Supporting Information

Photocatalytic selective conversion of furfural to γ-butyrolactone through tetrahydrofurfuryl alcohol intermediates over Pd NPs decorated g-C$_3$N$_4$

Rajat Ghalta[a] and Rajendra Srivastava[a]*

[a]*Catalysis Research Laboratory, Department of Chemistry, Indian Institute of Technology Ropar, Rupnagar, Punjab-140001, India*

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*Email: rajendra@iitrpr.ac.in

Phone: +91-1881-242175; Fax: +91-1881-223395

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Experimental section

Materials

All chemicals used in this study were of AR grade and used as received without any further treatment. Furfural (FAL), furfuryl alcohol (FOL), tetrahydrofurfuryl alcohol (THFA), palladium(II) chloride, methanol, ethanol, urea, IPA (propane-2-ol), nickel nitrate hexahydrate, cobalt nitrate hexahydrate, and acetonitrile were obtained from Merck India Pvt. Ltd., whereas NaOH, KI, and ammonium molybdate were procured from Loba Chemie, Pvt. Ltd., India. Nitro blue tetrazolium (NBT), and 2-hydroxyl terephthalic acid (THA) were purchased from Sigma Aldrich.

Catalyst preparation

Synthesis of g-C_3N_4
Urea-derived graphitic carbon nitride (g-C_3N_4) was synthesized using a previously reported method in which 15 g of urea was first crushed into a fine powder in a mortar pestle. Then the powder was kept in the capped crucible. The muffle furnace was programmed for heating at 550 ˚C for 3 h with a ramp of 2.5 ˚C per min. After the end of the program, the material was removed from the furnace and ground in a mortar pestle.

Synthesis of E-g-C_3N_4
The 400 mg of g-C_3N_4 was mixed with 100 ml of ethanol and stirred for 30 min. Then the suspension was sonicated for 3 h. The ethanol was evaporated at 80 ˚C, and obtained material was crushed in fine powder using a mortar pestle.

Synthesis of x% Pd@E-g-C_3N_4
400 mg of g-C_3N_4 was mixed with 100 ml of ethanol and stirred for 30 min. Then the suspension was sonicated for 3 h. Meanwhile, the required amount of $PdCl_2$ was mixed in 10 ml ethanol. After sonication, the $PdCl_2$ solution was added dropwise to the suspension with continuous stirring. After the addition, the ethanol was evaporated at 80 ˚C. The obtained greyish material was finely powdered using a mortar pestle and transferred into the boat-type crucible. The material was heated at 300 ˚C for 3 h in H_2 (5%)/Ar(95%) using a tube furnace. The obtained material is designated as x% Pd@E-g-C_3N_4, where x% is the percentage of Pd NPs in the material, i.e. (0.5%, 1%, 2%, and 3%).

Synthesis of 1% M@E-g-C₃N₄ (M = Co, and Ni)

For the synthesis of these materials, the same procedures were employed where 400 mg of g-C₃N₄ was first mixed with 100 ml ethanol and then sonicated for 3 h. Then the required amount of metal salt (cobalt nitrate hexahydrate) for 1% Co@E-g-C₃N₄, and nickel nitrate hexahydrate for 1% Ni@E-g-C₃N₄) was added in 10 ml of ethanol. The metal salt solution was dropwise added to the sonicated suspension, and finally, the ethanol was evaporated. The obtained material was reduced in a tube furnace in $H_2(5\%)/Ar(95\%)$ for 3 h at 450 ˚C.

Photocatalytic experiment

Photocatalytic FAL reduction

0.1 mM of FAL was taken in 5 mL of solvent in a glass reactor. 30 mg of catalyst was added and stirred in the dark for 30 min to establish equilibrium. H_2 (2 bar) was introduced in the glass reactor. The reaction was performed for different time intervals in a 150 W LED with continuous starring. The catalyst was removed from the reaction mixture using a centrifuge. Also, a similar reaction was carried out in the sunlight in March 2022 at IIT Ropar.

Photocatalytic THFA Oxidation

0.1 mM of THFA was taken in 3 mL of acetonitrile in a round bottom flask. 30 mg of catalyst was added and stirred in the dark for 30 min to establish equilibrium. After that, the solution was purged with oxygen gas for 10 min. The oxygen balloon was attached to the round bottom flask to maintain the oxygen atmosphere. The reaction was conducted using 150 W LED. The catalyst was removed from the reaction mixture using a centrifuge.

Thermal FAL reduction

0.1 mM of FAL was taken in 5 mL of solvent in a Büchi make liquid phase pressure reactor. 30 mg of catalyst was added to the reaction mixture. The reactor was purged with H_2 and then pressurized with H_2 (5 bar) at ambient temperature. The reactions were conducted at a desired temperature with continuous stirring for a desired time After the reaction, the reactor was cooled to ambient temperature, and the catalyst was removed from the reaction mixture using a centrifuge. The analysis was conducted in a similar manner as was conducted under photocatalytic conditions.

Analysis of the reaction mixture

The reaction mixture was analyzed with gas chromatography (GC) (Yonglin; 6100; GC column: BP-5; 30 m×0.25 mm×0.25 μm) fitted with FID detector. High-purity N$_2$ gas (99.99%, GC grade) was used as the carrier gas with a flow rate of 10 ml/min, whereas H$_2$ (99.99%, GC grade) and Air (99.99%, GC grade) were used as ignition gases. The injector and detector temperature were set at 280 ˚C. GC column oven temperature was programmed as follows: Initial temperature = 60 ˚C, hold time = 2 min followed by temperature ramping to a final temperature of 280 ˚C with a ramp rate of 5 ˚C /min. 0.2 μL of the sample was injected for the analysis. The calibration curves were constructed for pure FAL, FOL, THFA, and GBL (diluted in 2-propanol) for the determination of conversion and selectivity. Products were also confirmed using GC-MS (Shimadzu GCMS-QP 2010 Ultra; Rtx-5 Sil Ms; 30 m × 0.25 mm × 0.25 μm).

Apparent Quantum Yield Calculation

The quantum yield of the reaction was calculated using eq (4), which gives the ratio between the electron involved in the reaction and the molar flow of photons introduced into the reactor (eqn (1)). The apparent wavelength (λ) of photons was estimated from the band gap of the catalyst, determined from Tauc Plots. The quantum yield calculated in this manner was not the exact value but served as a reference to compare the photo efficiency of photocatalytic system.

$$Apparent\ Quantum\ Yeald\ (\phi) = \frac{Number\ of\ reacted\ electron\ \times 100}{Number\ of\ incident\ photon}$$

(1)

$$Number\ of\ reacted\ electron = Reactant\ conversion\ (mol^{-1})\ \times\ N_A\ (mol)$$

(2)

$$Number\ of\ incident\ photon = \frac{Light\ intansity\ (Wm^{-2})\ \times\ Area\ (m^2) \times\ Time\ (s)}{\dfrac{h\ (Js)\ c\ (ms^{-1})}{\lambda\ (m)}}$$

(3)

$$\phi = \frac{Reactant\ conversion\ (mol^{-1}) \times N_A\ (mol) \times h\ (Js)c\ (ms^{-1}) \times 100}{Light\ intansity\ (Js^{-1}m^{-2}) \times Area\ (m^2) \times Time\ (s) \times \lambda\ (m)}$$

(4)

Photoelectrochemical measurement

The photoelectrochemical analyses were performed with PGSTAT302N Autolab electrochemical workstation using standard three-electrode grouping in Pyrex cell with a 0.5 M aqueous solution of sodium sulfate as electrolyte. The coated photocatalyst acts as a photoanode (working electrode), Pt wire electrode as a counter electrode, and Ag/AgCl as a reference electrode. Photoanode was prepared by coating photocatalyst on glass slides containing fluorine-doped tin oxide (FTO), cleaned with acetone, rinsed with DI water, and oven-dried before use. The catalyst was coated over the FTO surface using the drop-casting method. To make a binder solution, 1.5 ml of ethanol was mixed with 1 ml of water and 40 µl of Nafion. 20 ml of the photocatalyst was added in binder solution and sonicated for 10 minutes. The binder solution was casted dropwise over the glass surface using a dropper and hotplate with a temperature of 45 °C. The electrochemical impedance spectroscopy was calculated in the frequency range of 1 MHz to 1 Hz using a sinusoidal AC perturbation signal of 5 mV. Mott-Schottky experiment was conducted at 1000 Hz constant frequency in dark conditions. LSV and transient photocurrent measurements were carried out under dark and illumination conditions using a 300 W Xenon arc lamp (Newport-R22) with a cut-off filter ($\lambda > 420$ nm and intensity of 100 mW.cm^{-2}).

Nitro blue tetrazolium (NBT) test

10 mL of 2.5×10^{-5} M aqueous solution of NBT was mixed with the 5 mg of the catalyst. This mixture was illuminated with light under continuous stirring. After 15 min, the catalyst was separated from the solution using a centrifuge. The solution was monitored using a UV-visible spectrophotometer, and its absorbance for NBT was compared with the neat stock solution. A decreased absorbance peak of NBT after light illumination confirms the capability of the photocatalyst for the generation of $^{\bullet}O_2^{-}$.

Terephthalic acid (THA) test

The 5×10^{-3} M aqueous solution of THA was prepared with a small amount of NaOH to make THA soluble in an aqueous medium. 20 mg of catalyst was mixed with the 20 ml of THA solution and exposed to light for 1 h. Then catalyst was separated from the solution using a centrifuge. The clear solution was monitored using a fluorescence spectrophotometer with an excitation wavelength of 330 nm. The emission peak at 425 nm confirms 2-hydroxyl terephthalic acid (THA) in the reaction mixture. The OH radicals react with THA (non-fluorescent) and convert it into THA (fluorescent). The emission peak of the reaction mixture was compared with the stock solution.

Iodometric test for H_2O_2 detection

The 0.1 M solution of potassium iodide (KI) and the 0.1 M solution of ammonium molybdate were prepared. The photocatalytic THFA oxidation reaction was performed for a different interval of time. The 3 ml of the reaction mixture was mixed with 0.5 ml of KI and 0.5 ml of ammonium molybdate solution. The obtained mixture was analysed using UV spectrophotometer. H_2O_2 in the solution oxidizes iodide to form iodine which gives a peak at 360 nm in UV visible spectrometer. The intensity of the 360 nm peak gives the amount of H_2O_2 present in the reaction mixture.

Materials characterization

Physicochemical characterization

X-ray diffraction (XRD) patterns were recorded from RIGAKU Mini-flex diffractometer using Cu Kα radiation on the 2θ scale 5° to 80°. The surface area and porosity were examined using N_2 adsorption-desorption measurements with the BELSORP instrument. All the materials were outgassed in the degassing port at 200 °C for 4 h before performing the N_2 adsorption. The relative pressure from 0.05 to 0.3 was chosen to analyze the specific surface area by applying Brunauer-Emmett-Teller (BET) equation. FT-IR spectra were recorded using Bruker Tenser 27 spectrometer. Joel instrument was used for the SEM analysis of the materials' morphology using an accelerating voltage of 10 kV. UV-DRS study of all catalysts was performed on a UV-visible spectrophotometer of Shimadzu (UV-2600) using $BaSO_4$ as standard reference material. The liquid UV analysis was also conducted using a UV-visible spectrophotometer of Shimadzu (UV-2600). Furthermore, the nanostructure level analysis was performed using a high-resolution transmission electron microscope (JEOL, Model: JEM 2100F) at an accelerating voltage of 200 kV (IIT Bombay). The X-Ray photoelectron spectroscopy (XPS)

was carried out on a Thermofisher scientific 'Nexsa Base' instrument (IIT Mandi). Fluorescence analysis was done by using Perkin Elmer LS55. The fluorescence decay time was evaluated using the TCSPC instrument of Fluorolog 3-221 fluorimeter equipped with an LED laser (INST Mohali). Electron paramagnetic resonance (EPR) spectroscopy was performed at room temperature by using a spectrometer (Bruker ER073, USA) equipped with an EPR magnet system (IIT Roorkee)

Table S1. The textural properties of catalysts prepared in this study.

Entry	Catalyst	BET surface area (m^2g^{-1})	Total pore volume ($cm^3\ g^{-1}$)
1	g-C$_3$N$_4$	38	0.260
2	E-g-C$_3$N$_4$	64	0.310
3	0.5% Pd@E-g-C$_3$N$_4$	71	0.325
4	1% Pd@E-g-C$_3$N$_4$	74	0.332
5	2% Pd@E-g-C$_3$N$_4$	79	0.339
6	3% Pd@E-g-C$_3$N$_4$	82	0.342

Table S2. The band edge potentials and bandgap of all the photocatalysts.

Entry	Catalyst	Slope value	Band Gap	Flat Band Potential vs. Ag/AgCl	Conduction Band edge Potential vs. Ag/AgCl	Conduction Band Edge Potential vs. NHE	Valance Band Edge Potential vs. NHE
1	g-C$_3$N$_4$	1.10×10^{10}	2.84	-1.11	-1.21	-1.01	1.83
2	E-g-C$_3$N$_4$	6.99×10^9	2.85	-1.14	-1.24	-1.04	1.81

3	0.5% Pd@E-g-C$_3$N$_4$	8.04 × 10^9	2.86	-1.25	-1.35	-1.15	1.71
4	1% Pd@E-g-C$_3$N$_4$	6.87 × 10^9	2.88	-1.29	-1.39	-1.19	1.69
5	2% Pd@E-g-C$_3$N$_4$	5.05× 10^9	2.89	-1.26	-1.36	-1.16	1.73
6	3% Pd@E-g-C$_3$N$_4$	4.50× 10^9	2.91	-1.20	-1.30	-1.10	1.80

Entry	Catalyst	Pre-exponential functions				Decay lifetimes (ns)					Fractional contribution			
		α_1	α_2	α_3	α_4	τ_1	τ_2	τ_3	τ_4	$\langle\tau\rangle$	f_1	f_2	f_3	f_4
1	g-C$_3$N$_4$	0.40	0.17	0.32	0.1	4.6	19.3	1.1	121.8	87.9	0.10	0.19	0.02	0.68
2	1% Pd @E-g-C$_3$N$_4$	0.33	0.11	0.08	0.47	6.1	29.3	174	1.6	125	0.10	0.16	0.69	0.04

Table S3. Best fitted parameters of multiexponential components for decay curve.

Table S4. AQY for the FAL reduction.

Entry	Catalyst	FAL Conversion	FOL Selectivity	THFA Selectivity	AQY
1	g-C$_3$N$_4$	2	100	0	12×10^{-3}
2	E-g-C$_3$N$_4$	4	100	0	25×10^{-3}
2	1% Pd @g-C$_3$N$_4$	45	93	7	289×10^{-3}
3	0.5% Pd @E-g-C$_3$N$_4$	38	100	0	241×10^{-3}
4	1% Pd @E-g-C$_3$N$_4$	60	89	11	383×10^{-3}
5	2% Pd @E-g-C$_3$N$_4$	70	85	15	448×10^{-3}
6	3% Pd @E-g-C$_3$N$_4$	75	67	33	484×10^{-3}
7	1% Ni @E-g-C$_3$N$_4$	11	100	0	70×10^{-3}
8	1% Co @E-g-C$_3$N$_4$	4	100	0	25×10^{-3}

Reaction conditions: Catalyst (25 mg), light source (150 W LED), FAL (0.1 mmol), IPA (5 ml), time (1 h), and H$_2$ (2 bar).

Entry	Catalyst	FOL Conversion	THFA Selectivity	AQY
1	g-C$_3$N$_4$	2	100	13×10^{-3}
2	E-g-C$_3$N$_4$	3	100	19×10^{-3}
2	1% Pd @g-C$_3$N$_4$	36	100	228×10^{-3}

3	0.5% Pd @E-g-C$_3$N$_4$	18	100	114×10^{-3}
4	1% Pd @E-g-C$_3$N$_4$	44	100	281×10^{-3}
5	2% Pd @E-g-C$_3$N$_4$	62	100	397×10^{-3}
6	3% Pd @E-g-C$_3$N$_4$	80	100	514×10^{-3}

reduction.

Reaction conditions: Catalyst (25 mg), light source (150 W LED), FOL (0.1 mmol), IPA (5 ml), time (1h), and H$_2$ (2 bar).

Table S6. AQY for the THFA oxidation.

Entry	Catalyst	THFA Conversion	GBL Selectivity	AQY
1	g-C$_3$N$_4$	35	100	28 × 10^{-3}
2	E-g-C$_3$N$_4$	40	100	32 × 10^{-3}
2	0.5% Pd @E-g-C$_3$N$_4$	53	100	42 × 10^{-3}
3	1% Pd @E-g-C$_3$N$_4$	62	100	50 × 10^{-3}
4	2% Pd @E-g-C$_3$N$_4$	70	100	56 × 10^{-3}
5	3% Pd @E-g-C$_3$N$_4$	75	100	59 × 10^{-3}

Reaction conditions: Catalyst (25 mg), light source (150W LED), THFA (0.1 mmol), ACN (3 ml), time (8 h), and O$_2$ atmosphere (1 bar).
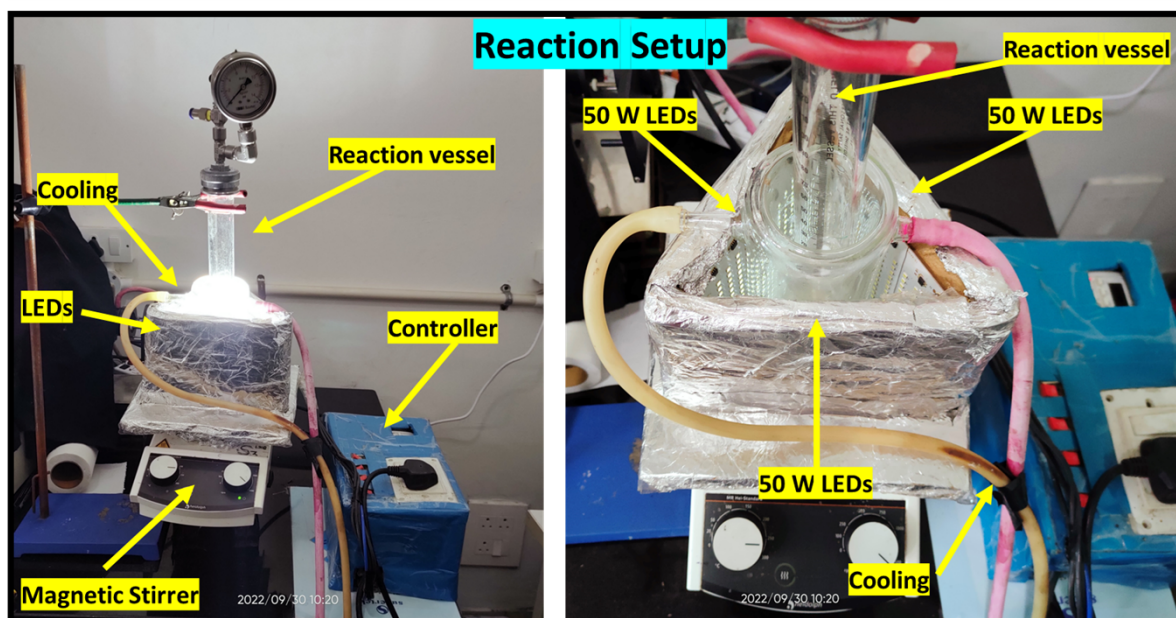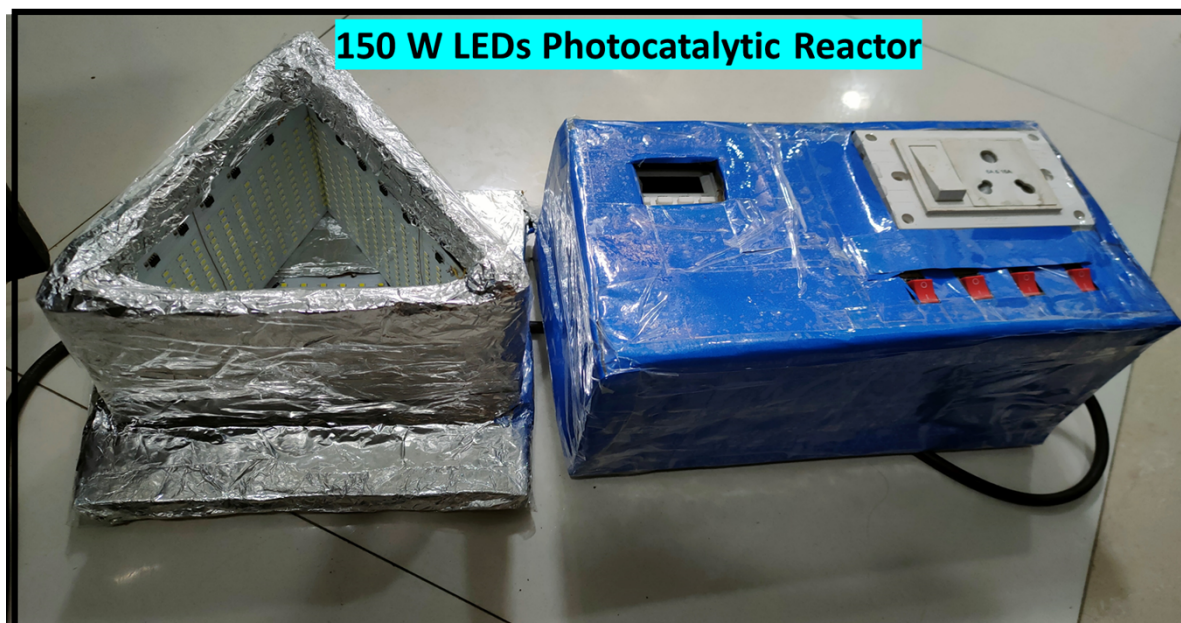
Fig. S1. The digital image of photocatalytic reactor and reaction setup for the photocatalytic reduction.
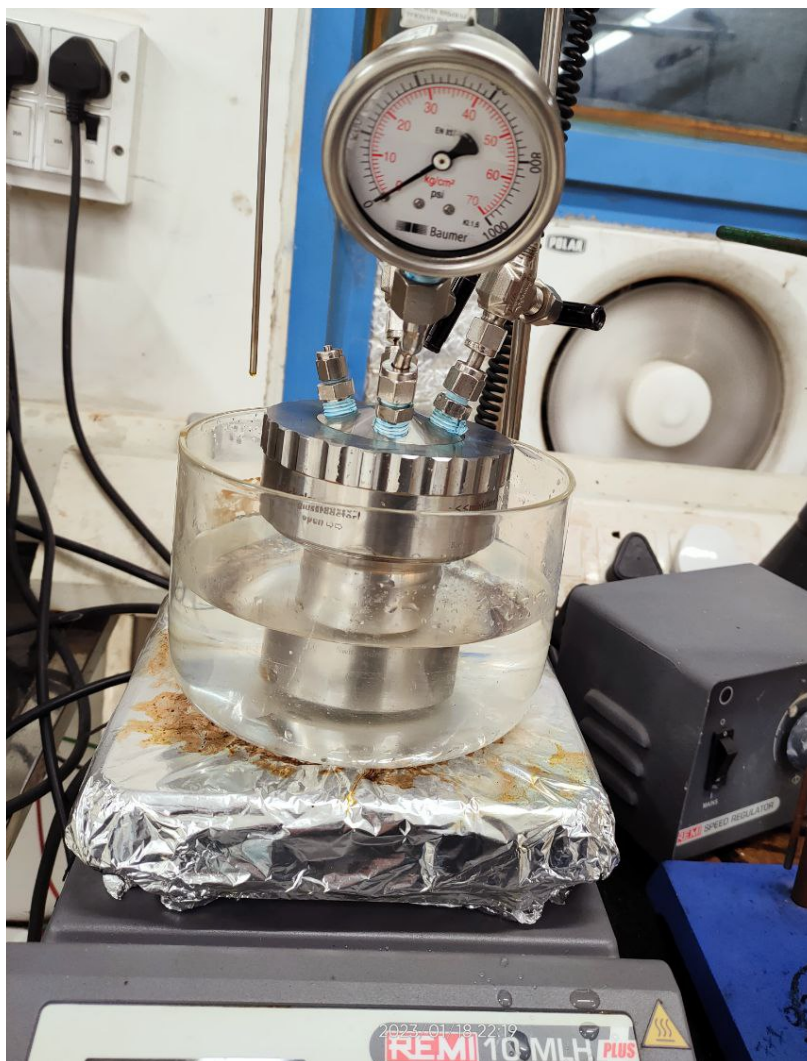
Fig. S2. The digital image of a liquid phase pressure reactor and reaction setup for the thermal catalytic reduction.
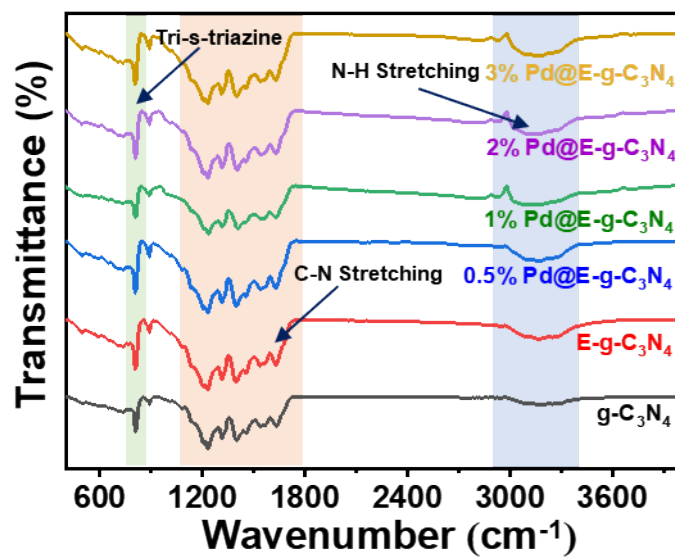
Fig. S3. FT-IR of all the synthesized photocatalysts.
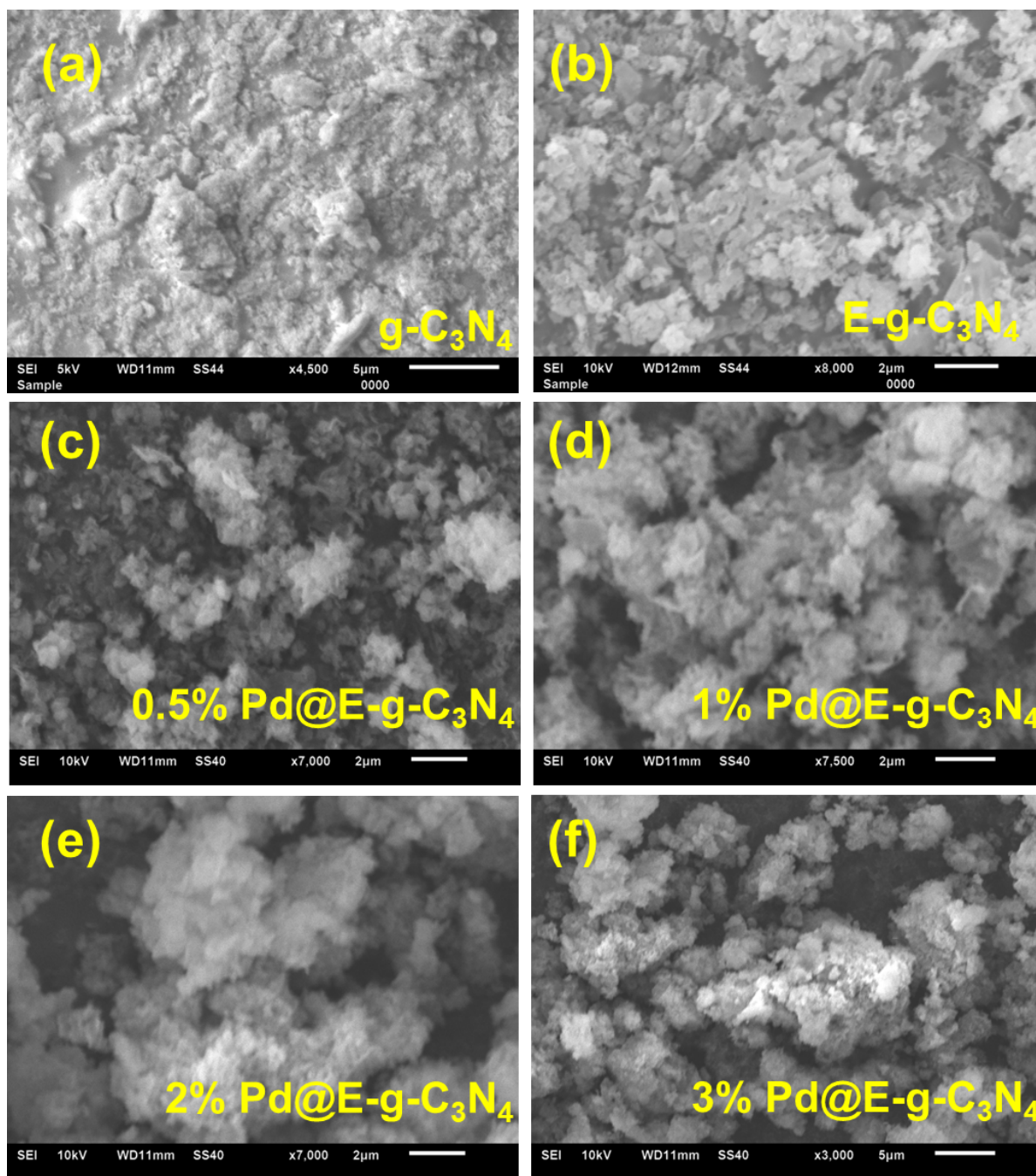
Fig. S4 SEM image of (a) g-C$_3$N$_4$, (b) E-g-C$_3$N$_4$, (c) 0.5% Pd@E-g-C$_3$N$_4$, (d) 1% Pd@E-g-C$_3$N$_4$, (e) 2% Pd@E-g-C$_3$N$_4$, and (f) 3% Pd@E-g-C$_3$N$_4$.
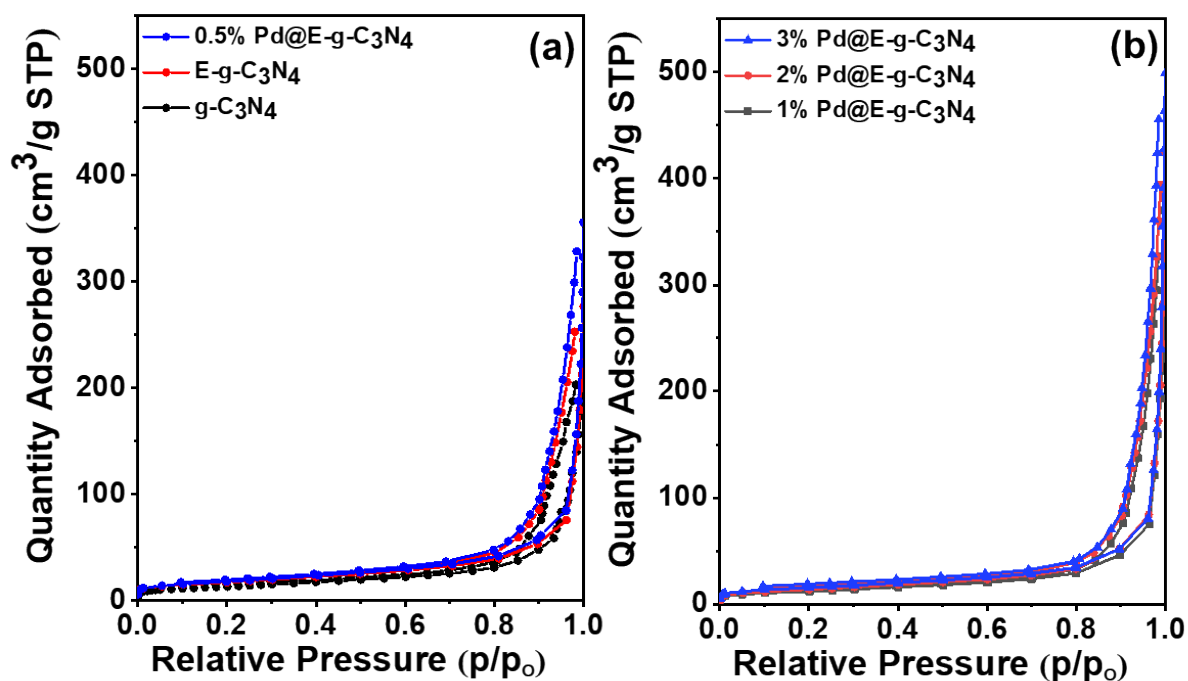
Fig. S5 N_2-adsorption-isotherms of (a) g-C_3N_4, E-g-C_3N_4, and 0.5% Pd@E-g-C_3N_4, (b) 1% Pd@E-g-C_3N_4, 2% Pd@E-g-C_3N_4, and 3% Pd@E-g-C_3N_4.
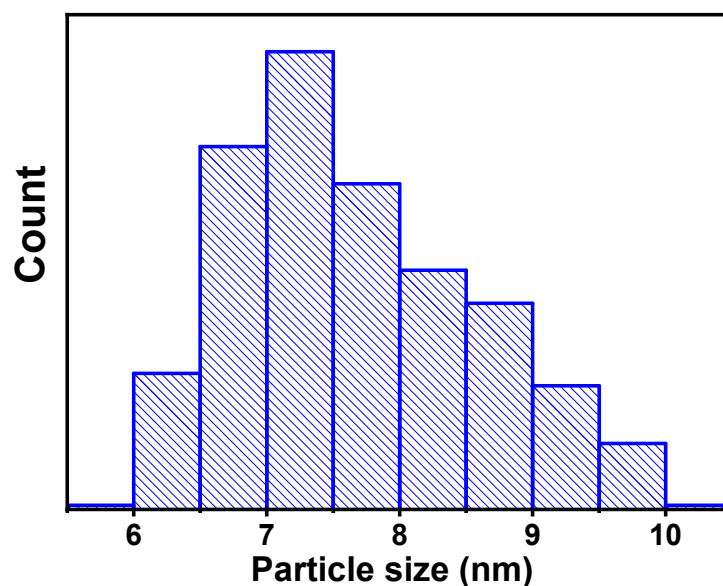
Fig. S6 Pd nanoparticles size distribution from TEM.
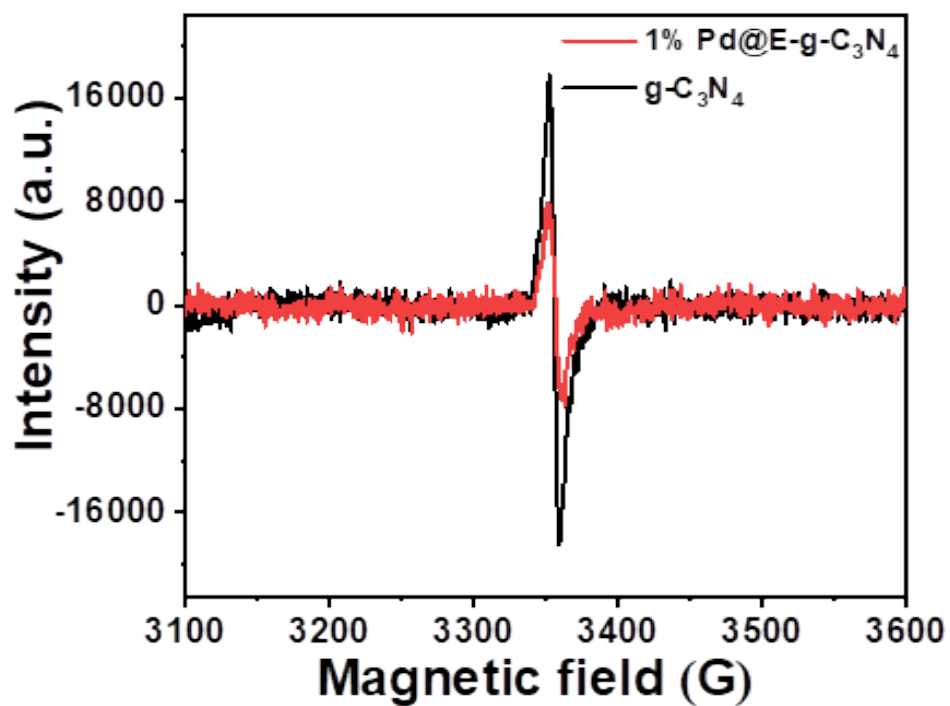
Fig. S7 EPR signals of bulk g-C$_3$N$_4$ and 1% Pd@E-g-C$_3$N$_4$.
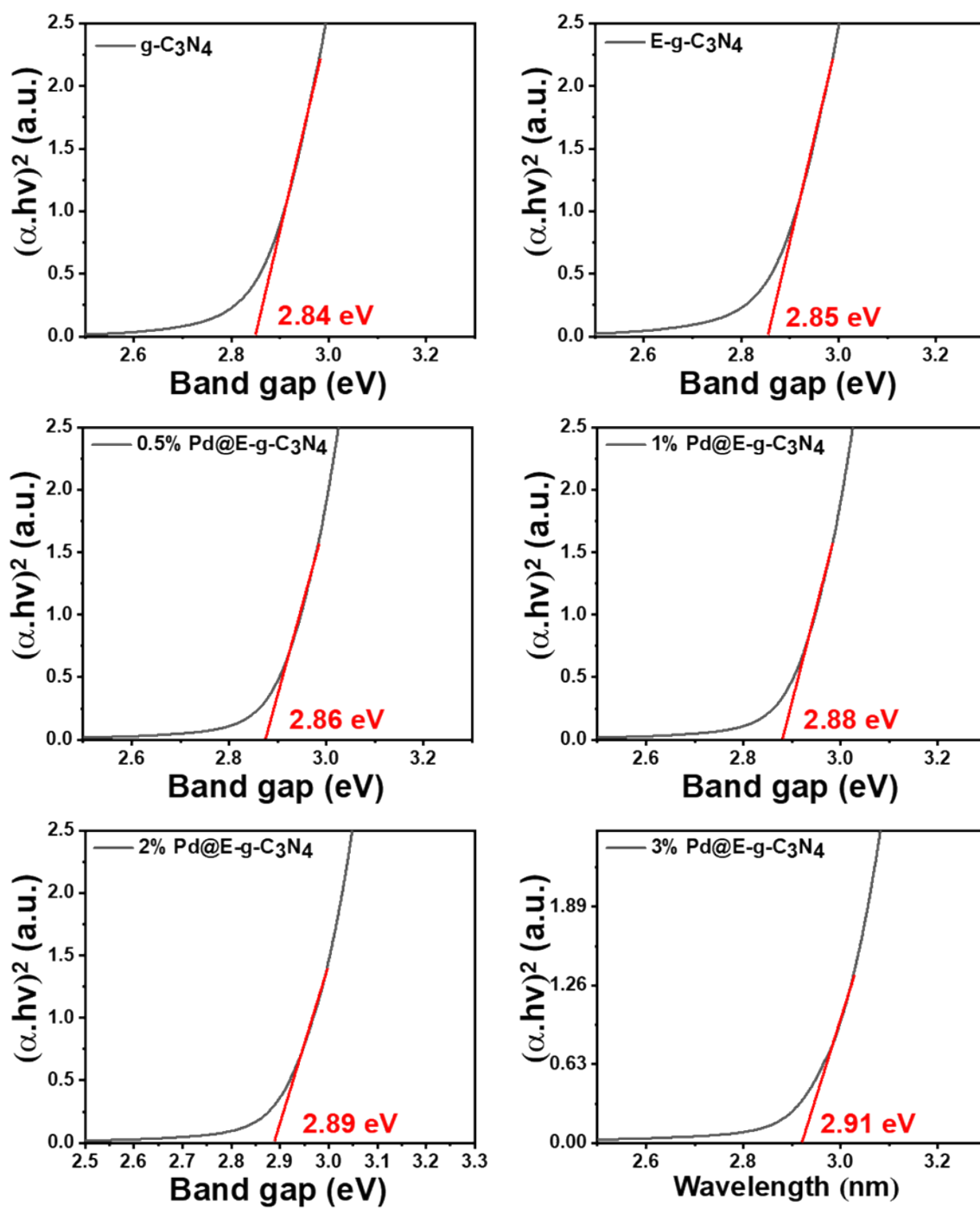
Fig. S8 Tauc plots of all synthesized photocatalyst.
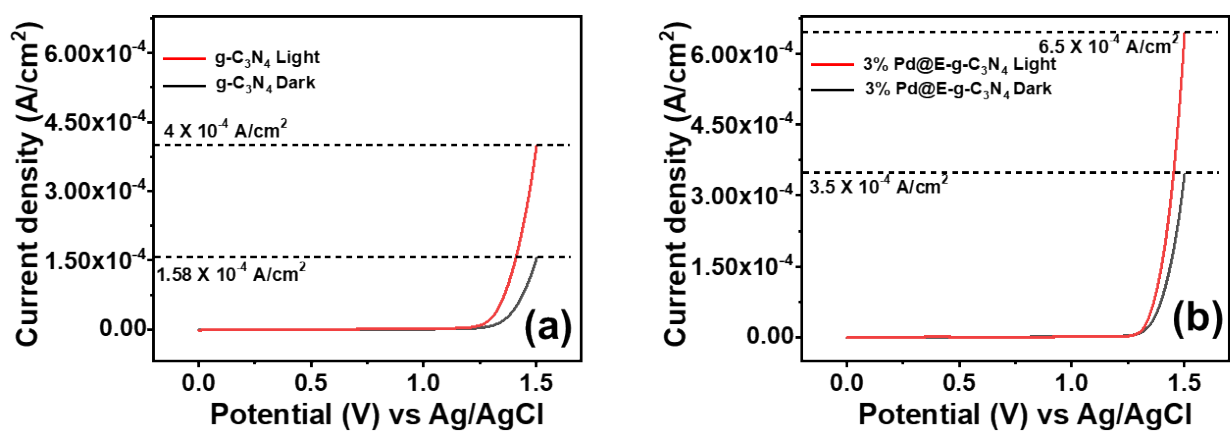
Fig. S9 LSV spectra of pristine g-C$_3$N$_4$ and 3% Pd@E-g-C$_3$N$_4$ in (a) dark and (b) light.
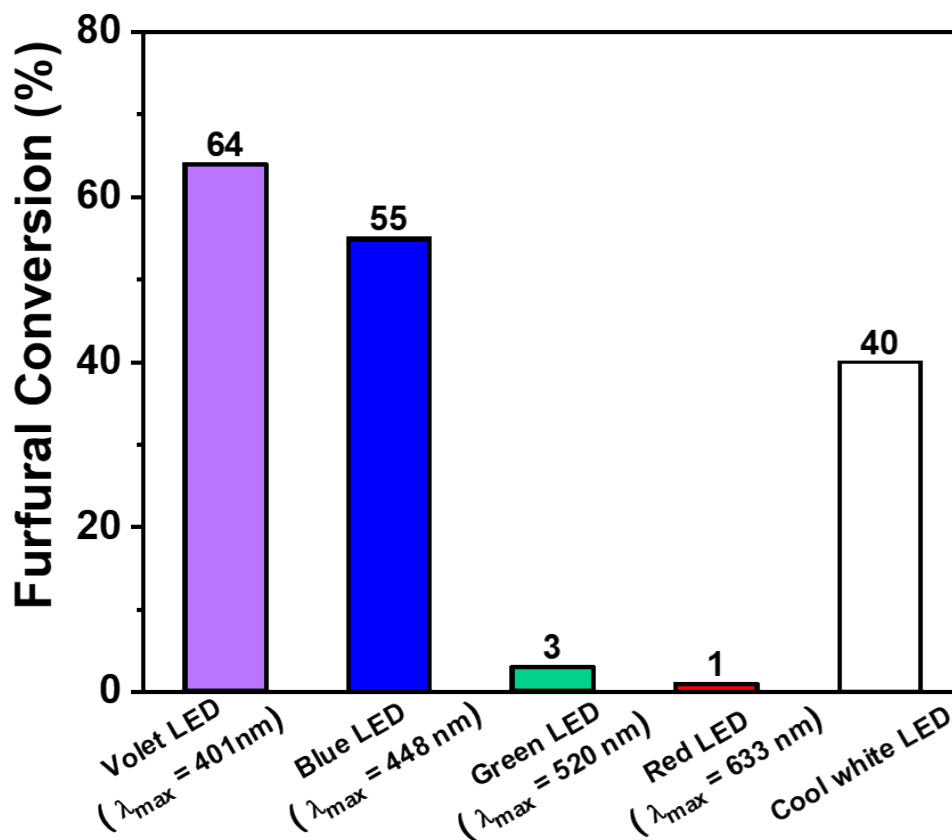
Fig. S10 FAL reduction using different colour 9 W LEDs (Reaction conditions: 1% Pd@E-C_3N_4 (25 mg), FAL (0.1 mmol), IPA (5 ml), time (5 h), and H_2 (2 bar)).
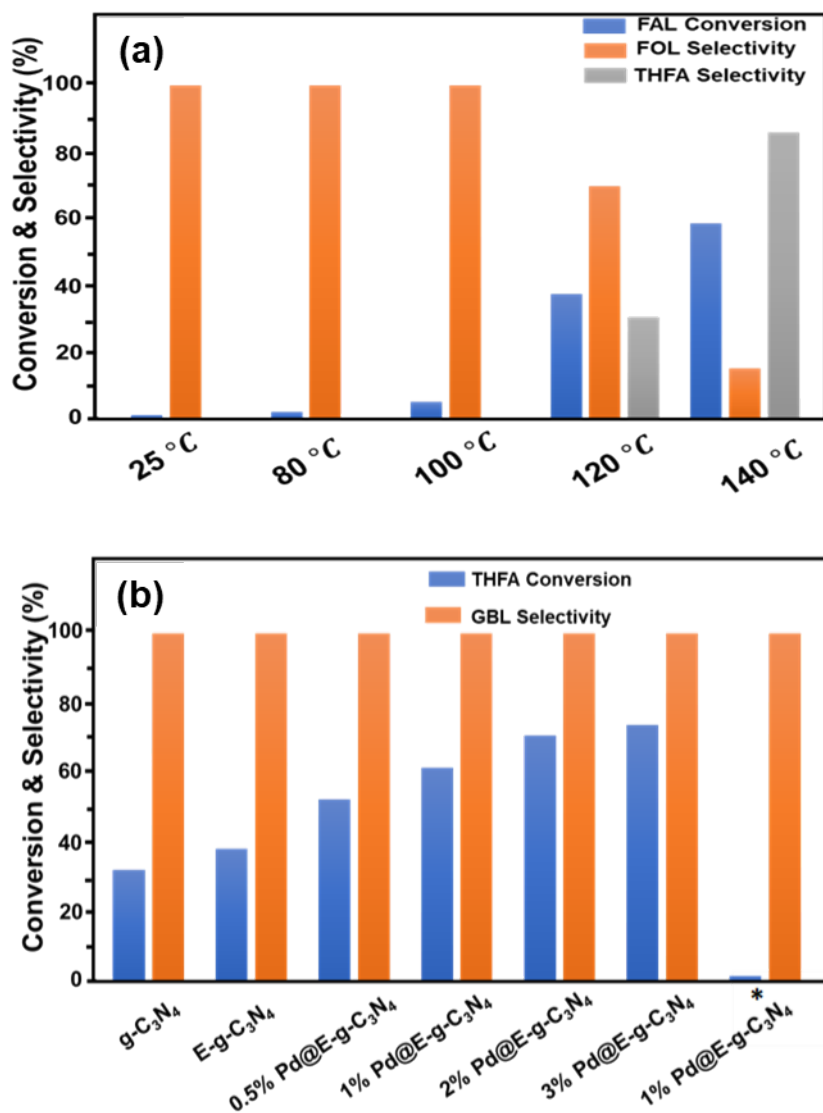
Fig. S11 (a) FAL reduction at different temperatures in dark (Reaction conditions: 1% Pd@E-C$_3$N$_4$ (25 mg), FAL (0.1 mmol), IPA (5 ml), time (1h), and H$_2$ (5 bar), and (b) THFA oxidation with different catalysts (Reaction conditions: catalyst (25 mg), light source (150W LED), THFA (0.1 mmol), ACN (3 ml), time (8 h), and O$_2$ atmosphere (1 bar).
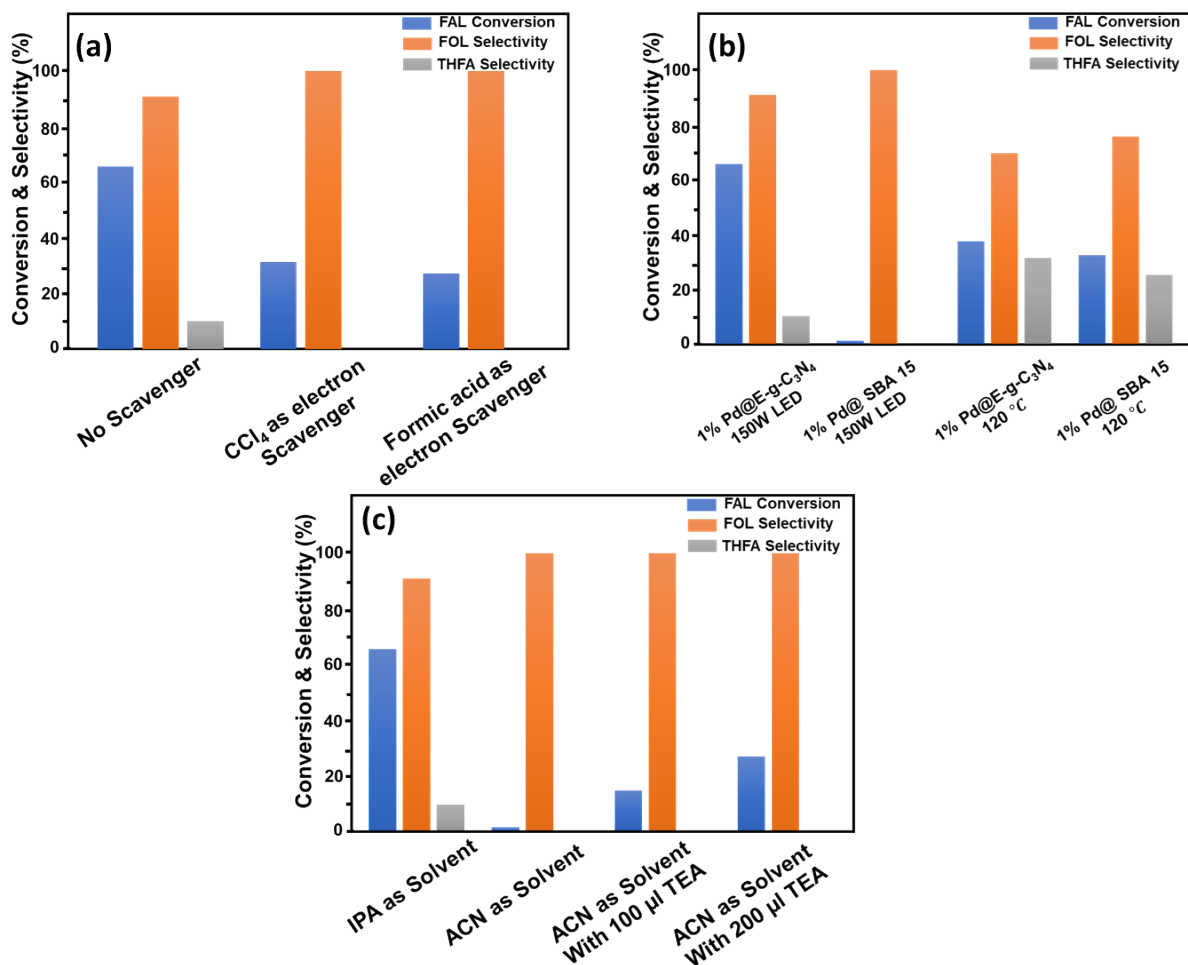
Fig. S12 Control experiments during FAL reduction (a) with using 1.5 mmol of electron scavenger, (b) by employing two different catalysts in light (rt) and in dark (at 120 °C), (c) in IPA, ACN and ACN + TEA. (Reaction conditions: 1% Pd@E-g-C$_3$N$_4$ (25 mg), light source (150W LED), FAL (0.1 mmol), IPA (5 ml), time (1 h), and H$_2$ (2 bar in light) and H$_2$ (5 bar in dark).
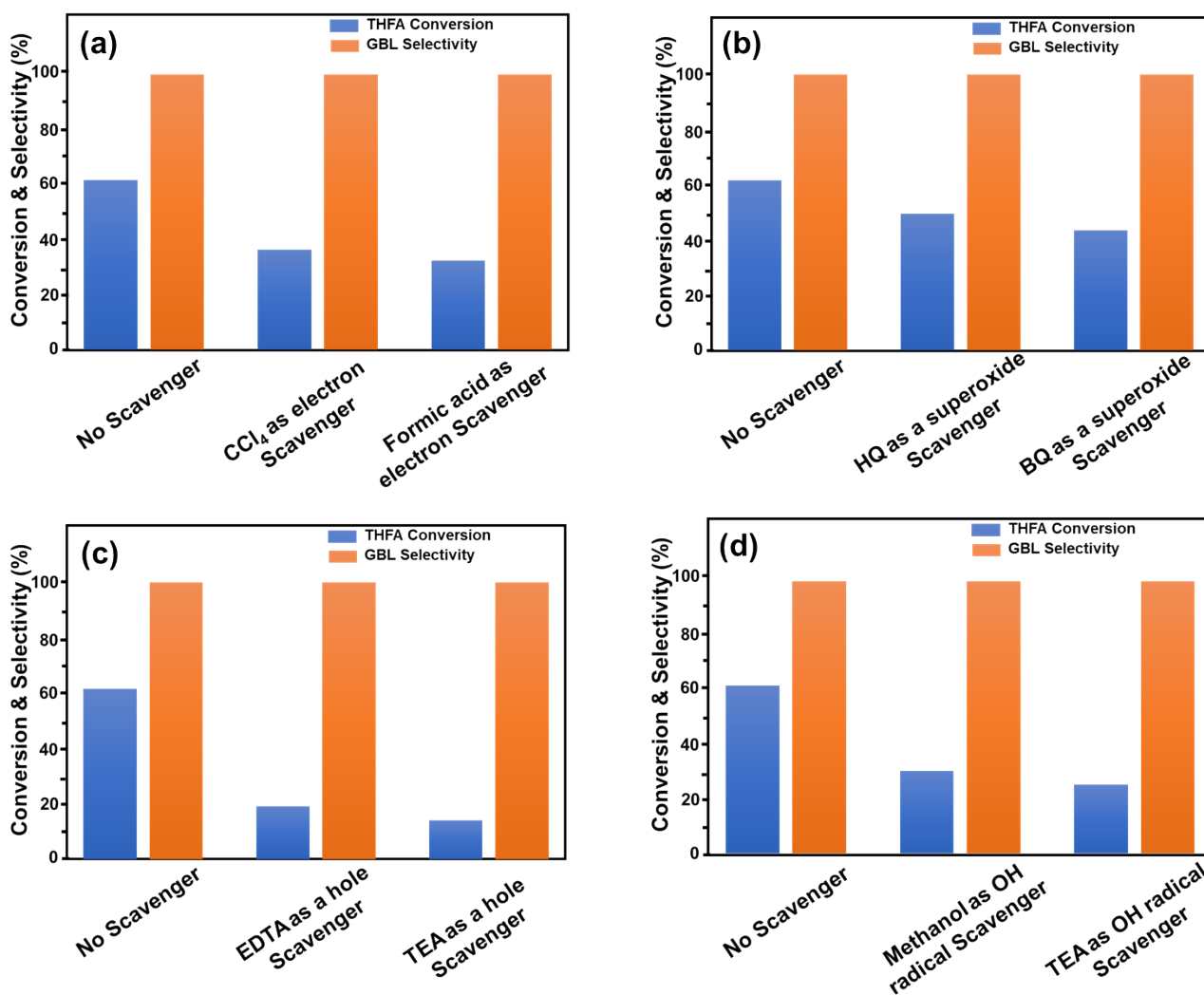
Fig. S13 Control experiments during THFA oxidation (a) using 1.5 mmol of electron scavenger, (b) using 1.5 mmol of superoxide scavenger, (c) 1.5 mmol of hole scavenger, and (d) using 1.5 mmol of OH• radical scavenger..(Reaction conditions: 1% Pd@E-g-C$_3$N$_4$ (25 mg), light source (150W LED), THFA (0.1 mmol), ACN (3 ml), time (8 h), and O$_2$ atmosphere (1 atm).
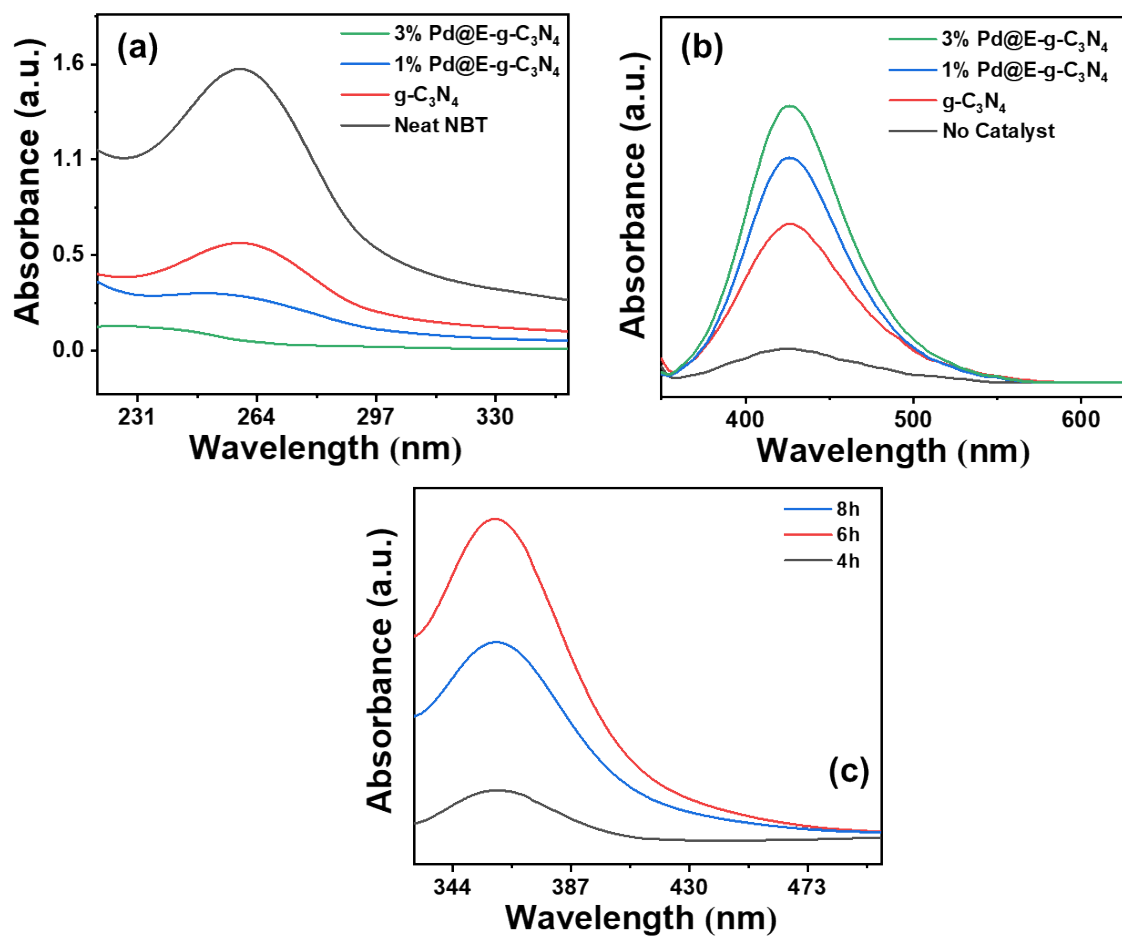
Fig. S14 (a) Comparative absorption spectra of NBT solution before and after light illumination for 15 min over pristine g-C$_3$N$_4$ and Pd decorated E-g-C$_3$N$_4$, (b) Comparative fluorescence spectra of THA solution before and after light illumination for 60 min over g-C$_3$N$_4$ and Pd decorated E-g-C$_3$N$_4$, and (c) UV-visible spectra recorded during the iodometric test for the analysis of photogenerated H$_2$O$_2$ during the reaction.
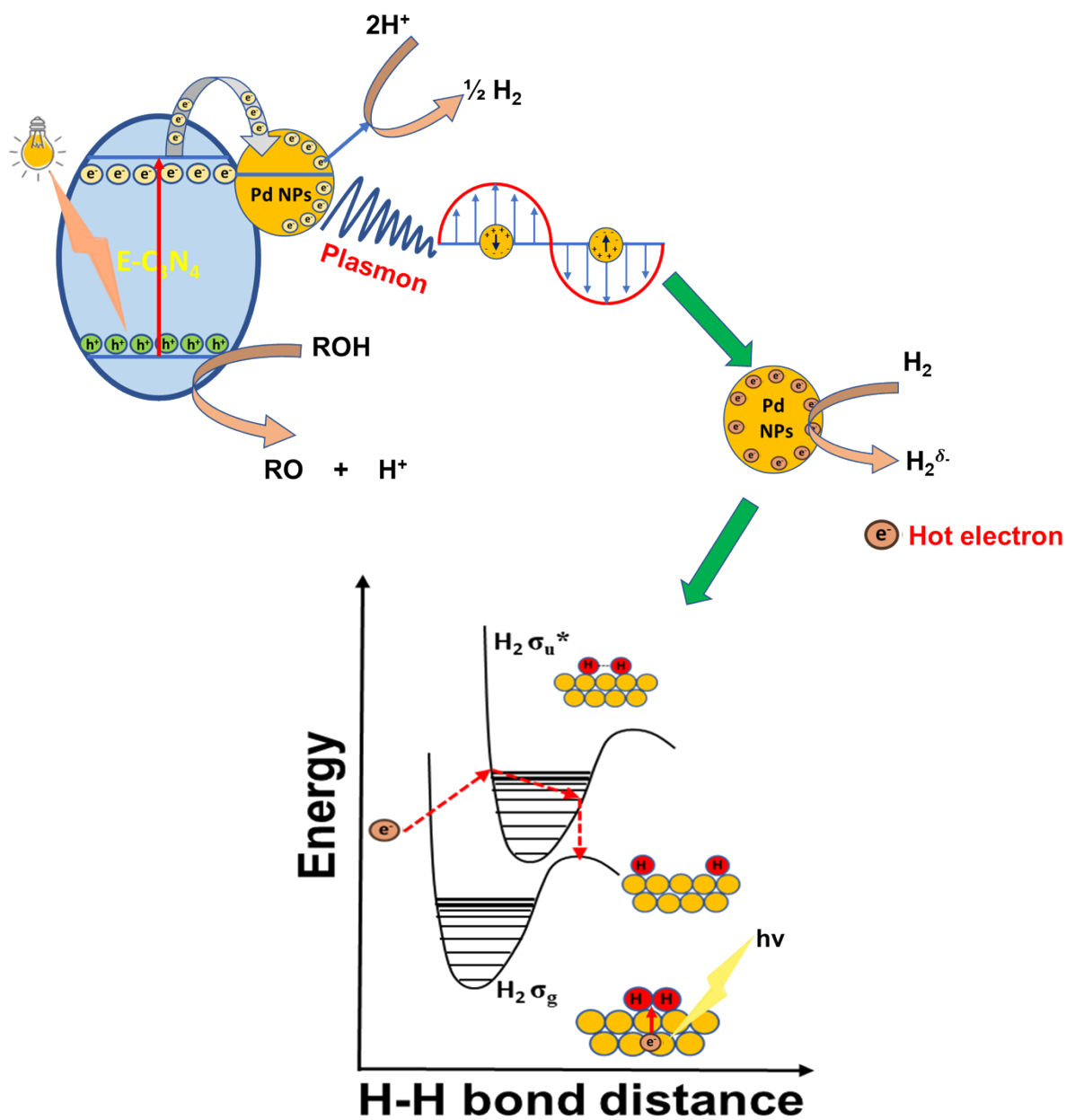
Fig. S15 Proposed mechanism for the hot electron mediated H_2 dissociative adsorption and desorption over Pd NPS.
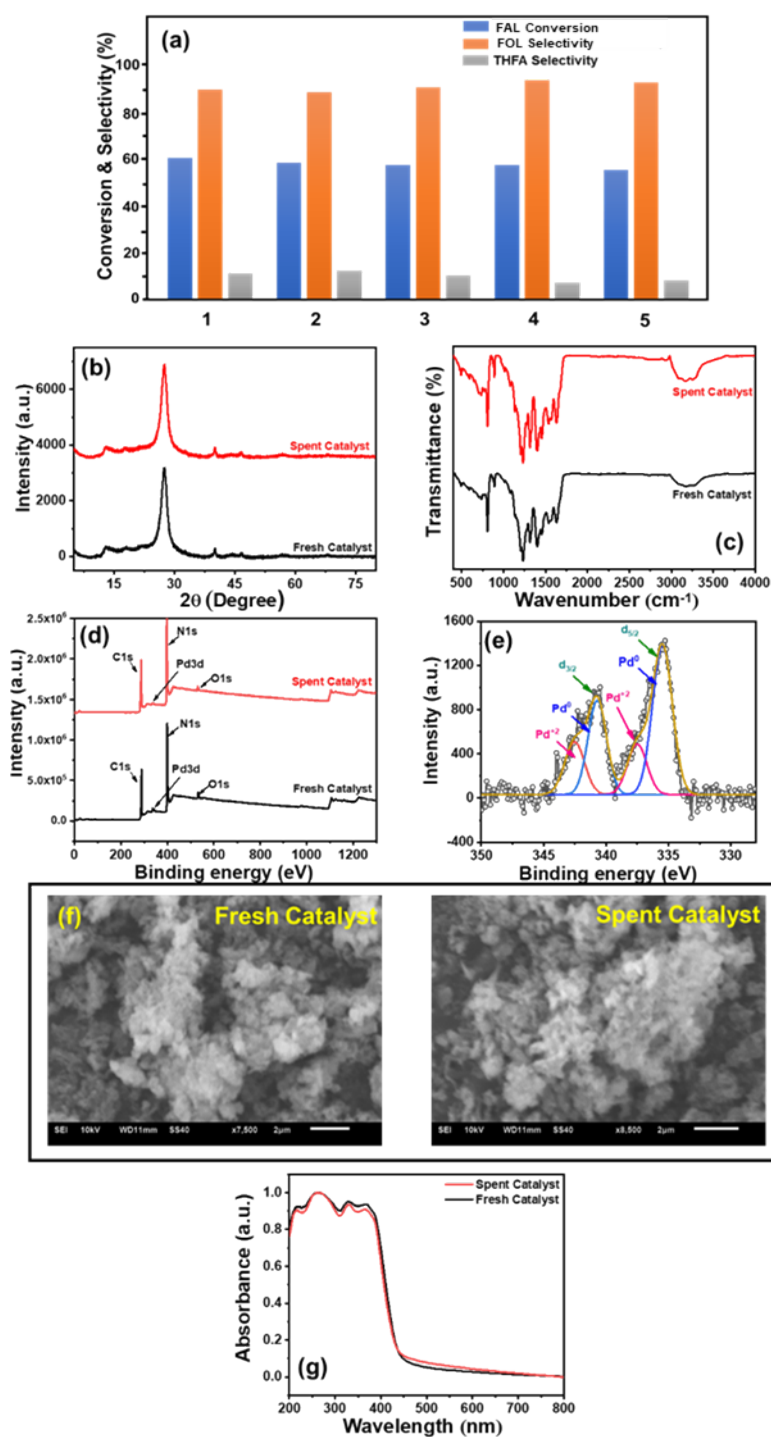
Fig. S16 (a) Recyclability data using 1% Pd@E-g-C$_3$N$_4$ for FAL reduction reaction (Reaction conditions: 1% Pd@E-g-C$_3$N$_4$ (25 mg), light source (150W LED), FAL (0.1 mmol), IPA (5 ml), time (1 h), and H$_2$ (2 atm) (b) XRD of fresh and spent 1% Pd@E-g-C$_3$N$_4$, (c) FT-IR of fresh and spent 1% Pd@E-g-C$_3$N$_4$, (d) XPS elemental survey for fresh and spent 1% Pd@E-g-C$_3$N$_4$, (e) deconvoluted XPS of Pd NPs in spent 1% Pd@E-g-C$_3$N$_4$, (f) SEM images of fresh and spent 1% Pd@E-g-C$_3$N$_4$, and (g) DR UV-vis spectra of fresh and spent 1% Pd@E-g-C$_3$N$_4$.
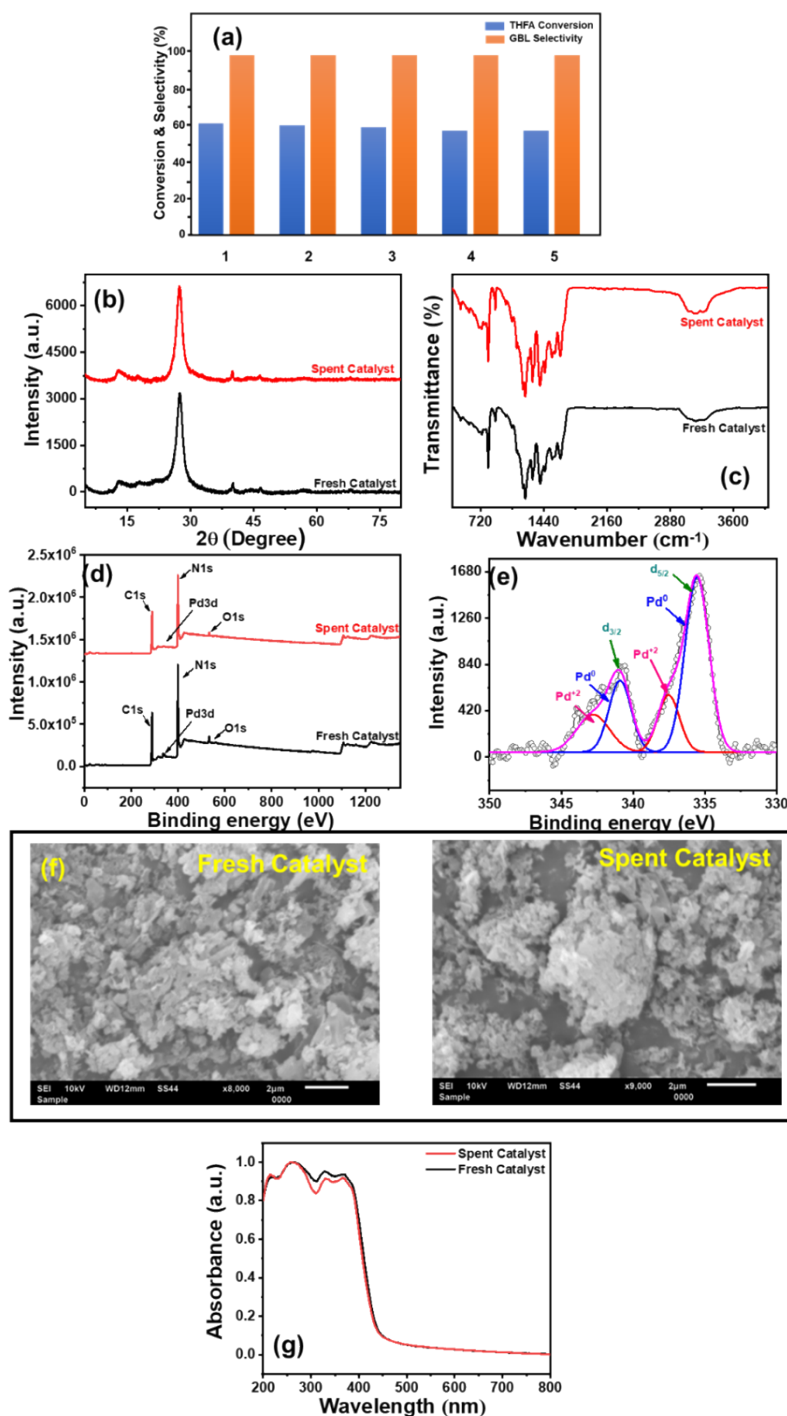
Fig. S17 (a) Recyclability data using 1% Pd@E-g-C$_3$N$_4$ in THFA oxidation (Reaction conditions: 1% Pd@E-g-C$_3$N$_4$ (25 mg), light source (150W LED), THFA (0.1 mmol), ACN (3 ml), time (8 h), and O$_2$ atmosphere (1 atm), (b) XRD of fresh and spent 1% Pd@E-g-C$_3$N$_4$, (c) FT-IR of fresh and spent 1% Pd@E-g-C$_3$N$_4$, (d) XPS elemental survey of fresh and spent 1% Pd@E-g-C$_3$N$_4$, (e) deconvoluted XPS of Pd NPs in spent 1% Pd@E-g-C$_3$N$_4$, (f) SEM images of fresh and spent 1% Pd@E-g-C$_3$N$_4$, and(g) DR UV-vis spectra of fresh and spent 1% Pd@E-g-C$_3$N$_4$.