

Supporting Information

Engineering of Highly Active Au/Pd Supported on Hydrogenated Urchin-like Yolk@Shell TiO₂ for Visible Light Photocatalytic Suzuki Coupling

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1. Experimental Section

1.1. Chemicals and apparatus

All the chemical reagents used in our experiments were purchased from Sigma-Aldrich and Merck and were used as received without further purification. Water was purified with a Milli-Q system ($\geq 18 \text{ M}\Omega\cdot\text{cm}$). UV-vis DRS of samples was obtained using AvaSpec-2048 TEC spectrometer. Microscopic morphology of products was visualized by SEM (Tescan, Mira3). Powder X-ray diffraction (XRD) was carried out on a Philips diffractometer (X'pert Company) with monochromatized Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$). XPS measurements were performed by a VG scientific photoelectron spectrometer ESCALAB-210 using Al K α radiation (1486.6 eV) from an X-ray source operating at 15 kV and 20 mA. Transmission electron microscopy (TEM) was obtained on Philips CM30 with an accelerating voltage of 150 kV. High resolution transmission electron microscopy (HRTEM) was obtained on JEOL JEM 2010 - TEM under 220 kV. ^1H NMR spectra were recorded in CDCl_3 on Bruker Avance-400 MHz spectrometers in the presence of tetramethylsilane as internal standard.

1.2. Catalysts synthesis

1.2.1. Synthesis of yolk@shell TiO_2 (Y@S-TO) structure

The Y@S-TO was synthesized according to our recently published article with some modifications [1].

1.2.2. Synthesis of hierarchical urchin like yolk@shell TiO₂ (HUY@S-TO):

A hydrothermal dissolution–re-crystallization method in alkaline media was used for the synthesis of (HUY@S-TO) microspheres. A solution of EtOH (6 mL), H₂O (6 mL) and NaOH (0.48 g) was mixed with 0.50 g of the above prepared Y@S-TiO₂ microspheres through vigorously stirred (20 min) and sonication for 10 min. Thereafter, the suspension was transferred into a Teflon-lined autoclave (18 mL in capacity). The autoclave was sealed and heated at 150 °C for 3 h in an oven. After reacting, the sample was allowed to cool to ambient temperature and the product was separated by centrifugation. Next, the precipitate was immersed in 0.10 M HCl solution (100 mL) for 5 min and then washed with deionized water until the pH value was near to 7, and then dried at 80 °C for 12 h.

1.2.3. Synthesis of hierarchical urchin like yolk@shell TiO_{2-x}H_x (HUY@S-TOH):

For the preparation of Y@HUS-TiO₂ structures, the synthesized HUY@S-TO microspheres were calcined at 500 °C for 3 h with a heating rate of 3 °C min⁻¹ at 200 psi flow rate under a H₂/Ar (1:1) atmosphere. After that, the system was cooled to ambient temperature under a H₂/Ar atmosphere

1.2.4. Preparation of bimetallic core-shell Au-Pd NPs:

The core-shell Au-Pd NPs were prepared *via* a seed-mediated growth technique.[2] Firstly, 0.10 mmol of HAuCl₄.3H₂O was dissolved in 10 mL of oleylamine. Next, the solution was heated to 150 °C and kept at this temperature for 1 h to reduce the Au³⁺ ions by use of oleylamine. Subsequently, for the direct growth of a thin Pd shell on the Au seeds, 0.015 mmol of Pd(OAc)₂ was swiftly added into the solution of Au seeds at 150 °C, under vigorous stirring and the mixture was continuously kept at this temperature for 2 h. The resulting core-shell Au-Pd NPs, were precipitated by MeOH (5 mL), followed by centrifugation and washing with MeOH several time, then re-dispersed in 10 mL of toluene.

1.2.5. Synthesis of hierarchical urchin like yolk@shell TiO_{2-x}H_x decorated by Au-Pd nanoparticles (HUY@S-TOH/AuPd):

0.25 g of the prepared HUY@S-TOH microspheres were dispersed in 20 mL THF and sonicated for 30 min. The resulting mixture was then transferred to a flask and the obtained Au-Pd solution was added under vigorous stirring for 5h. Subsequently, the precipitate was separated by centrifugation, washed with EtOH (3 times) and dried under vacuum at 50 °C for 12 h. For comparison, the procedure was repeated with P25 and Y@S-TO, HUY@S-TO) to yield P25/AuPd, Y@S-TO/AuPd and HUY@S-TO/AuPd respectively.

1.2.6. Synthesis of HUY@S-TOH/Au structure

The HUY@S-TOH/Au was synthesized according to a recently published article. The HUY@S-TOH structure was used instead of TiO₂ and amount of Au was tuned to 3 wt% [3].

1.2.7. Synthesis of HUY@S-TOH/Pd structure

The HUY@S-TOH/Pd was synthesized according to a recently published article. The HUY@S-TOH structure was used instead of TiO₂ and amount of Pd was tuned to 3 wt% [4].

1.3. Visible-light-induced Suzuki coupling reaction

The light-induced Suzuki reaction was performed in a reaction tube under the irradiation of a Xe lamp (300 W) equipped with mechanical fan and chiller to control the temperature during the experiments. In a typical reaction, aryl halide (1.0 mmol), phenylboronic acid (1.3 mmol), K₂CO₃ (3 mmol), and catalyst (5 mg) were added to a mixture of EtOH:H₂O (2:1, 6 mL). After completion of the reaction, (as monitored by TLC), the catalyst was easily separated by centrifugation and the reaction mixture was washed with H₂O to remove excess of boronic acids. Then the organic layer was extracted with ethyl acetate (3×5 mL), dried over anhydrous MgSO₄ and the solvent was evaporated under reduced pressure to give the corresponding biaryls. The purity of the products was checked by ¹H-NMR and ¹³C-NMR analysis.

2. Supplementary Figures and Tables

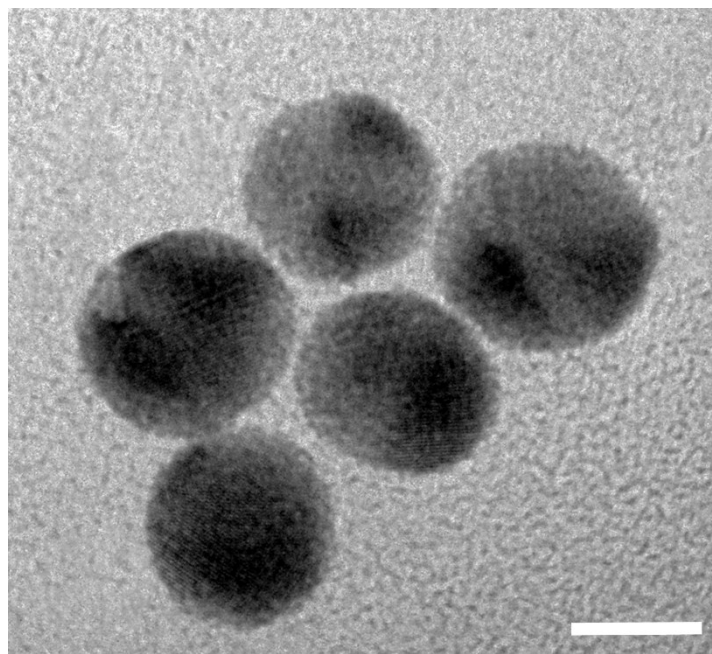


Fig. S1. HRTEM image of the AuPd nanoparticles. Scale bar is 5nm.

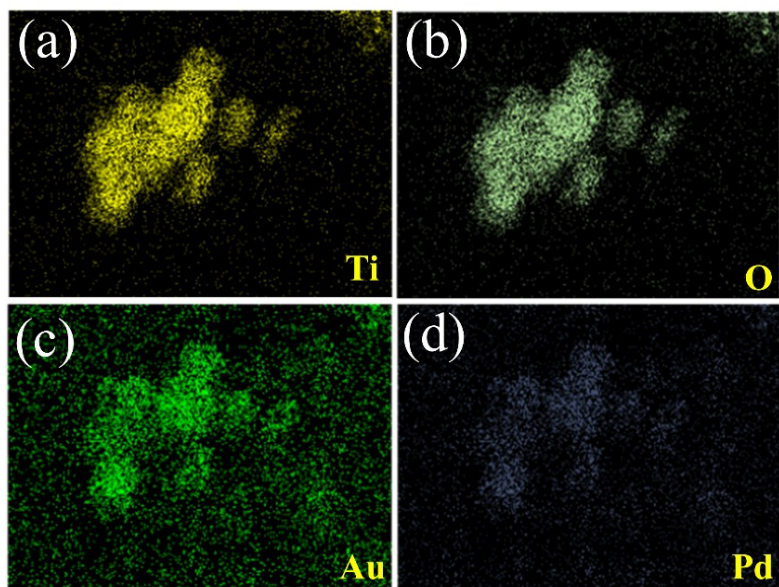


Fig. S2. EDS mapping images of the HUY@S-TOH/AuPd.

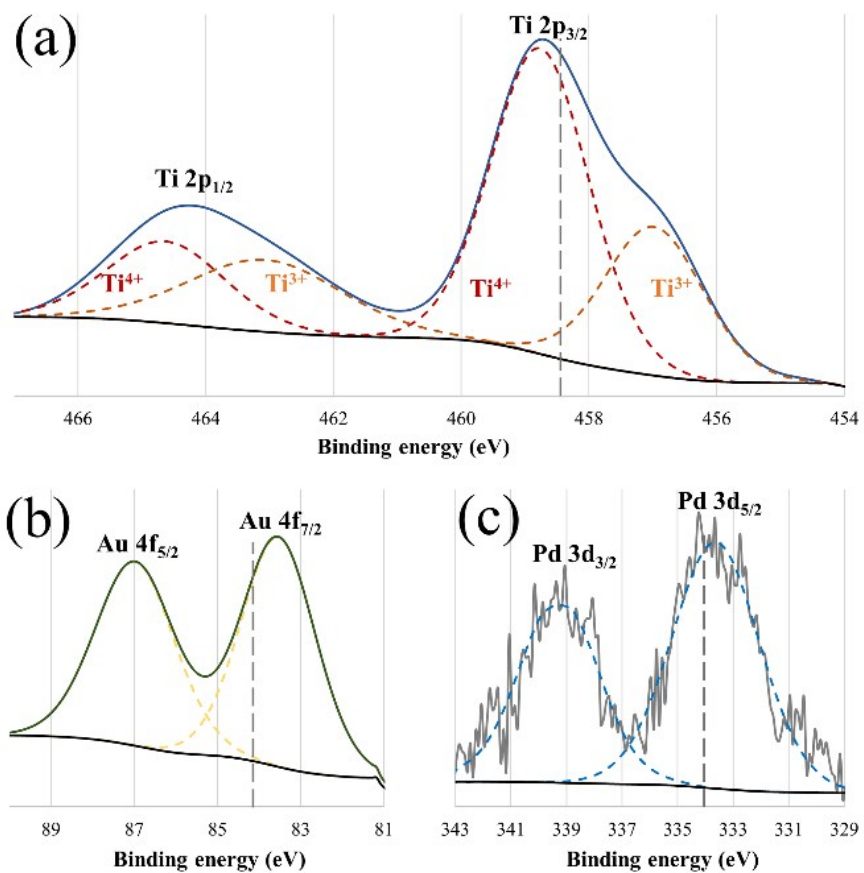


Fig. S3. High-resolution XPS spectra of Ti 2p (a), Au 4f (b) and Pd 3d (c) of the HUY@S-TOH/AuPd architecture.

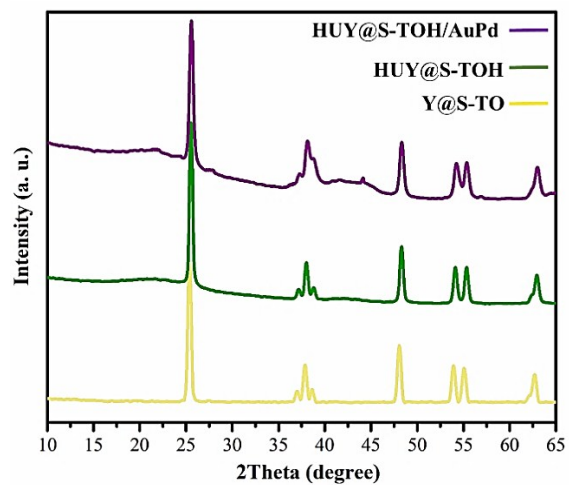


Fig. S4. XRD patterns and DRS spectra of Y@S-TO, HUY@S-TOH and HUY@S-TOH/AuPd architectures.

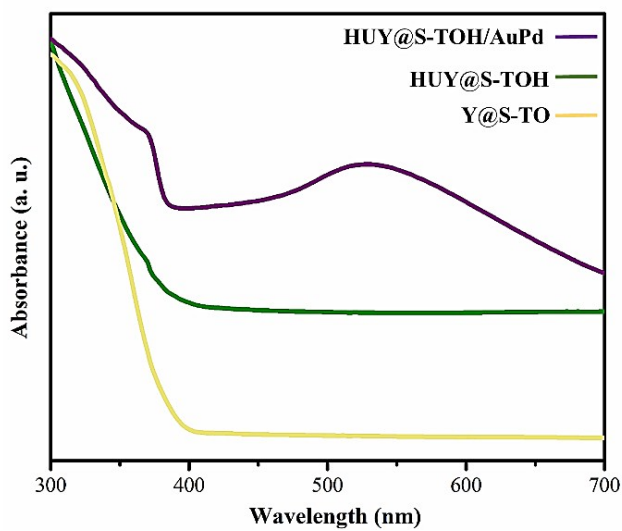


Fig. S5. DRS spectra of Y@S-TO, HUY@S-TOH and HUY@S-TOH/AuPd architectures.

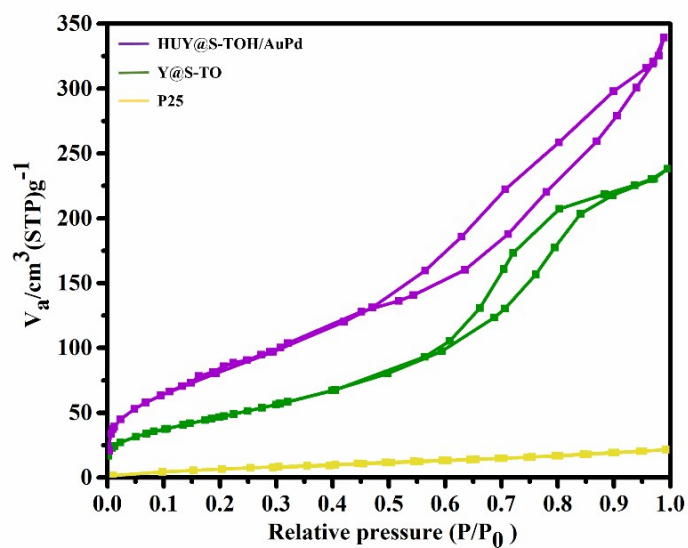


Fig. S6. N_2 adsorption-desorption of P25, Y@S-TO, HUY@S-TOH/AuPd structures.

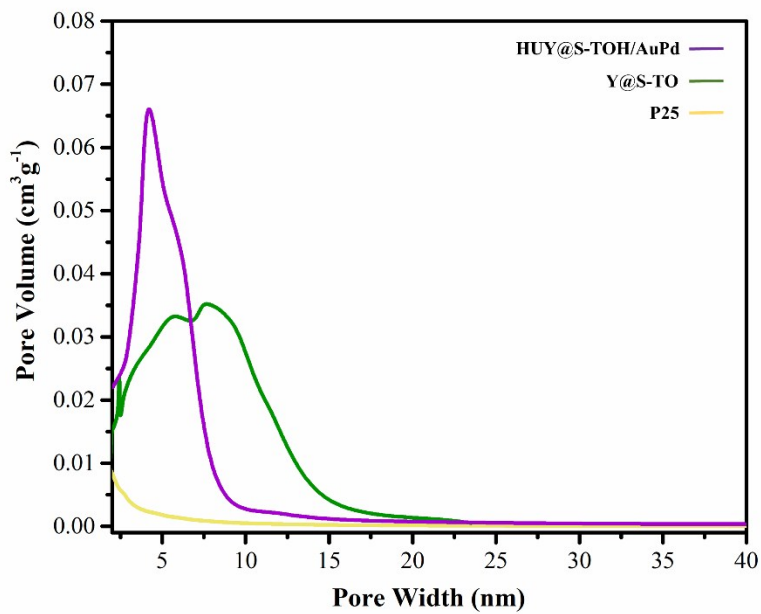


Fig. S7. Pore volume distribution of P25, Y@S-TO, HUY@S-TOH/AuPd structures.

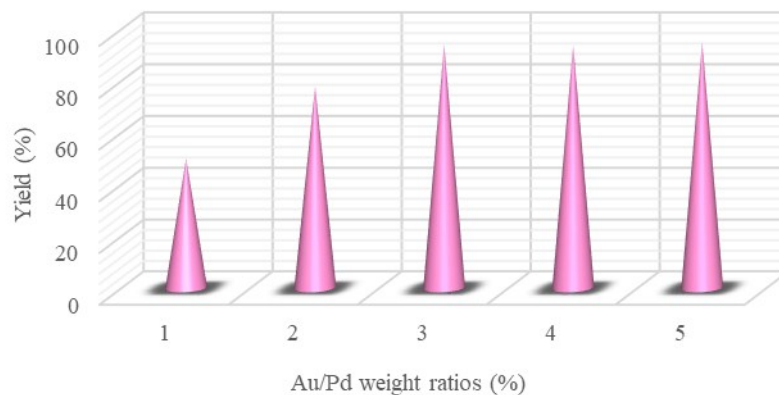


Fig. S8. Effect of loading ratios of Au/Pd nanoparticles on the HUY@S-TOH architecture in the Suzuki model reaction.

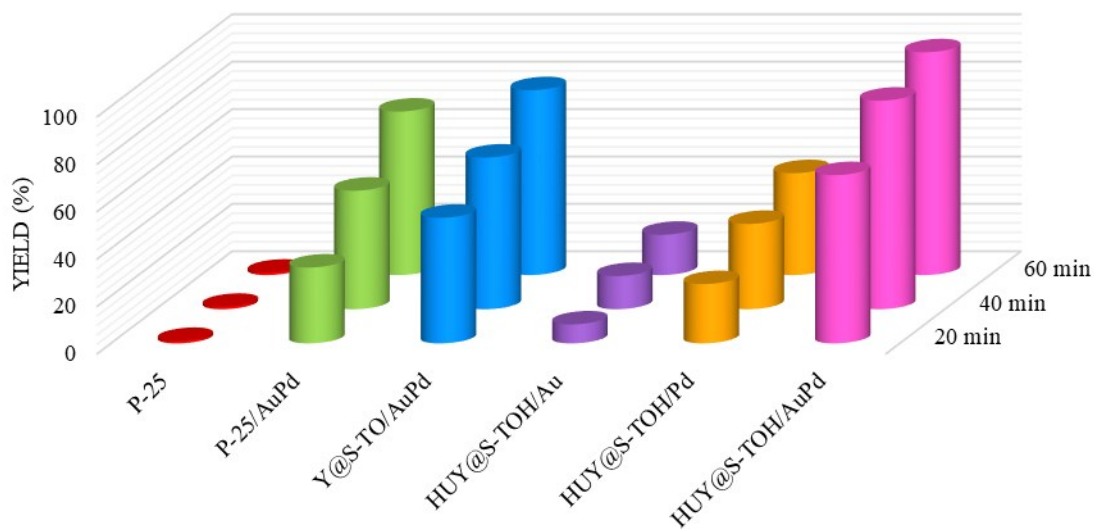


Fig. S9. Comparison the catalytic activity of P-25, P-25/AuPd, Y@S-TO/AuPd, HUY@S-TOH/Au, HUY@S-TOH/Pd and HUY@S-TOH/AuPd structures.

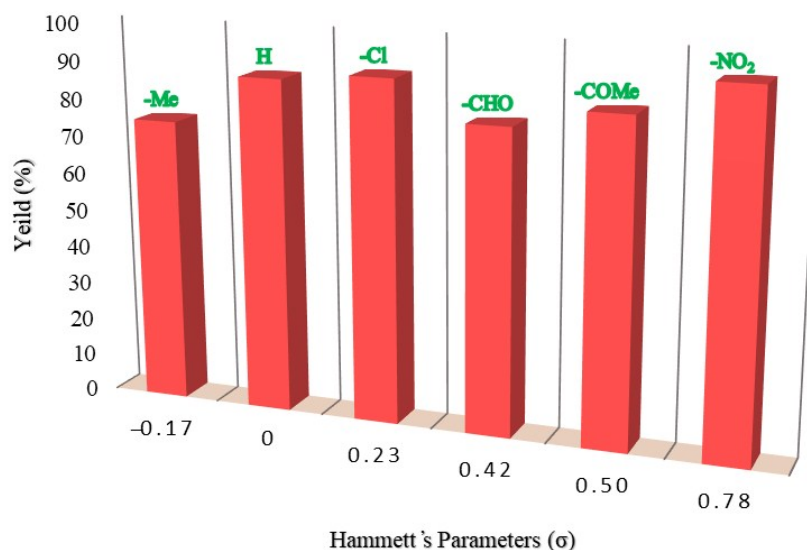


Fig. S10. Yield (%) of the Suzuki–Miyaura coupling products of *p*-substituted bromobenzenes vs. Hammett parameters (σ)

Table S1. Screening and control experiments for visible-light-promoted Suzuki coupling of 4-iodotoluene and phenylboronic acid.^a

Entry	Solvent	Base	<i>h</i> ν	Yield (%) ^b
1	EtOH:H ₂ O (2:1)	K ₂ CO ₃	–	Trace
2	EtOH:H ₂ O (2:1)	K ₂ CO ₃	–	68 ^c
3	Toluene	K ₂ CO ₃	+	Trace
4	DMF	K ₂ CO ₃	+	Trace
5	DMSO	K ₂ CO ₃	+	Trace
6	MeOH	K ₂ CO ₃	+	60
7	EtOH	K ₂ CO ₃	+	65
8	H ₂ O	K ₂ CO ₃	+	20
9	DMF:H ₂ O (1:1)	K ₂ CO ₃	+	12
10	DMSO:H ₂ O (1:1)	K ₂ CO ₃	+	10
11	MeOH:H ₂ O (1:1)	K ₂ CO ₃	+	70
12	EtOH:H ₂ O (1:1)	K ₂ CO ₃	+	78
13	EtOH:H ₂ O (2:1)	K ₂ CO ₃	+	94

14	EtOH:H ₂ O (1:2)	K ₂ CO ₃	+	54
15	EtOH:H ₂ O (2:1)	–	+	–
16	EtOH/H ₂ O (2:1)	NaOH	+	11
17	EtOH/H ₂ O (2:1)	K ₃ PO ₄	+	77
18	EtOH/H ₂ O (2:1)	Et ₃ N	+	Trace

^a Reaction conditions: 4-iodotoluene (1.0 mmol), phenylboronic acid (1.5 mmol), HUY@S-TOH/AuPd catalyst (5 mg), base (2 mmol) and solvent (6 mL), Xe lamp, room temperature, 1 h.

^b Isolated yield.

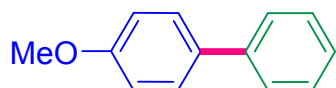
^c The reaction temperature was 80 °C, and other reaction conditions were as above.

Table S2. Comparison of the photocatalytic activity of HUY@S-TOH/AuPd architecture with other photocatalyzed Suzuki coupling reactions under visible light.

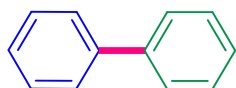
Entry	Photocatalyst	Catalyst amount	Light source	T (h)	Temp. (°C)	TOF (h ⁻¹)	Ref.
1	HUY@S-TOH/AuPd	5.0 mg of catalyst (0.6 wt% Pd)	Xe lamp (300 W)	0.5	r.t.	7095 (Iodoanisole)	This work
2	Pd-Azo-POP	200 μL of catalyst	Xe lamp (350 W)	2.5	25	6533.3 (Iodobenzene)	[5]
3	Pd–MoS ₂	75 μL of catalyst in distilled water 75μL	Xe-lamp, (4.3 W cm ⁻²)	2	25	5307 (Iodoanisole)	[6]
4	Pd@B-BO ₃	10 mg of catalyst (3 wt% Pd)	white LED lamp (1.2 W/cm ²)	2	r.t.	86.9 (Iodophenol)	[7]
5	Au–Pd/TiO ₂	0.002 mmol Pd	Blue LED lamp (5 W)	5	r.t.	98 (Iodobenzene)	[8]
6	2H-WS ₂ /Pd NPs	2.85 μg of Pd	White LED lamp (60 W)	4	r.t.	1244 (Iodotoluene)	[9]
7	Pd-NiFe ₂ O ₄ /rGO	0.5 mmol% Pd (0.005 mmol)	Xe lamp (300 W)	0.5	25	130.6 (Bromotoluene)	[10]
8	Pd@PDA-CL	0.6 mg of Pd	Two white LED lamps (12 W)	2	r.t.	85 (Iodotoluene)	[11]

9	Pd/SiC	10 mg of catalyst (3 wt% Pd)	Xe lamp (300 W)	1.3	30	1053 (Iodobenzene)	[12]
10	Au-Pd alloy NPs	50 mg, containing 3% of metals	White LED lamp (0.5 W cm ⁻²)	6	30	14.5 (Iodobenzene)	[13]
11	Pd/Au/PN-CeO ₂	15 mg of catalyst (2.95 wt% Au, 0.41 wt% Pd)	Xe lamp (150 W)	0.5	25	6.3 (Iodobenzene)	[14]
12	m-CNR-Pd	10 mg of catalyst (3 wt% Pd)	Xe lamp (150 W)	1	r.t.	51.6 (Iodobenzene)	[15]
13	Au-Pd NPs	0.49 μmol of catalyst	laser power (1.68 W)	1	25	162 (Iodobenzene)	[16]

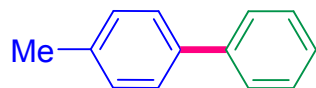
3. Characterization of cross-coupling products



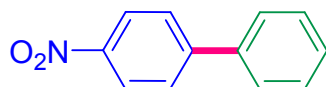
4-Methoxy-1,1'-biphenyl (3a) [17, 18]: ¹H NMR (400 MHz, CDCl₃): δ_H (ppm): 7.62-7.54 (m, 4H), 7.48-7.42 (t, 2H), 7.37-7.31 (t, 1H), 7.05-6.99 (d, 2H), 3.86 (s, 3H). ¹³C-NMR (100 MHz; CDCl₃): δ_C (ppm): 159.20, 140.89, 133.71, 128.67, 128.09, 126.76, 126.66, 114.21, 55.18.



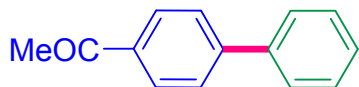
1,1'-Biphenyl (3b) [19]: ¹H NMR (400 MHz, CDCl₃): δ_H(ppm): 7.67-7.59 (m, 4H), 7.52-7.44 (m, 4H), 7.42-7.35 (t, 2H). ¹³C-NMR (100 MHz; CDCl₃): δ_C(ppm): 141.47, 128.74, 127.29, 127.20.



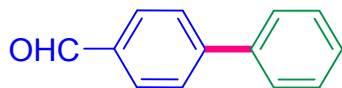
4-Methyl-1,1'-biphenyl (3c) [18, 20]: ^1H NMR (400 MHz, CDCl_3): δ_{H} (ppm): 7.63-7.59 (d, 2H), 7.55-7.50 (d, 2H), 7.48-7.42 (t, 2H), 7.38-7.33 (t, 1H), 7.30-7.25 (d, 2H), 2.50 (s, 3H). ^{13}C -NMR (100 MHz; CDCl_3): δ_{C} (ppm): 141.17, 138.35, 137.04, 129.48, 128.72, 127.05, 20.85.



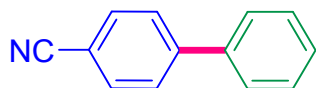
4-Nitro-1,1'-biphenyl (3d) [17, 21]: ^1H NMR (400 MHz, CDCl_3): δ_{H} (ppm): 8.29-8.36 (d, 2H), 7.80-7.74 (d, 2H), 7.68-7.62 (d, 2H), 7.56-7.44 (m, 3H). ^{13}C -NMR (100 MHz; CDCl_3): δ_{C} (ppm): 147.63, 147.10, 138.80, 129.19, 128.97, 127.83, 127.40, 124.15.



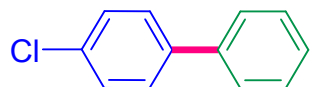
1-Biphenyl-4-yl-ethanone: (3e) [22]: ^1H NMR (400 MHz, CDCl_3): δ_{H} (ppm): $\delta = 8.05$ -8.10 (2H, d), 7.70-7.75 (2H, d), 7.63-7.68 (2H, d), 7.40-7.46 (3H, m), 2.65 (3H, s). ^{13}C -NMR (100 MHz; CDCl_3): δ_{C} (ppm): ^{13}C NMR (75 MHz, CDCl_3): $\delta = 198.00$, 145.90, 139.87, 135.90, 129.00, 128.96, 128.28, 127.31, 127.27, 26.73.



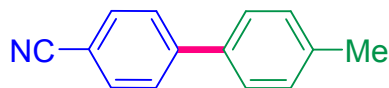
Biphenyl-4-carbaldehyde (3f) [18, 23] : ^1H NMR (400 MHz, CDCl_3): δ_{H} (ppm): 10.09 (s, 1H), 8.01-7.96 (d, 2H), 7.81-7.76 (d, 2H), 7.69-7.64 (m, 2H), 7.55-7.42 (m, 3H). ^{13}C -NMR (100 MHz; CDCl_3): δ_{C} (ppm): 192.03, 147.24, 139.75, 135.20, 130.32, 129.05, 128.51, 127.73, 127.41.



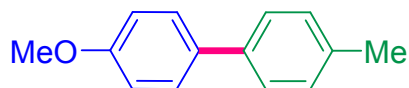
[1,1'-biphenyl]-4-carbonitrile: (3g) [18, 20]: ^1H NMR (400 MHz, CDCl_3): δ_{H} (ppm): 7.78-7.69 (m, 4H), 7.64-7.60 (m, $2\text{H}_{3,7}$), 7.54-7.48 (m, 2H), 7.48-7.42 (m, 1H). ^{13}C -NMR (100 MHz; CDCl_3): δ_{C} (ppm): 145.70, 139.21, 132.64, 129.15, 128.69, 127.77, 127.27, 119.00, 110.92.



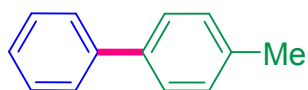
4-Chloro-1,1'-biphenyl (3j) [18]: ^1H NMR (400 MHz, CDCl_3): δ_{H} (ppm): 7.52-7.7.60 (m, 4H), 7.50-7.36 (m, 5H). ^{13}C -NMR (100 MHz; CDCl_3): δ_{C} (ppm): 142.99, 135.72, 133.54, 129.61, 128.88, 128.81, 128.21, 126.84.



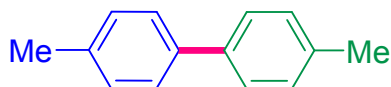
4'-Methyl-4-biphenylcarbonitrile (3l) [24]: ^1H NMR (400 MHz, CDCl_3): δ_{H} (ppm): 7.76-7.66 (dd, 4H), 7.54-7.49 (d, 2H), 7.34-7.29 (d, 2H), 2.44 (s, 3H). ^{13}C -NMR (100 MHz; CDCl_3): δ_{C} (ppm): 145.6, 138.78, 136.27, 132.60, 129.86, 127.47, 127.09, 119.10, 110.51, 21.20.



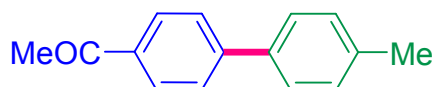
4-Methoxy-4'-methyl-1,1'-biphenyl (3m) [25]: ^1H NMR (400 MHz, CDCl_3): δ_{H} (ppm): 7.57-7.51 (d, 2H), 7.50-7.45 (d, 2H), 7.28-7.23 (d, 2H), 7.02-6.97 (d, 2H), 3.87 (s, 3H), 2.41 (s, 3H).



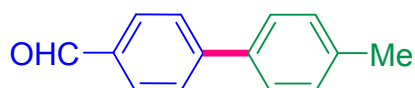
4-Methyl-1,1'-biphenyl (3n) [19]: ^1H NMR (400 MHz, CDCl_3): δ_{H} (ppm): 7.64-7.59 (d, 2H), 7.55-7.51 (d, 2H), 7.49-7.43 (t, 2H), 7.39-7.34 (t, 1H), 7.31-7.27 (d, 2H), 2.42 (s, 3H).



4,4'-Dimethyl-1,1'-biphenyl (3o) [26]: ^1H NMR (400 MHz, CDCl_3): δ_{H} (ppm): 2.41 (s, 6H), 7.28-7.23 (d, 4H), 7.53-7.48 (d, 4H). ^{13}C -NMR (100 MHz; CDCl_3): δ_{C} (ppm): 21.13, 126.84, 129.45, 136.73, 138.28.



1-(4'-methylbiphenyl-4-yl) ethanone (3q) [26]: ^1H NMR (400 MHz, CDCl_3): δ_{H} (ppm): 8.10-8.02 (d, 2H), 7.80-7.67 (d, 2H), 7.64-7.52 (d, 2H), 7.41-7.26 (d, 2H), 2.67 (s, 3H), 2.44 (s, 3H). ^{13}C -NMR (100 MHz; CDCl_3): δ_{C} (ppm): 197.94, 145.68, 138.23, 136.94, 135.65, 129.74, 128.93, 127.14, 127.00, 26.68, 21.19.



4'-Methyl-[1,1'-biphenyl]-4-carbaldehyde (3r) [26]: ^1H NMR (400 MHz, CDCl_3): δ_{H} (ppm): 10.07 (s, 1H), 7.99-7.94 (d, 2H), 7.80-7.74 (d, 2H), 7.60-7.54 (d, 2H), 7.34-7.29 (d, 2H), 2.44 (s, 3H).

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