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Supporting information

Visible-Light Driven Photochemical activity of ternary Ag/AgBr/TiO₂ Nanotubes for Oxidation C(sp³)-H and C(sp²)-H Bonds

Mona Hosseini-Sarvari, *a Abdulhamid Dehghani b

^a Department of Chemistry, Shiraz University, Shiraz 7194684795, I.R. Iran

* Corresponding author, E-mail address: hossaini@shirazu.ac.ir (Mona Hosseini-Sarvari)

Contents

Table S1 . An overview on the different catalysts for the oxidation of C(sp bonds	³)-H and C(sp ²)-H
Optimization of reaction conditions	III
Characterization of products	IV
¹ H, ¹³ C NMR Spectra of Synthesized Compounds	VIII
REFERENCES	XXXV

Oxidation C(sp ³)-H							
Entry	Catalyst/ Base	Oxidant	Time	Temperature	Solvent	Irradiation	Ref.
1	Ag/AgBr/TiO ₂	aerobic oxidation	4-12 h	R.T	Solvent-Free	CFL White 15 W	this work
2	Co, Mn, Br ₂	O ₂ (27atm)	-	205 °C	HOAC	-	1
3	KAuCl4. 0.5 H2O	TBHP, Air	24h	90 °C	Pyridine	-	2
4	I ₂	TBHP	18 h	70 °C	CH ₃ CN	-	3
5	LiHDMS (base)	O ₂ (1 atm)	12 h	60 °C	THF	-	4
6	Flavin scandium complex (RFT)	Air	0.5-7 h	R.T	CH ₃ CN/HCl	Blue LED (440nm)	5
7	TBADT	O ₂	45 min	R.T	CH ₃ CN/HCl	LED (365nm)	6
			Ox	idation C(sp ²)	-H		
8	Ag/AgBr/TiO ₂	aerobic oxidation	24-48 h	R.T	CH ₃ CN	CFL White 15 W	this work
9	Fe ₃ O ₄	H ₂ O ₂	2 h	80 °C	Solvent-Free	-	7
10	Co-Ag/ZnO	H ₂ O ₂	24 h	80 °C	АсОН	-	8
11	Pd(II)-L complex	0.5 MPa O ₂	12 h	80 °C	Water	-	9
12	Alumina-supported VxOy Nanoparticle	Anhydrous Hydrogen Peroxide	4.96 h	63 °C	Ethyl acetate	-	10
13	[Cu₂(bipy)₂(btec)]∞	TBHP	6 h	75 °C	Dichloroethane, n-decane,	-	11
14	Eosin Y	air	9- 12 h	-	DMSO	Green LED	12

Table S1. An overview on the different catalysts for the oxidation of $C(sp^3)$ -H and $C(sp^2)$ -H bonds.

Optimization of reaction conditions:

Optimization of reaction conditions

Table S2. Optimization of the reaction parameters for the preparation of benzaldehyde ^a



Entry	photocatalyst	Amount of photocatalyst	Solvent	Light	Atmosphere	Conversion %
1	Ag/AgBr/TiO ₂	0.005	EtOH	CFL 15 W White	air	5
2	Ag/AgBr/TiO ₂	0.005	CH ₃ CN	CFL 15 W White	air	83
3	Ag/AgBr/TiO ₂	0.005	EtOAC	CFL 15 W White	air	79
4	Ag/AgBr/TiO ₂	0.005	Acetone	CFL 15 W White	air	82
5	Ag/AgBr/TiO ₂	0.005	THF	CFL 15 W White	air	52
6	Ag/AgBr/TiO ₂	0.005	MeOH	CFL 15 W White	air	51
7	Ag/AgBr/TiO ₂	0.005	H ₂ O	CFL 15 W White	air	5
8	Ag/AgBr/TiO ₂	0.005	CH_2Cl_2	CFL 15 W White	air	45
9	Ag/AgBr/TiO ₂	0.005	CHCl ₃	CFL 15 W White	air	55
10	Ag/AgBr/TiO ₂	0.005	DMF	CFL 15 W White	air	51
11	Ag/AgBr/TiO ₂	0.005	DMSO	CFL 15 W White	air	52
12	Ag/AgBr/TiO ₂	0.005	CH ₃ CN/H ₂ O	CFL 15 W White	air	80
13	Ag/AgBr/TiO ₂	0.005	Solvent Free	CFL 15 W White	air	0
14	Ag/AgBr/TiO ₂	0.001	CH ₃ CN	CFL 15 W White	air	15
15	Ag/AgBr/TiO ₂	0.003	CH ₃ CN	CFL 15 W White	air	25
16	Ag/AgBr/TiO ₂	0.007	CH ₃ CN	CFL 15 W White	air	84
17	Ag/AgBr/TiO ₂	0.01	CH ₃ CN	CFL 15 W White	air	90
18	Ag/AgBr/TiO ₂	0.015	CH ₃ CN	CFL 15 W White	air	90
19	Ag/AgBr/TiO ₂	0.02	CH ₃ CN	CFL 15 W White	air	95
20	Ag/AgBr/TiO ₂	0.01	CH ₃ CN	Blue LED 12 W	air	20
21	Ag/AgBr/TiO ₂	0.01	CH ₃ CN	Green LED 12 W	air	10
22	Ag/AgBr/TiO ₂	0.01	CH ₃ CN	Red LED 12 W	air	5
23	Ag/AgBr/TiO ₂	0.01	CH ₃ CN	Dark	air	trace
24	Ag/AgBr/TiO ₂	0.01	CH ₃ CN	Sun Light	air	50
25	Ag/AgBr/TiO ₂	0.01	CH ₃ CN	CFL 15 W White	O ₂ balloon	90
26	Ag/AgBr/TiO ₂	0.01	CH ₃ CN	CFL 15 W White	Argon	50
27	AgBr	0.01	CH ₃ CN	CFL 15 W White	air	35
28	Ag/AgBr	0.003	CH ₃ CN	CFL 15 W White	air	50
29	TiO ₂ (P25)	0.003	CH ₃ CN	CFL 15 W White	air	20
30	Ag/TiO ₂	0.003	CH ₃ CN	CFL 15 W White	air	70

^a Reaction conditions: Styrene (0.3 mmol), catalyst in 2 mL of solvent for 24h, at room temperature.

Characterization of products:

Acetophenone (2a, 4m)



Clear colorless liquid; Yield: 96%(2a), 78%(4m); ¹H NMR (300 MHz, CDCl₃): δ (ppm) = 7.97 (d, 2H, *J*= 7.2 Hz, ArH), 7.57 (dd, 1H, *J* = 7.2 Hz, ArH), 7.46(dd, 2H, *J* = 7.2 Hz, ArH), 2.61 (s, 3H, CH₃); ¹³C NMR (75 MHz, CDCl₃): δ (ppm) = 198.9, 137.1, 133.1, 128.6, 128.3, 26.6.

Benzophenone (2b)



White solid; Yield: 82%; m.p. 48.5 °C (Lit¹³. 48.5 °C); ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.82 (d, 4H, *J* = 7.6 Hz, ArH), 7.60 (dd, 2H, *J* = 7.6 Hz, ArH), 7.49 (dd, 4H, *J* = 7.6 Hz, ArH); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 196.7, 137.6, 132.4, 130.1, 128.3. **9H-fluoren-9-one (2c)**



Yellow solid; Yield:41%; m.p. 84 °C (Lit¹⁴. 84 °C); ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.65 (d, 2H, J = 7.2 Hz, ArH), 7.09-7.45 (m, 4H, ArH), 7.30 (d, 2H, J = 7.2 Hz, ArH); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 193.9, 144.4, 134.7, 134.1, 129.1, 124.9, 120.3.

Phenyl(pyridin-2-yl) methanone (2d)



light yellow crystalline; Yield: 66%; mp 41–46 °C (lit¹⁴. 41– 46 °C); ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 8.81 (d, 2H, *J* = 4.4 Hz, ArH), 7.82 (d, 2H, *J* = 7.6 Hz, ArH), 7.64 (dd, 1H, *J* = 7.6 Hz, ArH), 7.58 (dd, 2H, *J* = 4.4 Hz, ArH), 7.52 (dd, 2H, *J* = 7.6 Hz, ArH); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) =195.2, 166.0, 152.7, 150.4, 144.4, 135.9, 133.5, 130.1, 128.7, 122.3.

4-Chlorobenzophenone (2e)



White solid; Yield: 71%; m.p. 75 °C (Lit¹³. 75 °C); ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.85-7.73 (m, 4H, ArH), 7.63 (dd, 1H, J = 6.4 Hz, ArH), 7.52 (d, 2H, J = 6.4 Hz, ArH), 7.48

(dd, 2H, J = 6.4 Hz, ArH); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 195.5, 138.9, 137.2, 135.9, 132.7, 131.5, 129.9, 128.6, 128.4. **1-([1,1'-Biphenyl]-4-yl)ethan-1-one (2f)**



White solid; Yield: 84%, m.p. 119-120 °C (Lit¹⁵. 119-120 °C); ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 8.06 (d, 2H, *J* = 8.4 Hz, ArH), 7.72 (d, 2H, *J* = 10.4 Hz, ArH), 7.66 (d, 2H, *J* = 10.4 Hz, ArH), 7.49 (dd, 2H, *J* = 8.4 Hz, ArH), 7.43 (dd, 1H, *J* = 8.4 Hz, ArH), 2.67 (s, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 197.8, 145.8, 139.9, 135.9, 129.0, 128.9, 128.2, 127.3, 127.2, 26.7.

1-(4-methoxyphenyl) ethan-1-one (2g)



White solid; Yield: 92%, m.p. 35-36 °C (Lit¹⁶. 36-38 °C); ¹H NMR (250 MHz, DMSO-d₆): δ (ppm) = 7.52 (d, 2H, *J* = 9 Hz, ArH), 6.97 (d, 2H, *J* = 9 Hz, ArH), 3.76 (s, 3H, OMe), 2.48 (s, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 196.6, 163.5, 130.5, 113.7, 55.4, 26.2 **1-(4-aminophenyl) ethan-1-one (2h)**



Yellow solid; Yield:48%; m.p. 103-105 °C (Lit¹⁷. 102.8-104.1 °C); ¹H NMR (250 MHz, DMSO-d₆): δ (ppm) = 7.65 (d, 2H, J = 8.7 Hz, ArH), 6.55 (d, 2H, J = 8.7 Hz, ArH), 6.01(s, 2H, NH₂), 2.36 (s, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 196.4, 150.3, 141.4, 129.3, 123.8, 27.0.

1-(4-bromophenyl) ethan-1-one (2i)



White solid; Yield: 65%; m.p. 50 °C (Lit¹⁸. 52-54 °C); ¹H NMR (250 MHz, DMSO-d₆): δ (ppm) = 7.86 (d, 2H, *J* = 8.7 Hz, ArH), 7.86 (d, 2H, *J* = 8.7 Hz, ArH), 2.55 (s, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 197.0, 135.8, 131.9, 129.8, 128.3, 26.5.

Propiophenone (2j)

Colorless liquid; Yield: 83%; ¹H NMR (300 MHz, CDCl₃): δ (ppm) = 7.94 (d, 2H, *J* = 7.2 Hz, ArH), 7.51 (dd, 1H, *J* = 7.2 Hz, ArH), 7.41 (dd, 2H, *J* = 7.2 Hz, ArH), 2.96 (q, 4H, J = 7.2 Hz, CH₂), 1.19 (t, 3H, *J* = 7.2 Hz, CH₃); ¹³C NMR (75 MHz, CDCl₃): δ (ppm) = 200.7, 136.8, 132.8, 128.52, 127.9, 31.7, 8.9.

1-(4-nitrophenyl) ethan-1-one (2k)



Light yellow solid, Yield: 91%; m.p. 79 °C (Lit¹⁹. 80-81 °C); ¹H NMR (250 MHz, DMSO-d₆): δ (ppm) = 8.27 (d, 2H, J = 8.5 Hz, ArH), 8.12 (d, 2H, J = 8.5 Hz, ArH), 2.63 (s, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 196.4, 150.3, 141.4, 129.3, 123.8, 27.0.

1-(4-fluorophenyl) ethan-1-one (2l)



Clear colorless liquid¹⁷; Yield:86%, ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 7.88 (d, 2H, *J* = 4 Hz, ArH), 7.02 (d, 2H, *J* = 4Hz, ArH), 2.49 (s, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 191.1, 134.2, 134.0, 129.0, 128.5, 46.1.

1-(3-nitrophenyl) ethan-1-one (2m)



Yellow solid; Yield: 62%; m.p. 78°C (Lit¹⁹. 76-80 °C); ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 8.46 (s, 1H), 7.52 (dd, 1H, *J* = 7.6 Hz, ArH), 7.11 (d, 1H, *J* = 7.6 Hz, ArH), 7.05 (d, 1H, *J* = 7.6 Hz, ArH), 2.52 (s, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 190.6, 131.8, 129.8, 115.9, 114.2, 55.4.

1-(2-nitrophenyl) ethan-1-one (2n)



Brown oil¹⁹; Yield: 53%; ¹H NMR (250 MHz, CDCl₃): δ (ppm) = 8.16 (d, 1H, *J* = 7.5 Hz, ArH), 7.61 (dd, 1H, *J* = 7.5 Hz, ArH), 7.52 (dd, 1H, *J* = 7.5 Hz, ArH), 7.24 (d, 1H, J = 7.5 Hz, ArH), 1.18 (s, 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 197.8, 145.8, 139.9, 135.9, 129.0, 128.2, 127.3, 26.7.

1-(pyridin-3-yl) ethan-1-one (20)



Liquid¹⁷; Yield:74%; ¹H NMR (400 MHz, CDCl₃); δ (ppm) = 8.94 (d, 1H, *J* = 8 Hz, ArH), 8.55 (dd, 1H, *J* = 4 Hz, ArH), 8.01 (d, 1H, *J* = 8 Hz, ArH), 7.22 (s, 1H, ArH), 2.44 (s. 3H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 196.4, 167.0, 164.4, 133.6, 133.5, 130.9, 130.9, 115.7, 115.5, 26.4.

Benzaldehyde (4a, 4b)



Colorless liquid; Yield: 90% (4a), 12% (4b); ¹H NMR (300 MHz, CDCl₃): δ (ppm) = 10.03 (s, 1H), 7.89 (d, 2H, *J* = 7.8 Hz, ArH), 7.64 (dd, 1H, *J* = 6.9 Hz, ArH), 7.54 (dd, 2H, *J* = 7.8 Hz, ArH); ¹³C NMR (75 MHz, CDCl₃): δ (ppm) = 192.4, 136.4, 134.5, 129.7, 129.0. **4-methoxybenzaldehyde (4c)**

Clear liquid²⁰; Yield: 52%; ¹H NMR (300 MHz, CDCl₃): δ (ppm) = 9.88 (s, 1H), 7.84 (d, 2H, J = 8.7 Hz, ArH), 7.00 (d, 2H, J = 8.7 Hz, ArH), 3.89 (s, 3H, OMe); ¹³C NMR (75 MHz, CDCl₃): δ (ppm) = 190.8, 164.6, 132.0, 129.9, 114.3, 55.6.

4-fluorobenzaldehyde (4d)



Yellow liquid; Yield:42%; ¹H NMR (300 MHz, CDCl₃): δ (ppm) = 9.97 (s,1H), 7.91 (dd, 2H, *J*= 8.4, 5.4 Hz, ArH), 7.21 (dd, 2H, *J*= 8.7, 8.1 Hz, ArH); ¹³C NMR (75 MHz, CDCl₃): δ (ppm) = 190.5, 166.5 (d, *J* = 255.0 Hz), 132.9(d, *J* = 2.0 Hz), 132.2 (d, *J* = 9.6 Hz), 116.3 (d, *J* = 22.3 Hz).

4-bromobenzaldehyde (4e)



White solid; Yield: 49%; m.p. 56-58 °C; ¹H NMR (250 MHz, CDCl₃): δ (ppm) = 10.00 (s, 1H), 7.74 (d, 2H, J = 8.2 Hz, ArH), 7.69 (d, 2H, J = 8.2 Hz, ArH); ¹³C NMR (75 MHz, CDCl₃) δ 191.1, 135.0, 132.5, 131.0, 129.8.

4-chlorobenzaldehyde (4f)

0



White solid; Yield: 56%; m.p. 47 °C (Lit²¹. 47.5 °C); ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 9.99 (s, 1H), 7.83 (d, 2H, J = 8.4 Hz, ArH), 7.52 (d, 2H, J = 8.4 Hz, ArH); ¹³C NMR (100 MHz, CDCl₃): δ (ppm) = 190.9, 141.0, 134.7, 130.9, 129.5.

4-(chloromethyl)benzaldehyde and 2-(chloromethyl)benzaldehyde (4g)



Colorless liquid; Yield: 88%, ¹H NMR (300 MHz, CDCl₃): δ (ppm) = 9.95 (s, 1H), 9.94 (s, 1H), 7.83- 7.76 (m, 4H, ArH), 7.60 (d, 1H, *J* = 7.5 Hz, ArH), 4.48-7.50 (m, 3H, ArH), 4.58 (s, 2H, CH₂), 4.56 (s, 2H, CH₂); ¹³C NMR (75 MHz, CDCl₃): δ (ppm) = 191.8, 191.7, 143.8, 138.6, 136.8, 136.1, 134.5, 130.1, 129.8, 129.5, 129.5, 129.1, 50.9, 45.3.

4-nitrobenzaldehyde (4h)



Yellow to brown crystalline powder; Yield: 62%; m.p. 103-105.5 °C (Lit²². 103-106 °C); ¹H NMR (300 MHz, CDCl₃): δ (ppm) = 10.18 (s, 1H), 8.42 (d, 2H, *J* = 8.7 Hz, ArH), 8.10 (d, 2H, *J* = 8.7 Hz, ArH); ¹³C NMR (75 MHz, CDCl₃): δ (ppm) = 190.3, 151.1, 140.0, 130.5, 124.3. **Isonicotinaldehyde (4i)**



Slight brown liquid²⁰; Yield: 54%, ¹H NMR (400 MHz, CDCl₃): δ (ppm) = 10.02 (s, 1H), 8.81 (d, 2H, *J* = 5.6Hz, ArH), 7.64 (d, 2H, *J* = 5.6Hz, ArH); ¹³C NMR (75 MHz, CDCl₃): δ (ppm) = 191.5, 151.2, 141.3, 122.1.

Picolinaldehyde (4j)



Brown liquid²⁰; Yield: 57%; ¹H NMR (400 MHz CDCl₃): δ (ppm) = 10.07 (s, 1H), 8.78 (d, 1H, J = 4.8 Hz, ArH), 7.95 (d, 1H, J = 8 Hz, ArH), 7.87 (dd, 1H, J = 10.8, 8 Hz, ArH), 7.51 (dd, 1H, J = 10.8, 4.8 Hz, ArH); ¹³CNMR (100 MHz, CDCl₃ ppm): δ (ppm) = 193.4, 152.8, 150.2, 137.1, 127.9, 121.7.

Isophthalaldehyde (4k)



White to yellow solid, Yield: 69%; m.p. 86-88 °C; ¹H NMR (300 MHz, CDCl₃): δ (ppm) = 10.05 (s, 2H), 8.32 (s, 1H, ArH), 8.10 (d, 2H, *J* = 7.5 Hz, ArH), 7.67 (dd, 1H, *J* = 7.5 Hz, ArH); ¹³C NMR (75 MHz, CDCl₃): δ (ppm) = 191.1, 137.0, 134.7, 131.0, 129.9.

Terephthalaldehyde (4l)



Light yellow solid, Yield: 55%; m.p. 114.5-116 °C; ¹H NMR (300 MHz, CDCl₃): δ (ppm) = 10.14 (s, 2H), 8.06 (s, 4H); ¹³C NMR (75 MHz, CDCl₃): δ (ppm) = 191.5, 140.0, 130.1.

¹H, ¹³C NMR Spectra of Synthesized Compounds:



Figure 2:¹³C NMR spectrum of Acetophenone (2a, 4m).



Figure 4:¹³C NMR spectrum of Benzophenone (2b).



Figure 6:¹³C NMR spectrum of 9H-fluoren-9-one (**2c**).



Figure 8:¹³C NMR spectrum of Phenyl(pyridin-2-yl) methanone (**2d**).



Figure 9:¹H NMR spectrum of 4-Chlorobenzophenone (2e).



Figure 10:¹³C NMR spectrum of 4-Chlorobenzophenone (2e).



Figure 12:¹³C NMR spectrum of 1-([1,1'-Biphenyl]-4-yl)ethan-1-one (**2f**).



Figure 14:¹³C NMR spectrum of 1-(4-methoxyphenyl) ethan-1-one (2g).



Figure 16:¹³C NMR spectrum of 1-(4-aminophenyl) ethan-1-one (2h).



Figure 18:¹³C NMR spectrum of 1-(4-bromophenyl) ethan-1-one (2i).



Figure 20:¹³C NMR spectrum of Propiophenone (2j).



Figure 22:¹³C NMR spectrum of 1-(4-nitrophenyl) ethan-1-one (**2k**).



Figure 24:¹³C NMR spectrum of 1-(4-fluorophenyl) ethan-1-one (2l).



Figure 26:¹³C NMR spectrum of 1-(3-nitrophenyl) ethan-1-one (2m).



Figure 28:¹³C NMR spectrum of 1-(2-nitrophenyl) ethan-1-one (**2n**).



Figure 30:¹³C NMR spectrum of 1-(pyridin-3-yl) ethan-1-one (20).

Figure 32:¹³C NMR spectrum of Benzaldehyde (**4a**, **4b**).

Figure 34:¹³C NMR spectrum of 4-methoxybenzaldehyde (**4c**).

Figure 36:¹³C NMR spectrum of 4-fluorobenzaldehyde (**4d**).

Figure 38:¹³C NMR spectrum of 4-bromobenzaldehyde (**4e**).

Figure 40:¹³C NMR spectrum of 4-chlorobenzaldehyde (4f).

Figure 41:¹H NMR spectrum of 4-(chloromethyl)benzaldehyde and 2-(chloromethyl)benzaldehyde (**4g**).

Figure 42:¹³C NMR spectrum of 4-(chloromethyl)benzaldehyde and 2-(chloromethyl)benzaldehyde (**4g**).

Figure 44:¹³C NMR spectrum of 4-nitrobenzaldehyde (4h).

Figure 46:¹³C NMR spectrum of Isonicotinaldehyde (4i).

Figure 48:¹³C NMR spectrum of Picolinaldehyde (**4j**).

Figure 50:¹³C NMR spectrum of Isophthalaldehyde (4k).

Figure 52:¹³C NMR spectrum of Terephthalaldehyde (41).

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