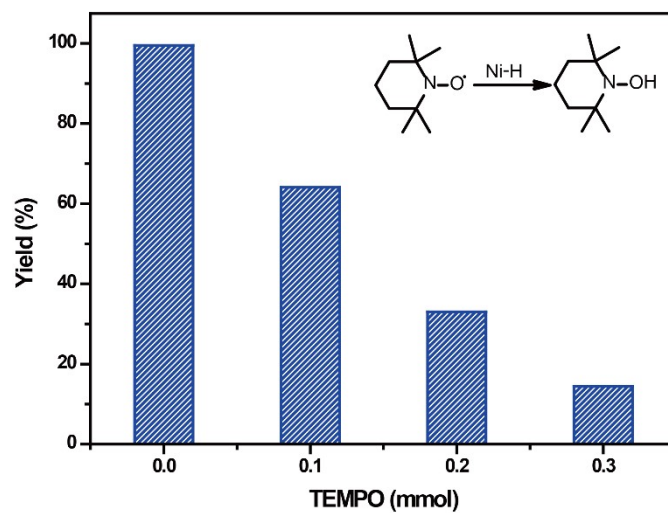


Fig. S7 Effect of TEMPO as the hydrogen abstractor on the catalytic activity for selective hydrogenation of phenylacetylene. Reaction conditions: 20 mg Ni₂P/TiO₂, 0.1 mmol phenylacetylene, 2 mL, i-PrOH, 12 h, 80 °C, 1 atm H₂, LED white light (0.5 W cm⁻²).

Table S1 The structural properties of various catalysts.

Sample	S_{BET} ($\text{m}^2 \text{g}^{-1}$)	Pore volume ($\text{cm}^3 \text{g}^{-1}$)	Average pore diameter (nm)
Ni_2P	1.7	0.01	51.6
$\text{Ni}_2\text{P}/\text{TiO}_2$	19.8	0.12	23.22
Ni/TiO_2	15.3	0.11	20.59
TiO_2	31.8	0.12	16.6

Table S2 The concentrations of PA, PE and PEA in the reaction system at different reaction times.

Reaction time (h)	C _{PA} (mmol)	C _{PE} (mmol)	C _{PEA} (mmol)	C _{all} (mmol)
0	0.0496	0	0	0.0496
1	0.0400	0.0060	0.0035	0.0495
4	0.0133	0.0332	0.0042	0.0507
8	0.0008	0.0371	0.0127	0.0506
12	0	0	0.05223	0.0483

Reaction conditions: 20 mg Ni/TiO₂, 0.1 mmol phenylacetylene, 2 mL, i-PrOH, 12 h, 80 °C, 1 atm H₂, LED white light (0.5 W cm⁻²).

Table S3 Effect of Ni₂P loading on selective hydrogenation of PA.

Entry	Ni ₂ P loading (wt%)	Light source	Conv. (%)	Sel. (%)
1	2	0.5W/cm ² LED white	19.1	99.7
2	2	dark	5.0	97.2
3	5	0.5W/cm ² LED white	48.0	97.0
4	5	dark	22.2	96.7
5	10	0.5W/cm ² LED white	99.9	96.0
6	10	dark	33.1	97.3
7	15	0.5W/cm ² LED white	76.5	97.3
8	15	dark	29.0	97.2

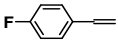
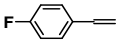
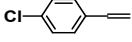
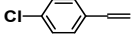
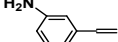
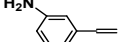
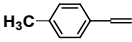
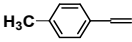
Reaction conditions: 20 mg catalyst, 0.1 mmol PA, 2 mL i-PrOH, 12 h, 80 °C, 1 atm H₂.

Table S4 The TiO₂ with different R value for selective hydrogenation of PA.

Entry	R	Light Source	Conv. (%)	Sel. (%)
1	1	0.5W/cm ² LED white	46.2	97.5
2	1	Dark	23.2	97.3
3	3	0.5W/cm ² LED white	70.3	97.5
4	3	Dark	30.4	96.9
5	5	0.5W/cm ² LED white	99.9	96.0
6	5	Dark	33.1	97.3
7	7	0.5W/cm ² LED white	54.3	97.2
8	7	Dark	23.1	97.5

Reaction conditions: 20 mg catalyst, 0.1 mmol PA, 2 mL i-PrOH, 6 h, 80 °C, 1 atm H₂.
R=m(NaH₂PO₂):m(NiCl₂·6H₂O).

Table S5 The Ni₂P/TiO₂ photocatalyst selective hydrogenation of various alkynes.

Entry	Product	Light Source	Conv. (%)	Sel. (%)
1		0.5W white cm ⁻² LED	97.4	99.0
2		dark	27.5	99.5
3		0.5W/cm ² LED white	78.1	98.1
4		dark	21.4	99.0
5		0.5W/cm ² LED white	13.4	99.5
6		dark	1.7	98.3
7		0.5W/cm ² LED white	64.3	97.1
8		dark	23.8	97.6

Reaction conditions: 20 mg catalyst, 0.1 mmol substrate, 2 mL i-PrOH, 12 h, 80 °C, 1 atm H₂.

Table S6 Photocatalytic activity over various catalysts for selective hydrogenation.

Entry	Catalyst	Conditions	Light and performance	Ref.
			white LED, 0.5 W cm ⁻²	
1	Ni ₂ P/TiO ₂	80 °C, 12 h, 1 atm H ₂ , 2mL i-PrOH, 0.1 mmol PA	Conv.=99.9% Sel.=97.2%	This work
2	Au/BT	25 °C, 2 h, 2 MPa H ₂ , 2mL ethanol, 4.42 mmol PA	1.0 W cm ⁻² (λ≥400 nm) Conv.=51.4% Sel.=90.3%	41
3	Au@CeO ₂	25 °C, 3h, 30 atm H ₂ , 5 mL toluene, 0.4 mmol PA	- Conv=99% Sel.=98%	42
4	Cu ₃ Pd ₁ /SN	25 °C, 1 h methanol 2 mL, , 1 atm H ₂ , 0.1 mmol PA	300 W Xe lamp (λ≥400 nm) Conv.=99.6% Sel.=99.4%	37
5	Pd ₁ /Ni@G	25 °C, 1.25h, 2 bar H ₂ , 10 mL ethanol, 1.85 mmol PA	- Conv.=100% Sel.=93%	43
6	H ₃₅₀ Ni/COF	100 °C, 1h, 1 Mpa H ₂ , 0.4 mmol PA, 3 mL methanol	- Conv.=99% Sel.=85%	44
7	NiZn ₃ /AISBA-15	40 °C, 15.5 h, 0.1 MPa, 4.5 g PA, 90 g methanol	- Conv.=99.6% Sel.=90.3%	5
			white LED, 0.6 W cm ⁻²	
8	Llysine/Ni/Nb ₂ O ₅	80 °C, 12 h, 1 atm H ₂ , 2mL i-PrOH 0.1 mmol PA	Conv.=99.9% Sel.=96.7%	39
9	Ni–Ga IMCs	50 °C, 5h 0.5 MPa H ₂ , 30 mL i-PrOH, 1.0 mL PA	- conv=95.1% sel.=92.2%	45
10	Ni ₂ Si/SBA-15	40 °C, 2h ,1.0 MPa H ₂ , 10 mL methanol, 1 mmol PA	- Conv.=81.5% Sel.=91.5%	46
11	Co/NC	120 °C, 2 h, 2 MPa H ₂ , 100 μL PA, 6 mL ethanol	- Conv.=88.7% Sel.=91.2%	47

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Fe/NC

120 °C, 2 h, 2 MPa H₂, 100 μL
PA, 6 mL ethanol

-
Conv.=20.5%
Sel.=82.2%

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