

Synthesis of silver-graphene nanocomposite and its catalytic application for the one-pot three-component coupling reaction and one-pot synthesis of 1,4-disubstituted 1,2,3-triazoles in water

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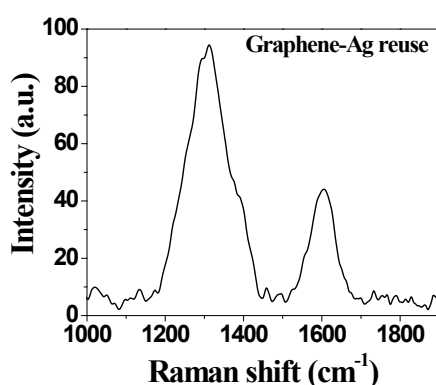


Figure S1 Raman spectra of graphene-Ag reused materials shows two well documented D and G band at 1308 and 1607 cm⁻¹.

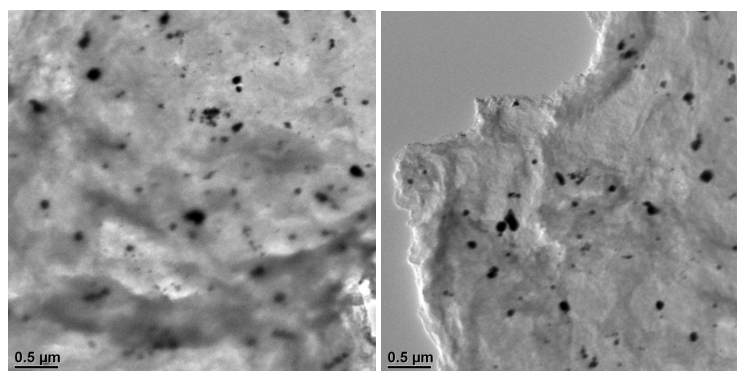


Figure S2 TEM image of reused graphene-Ag composite materials.

Spectral data of the isolated product:

The three-component (A^3) coupling reaction:

1-(1-Cyclohexyl-3-phenyl-2-propynyl)piperidine.¹

¹H-NMR (CDCl₃, 400MHz, ppm): δ 7.46-7.44 (m, 2H), 7.30-7.24 (m, 3H), 3.09 (d, 1H), 2.65-2.60 (m, 2H), 2.44-2.40 (m, 2H), 2.14-2.04 (m, 2H), 1.80-1.70 (m, 2H), 1.65-1.51(m, 6H), 1.45-1.40 (m, 2H), 1.34-1.15 (m, 3H), 1.07-0.88 (m, 2H).

N-(3-Phenyl)-prop-2-ynyl piperidine¹

¹H-NMR (CDCl₃, 400MHz, ppm): δ = 7.44-7.42 (m, 2H), 7.31-7.28 (m, 3H), 3.47 (s, 2H), 2.56 (br, 4H), 1.69-1.60 (m, 4H), 1.45 (br, 2H).

1-(5-cyclohexyl-1-phenylpent-1-yn-3-yl) piperidine¹

¹H-NMR (CDCl₃, 400MHz, ppm): δ = 7.42-7.41 (m, 2H), 7.30-7.25 (m, 3H), 3.49-3.46 (t, 1H), 2.70-2.65 (m, 2H), 2.47 (br, 2H), 1.75-1.53 (m, 10H), 1.51-1.40 (m, 3H), 1.36-1.08 (m, 6H), 0.96-0.87 (m, 2H).

N-(1-Isopropyl-3-phenyl-2-propynyl) piperidine¹

¹H-NMR (CDCl₃, 400MHz, ppm): δ = 7.47-7.44 (m, 2H), 7.34-7.30 (m, 3H), 3.01 (d, 1H), 2.69-2.65 (m, 2H), 2.43 (br, 2H), 1.97-1.93 (m, 1H), 1.68-1.57 (m, 4H), 1.49-1.46 (m, 2H), 1.10 (d, 3H), 1.02 (d, 3H).

1-[1-(1-Ethylpropyl)-3-phenyl-2-propynyl] piperidine¹

¹H-NMR (CDCl₃, 400MHz, ppm): δ = 7.44-7.41 (m, 2H), 7.33-7.23 (m, 3H), 3.21 (d, 1H), 2.65-2.61 (m, 2H), 2.43-2.39 (m, 2H), 1.78-1.65 (m, 1H), 1.61-1.50 (m, 6H), 1.51-1.40 (m, 4H), 0.91 (t, 3H), 0.83 (t, 3H).

1-(1, 3-diphenylprop-2-ynyl) piperidine²

¹H-NMR (CDCl₃, 400MHz, ppm) δ = 7.68-7.60 (m, 2H), 7.57-7.52(m, 2H), 7.41-7.25(m, 6H), 4.80(s, 1H), 2.63-2.52(m, 4H), 1.70-1.52(m, 4H), 1.51-1.42(m, 2H)

N-[1-(4-Methoxyphenyl)-3-phenyl-2-propynyl]piperidine²

¹H-NMR (CDCl₃, 400MHz, ppm) δ = 7.60-7.55(m, 4H), 7.36-7.30(m, 3H), 6.95-6.92(m, 2H), 4.75(s, 1H), 3.80(s, 3H), 2.65-2.50(m, 4H), 1.71-1.55(m, 4H), 1.55-1.40(m, 2H)

N-[1-(4-Methylphenyl)-3-phenyl-2-propynyl]piperidine²

¹H-NMR (CDCl₃, 400MHz, ppm) δ = 7.60-7.57(m, 4H), 7.44-7.33(m, 3H), 7.20(d, 2H), 4.81(s, 1H), 2.65-2.57(m, 4H), 2.40(s, 3H), 1.72-1.56(m, 4H), 1.55-1.46(m, 2H)

N-[1-(4-Ethylphenyl)-3-phenyl-2-propynyl]piperidine²

¹H-NMR (CDCl₃, 400MHz, ppm) δ = 7.54-7.46(m, 4H), 7.34-7.26(m, 3H), 7.19(d, 2H), 4.76(s, 1H), 2.63(q, 2H), 2.61-2.48(m, 4H), 1.64-1.51(m, 4H), 1.51-1.35(m, 2H), 1.25(t, 3H)

N-[1-(4-Chlorophenyl)-3-phenyl-2-propynyl]piperidine²

¹H-NMR (CDCl₃, 400MHz, ppm) δ= 7.64-7.60(m, 2H), 7.58-7.55(m, 2H), 7.41-7.35(m, 5H), 4.82(s, 1H), 2.60-2.55(m, 4H), 1.72-1.55(m, 4H), 1.53-1.47(m, 2H)

N-[1-(4-Bromophenyl)-3-phenyl-2-propynyl]piperidine²

¹H-NMR (CDCl₃, 400MHz, ppm) δ= 7.65-7.58(m, 4H), 7.54-7.50(m, 2H), 7.41-7.37(m, 3H), 4.80(s, 1H), 2.67-2.58(m, 4H), 1.75-1.59(m, 4H), 1.55-1.49(m, 2H)

1-(1,3-Diphenylprop-2-ynyl)pyrrolidine³

¹H-NMR (CDCl₃, 500MHz, ppm): δ= 1.85 (s, 4H), 2.74 (s, 4H), 4.92 (s, 1H), 7.30–7.36 (m, 4H), 7.41 (t, 2H), 7.52–7.55 (m, 2H), 7.62-7.67 (m, 2H).

1-[1-Cyclohexyl-3-(4-methylphenyl)-2-propynyl] piperidine⁴

¹H-NMR (CDCl₃, 400MHz): δ 7.32 (d, 2H), 7.07 (d, 2H), 3.09 (d, 1H), 2.67-2.59 (m, 2H), 2.41-2.30 (m, 2H), 2.33 (s, 3H), 2.13-1.95 (m, 2H), 1.77-1.67 (m, 2H), 1.66-1.45 (m, 6H), 1.45-1.35 (m, 2H), 1.33-1.10(m, 3H), 1.06-0.87 (m, 2H)

1-(1-cyclohexyl-3-(4-methoxyphenyl) prop-2-ynyl) piperidine¹

¹H-NMR (400MHz, CDCl₃): δ= 7.38 (d, 2H), 6.83 (d, 2H), 3.84 (s, 3H), 3.09 (d, 1H), 2.63-2.55 (m, 2H), 2.37 (br, 2H), 2.13-2.04 (m, 2H), 1.75-1.58 (m, 8H), 1.45-1.40 (m, 2H), 1.33-1.04 (m, 3H), 1.00-0.88 (m, 2H)

1-(1-cyclohexyl-3-(3-methoxyphenyl) prop-2-ynyl) piperidine¹

¹H-NMR (400MHz, CDCl₃): δ= 7.24-7.17 (m, 1H), 7.06 (d, 1H), 6.97 (d, 1H), 6.85-6.82 (d, 1H), 3.83 (s, 3H), 3.10(d, 1H), 2.65-2.56 (m, 2H), 2.35 (br, 2H), 2.10-2.00(m, 2H), 1.78-1.55 (m, 8H), 1.47-1.40 (m, 2H), 1.30-1.01 (m, 3H), 1.04-0.85 (m, 2H)

1-(3-(4-chlorophenyl)-1-cyclohexylprop-2-ynyl) piperidine¹

¹H-NMR (400MHz, CDCl₃): δ= 7.38 (d, 2H), 7.25 (d, 2H), 3.06 (d, 1H), 2.60-2.55(m, 2H), 2.36 (br, 2H), 2.07-2.03 (m, 2H), 1.85-1.75 (m, 2H), 1.69-1.53 (m, 6H), 1.46-1.42 (m, 2H), 1.32-1.13 (m, 3H), 1.04-0.82 (m, 2H)

One-pot click reaction:

1, 4-diphenyl-1H-1,2,3-triazole⁵

¹H-NMR (500MHz, CDCl₃): δ=7.35 (t, 1H), 7.45-7.46 (m, 3H), 7.56 (t, 2H), 7.78 (d, 2H), 7.91 (d, 2H), 8.4 (s, 1H)

1-(2-iodophenyl)-4-phenyl-1H-1,2,3-triazole⁵

¹H-NMR (400MHz, CDCl₃): δ= 7.24-7.27 (m, 2H), 7.38 (t, 1H), 7.46-7.55 (m, 3H), 7.9 (d, 2H), 8.016 (d, 1H), 8.1 (s, 1H)

1-(4-methoxyphenyl)-4-phenyl-1H-1,2,3-triazole⁶

¹H-NMR (300MHz, CDCl₃): δ= 3.91 (s, 3H), 7.04 -7.06 (m, 2H), 7.37-7.49 (m, 3H), 7.68-7.71 (m, 2H), 7.89-7.93 (m, 2H), 8.12 (s, 1H)

3-(4-phenyl-1H-1,2,3-triazol-1-yl)phenol⁷

¹H-NMR (300MHz, CDCl₃): δ= 6.9 (d, 1H), 7.34-7.42 (m, 3H), 7.46-7.49 (m, 2H), 7.93-7.94 (m, 3H), 9.3 (s, 1H), 10.1 (s, 1H)

1-(2-nitrophenyl)-4-phenyl-1H-1,2,3-triazole⁵

¹H-NMR (300MHz, CDCl₃): δ= 7.27 (s, 1H), 7.38 (t, 2H), 7.47 (t, 2H), 7.69-7.74 (m, 1H), 7.82 (t, 2H), 8.07 (s, 1H), 8.10 (d, 1H)

1-(3-chlorophenyl)-4-phenyl-1H-1,2,3-triazole⁵

¹H-NMR (400MHz, CDCl₃): δ= 7.37-7.39 (m, 1H), 7.44-7.51 (m, 4H), 7.71 (d, 1H), 7.84 (s, 1H), 7.90 (d, 2H) 8.2 (s, 1H)

1-benzyl-4-phenyl-1H-1,2,3-triazole⁶

¹H-NMR (400MHz, CDCl₃): δ= 5.58 (s, 2H), 7.27-7.34 (m, 4H), 7.37-7.42 (m, 4H), 7.67 (s, 1H), 7.8 (d, 1H)

4-(6-Methoxynaphthalen-2-yl)-1-(2-nitrophenyl)-1H-1,2,3-triazole

¹H NMR (300 MHz, DMSO-d₆) δ= 3.87 (s, 3H), 7.21 (dd, 1H), 7.39 (s, 1H), 7.89 (t, 1H) 7.90-7.97 (m, 3H) 7.95-8.03 (m, 3H), 8.26 (d, 1H), 8.43 (s, 1H)

3-(4-(4-methoxyphenyl)-1H-1,2,3-triazol-1-yl)phenol⁸

¹H NMR (400 MHz, DMSO-d₆) δ= 3.82 (s, 3H), 6.9 (d, 1H), 7.06-7.07 (m, 2H), 7.34-7.43 (m, 3H), 7.86 (d, 2H), 9.14 (s, 1H) 10.1 (s, 1H)

4-(1-(4-methoxyphenyl)-1H-1,2,3-triazol-4-yl)benzonitrile⁹

¹H NMR (400 MHz, CDCl₃) δ= 3.88 (s, 3H), 7.04 (dd, 2H), 7.64-7.68 (m, 2H), 7.69-7.73 (m, 2H), 7.98-8.02 (m, 2H), 8.2 (s, 1H)

4-(4-ethynylphenyl)-1-(2-nitrophenyl)-1H-1,2,3-triazole

¹H NMR (400 MHz, CDCl₃) δ= 3.17 (s, 1H), 7.54 (d, 2H), 7.65-7.70 (m, 2H), 7.75-7.86 (m, 3H), 8.05-8.08 (m, 1H), 8.11 (s, 1H)

1-(3-chlorophenyl)-4-(4-fluorophenyl)-1H-1,2,3-triazole

¹H NMR (500 MHz, CDCl₃) δ= 7.17 (s, 2H), 7.45-7.51 (m, 2H), 7.71 (s, 1H), 7.84 (d, *J* = 22 Hz, 3H), 8.17 (s, 1H)

2-(1-benzyl-1H-1,2,3-triazol-4-yl)pyridine⁵

¹H NMR (400 MHz, CDCl₃) δ= 5.55 (s, H), 7.18 (t, 1H), 7.27- 7.35 (m, 5H), 7.73 (t, 1H), 8.06 (s, 1H), 8.14-8.16 (m, 1H), 8.51 (s, 1H)

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