Supporting information

Selective Synthesis of Cu-Cu_2O/C and CuO-Cu_2O/C Catalysts for Pd Free C-C, C-N Coupling and Oxidation Reactions

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Table S5. Comparative catalytic activity of CuO-Cu_2O/C with various catalysts reported in the literature for the diphenylmethane oxidation.

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Materials synthesis

Synthesis of CuO

CuO was prepared by following the reported procedure.[1] 1.33 mmol citric acid was added to the 50 mL of aqueous solution of $CuSO_4.5H_2O$ (4 mmol). Then 7 mL of aqueous NH_3 (25%) was added drop-wise under continuous stirring. After that, 15 mL aqueous NaOH (1 M) was added to the reaction mixture. Then it was transferred into a Teflon-lined autoclave and hydrothermally treated for 4 h at 130 °C. After the reaction, the resulting black precipitate was filtered and washed with distilled water. The product was dried at 80 °C for 24 h to obtain the black CuO. The product was confirmed from XRD (Figure S1).

Synthesis of Cu_2O

Cu_2O was prepared by following the reported procedure.[2] In a typical synthesis, 5.0 mmol of $CuCl_2.2H_2O$ was dissolved in 100 mL deionized water to obtain a blue colour solution. Then 3 mL of aqueous NH_3 (25 %) was added under constant stirring. After that, 10 mL aqueous NaOH (1 M) was added drop-wise to the above solution. Then 1 mL of $N_2H_4.H_2O$ was added drop-wise into the precipitate suspension, with constant stirring for another 10 min. After addition of $N_2H_4.H_2O$, the blue colour precipitation was turned to red. Then the red precipitate was filtered, washed with deionized water and ethanol several times, and dried at 80 °C for 4 h to obtain Cu_2O. The product was confirmed from XRD (Figure S1).

Synthesis of Cu-Cu_2O

Cu-Cu_2O was prepared by following the reported procedure.[3] 4.0 mmol of $Cu(NO_3)_2{\cdot}3H_2O$ was dissolved in 4 mL of deionized water to obtain a blue colour solution. Then 4.0 mmol of hydrazine $N_2H_4{\cdot}H_2O$ and 55 mmol of tetraethylene glycol were added. After the addition, the blue colour solution was turned to brown and it was transferred into a Teflon-lined autoclave and hydrothermally treated for 2 h at 120 °C. After this, the reaction mixture was centrifuged and the supernatant liquid was decanted and the precipitates were washed with ethanol several times and dried at 80 °C to obtain Cu-Cu_2O. The product was confirmed from XRD (Figure S1).

Synthesis of Cu NPs

Cu NPs was prepared by following the reported procedure.[4] 2.5 mmol of $Cu(NO_3)_2.3H_2O$ was added to 150 mL ethanol and stirred at 2–3 °C for 10 minutes to get a solution A. 3.5 mmol of sodium dodecyl sulphate and 15.0 mmol of sodium borohydride were dissolved in 100 mL of ethanol and stirred at 2–3 °C for 10 minutes to get a solution B. Then, solution A was slowly added to solution B and the resultant solution was stirred at 2–3 °C for 30 minutes. Further, the solution was kept under ambient conditions for 45 minutes. The resultant black solution confirmed the formation of Cu NPs. Then it was filtered and washed with ethanol for three times to get Cu NPs. The product was confirmed from XRD (Figure S1).

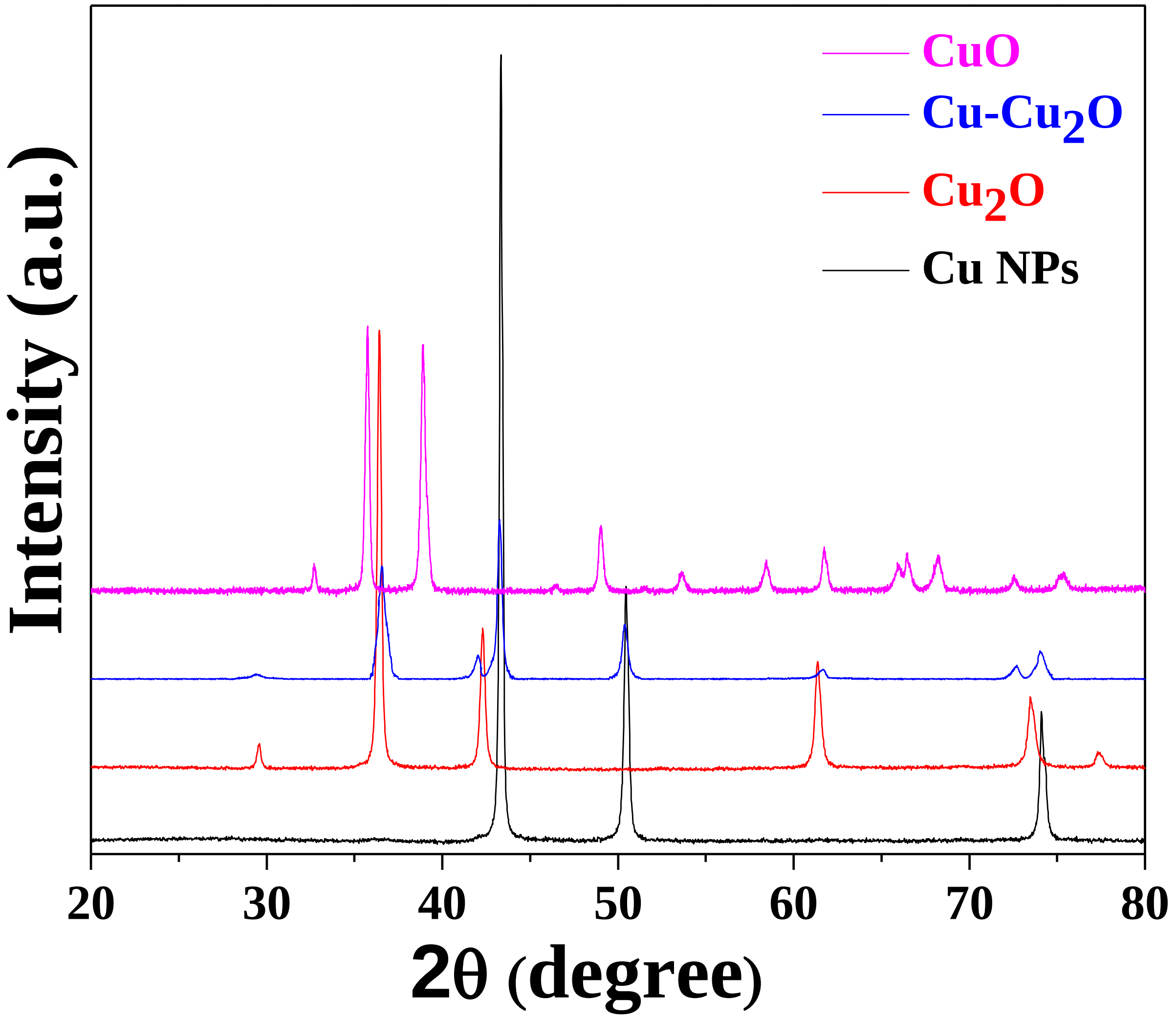


Fig. S1. XRD patterns of Cu NPs, CuO, Cu_2O, and $Cu\text{-}Cu_2O$.

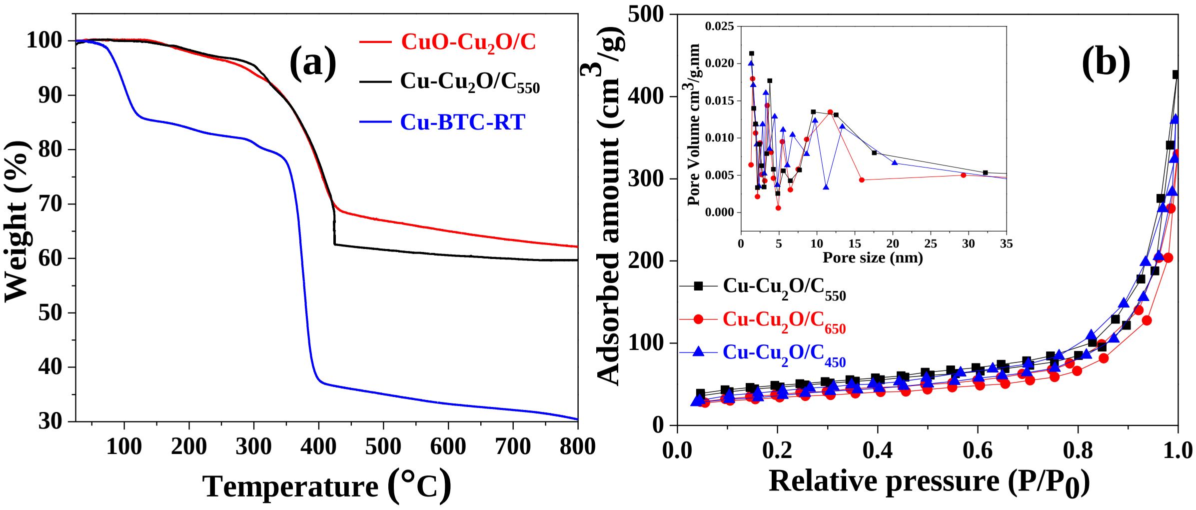


Fig. S2. (a) TGA profiles of CuO-Cu_2O/C, Cu-Cu_2O/C_{550}, Cu-BTC-RT and (b) N_2-adsorption isotherms of Cu-Cu_2O/C_{450}, Cu-Cu_2O/C_{550}, and Cu-Cu_2O/C_{650}.

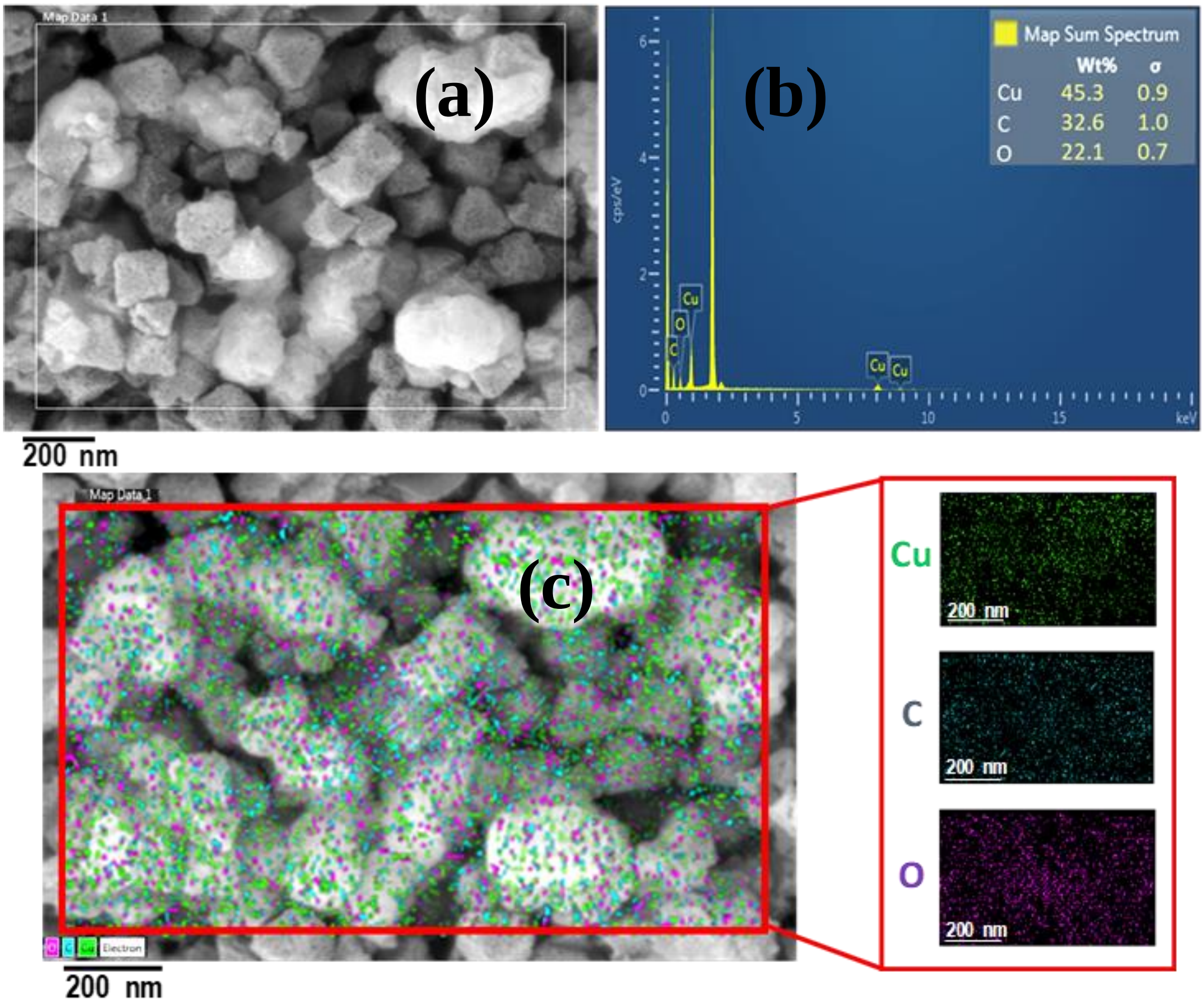


Fig. S3. (a) FE-SEM image, (b) EDS spectrum, and (c) elemental mapping from the selected FE-SEM region of CuO-Cu_2O/C.

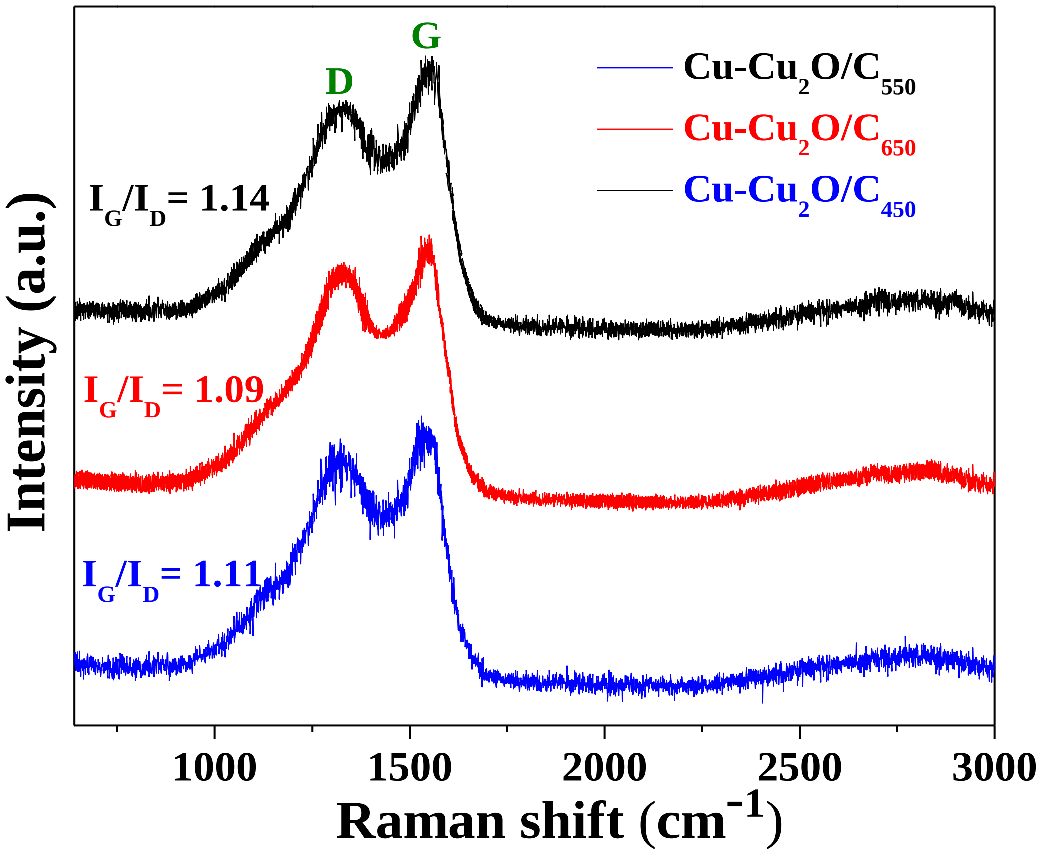


Fig. S4. Raman spectra of Cu-Cu_2O/C_{450}, Cu-Cu_2O/C_{550}, and Cu-Cu_2O/C_{650}.

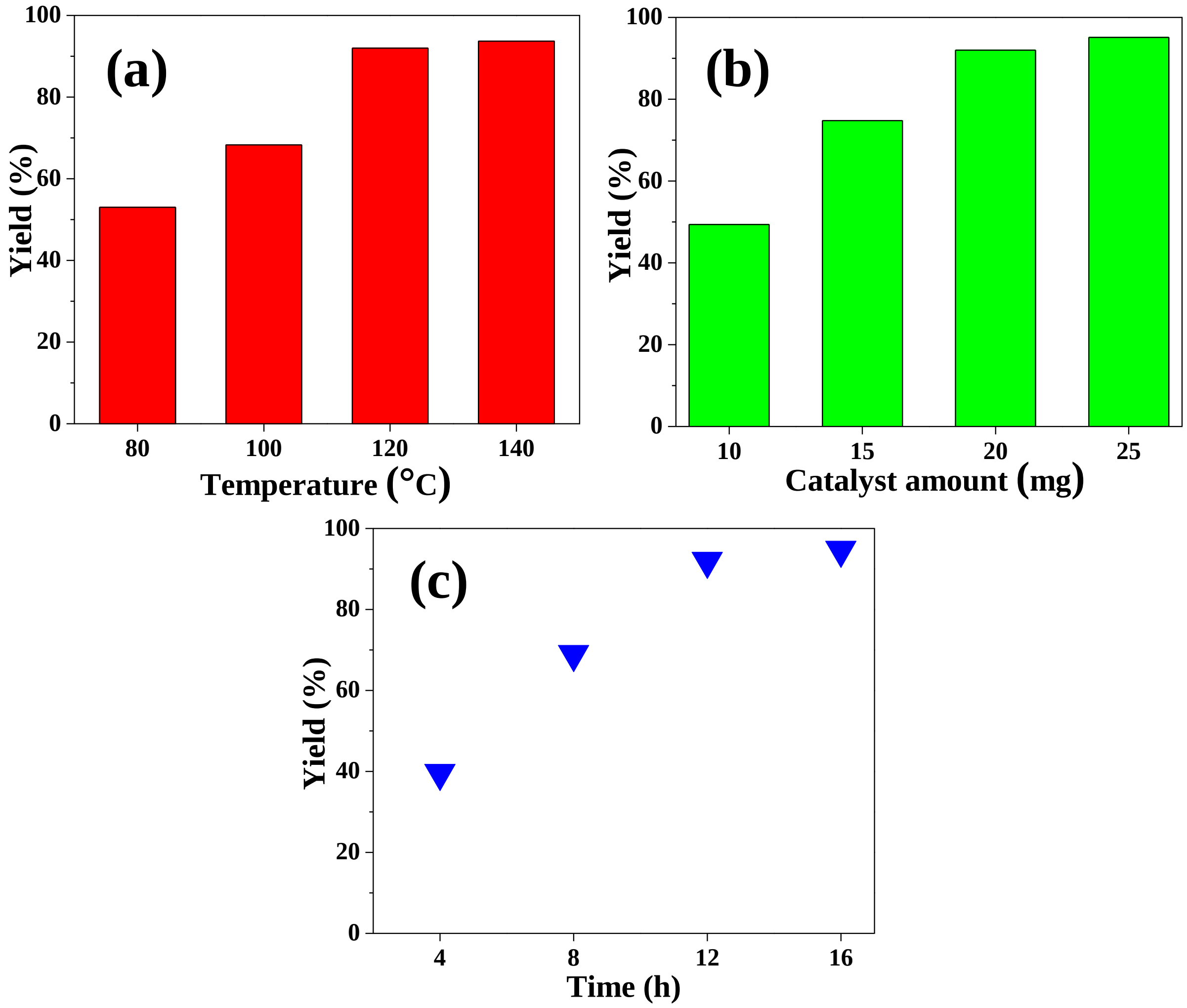


Fig. S5. Influence of (a) temperature, (b) catalyst amount, and (c) reaction time for the Sonogashira cross-coupling over Cu-Cu_2O/C_{550}.

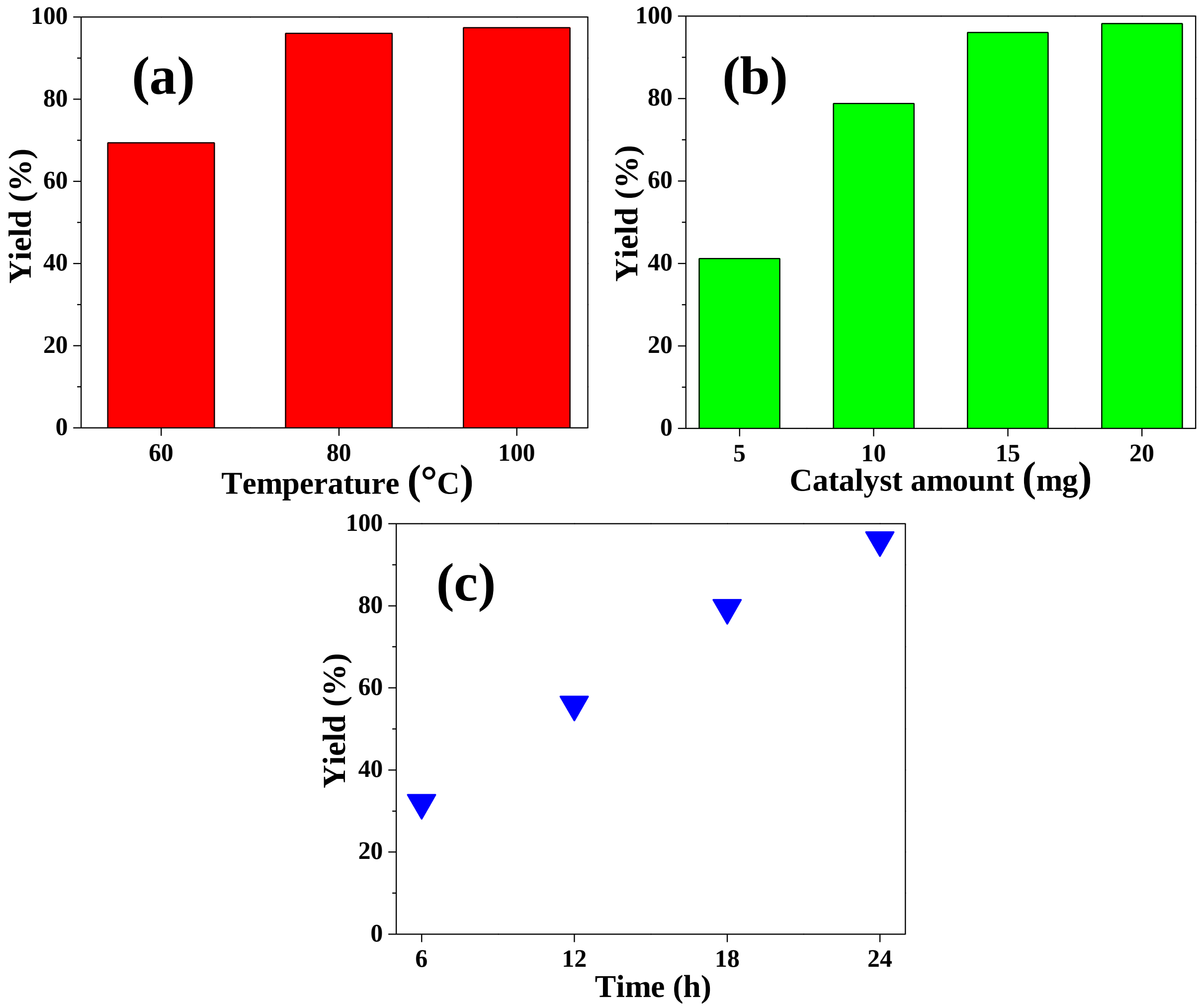


Fig. S6. Influence of (a) temperature, (b) catalyst amount, and (c) reaction time for the Ullmann amination over Cu-Cu_2O/C_{550}.

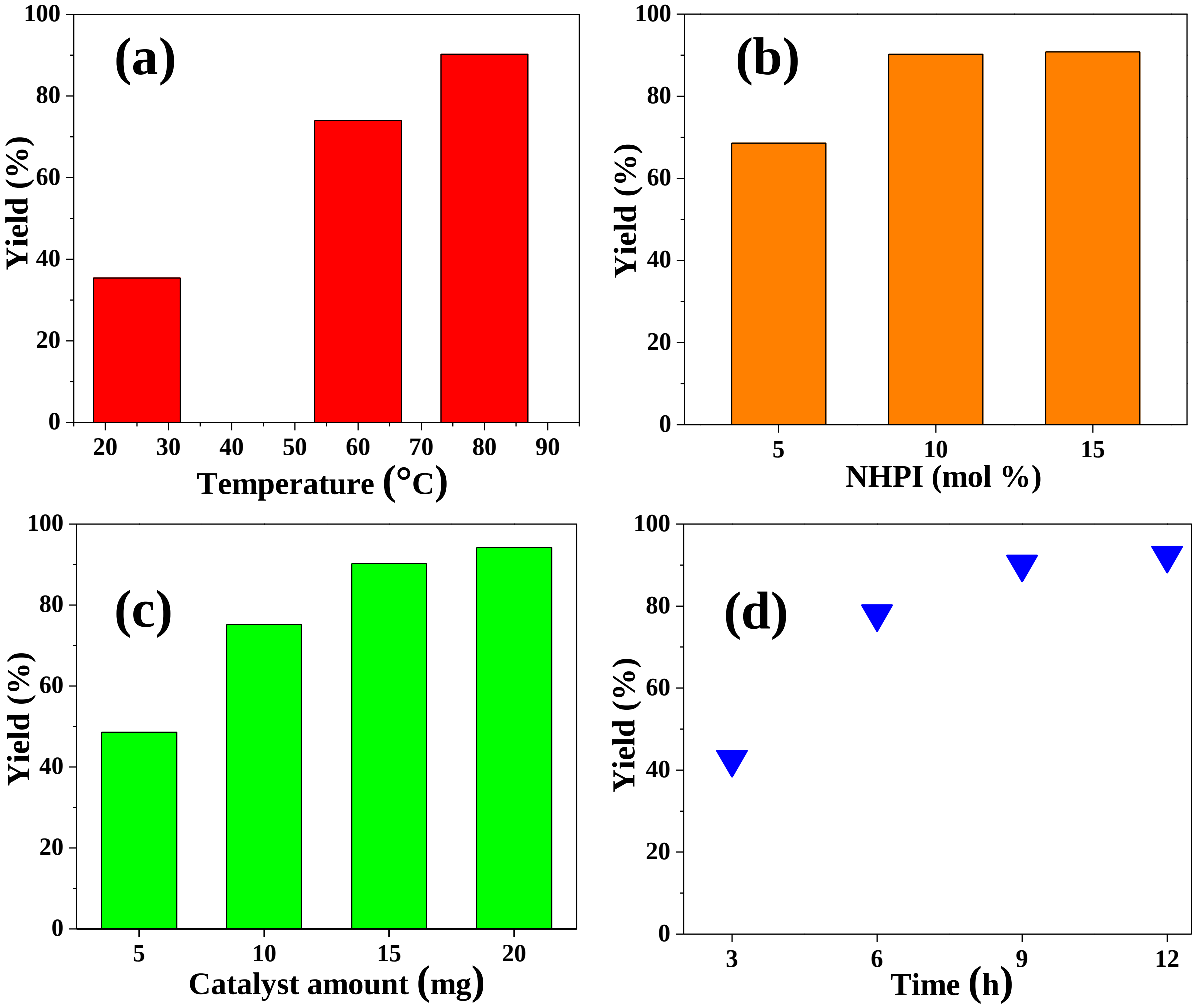
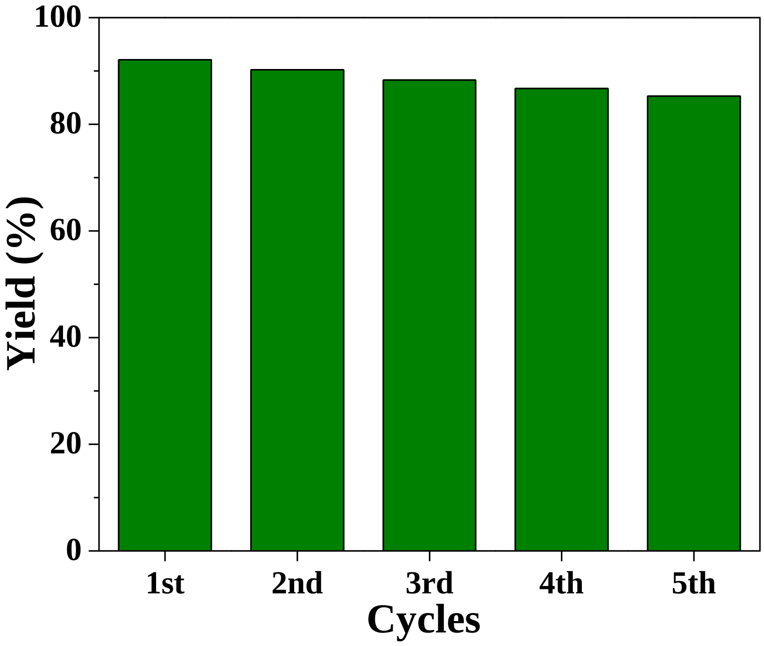


Fig. S7. Influence of (a) temperature, (b) NHPI amount, (c) catalyst amount, and (d) reaction time for the diphenylmethane oxidation over CuO-Cu_2O/C.



Cycles	Yield (%)	Cu leaching (%)
1st cycle	92	0.9
2nd cycle	90.1	1.1
3rd cycle	88.4	1.5
4th cycle	86.7	1.8
5th cycle	85.3	2.1

Fig. S8. Recycling of Cu-Cu_2O/C_{550} in the Sonogashira cross coupling reaction.

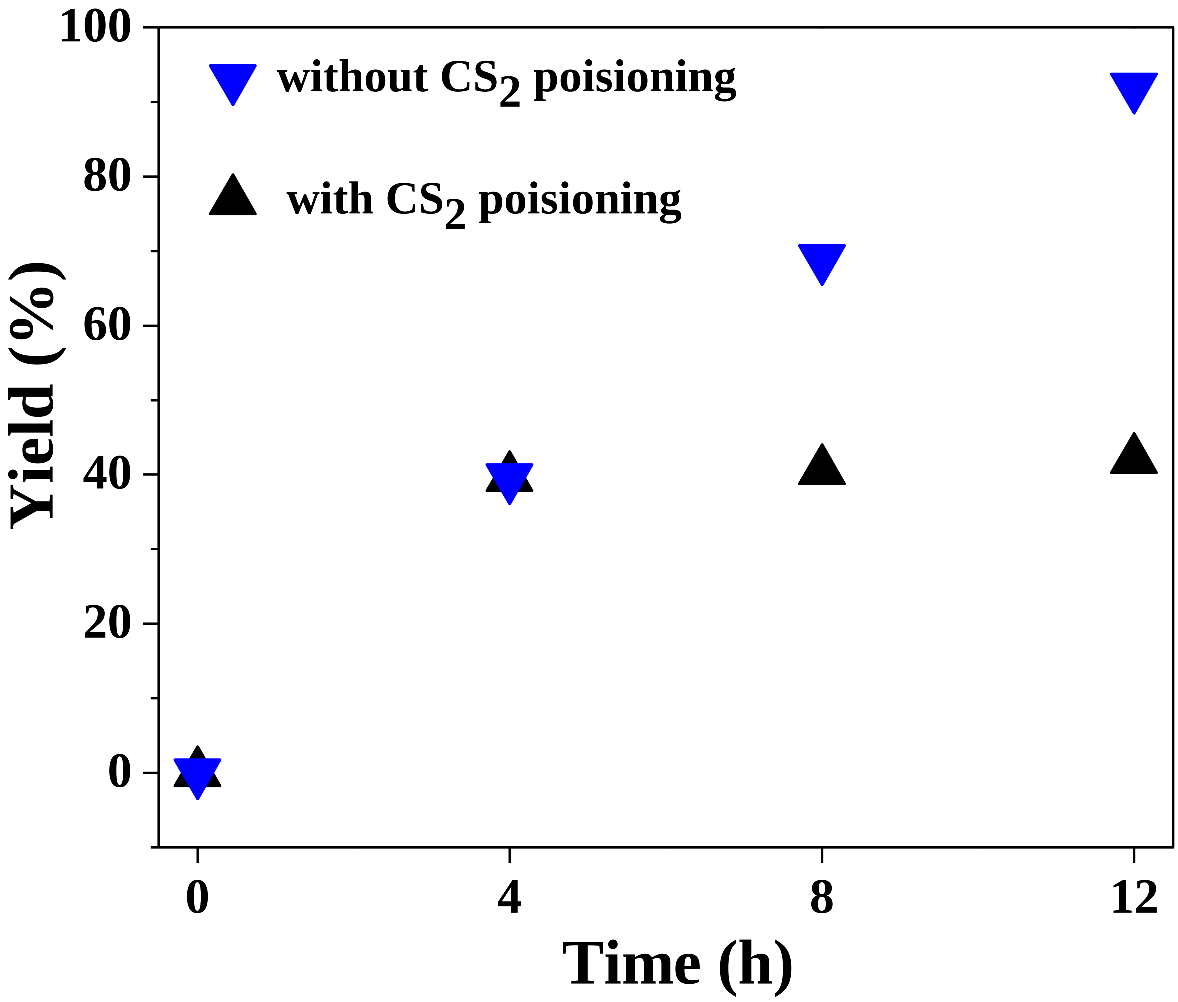
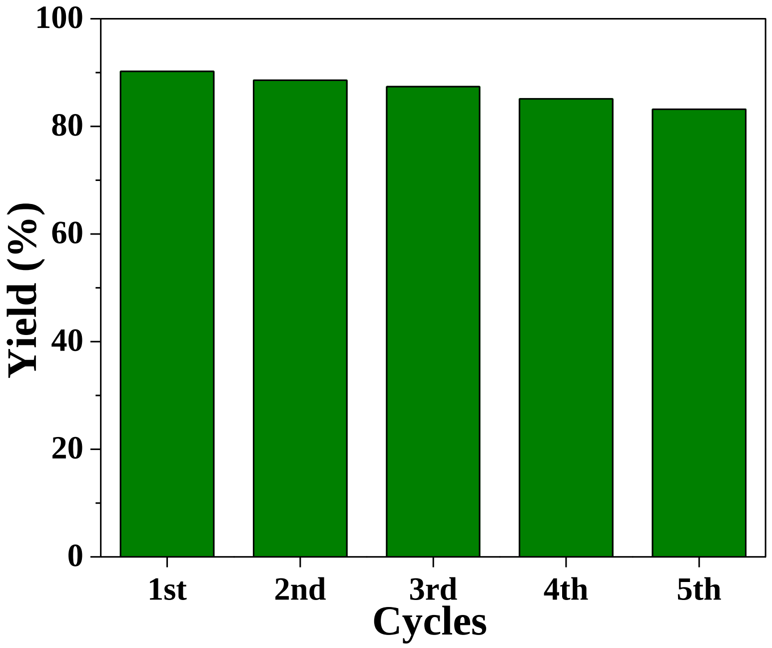


Fig. S9. Influence of CS_2 addition on the poisoning of Cu-Cu_2O/C_{550} in the Sonogashira cross coupling reaction.



Cycles	Yield (%)	Cu leaching (%)
1st cycle	90.2	0.7
2nd cycle	88.6	0.9
3rd cycle	87.4	1.4
4th cycle	85	1.5
5th cycle	83.2	1.7

Fig. S10. Recycling of CuO-Cu_2O/C in the diphenylmethane oxidation.

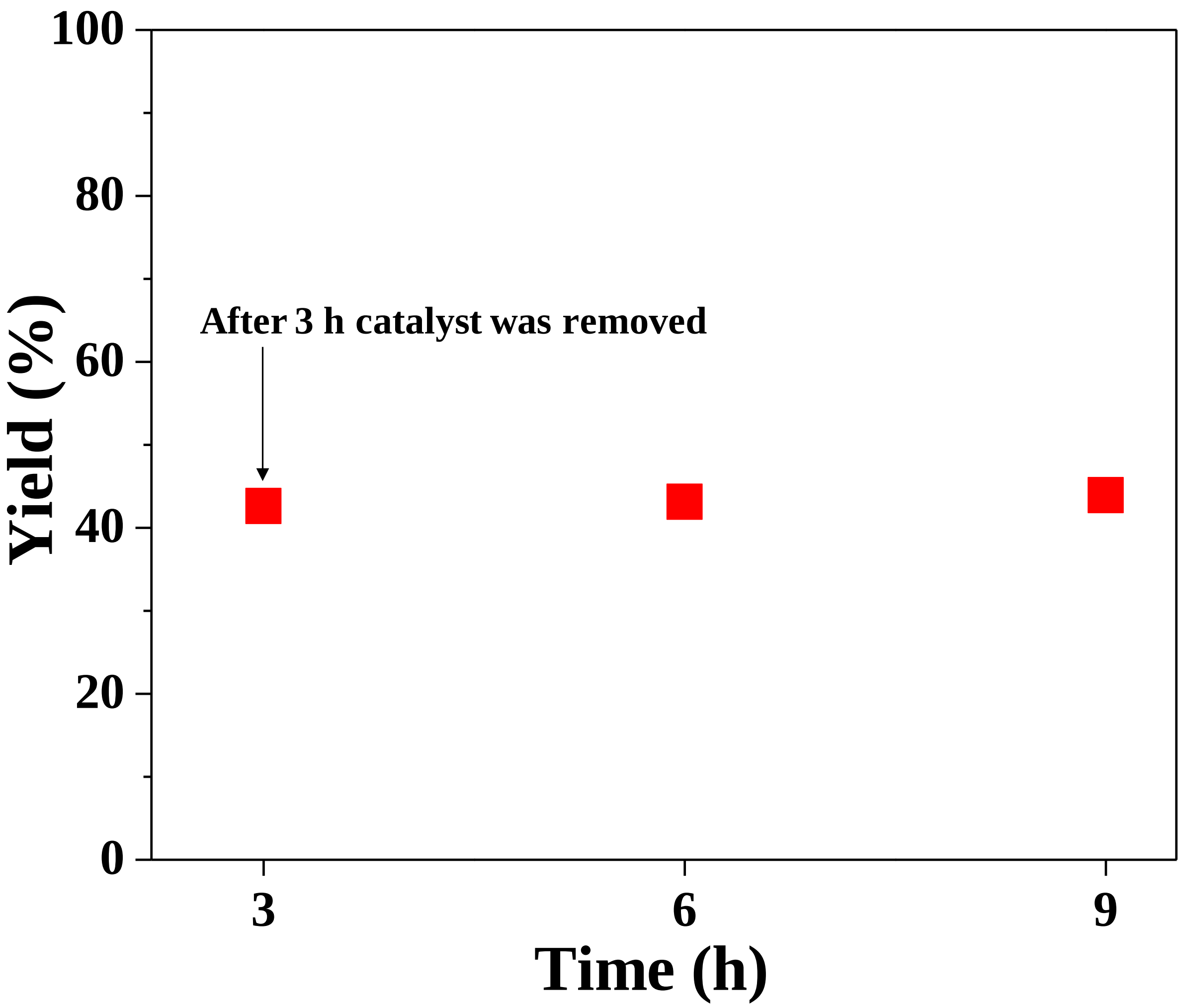
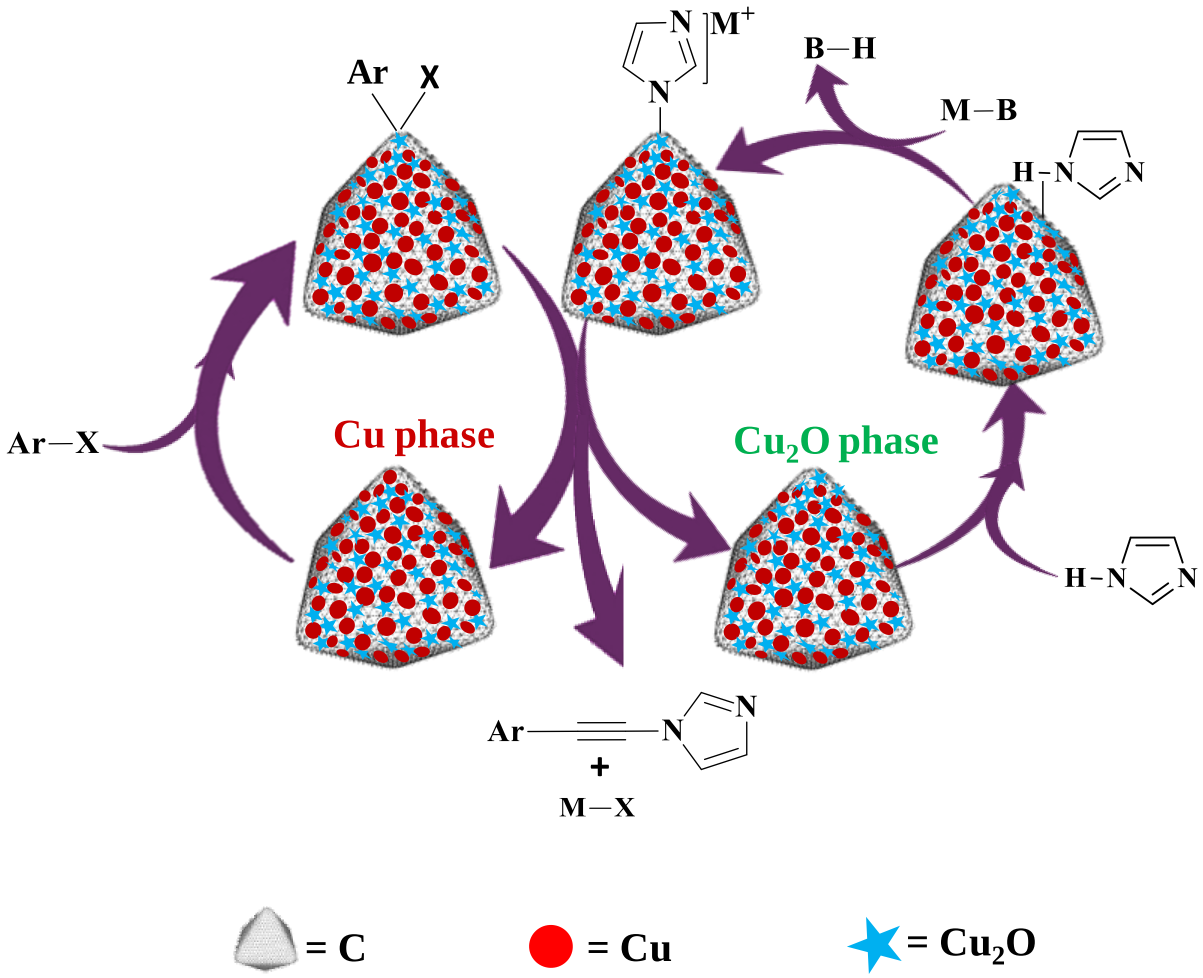
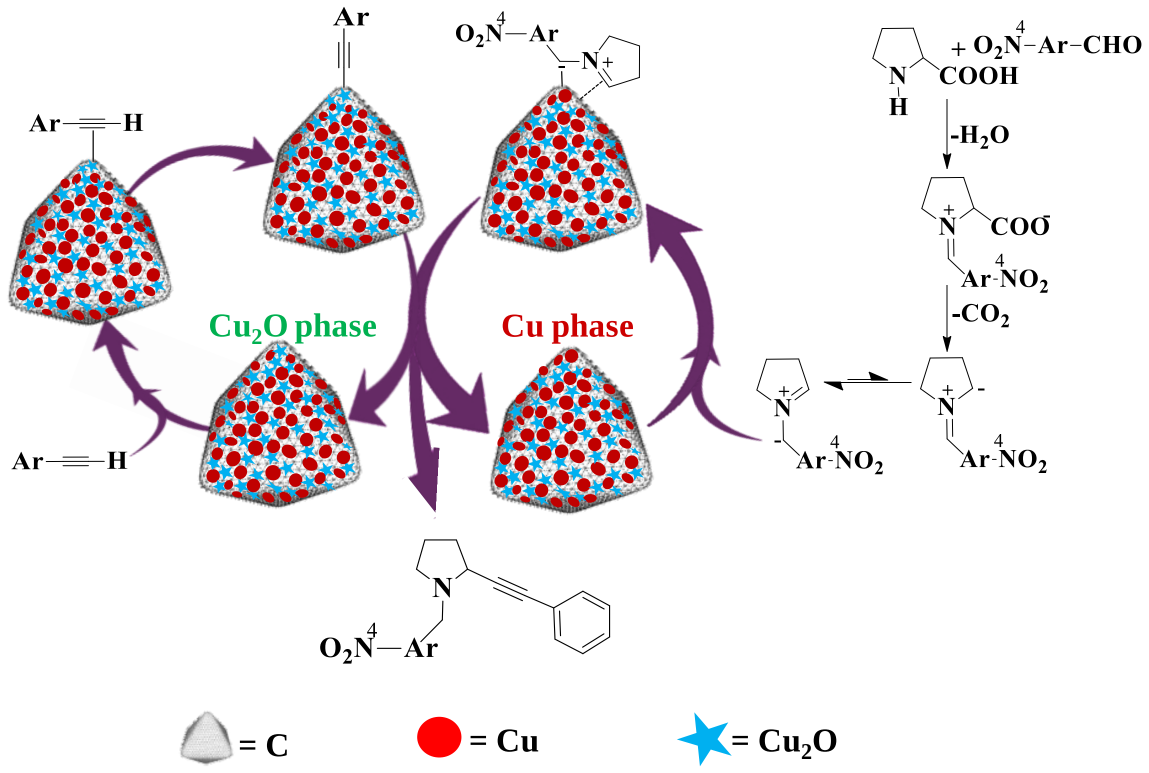


Fig. S11. Progress of reaction after the removal of the catalyst after 3 h during the hot filtration test in the diphenylmethane oxidation over CuO-Cu_2O/C.

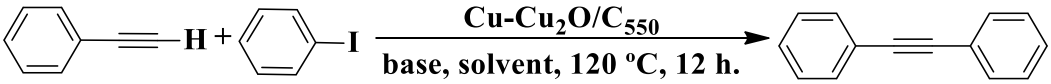


Scheme S1. Plausible mechanism for the Ullmann amination over Cu-Cu$_2$O/C$_{550}$.



Scheme S2. Plausible mechanism for the A^3 coupling reaction over Cu-Cu_2O/C_{550}.

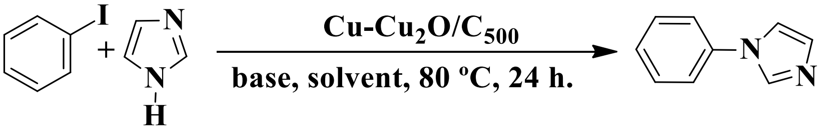
Table S1. Influence of solvent, base, and aryl halide in the Sonogashira cross coupling using Cu-Cu_2O/C_{550}.



Entry	Ar-X	Solvent	Base	Yield (%)
1	Ar-I	CH_3OH	Cs_2CO_3	45.3
2	Ar-I	CH_3CN	Cs_2CO_3	58.1
3	Ar-I	Dioxane	Cs_2CO_3	65.4
4	Ar-I	DMF	Cs_2CO_3	92.1
5	Ar-I	Toluene	Cs_2CO_3	Trace
6	Ar-I	DMF	Na_2CO_3	62.2
7	Ar-I	DMF	K_2CO_3	67.4
8	Ar-I	DMF	NaOH	34.2
9	Ar-I	DMF	KOH	41.3
10	Ar-I	DMF	K_3PO_4	Trace
11	Ar-Br	DMF	Cs_2CO_3	45.4
12	Ar-Cl	DMF	Cs_2CO_3	19.7

Reaction condition: Phenylacetylene (2 mmol), iodobenzene (2 mmol), catalyst (20 mg), Cs_2CO_3 (4 mmol), solvent (1 mL), temperature (120 °C), and time (12 h). Product yield was determined from the GC (average of three measurements) based on the phenylacetylene conversion because no side product was obtained.

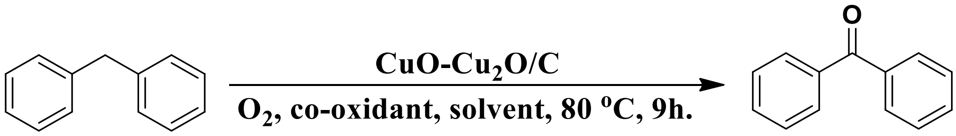
Table S2. Influence of solvent, base, and aryl halide in the Ullmann amination (C-N coupling reaction) using Cu-Cu_2O/C_{550}.



Entry	Ar-X	Solvent	Base	Yield (%)
1	Ar-I	CH_3OH	KOH	42.3
2	Ar-I	CH_3CN	KOH	Trace
3	Ar-I	Dioxane	KOH	Trace
4	**Ar-I**	**DMSO**	**KOH**	**96.2**
5	Ar-I	DMF	KOH	88.5
7	Ar-I	Toluene	KOH	58.3
7	Ar-I	DMSO	Na_2CO_3	45.4
8	Ar-I	DMSO	Cs_2CO_3	90.1
9	Ar-I	DMSO	K_3PO_4	78.4
10	Ar-I	DMSO	NaOH	92.3
11	Ar-Br	DMSO	KOH	50.4
12	Ar-Cl	DMSO	KOH	20.9

Reaction condition: Halobenzene (1 mmol), imidazole (1.2 mmol), base (2 mmol), catalyst (15 mg), solvent (2 mL), temperature (80 ºC), time (24 h). Product yield was determined from the GC (average of three measurements) based on the iodobenzene conversion because no side product was obtained.

Table S3. Influence of solvent and co-oxidant on the diphenylmethane oxidation using Cu-Cu_2O/C_{550}.



Entry	Co-oxidant	Solvent	DPM conversion (%)	Benzophenone selectivity (%)
1	None	CH_3CN	22	>99
2	**NHPI**	**CH_3CN**	**90.2**	**>99**
3	H_2O_2	CH_3CN	41.8	90.7
4	TBHP	CH_3CN	62.1	88.5
5	NHPI	Ethylacetate	40.5	>99
6	NHPI	Acetone	71.4	>99

Reaction condition: Diphenylmethane (1.0 mmol), co-oxidant (0.1 mmol), CuO-Cu_2O/C (15 mg), oxidant (O_2 (1 atm, balloon), solvent (5.0 mL), temperature (80 °C), and time (9 h),

Table S4. Comparative catalytic activity of Cu-Cu_2O/C_{550} with various catalysts reported in the literature for the Sonogashira cross coupling reaction.

S.N	**Catalyst**	**Reaction condition**	**Y_{DPA} (%)**	**References**
1	Nano Fe_3O_4	Phenylacetylene (2 mmol), iodobenzene (1 mmol), catalyst (5 mol %), K_2CO_3 (2 mmol), DMF (3 mL), temperature (125 °C), and time (35 h).	68	5
2	Silica supported $Pd(OAc)_2$ complex	Phenylacetylene (1 mmol), iodobenzene (2 mmol), catalyst (1 mol %), TEA (3 mmol), undecane (1 mmol), temperature (70 °C), and time (12 h).	87	6
3	Pd^0/MgLa mixed oxide	Phenylacetylene (1.2 mmol), iodobenzene (1 mmol), catalyst (20 mg), TEA (1.2 mmol), DMF (3 mL), temperature (145 °C), and time (10 h).	85	7
4	$Cu(OAc)_2$	Phenylacetylene (0.8 mmol), iodobenzene (0.5 mmol), catalyst (50 mol %), TEA (3 mL), temperature (150 °C), and time (24 h)	65	8
5	N,N'-dibenzyl BINAM–CuI complex	Phenylacetylene (1.5 mmol), iodobenzene (1 mmol), catalyst (20 mol %), K_2CO_3 (3 mmol), DMF (5 mL), temperature (80 °C), and time (6 h).	90	9
6	Cu(I)-MOF	Phenylacetylene (0.5 mmol), iodobenzene (0.5 mmol), CuI (0.05 mmol), ligand (0.1 mmol), K_2CO_3 (1 mmol), DMF (3 mL), temperature (140 °C), and time (12 h).	81	10
7	$FeCl_3$-N,N'-dimethylethylenediamine	Phenylacetylene (1 mmol), iodobenzene (1.5 mmol), $FeCl_3$ (0.1 equiv), N,N'-dimethylethylenediamine (0.2 equiv), Cs_2CO_3 (3 mmol), Toluene (1 mL $mmol^{-}1$), temperature (135 °C), and time (120 h).	95	11
8	CuBr/Phen, n-Bu_4NBr	Phenylacetylene (2 mmol), iodobenzene (1 mmol), catalyst (0.1 mmol), 1,10-phenanthroline (0.2 mmol), n-Bu_4NBr (0.2 mmol), NaOH (2	80	12

		mmol), H_2O (1.5 mL), temperature (120 °C), and time (24 h).		
9	Pd/Fe_3O_4 NPs	Phenylacetylene (1 mmol), iodobenzene (1 mmol), catalyst (0.2 mol %), piperidine (1.5 mmol), DMF (4 mL), temperature (110 °C), and time (24 h).	88	13
10	TbPo-Pd(II)	Phenylacetylene (1 mmol), iodobenzene (1 mmol), catalyst (0.35 mmol % Pd)), NaOH (2 mmol), MeOH:H_2O (3:2) (5 mL), temperature (80 °C), and time (12 h).	93	14
11	Au−Pd_2Sn NRs	Phenylacetylene (1.5 mmol), iodobenzene (1 mmol), catalyst (10 mg), KOH (2 mmol), DMF (10 mL), temperature (130 °C), and time (1 h).	65	15
12	CuI/DABCO	Phenylacetylene (0.6 mmol), iodobenzene (0.5 mmol), CuI (10 mol %), DABCO (20 mol %), Cs_2CO_3 (1 mmol), DMF (3 mL), temperature (140 °C), and time (10 h).	94	16
13	Pd-$CuFe_2O_4$@SiO_2	Phenylacetylene (1.5 mmol), iodobenzene (1 mmol), catalyst (10 mg containing 0.3 mol % Pd), DABCO (2 mmol), DMA (2 mL), temperature (50 °C), and time (24 h).	80	17
14	Cu-Cu_2O@rGO	Phenylacetylene (2 mmol), iodobenzene (2 mmol), catalyst (1 wt %), Cs_2CO_3 (4 mmol), DMF (1 mL), temperature (80 °C), and time (8 h).	91	18
15	**Cu-Cu_2O/C_{550}**	Phenylacetylene (2 mmol), iodobenzene (2 mmol), catalyst (20 mg), Cs_2CO_3 (4 mmol), DMF (1 mL), temperature (120 °C), and time (12 h).	**92**	This work

Table S5 . Comparative catalytic activity data of Cu-Cu_2O/C_{550} reported in literature for the Ullmann reaction for imidazole arylation over various reported catalyst.

S.N	Catalyst	Reaction condition	$Y_{1\text{-phenyl-imidazole}}$ (%)	References
1	Cu_2O/ninhydrin	Iodobenzene (1 mmol), imidazole (1.5 mmol), KOH (2 mmol), [Cu] (0.1 mmol), Ninhydrin (0.2 mmol), DMSO (2 mL), temperature (90 ºC), time (24 h).	92	19
2	SWCNT-Met/Pd	Iodobenzene (2 mmol), imidazole (2 mmol), K_2CO_3 (4 mmol), catalyst (20 mg), DMSO (2 mL), temperature (110 ºC), time (10 h).	96	20
3	CELL-Cu(0)	Iodobenzene (1.1 mmol), imidazole (1.5 mmol), NEt_3 (2 mmol), catalyst (20 mg), DMSO (2 mL), temperature (130 ºC), time (12 h).	95	21
4	Cu_2O, 4,7-dimethoxy-1,10-phenanthroline	Iodobenzene (1 mmol), imidazole (1.2 mmol), Cs_2CO_3 (1.4 mmol), (PEG 200 mg), butyronitrile (1 mL), Cu_2O (0.025), 4,7-dimethoxy-1,10-phenanthroline (0.075 mmol), (20 mg), DMSO (2 mL), temperature (110 ºC), time (24 h).	95	22
5	CuI	Iodobenzene (1 mmol), imidazole (1.4 mmol), K_3PO_4 (2 mmol), catalyst (20 mol %), DMSO (2 mL), temperature (40 ºC), time (40 h).	84	23
6	CuI/*N*-hydroxysuccinimide	Iodobenzene (1.5 mmol), imidazole (1.5 mmol), K_3PO_4 (2 mmol), CuI (5 mol %)/ligand (10 mol %), DMSO (5 mL), temperature (90 ºC), time (12 h).	98	24
7	CuCl	Iodobenzene (5 mmol), imidazole (6.5 mmol), K_2CO_3 (5.5 mmol), CuI (2.5 mol %)/ligand (10 mol %), NMP (1 mL), temperature (160 ºC), time (16 h).	61	25

8	**Cu-Cu$_2$O/C$_{550}$**	Iodobenzene (1 mmol), imidazole (1.2 mmol), KOH (2 mmol), catalyst (15 mg), DMSO (2 mL), temperature (80 ºC), time (24 h)	**96**	This work

Table S6 . Comparative catalytic activity data of Cu-Cu_2O/C_{550} reported in literature for the A^3 coupling (decarboxylative C-C and C-N coupling) over various reported catalyst.

S.N	**Catalyst**	**Reaction condition**	**$Y_{A3\ Coupled\ Pdt}$ (%)**	**References**
1	CuI	4-nitrobenzaldehyde (0.42 mmol), proline (0.45 mmol), phenylacetylene (0.3 mmol), catalyst (0.045 mmol), Toluene (1.5 mL), temperature (130 °C), and time (12 h).	95	26
2	RGO@CuO	4-nitrobenzaldehyde (1 mmol), proline (1 mmol), phenylacetylene (1 mmol), catalyst (3.7 mol %), PEG-600 (3 mL), temperature (110 °C), and time (1 h).	99	27
3	**Cu-Cu_2O/C_{550}**	4-nitrobenzaldehyde (1 mmol), proline (1 mmol), phenylacetylene (1 mmol), catalyst (20 mg), PEG-400 (2 mL), temperature (120 °C), and time (2 h).	**>99**	This work

Table S7. Comparative catalytic activity of CuO-Cu_2O/C with various catalysts reported in the literature for the diphenylmethane oxidation.

S.N	**Catalyst**	**Reaction condition**	**$Y_{Benzophenone}$ (%)**	**References**
1	NHPI/xanthone/TMAC	Diphenylmethane (5 mmol), NHPI (10 mol %), xanthone (10 mol %), TMAC (0.5 mol %), O_2 (0.5 MPa), acetonitrile (10 mL), temperature (60 °C) and time (4 h).	65	28
2	silica gel supported cobalt(II) Schiff base complex	Diphenylmethane (20 mmol), NHPI (2 mmol), catalyst (0.1 g), O_2 (1 atm), acetic acid (40 mL), temperature (100 °C) and time (24 h).	83	29
3	$Fe(NO_3)_3·9H_2O$	Diphenylmethane (10 mmol), NHPI (10 mol %), catalyst (0.125 g), air (5 atm), acetonitrile (20 mL), temperature (80 °C) and time (24 h).	84	30
4	$[CoTSPc]^{4-}/SiO_2$	Diphenylmethane (1 mmol), NHPI (10 mol %), catalyst (8 mol %), O_2 (1 atm), acetonitrile (1.5 mL), temperature (25 °C) and time (40 h).	68	31
5	NHPI–acridine yellow–Br_2	Diphenylmethane (1 mL), NHPI (7.5 mol %), acridine yellow (2.5 mol %), Br_2 (2.5 mol %), O_2 (0.3 MPa), acetonitrile (10 mL), temperature (100 °C) and time (20 h).	65	32
6	$Pd(OAc)_2$	Diphenylmethane (0.5 mmol) TBN (1.5 mmol), $Pd(OAc)_2$ (5 mol %), and NHPI (30 mol %) N_2 (1 atm), acetonitrile (0.5 mL), temperature (70 °C) and time (24 h).	80	33
7	aryl-tetrahalogenated N-hydroxyphthalimides and 1,4-diamino-2,3-dichloroanthraquinone (TCNHPI/DADCAQ)	Diphenylmethane (5 mmol) TCNHPI (5 mol %), DADCAQ (1 mol %), O_2 (0.3 MPa), acetonitrile (7 mL), temperature (100 °C) and time (5 h).	49	34
8	NHPI/DMG	Diphenylmethane (2 mmol) NHPI (10mol %), DMG (10 mol %), O_2 (0.5 MPa),	66	35

		acetonitrile (10 mL), temperature (80 °C) and time (6 h).		
9	Au–pDA–rGO	Diphenylmethane (1 mmol), NHPI (10 mol %), catalyst (20 mg), O_2 (10 bar), acetonitrile (5 mL), temperature (60 °C) and time (12 h).	92	36
10	CoPcTs-Zn_2Al -LDH	Diphenylmethane (2 mmol), NHPI (0.4 mmol), catalyst (30 mg), O_2 (10 mL/min.), benzonitrile (6 mL), temperature (120 °C) and time (12 h).	86	37
11	**CuO-Cu_2O/C**	Diphenylmethane (1 mmol), NHPI (10 mol %), catalyst (15 mg), O_2 (1 atm), acetonitrile (5 mL), temperature (80 °C) and time (9 h).	**90**	This work

Notes and references

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