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Supporting Information

Visible light-induced recyclable porous $g-C_3N_4$ catalyzed one-pot

alcohol oxidation-Wittig tandem reactions

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1. General information

All glassware was thoroughly oven-dried. Chemicals and solvents were either purchased from commercial suppliers or purified by standard techniques. Thin-layer chromatography plates were visualized by exposure to ultraviolet light and/or staining with phosphomolybdic acid followed by heating on a hot plate. Flash chromatography was carried out using silica gel (200-300 mesh). The Ultraviolet visible (UV-vis) absorption spectras were recorded on a UV-2550 UV spectrophotometer (Shimadzu, Japan) at room temperature. Fourier transform infrared (FT-IR) analysis was characterized on a Magna 750 FT-IR spectrophotometer in the 400–4000 cm⁻¹ range (Thermo Nicolet Corporation, USA). Transmission electron microscope (TEM) images were gained with a JEM-100SX (Japan Electronics, Japan) transmission electron microscope under an acceleration voltage of 200kV. Scanning electron microscope (SEM) images were recorded on Field Emission Scanning Electron Microscopy (Hitachi SU8010). The photoluminescence (PL) spectra were measured with fluorescence spectrophotometer (Hitachi, F7100). The nitrogen adsorption-desorption experiment was on the specific surface area and porosity analyzer (Micromeritics, ASAP2460). The phase structure was analyzed by X-ray diffractometer (Bruker, D8 ADVANCE). ¹H NMR and ¹³C NMR spectra were recorded on a Bruker 400 Avance spectrometer (500 MHz). The spectra were recorded in CDCl₃ as solvent at room temperature, ¹H and ¹³C NMR chemical shifts are reported in ppm relative to the residual solvent peak. The photocurrent density and electrochemical impedance spectroscopy (EIS) were collected on the electrochemical workstation (Chenhua instrument, CHI660B) with a standard three-electrode system.

2. experimental section

2.1 Preparation of Bulk g-C₃N₄

Melamine (10.0 g) was added into a crucible, ensuring that the crucible was capped with a cover. Gradually heated the crucible to the temperature of 550 °C at a heating rate of 2°C/min and maintained at this temperature for 4 hours. Upon completion of the heating process, the crucible was allowed to cool naturally to room temperature. After grinding, the bulk g-C₃N₄ was obtained as yellow powder.

2.2 Preparation of porous g- C_3N_4

To a three-necked flask equipped with a stir bar, melamine (2.0 g), F127 (1.0 g) and 100 mL deionized water was added successively. Then vigorous stirring was conducted at room temperature until F127 was completely dissolved. The resulting mixture was subsequently refluxed at 100 °C under continuous stirring. Then 2 mL of 98% sulfuric acid was added dropwise, and the reflux was continued for an additional 5 hours. After the completion of heating process, the mixture was allowed to cool to room temperature naturally. Subsequently, the white solid was collected by centrifugation and washed several times with deionized water and EtOH until the pH of the supernatant reached 7. The solid was then transferred to a vacuum oven and dried

for 12 hours. The white solid was subjected to calcination in a muffle furnace, where it was heated at a rate of 2 °C/min until it reached 500 °C and was maintained at this temperature for 2 hours. Subsequently, the temperature was increased to 550 °C at the same heating rate and maintained for another 2 hours. After cooling to room temperature, the porous $g-C_3N_4$ was obtained as yellow-brown powder.

2.3 Experimental procedure for photocatalytic alcohol oxidation-Wittig reactions:

The photocatalytic alcohol oxidation-Wittig reaction was conducted under visible light irradiation in an oxygen atmosphere (balloon). Specifically, to a 10 ml Schlenk flask equipped with a stir bar, benzyl alcohol (22 μ L, 0.2 mmol), ylide (139.2 mg, 0.4 mmol), porous g-C₃N₄ (10 mg) and toluene (1 ml) was added sequentially. The resulting mixture was stirred at room temperature under the irradiation of blue LEDs. The reaction process was monitored by TLC. After the starting materials was completely consumed, the crude mixture was purified by flash chromatography (silica gel, petroleum/ethyl acetate) to give the pure α , β -unsaturated ester.



Figure S1. SEM images of bulk $g-C_3N_4$ (A) and porous $g-C_3N_4$ (B).



Figure S2. photographs of bulk and porous $g-C_3N_4$ with same mass. Table S1. The oxidation reactions of benzyl alcohol to benzaldehyde^a

	<u>ОН (</u>	$g-C_3N_4$, blue LEDs toluene, O_2 , rt.	0
Entry	Solvent	catalyst	Yield ^b (%)
1	Toluene	bulk $g-C_3N_4$	41
2	Toluene	porous $g-C_3N_4$	52
3°	Toluene	bulk $g-C_3N_4$	57
4°	Toluene	porous g-C ₃ N ₄	69

^{*a*}Reaction conditions: benzyl alcohol (0.2 mmol), solvent (1 ml), catalyst (10 mg) at rt. ^{*b*} Isolated yield. ^{*c*}The reaction was conducted at 40 °C.



Figure S3. The impacts of light power density on the one-pot tandem reaction.



Figure S4. Free radical trapping experiments by TEMOP and BHT.



Figure S5. The analysis of H_2O_2 by HPLC (mobile phase: MeOH/ $H_2O = 0.6/0.4$, 1mL/min, $\lambda = 220 \text{ nm}$).

3. Spectra data of all products

Ethyl cinnamate (3a) :

Colorless oil. Yield: 87%. ¹H NMR (500 MHz, CDCl₃): δ 7.69 OEt (d, J = 20.0 Hz, 1H), 7.54-7.52 (m, 2H), 7.39-7.38 (m, 3H), 6.44 (d, J = 15.0 Hz, 1H), 4.27 (q, J = 5.0 Hz, 2H), 1.34 (t, J = 5.0 Hz, 2H)

3H). ¹³C NMR (125 MHz, CDCl₃): δ 167.0, 144.6, 134.5, 133.7, 130.2, 128.9, 128.1, 118.3, 60.5, 14.3.

Ethyl (E)-3-(p-tolyl)acrylate (3b):

Colorless oil. Yield: 84%.¹H NMR (500 MHz, CDCl₃): δ 7.66 (d, J = 20.0 Hz, 1H), 7.44-7.41 (m, 2H), 7.19-7.18 (m, 2H), 6.39 (d, J = 20.0 Hz, 1H), 4.25 (d, J = 5.0 Hz, 2H), 2.37 (s, 3H), 1.31

(t, *J* = 5.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 167.2, 144.6, 140.6, 133.7, 131.8, 129.6, 129.3, 128.5, 128.1, 117.2, 60.4, 21.5, 14.4.

Ethyl (E)-3-(4-methoxyphenyl)acrylate (3c) :



Colorless oil. Yield: 82%. ¹H NMR (500 MHz, CDCl₃): δ 7.62 (d, J = 20.0 Hz, 1H), 7.41 (s, 2H), 6.86 (d, J = 5.0 Hz, 2H), 6.28 (d, J = 15.0 Hz, 1H), 4.23 (q, J = 5.0 Hz, 2H),

3.77 (s, 3H), 1.31 (t, *J* = 5.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 167.2, 161.3, 144.2, 129.6, 127.1, 115.7, 114.3, 60.2, 55.2, 14.3.

Ethyl (E)-3-(4-(trifluoromethyl)phenyl)acrylate (3d):



Colorless oil. Yield: 82%.¹H NMR (500 MHz, CDCl₃): δ 7.69 (d, J = 15.0 Hz, 1H), 7.64-7.59 (m, 4H), 6.50 (d, J = 15.0 Hz, 1H), 4.28 (q, J = 10.0 Hz, 2H), 1.34 (s, 3H). ¹³C

NMR (125 MHz, CDCl₃): δ 166.5, 142.8, 138.0, 132.2, 131.9, 131.7, 131.4, 128.6, 126.0, 125.9, 125.9, 125.9, 121.0, 60.9, 14.3.

Ethyl (E)-3-(4-fluorophenyl)acrylate (3e):



Colorless oil. Yield: 81%.¹H NMR (500 MHz, CDCl₃): δ 7.65 (d, J = 15.0 Hz, 1H), 7.52-7.51 (m, 2H), 7.09-7.07 (m, 2H), 6.36 (d, J = 20.0 Hz, 1H), 4.26 (q, J = 10.0 Hz, 2H), 1.34 (t, J

= 10.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 166.9, 164.9, 162.9, 143.3, 130.7, 129.9, 129.9, 118.1, 118.1, 116.1, 116.0, 60.6, 14.3.

Ethyl (E)-3-(4-bromophenyl)acrylate (3f):



Colorless oil. Yield: 89%. ¹H NMR (500 MHz, CDCl₃): δ 7.62 (d, J = 15.0 Hz, 1H), 7.53-7.47 (m, 2H), 7.39-7.38 (m, 2H), 6.42 (d, J = 15.0 Hz, 1H), 4.26 (q, J = 10.0 Hz, 2H), 1.34

(t, *J* = 10.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 166.8, 143.2, 133.4, 132.1, 129.4, 124.5, 119.0, 60.7, 14.3.

Ethyl (E)-3-(4-iodophenyl)acrylate (3g):



Yellow oil. Yield: 76%. ¹H NMR (500 MHz, CDCl₃): δ 7.66 (d, J = 10.0 Hz, 2H), 7.55 (d, J = 15.0 Hz, 1H), 7.19 (d, J = 10.0 Hz, 2H), 6.40 (d, J = 20.0 Hz, 1H), 4.24 (q, J = 10.0 Hz, 2H),

1.32 (t, J = 5.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 166.5, 143.2, 138.1, 133.9,

129.5, 119.0, 96.6, 60.6, 14.4.

Ethyl (E)-3-(4-isopropylphenyl)acrylate (3h):



(125 MHz, CDCl₃): δ 167.0, 151.4, 144.5, 132.2, 128.2, 127.0, 117.3, 60.3, 34.0, 23.7, 14.3.

Ethyl (E)-3-(4-(tert-butyl)phenyl)acrylate (3i):



Yellow oil. Yield: 84%. ¹H NMR (500 MHz, CDCl₃): δ 7.68 OEt (d, J = 15.0 Hz, 1H), 7.42 (d, J = 10.0 Hz, 2H), 7.36 (d, J = 5.0 Hz, 2H), 6.41 (d, J = 15.0 Hz, 1H), 4.24 (q, J = 5.0 Hz, 2H), 1.32 – 1.29 (m, 12H). ¹³C NMR (125 MHz, CDCl₃): δ

 $167.0,\,153.6,\,144.5,\,131.8,\,127.9,\,125.8,\,117.4,\,60.3,\,34.8,\,31.1,\,14.4.$

Ethyl (E)-3-(4-nitrophenyl)acrylate (3j) :



Yellow solid. Yield: 56%. ¹H NMR (500 MHz, CDCl₃): δ 8.25 (d, *J* = 10.0 Hz, 2H), 7.73-7.67 (m, 3H), 6.57 (d, *J* = 15.0 Hz, 1H), 4.30 (q, *J* = 5.0 Hz, 2H), 1.36 (t, *J* = 10.0 Hz,

3H). ¹³C NMR (125 MHz, CDCl₃): δ 166.1, 148.5, 141.6, 140.6, 130.2, 128.6, 124.2, 123.2, 122.6, 61.0, 14.3.

Ethyl (E)-3-(m-tolyl)acrylate (3k):



OMe

Colorless oil. Yield: 86%.¹H NMR (500 MHz, CDCl₃): δ 7.66 (d, J = 15.0 Hz, 1H), 7.33-7.32 (m, 2H), 7.28-7.25 (m, 1H), 7.20-7.18 (m, 1H), 6.43 (d, J = 15.0 Hz, 1H), 4.25 (q, J = 5.0 Hz, 2H), 2.36 (s, 3H), 1.33 (t, J = 5.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ

167.1, 144.8, 138.5, 134.4, 133.6, 131.1, 128.9, 128.8, 128.7, 128.1, 125.2, 118.1, 60.5, 21.3, 14.3.

Ethyl (E)-3-(3-methoxyphenyl)acrylate (3l):

Colorless oil. Yield: 85%. ¹H NMR (500 MHz, CDCl₃): δ 7.63 t (d, J = 15.0 Hz, 1H), 7.25 (d, J = 10.0 Hz, 1H), 7.08 - 7.06 (m, 1H), 7.01 (d, J = 5.0 Hz, 1H), 6.89 – 6.87 (m, 1H), 6.40 (d, J = 15.0 Hz, 1H), 4.24 (q, J = 10.0 Hz, 2H), 3.76 (s, 3H), 1.31 (t, J = 5.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 166.8, 159.9, 144.4, 135.8, 129.8, 120.6, 118.5, 116.0, 112.9, 60.4, 14.3.

Ethyl (E)-3-(3-(trifluoromethyl)phenyl)acrylate (3m):



Colorless oil. Yield: 84%. ¹H NMR (500 MHz, CDCl₃): δ 7.75 (s, 1H), 7.72 – 7.65 (m, 2H), 7.60 (d, J = 5.0 Hz, 1H), 7.48 (t, J = 5.0 Hz, 1H), 6.49 (d, J = 15.0 Hz, 1H), 4.28 (q, J = 10.0 Hz, 2H), 1.34 (t, J = 5.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 166.3,

142.6, 135.3, 130.9, 129.3, 126.5, 126.4, 124.5, 124.5, 120.2, 60.6, 14.1.

Ethyl (E)-3-(3-fluorophenyl)acrylate (3n):



Colorless oil. Yield: 83%. ¹H NMR (500 MHz, CDCl₃): δ 7.62 (d, J = 15.0 Hz, 1H), 7.32 (t, J = 5.0 Hz, 1H), 7.26 (d, J = 5.0 Hz, 1H), 7.19 (t, J = 5.0 Hz, 1H), 7.07-7.03 (m, 1H), 6.41 (d, J = 20.0 Hz, 1H), 4.26 (q, J = 5.0 Hz, 2H), 1.32 (t, J = 5.0 Hz, 3H). ¹³C

NMR (125 MHz, CDCl₃): δ 166.5, 164.0, 162.0, 143.1, 143.0, 136.8, 136.7, 130.4, 130.3, 124.0, 124.0, 119.7, 117.1, 116.9, 114.3, 114.1, 60.6, 14.2.

Ethyl (E)-3-(3-bromophenyl)acrylate (30):



Colorless oil. Yield: 80%. ¹H NMR (500 MHz, CDCl₃): δ 7.63 (s, 1H), 7.57 (d, J = 15.0 Hz, 1H), 7.46 (d, J = 5.0 Hz, 1H), 7.40 (d, J = 10.0 Hz, 1H), 7.22 (t, J = 10.0 Hz, 1H), 6.40 (d, J = 15.0 Hz, 1H), 4.26 (q, J = 10.0 Hz, 2H), 1.33 (t, J = 10.0 Hz, 3H). ¹³C

NMR (125 MHz, CDCl₃): δ 166.4, 142.8, 136.6, 133.0,130.7, 130.4, 126.6, 123.0, 119.8, 60.6, 14.3.

Ethyl (E)-3-(3-iodophenyl)acrylate (3p):

Yellow oil. Yield: 80%. ¹H NMR (500 MHz, CDCl₃): δ 7.81 (d, J = 5.0 Hz, 1H), 7.64 (d, J = 10.0 Hz, 1H), 7.52 (d, J = 15.0 Hz, 1H), 7.41 (s, 1H), 7.06 (t, J = 10.8 Hz, 1H), 6.38 (d, J = 20.0 Hz, 1H), 4.25 (q, J = 10.0 Hz, 2H), 1.32 (t, J = 5.0 Hz, 3H). ¹³C NMR (125)

MHz, CDCl₃): δ 166.3, 142.7, 138.9, 136.7, 136.6, 130.5, 127.2, 119.6, 94.9, 60.6, 14.4.

Ethyl (E)-3-(o-tolyl)acrylate (3q) :



Colorless oil. Yield: 87%. ¹H NMR (500 MHz, CDCl₃): δ 7.95 (d, J = 15.0 Hz, 1H), 7.49 (d, J = 10.0 Hz, 1H), 7.21 (d, J = 5.0 Hz, 1H), 7.15 (t, J = 10.0 Hz, 2H), 6.33 (d, J = 15.0 Hz, 1H), 4.24

(q, *J* = 5.0 Hz, 2H), 2.38 (s, 3H), 1.31 (t, *J* = 5.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 166.9, 142.2, 137.6, 133.4, 130.8, 130.0, 126.4, 126.3, 119.3, 60.4, 19.7, 14.3.

Ethyl (E)-3-(2-methoxyphenyl)acrylate (3r):



1H), 4.23 (q, J = 5.0 Hz, 2H), 3.80 (s, 3H), 1.31 (t, J = 5.0 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃): δ 167.4, 158.3, 139.9, 131.5, 128.8, 123.3, 120.7, 118.7, 111.1, 60.2, 55.3, 14.3.

Ethyl (E)-3-(2-(trifluoromethyl)phenyl)acrylate (3s) :



Yellow oil. Yield: 79%. ¹H NMR (500 MHz, CDCl₃): δ 7.94 (d, J = 5.0 Hz, 1H), 7.57 – 7.51 (m, 2H), 7.40 (t, J = 5.0 Hz, 1H), 7.30 (d, J = 5.0 Hz, 1H), 6.27 (d, J = 15.0 Hz, 1H), 4.14 (q, J = 5.0 Hz, 1H), 6.27 (d, J = 15.0 Hz, 1H), 4.14 (q, J = 5.0 Hz, 1H), 6.27 (d, J = 15.0 Hz, 1H), 4.14 (q, J = 5.0 Hz, 1H), 5

2H), 1.20 (t, *J* = 5.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 166.0, 139.9, 139.9, 132.1, 129.5, 127.8, 126.0, 126.0, 122.5, 60.7, 14.0.

Ethyl (E)-3-(2-fluorophenyl)acrylate (3t) :



Colorless oil. Yield: 87%. ¹H NMR (500 MHz, CDCl₃): δ 7.80 (d, J = 15.0 Hz, 1H), 7.50 (t, J = 5.0, Hz, 1H), 7.34 – 7.28 (m, 1H), 7.12 (t, J = 10.0 Hz, 1H), 7.08-7.04 (m, 1H), 6.52 (d, J = 20.0 Hz,

1H), 4.26 (q, J = 5.0 Hz, 2H), 1.32 (t, J = 10.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 166.6, 162.3, 160.3, 137.0, 137.0, 131.6, 131.6, 129.0, 128.9, 124.4, 124.4, 122.5, 122.4, 120.8, 120.8, 116.2, 116.0, 60.5, 14.2.

Ethyl (E)-3-(2-iodophenyl)acrylate (3u):



Yellow oil. Yield: 75%. ¹H NMR (500 MHz, CDCl₃): δ 7.89 – 7.84 (m, 2H), 7.51 (d, *J* = 5.0 Hz, 1H), 7.32 (d, *J* = 10.0 Hz, 1H), 7.00 (t, *J* = 5.0 Hz, 1H), 6.29 (d, *J* = 20.0 Hz, 1H), 4.26 (q, *J* = 10.0 Hz, 2H), 1.33 (t, *J* = 10.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 166.2, 147.6, 140.0, 137.7, 131.2, 128.6, 127.4, 121.2, 101.3, 60.7, 14.4.

Ethyl (E)-3-(4-bromo-2-fluorophenyl)acrylate (3v) :

Colorless oil. Yield: 78%. ¹H NMR (500 MHz, CDCl₃): δ 7.68 (d, J = 20.0 Hz, 1H), 7.37 (t, J = 5.0 Hz, 1H), 7.28 – 7.23 (m, 2H), 6.49 (d, J = 20.0 Hz, 1H), 4.26 (q, J = 10.0 Hz, 2H),

1.33 (t, *J* = 10.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 166.3, 161.8, 159.7, 135.8, 129.8, 129.8, 127.9, 127.8, 124.3, 124.2, 121.6, 121.5, 121.4, 121.3, 119.9, 119.7, 60.6, 14.2.

Ethyl (E)-3-(3,5-difluorophenyl)acrylate (3w) :



Colorless oil. Yield: 83%. ¹H NMR (500 MHz, CDCl₃): δ 7.56 (d, J = 20.0 Hz, 1H), 7.03 – 7.02 (m, 2H), 6.83-6.79 (m, 1H), 6.42 (d, J = 15.0 Hz, 1H), 4.28 (q, J = 10.0 Hz, 2H), 1.34 (t, J = 5.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 166.0, 164.2,

164.1, 162.2, 162.1, 141.8, 141.8, 141.8, 137.7, 121.0, 110.7, 110.6, 110.5, 110.5, 105.3, 105.1, 104.9, 60.7, 14.1.

Ethyl (E)-3-(perfluorophenyl)acrylate (3x):



Yellow oil. Yield: 76%. ¹H NMR (500 MHz, CDCl₃): δ 7.63 (d, J = 15.0 Hz, 1H), 6.73 (d, J = 15.0 Hz, 1H), 4.30 (q, J = 5.0 Hz, 2H), 1.37 (t, J = 5.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ 165.8, 132.1, 126.4, 61.0, 13.9.

4. Copies of NMR spectra for products

¹H NMR and ¹³C NMR of compound **3a**







 $^1\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR of compound $\mathbf{3b}$

-237

-0.00

426

OEt 3b







3b



 $^1\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR of compound 3c



¹H NMR and ¹³C NMR of compound **3d**







 $^1\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR of compound 3e



 $^1\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR of compound $\mathbf{3f}$



¹H NMR and ¹³C NMR of compound **3g**







160 140 120 100 80 60 40 20 0

 $^1\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR of compound $\mathbf{3h}$











¹H NMR and ¹³C NMR of compound **3i**



 $^1\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR of compound 3j



 $^1\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR of compound 3k







 $^1\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR of compound $\boldsymbol{3l}$





¹H NMR and ¹³C NMR of compound **3m**









¹H NMR and ¹³C NMR of compound **3n**











AMA

 $\begin{pmatrix} 1.34 \\ 1.32 \\ 1.31 \end{pmatrix}$











¹H NMR and ¹³C NMR of compound **3p**















 $^1\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR of compound $\mathbf{3q}$













 $^1\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR of compound 3r





¹H NMR and ¹³C NMR of compound **3s**

---0.00





¹H NMR and ¹³C NMR of compound **3**t



 $^1\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR of compound $\boldsymbol{3u}$







 $^1\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR of compound 3v



 $^1\mathrm{H}$ NMR and $^{13}\mathrm{C}$ NMR of compound $\mathbf{3w}$



¹H NMR and ¹³C NMR of compound 3x

$$-0.00$$









