Triazine functionalized ordered mesoporous polymer: a novel solid support for Pd-mediated C-C cross coupling reactions in water

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Supporting information

Spectroscopic data of ^{1}H & ^{13}C NMR of Mizoroki-Heck coupling products:

Product,(E)-Stilbene:(Table-1, Entry 1, 2, 16 ; S_1):

White crystalline solid, mp = 120-122 ^{0}C.

^{1}H NMR(300 MHz $CDCl_3$) δ = 7.59-7.56 (d, 4H, Ar-H) , 7.44-7.39 (t, 4H, Ar-H) , 7.35-7.18 (m,4H,Ar-H & vinyl-H) ppm.
^{13}C NMR (75 MHz $CDCl_3$) : 137.4,128.7,127.6,126.6 ppm

Product,(E)-4-methoxy Stilbene (Table-1, Entry 7, S_4) :

Pale yellow crystalline solid, mp = 135-138 ^{0}C.

^{1}H NMR (300 MHz DMSO-d_6) δ = 7.57 (d, J= 4.36 Hz, 2H, ArH),7.54 (d, J=5.9 Hz, 2H, ArH), 6.96 (d, J=8.68 Hz, 2H, ArH), 7.38 (t, J=7.37 Hz, 2H, ArH), 7.25 (t, J=7.33 Hz, 1H, ArH),7.22 (d, J=16.4 Hz, 1H, Vinyl-H),7.11 (d, J=16.4 Hz, 1H, Vinyl-H), 3.77 (s, 3H, -OCH_3) ppm

^{13}C NMR (75 MHz, DMSO-d_6) : δ159.4,137.8,130.1,129.1,128.5,127.6,126.5,114.6,55.62 ppm

Product, (E)-4-nitro Stilbene (Table-1, Entry 3, 4,17 ; S_2) :

Yellow orange crystalline solid, mp = 155 ^{0}C.

^{1}H NMR (300 MHz DMSO-d_6) δ = 8.23 (d, J=8.57 Hz, 2H, ArH), 7.86 (d, 2H, J=8.62 Hz, ArH), 7.67 (d, J=7.56, 2H, ArH) 7.54 (d, 1H, J=16.4 Hz, Vinyl-H), 7.43-7.33(m, 4H, 3ArH, 1 Vinyl-H) ppm.

^{13}C NMR (75 MHz, DMSO-d_6) : δ 146.17, 143.9,136.25,133.2, 128.8,127.27,126.3,124.16 ppm.

Product, (E)-para formyl Stilbene (Table-1, Entry 12,18 ; S_7):

White crystalline solid, mp = 114 ^{0}C.

^{1}H NMR (300 MHz DMSO-d_6) δ = 7.90 (d, 2H, J=8.25 Hz, ArH), δ = 7.81 (d, 2H, J=8.25 Hz, ArH) , δ = 7.65 (d, 2H, J=7.24 Hz, ArH), 7.49-7.27 (m, 5H, 3ArH, 2 Vinyl-H) ppm.

^{13}C NMR (75 MHz, DMSO-d_6) :
δ = 192.3,143,136.5,135,131.9,129.9,128.7,127.3,126.9 ppm.

Product, trans-cinamic acid (Table-1, Entry 13,14 ; S_8) :

White crystalline solid, mp = 133 ^{0}C.

^{1}H NMR (300 MHz, $CDCl_3$) : δ= 7.86 (d, 1H, J=9.6 MHz, Vinyl-H), δ= 7.60 (d, 2H, ArH), δ= 7.46 (m, 3H, ArH), δ= 6.52 (d, J= 9.6 Hz, 1H, Vinyl-H) δ= 11.77 (s, 1H, COOH) ppm.

^{13}C NMR (75 MHz, $CDCl_3$) : δ= 172,147,134,130,128,117 ppm.

Product, (E)-4-methoxy cinamic acid (Table-1, Entry 20, S_{10}) :
Pale brown fine crystalline powder, mp = 173 ^{0}C.

^{1}H NMR (300 MHz, $CDCl_3$) : δ =7.82 (d, 1H, J= 9.6 Hz, Vinyl-H), δ= 6.48 (d, 1H, J= 9.6 Hz, Vinyl-H), δ= 7.57-7.37 (m, 4H, ArH) ppm.

^{13}C (75 MHz, $CDCl_3$) : δ = 172,147,134,130,128,117,51.8 ppm.

Product, (E)-4-nitro cinamic acid (Table-1, Entry 15, 19 ; S_9) :

Off white to pale yellow powder, mp = 204 ^{0}C.

^{1}H NMR (300 MHz,DMSO-d_6) δ = 8.16 (d, 2H, J=6.9 Hz, ArH), δ = 7.86 (d, 2H, J=5.4 Hz, ArH), δ = 7.59 (d, 1H, J= 9.9 Hz, Vinyl-H), δ = 6.64 (d, 1H, J=9.9 Hz, Vinyl-H) ppm.

^{13}C (75 MHz, DMSO-d_6) : δ = 167.5, 148.4, 141.8, 129.9, 124.4 ppm.

Product, (E)-4-Acetyl Stilbene (Table-1, Entry 5, 6 ; S_3) :

^{1}H NMR (300 MHz, $CDCl_3$) δ = 2.6 (s, 3H, $COCH_3$), δ = 7.97 (d, 2H, J= 8.3 Hz, ArH), δ = 7.60 (d, 2H, J= 8.4 Hz, Ar-H), δ = 7.55 (d, 2H, J= 7.24 Hz, Ar-H), δ = 7.40 (t, 2H, J= 7.59 Hz, Ar-H), δ = 7.32 (t, 1H, J= 7.16 Hz), δ = 7.15 (d, 1H, J=15.8 Hz, Vinyl-H), 7.10 (d, 1H, J= 15.8 Hz, Vinyl-H) ppm.

^{13}C (75 MHz, $CDCl_3$) : δ = 197.6, 142.17, 136.8, 136.12, 131.63, 129.03, 128.94, 128.46, 127.60, 126.96, 29.8 ppm.

Product, (E)-4-Methyl Stilbene (Table-1, Entry 8, 9 ; S_5) :

^{1}H NMR (300 MHz, $CDCl_3$) δ = 2.34 (s, 3H, -CH_3), δ = 7.16 (d, 2H, J = 7.8 Hz, ArH), δ = 7.25 (t, 1H, J= 7.29 Hz, ArH), δ = 7.25 (t, 2H, J= 7.7 Hz, ArH), δ = 7.41 (d, 2H, J= 8.07 Hz, ArH), δ = 7.50 (d, 2H, J= 7.17 Hz, ArH), δ = 7.06 (s, 2H, Vinylic-H) ppm.

^{13}C (75 MHz, $CDCl_3$) : δ = 137.4, 134.5, 129.3, 128.8, 128.7, 128.58, 128.4, 127.7, 127.6, 127.3, 126.39, 21.19 ppm.

Spectroscopic data of ^{1}H & ^{13}C NMR of Sonogashira coupling products:

Product, Di-phenyl acetylene:(Table-2, Entry 1, 2 ; S_{11}):

Pale yellow crystal, mp = 57 ^{0}C .

^{1}H NMR(300 MHz, $CDCl_3$) δ = 7.60 (d, 4H, J= 6.4 Hz, ArH), δ = 7.45 (m, 6H, ArH) ppm
^{13}C (75 MHz, $CDCl_3$) : δ = 132.6, 129.3, 128.58, 121.96, 81.7 ppm.

Product, 4-nitro-di-phenylacetylene (Table-2, Entry 3 , 4 ; S_{12}) :

Dark yellow needles, mp = 114 ^{0}C.

^{1}H NMR (300 MHz, $CDCl_3$) δ = 8.25 (d, 2H, J= 8.88 Hz, ArH), δ = 7.69 (d, 2H, J= 8.88 Hz, ArH), δ = 7.59 (d, 2H, J= 6.14 Hz, ArH), δ = 7.42 (m, 3H, ArH) ppm.

^{13}C (75 MHz, $CDCl_3$) : δ = 147.1, 132.6, 131.9, 130.4, 129.4,128.68,128.58,123.77, 122.25, 94.8 ppm.

Product, 4-acetyl-di-phenylacetylene (Table-2, Entry 5, 6 ; S_{13}) :

Light yellow solid, mp = 98 ^{0}C.

^{1}H NMR (300 MHz, $CDCl_3$) δ = 2.61 (s, 3H, $COCH_3$), δ = 7.937 (d, 2H, J=8.6 Hz, ArH) δ = 7.356 (m,3H, ArH), δ = 7.543 (m, 4H, ArH) ppm.

^{13}C (75 MHz, $CDCl_3$) : δ = 196.5, 137.1, 128, 94, 132.2, 121, 125, 128.4, 132.3 ppm

Product, 4-methyl-di-phenylacetylene (Table-2, Entry 7, S_{14}) :

^{1}H NMR (500 MHz, $CDCl_3$) δ = 2.61 (s, 3H, -CH_3), δ = 7.36 (m, 4H, ArH), δ = 7.84 (d, 2H, J= 9 Hz, ArH), δ = 7.65 (d, 2H, J=8.5 Hz, ArH), 7.54 (m, 1H, ArH) ppm.

^{13}C (125 MHz, $CDCl_3$): δ = 128.8, 139.5, 132.1, 119, 94, 125.3, 132.1, 129.3, 128.2, ppm

Spectroscopic data of (^{1}H & ^{13}C NMR) of Suzuki-Miyaura coupling products:

Product, Biphenyl (Table-3, Entry 1, 2, 16 ; S_{15}) :

Colourless crystalline solid, m.p= 65^{0}C

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.53 (d, 4H, J=8.7 Hz, ArH), δ = 7.38 (t, 4H, J=7.77 Hz, ArH), δ = 7.26 (t, 2H, J=7.32 Hz) ppm.

^{13}C (75 MHz, $CDCl_3$) : δ = 141.39, 128.89, 127.39, 127.31 ppm.

Product, 4-Nitro-Biphenyl (Table-3, Entry 3, 4, 17 ; S_{16}) :

Yellow needle like crystalline solid with sweetish odour, m.p= 113-115^{0}C

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.31 (d, 2H, J=8.84 Hz, ArH), 7.76 (d, 2H, J=8.85 Hz, ArH), 7.64 (d, 2H, J=7.85), 7.53 (m, 3H, ArH) ppm.

^{13}C (75 MHz, $CDCl_3$) : δ = 147.74, 147.21, 138.8, 129.25, 129, 127.904, 127.48, 124.20 ppm.

Product, 4-Acetyl-Biphenyl (Table-3, Entry 5, 6, 18 ; S_{17}) :

White to yellowish powder, m.p= 118-112^{0}C

^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.64 (s, 3H, -CH_3), 8.05 (d, 2H, J=8.30 Hz, ArH), 7.70 (d, 2H, J=8.30 Hz, ArH), 7.64 (d, J=7.13 Hz, 2H, ArH), 7.47 (m, 3H, ArH) ppm.

^{13}C (75 MHz, $CDCl_3$) : δ = 26.8, 197.9, 145, 140, 136, 129.1, 128.37, 127.42, 127.35 ppm.

Product, 4-Methoxy-Biphenyl (Table-3, Entry 7 ; S_{18}) :

White powder, m.p= 88-92^{0}C

^1H NMR (300 MHz, $CDCl_3$): δ = 3.77 (s, 3H, $-CH_3$), 6.92 (d, 2H, J=8.76 Hz, ArH), 7.25 (t, 2H, J=7.24 Hz, ArH), 7.36 (t, 1H, J=7.29 Hz, ArH), 7.49 (m, 4H, ArH) ppm.

^{13}C (75 MHz, $CDCl_3$) : δ = 159.3, 140.9, 133.9, 128.8, 128.3, 126.8, 126.7, 114.3, 55.48 ppm.

Product, 4-Methyl-Biphenyl (Table-3, Entry 8, 9 ; S_{19}) :

White crystalline solid, m.p= 49-51^{0}C

^1H NMR (300 MHz, $CDCl_3$): δ = 2.40 (s, 3H, $-CH_3$), 7.60 (d, 2H, J= 7.10 Hz, ArH), 7.51 (d, 2H, J= 8.13 Hz), 7.45 (t, 2H, J=7.19 Hz), 7.35 (t, 1H, J=7.23 Hz), 7.27 (d, 2H, J= 7.8 Hz)

^{13}C (75 MHz, $CDCl_3$) : δ = 141.3, 138.5, 137.16, 135.80, 133.59, 129.62, 128.85, 128.14, 127.12 ppm.

Product, 4-Phenyl-Benzonitrile (Table-3, Entry 10, 11, 19 ; S_{20}) :

Pale yellow solid, m.p= 85-87^{0}C

^1H NMR (300 MHz, $CDCl_3$): δ = 7.74 (d, 4H, J= 8.62 Hz, ArH), 7.61 (d, 2H, J=7.82 Hz, ArH), 7.57 (m, 3H, ArH)

^{13}C (75 MHz, $CDCl_3$): δ = 145.84, 139.3, 132.75, 129.26, 128.81, 127.89, 127.38, 119.08, 111.08 ppm.

Product, 4-Phenyl-Salicylaldehyde (Table-3, Entry 12 ; S_{21}) :

White solid, m.p= 79-80^{0}C, IR spectrum: 1670 (-C=0)

^1H NMR (300 MHz, $CDCl_3$) δ = 10.9 (s, 1H, CHO), 9.9 (S, 1H, -OH), 7.68 (s, 1H, ArH), 7.02 (d, 1H, J= 9.45 Hz, ArH), 7.30 (m, 1H, ArH), 7.38 (t, 2H, J= 7.17 Hz, ArH), 7.49 (d, 2H, J= 7.23 Hz, ArH), 7.71 (d, 1H, J= 7.59 Hz, ArH) ppm.

^{13}C (75 MHz, $CDCl_3$): δ = 196.7, 161.09, 139.45, 136.42, 135.87, 133.46, 131.97, 129.09, 127.51, 126.71, 120.83, 118.25 ppm.

Product, Para-terphenyl (Table-3, Entry 14, 15 ; S_{23}) :

White powder, m.p= 212-214^{0}C

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.46 (m, 8H, ArH), 7.56 (m, 6H, ArH) ppm.

^{13}C (75 MHz, $CDCl_3$): δ = 129, 127.4, 135.5, 137 ppm.

Product, Biphenyl-4-carboxylic acid (Table-3, Entry 13 ; S_{22}):

White solid powder, m.p= 224-228^{0}C

^{1}H NMR (300 MHz, DMSO-d_6): δ = 7.77 (d, 2H, J= 8.49 Hz, ArH), 7.61 (d, 2H, J= 8.46 Hz, ArH), 7.68 (d, 2H, J= 6.44 Hz), 7.23 (m, 3H, ArH) ppm.

^{13}C (75 MHz, DMSO-d_6): δ = 167, 134.6, 132.25, 131.8, 130.5, 127.8, 127.4 ppm.