

Electronic Supplementary Information (ESI†)

Ni–rGO–zeolite nanocomposite: An efficient heterogeneous catalyst for one-pot synthesis of triazoles in water†

Prasun Choudhury,^a Shreyasi Chattopadhyay,^b Goutam De^{*bc} and Basudeb Basu^{*ad}

^aDepartment of Chemistry, North Bengal University, Darjeeling 734013, India. E-mail: basu_nbu@hotmail.com

^bCSIR-Central Glass & Ceramics Research Institute, 196, Raja S. C. Mullick Road, Jadavpur, Kolkata 700032, India

^cS. N. Bose National Centre for Basic Sciences, Block – JD, Sector – III, Salt Lake, Kolkata 700106, India. E-mail: g.de@bose.res.in

^dRaiganj University, Raiganj 733134, India

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SI-1. Materials and Methods

SI-1.1. Chemicals

All reagents were purchased from Sigma–Aldrich and used directly without further purification. NaY zeolite was purchased from Sigma–Aldrich. The solvents were purchased from commercial suppliers and used after distillation. For TLC, Merck plates coated with silica gel 60, F₂₅₄ were used. The products were purified by column chromatography on 60–120 mesh silica gel (Merck, India).

SI-1.2. Characterization

FT–IR spectra were recorded in FT–IR 8300 SHIMADZU spectrophotometer. The ¹H & ¹³C NMR spectra were recorded at 400 MHz and 100 MHz respectively on Bruker Ascend 400 spectrometer in CDCl₃ and DMSO–d₆. Splitting patterns of protons were described as s (singlet), d (doublet), dd (doublet of doublet) and m (multiplet). Chemical shifts (δ) were reported in parts per million (ppm) relative to TMS as internal standard. *J* values (coupling constant) were reported in Hz (Hertz). ¹³C NMR spectra were recorded with complete proton decoupling (CDCl₃: δ 77.0 ppm and DMSO–d₆: 39.5 ppm). Centrifugation was done in REMI R–8C DX centrifuge. Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP–AES) was measured by using SPECTRO analytical instruments GmbH, Germany. The X–ray diffraction studies (PXRD) were done by the Rigaku SmartLab (9 kW) diffractometer using CuK α radiation. Raman spectra of the samples were obtained with Renishaw InVia micro Raman spectroscopy with 514 nm laser source. Scanning Electron Microscopy (SEM) and Electron–Dispersive X–ray Spectroscopy (EDS) were performed using JEOL JSM–IT 100 electron microscope. EDS elemental analysis was done in 3 different areas of the samples, and average values were reported. Transmission electron microscopy (TEM) measurements were carried out using a JEOL JEM–2100F electron microscope. X–ray photoelectron spectroscopic (XPS) measurements were done on a PHI 5000 Versaprobe II XPS system with an Al K α source and a charge neutralizer at room temperature.

SI-2. Characterization of Ni–zeolite and Ni–rGO nanocomposites

SI-2.1. FT–IR spectrum of Ni–rGO

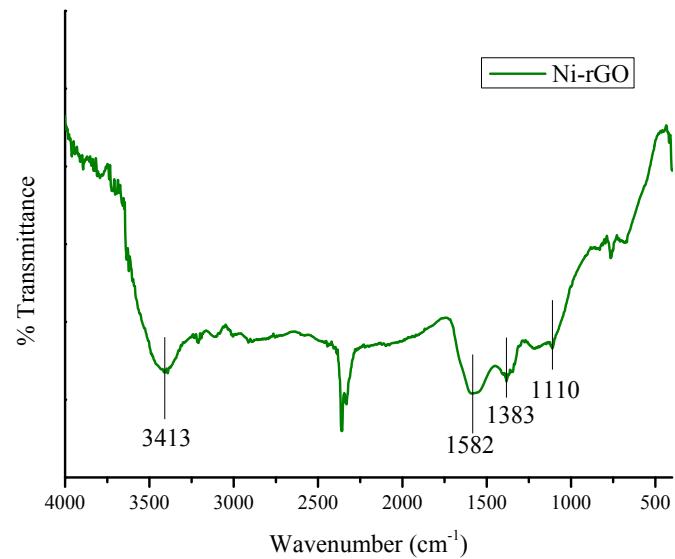


Fig. S1 FT–IR spectrum of Ni–rGO nanocomposite.

SI-2.2. FT–IR spectrum of Ni–zeolite

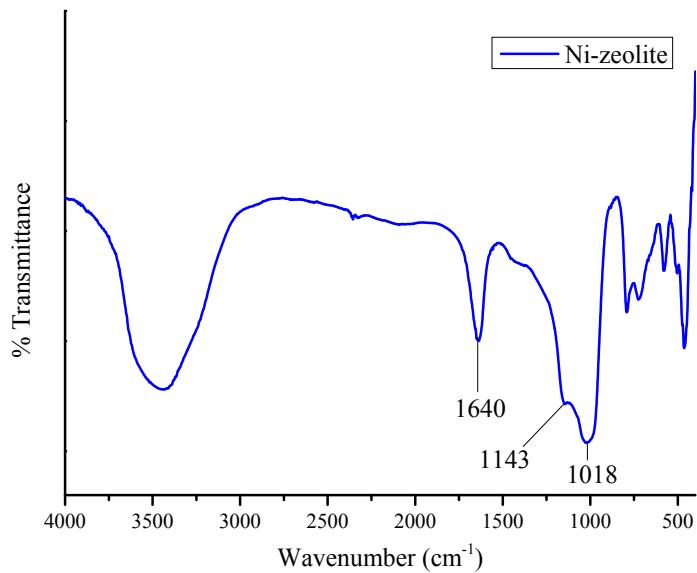


Fig. S2 FT–IR spectrum of Ni–zeolite nanocomposite.

SI-3. Characterization of Ni-rGO–zeolite nanocomposite fresh and after reaction

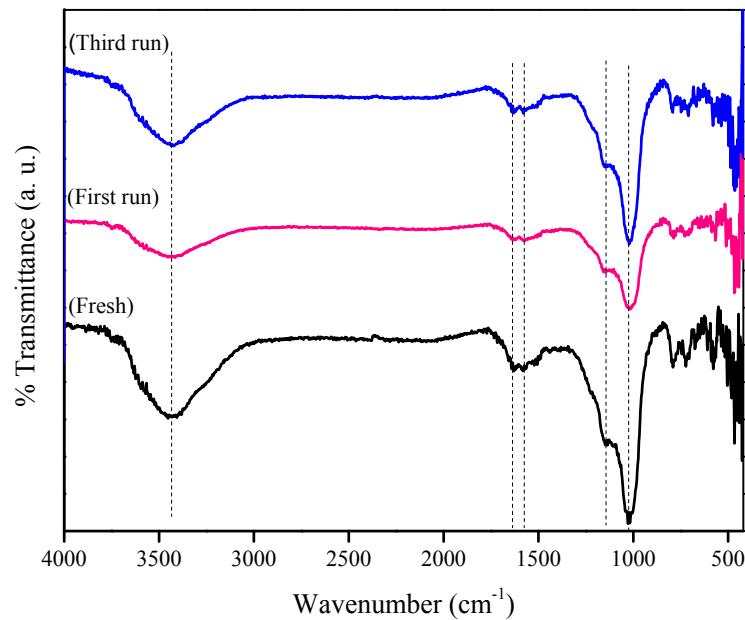


Fig. S3 FT-IR spectra of Ni-rGO–zeolite nanocomposite of fresh, after first and after third run.

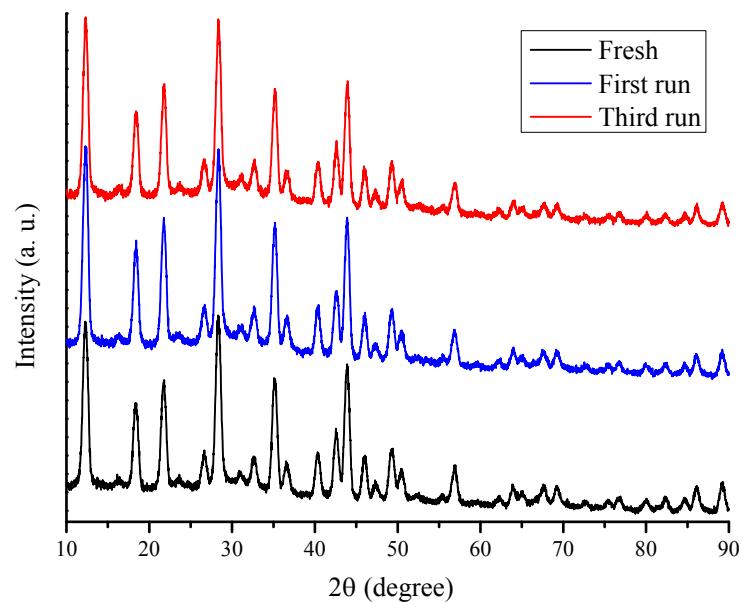


Fig. S4 XRD patterns of Ni-rGO–zeolite nanocomposite of fresh, after first and after third run.

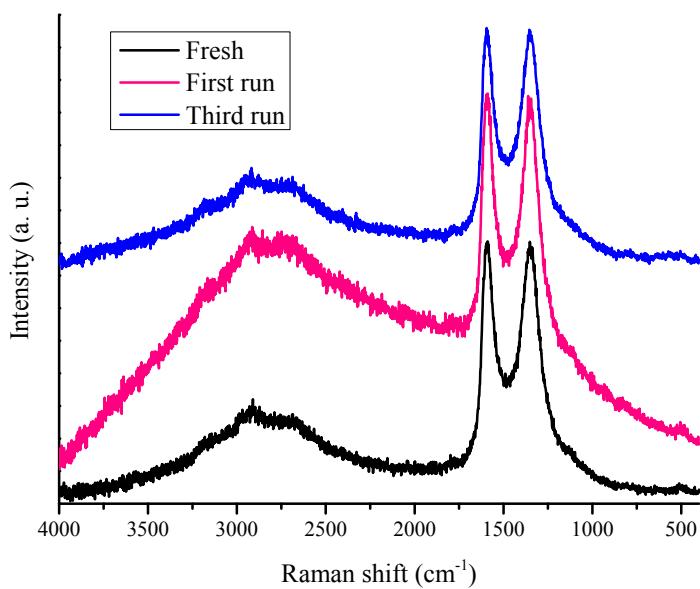
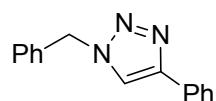


Fig. S5 Raman spectra of Ni–rGO–zeolite nanocomposite of fresh, after first and after third run.

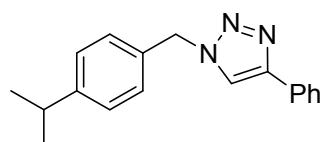
SI-4. Characterization data of various 1,2,3-triazole derivatives listed in Table 2

1-Benzyl-4-phenyl-1*H*-1,2,3-triazole (**3a**)¹



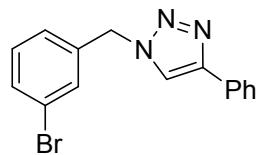
White crystalline solid; m.p.: 128–130 °C (Lit. m.p.: 128–129 °C)¹; **1H NMR (400 MHz, CDCl₃)**: δ 5.53 (s, 2H), 7.29–7.37 (m, 8H), 7.67 (s, 1H), 7.78–7.79 (m, 2H); **13C NMR (100 MHz, CDCl₃)**: δ 54.2, 119.7, 125.7, 128.0, 128.2, 128.8, 128.9, 129.2, 130.5, 134.7, 148.2.

1-(4-Isopropylbenzyl)-4-phenyl-1*H*-1,2,3-triazole (**3b**)²



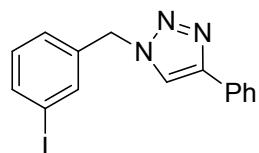
White solid; m.p.: 177–178 °C (Lit. m.p.: 175 °C)²; **1H NMR (400 MHz, CDCl₃)**: δ 1.20–1.58 (m, 6H), 2.97–2.99 (m, 1H), 5.52 (s, 2H), 7.00–7.15 (m, 1H), 7.23–7.27 (m, 2H), 7.38–7.42 (m, 2H), 7.65 (m, 3H), 7.73–7.82 (m, 2H); **13C NMR (100 MHz, CDCl₃)**: δ 23.9, 26.2, 33.9, 54.1, 119.4, 125.7, 126.9, 127.29, 128.1, 128.8, 128.9, 129.4, 131.9, 133.1.

1-(3-Bromo benzyl)-4-phenyl-1*H*-1,2,3-triazole (3c)³



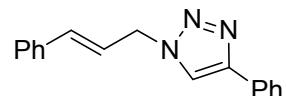
Light yellow solid; m.p.: 92–94 °C (Lit. m.p.: 91–93 °C)³; **1H NMR (400 MHz, CDCl₃)**: δ 5.53 (s, 2H), 7.22–7.25 (m, 2H), 7.32 (d, *J* = 7.2 Hz, 1H), 7.38–7.49 (m, 4H), 7.69 (s, 1H), 7.79–7.81 (m, 2H); **13C NMR (100 MHz, CDCl₃)**: δ 53.5, 119.6, 123.1, 125.7, 126.6, 128.3, 128.9, 130.3, 130.7, 131.0, 132.0, 136.9, 148.5.

1-(3-Iodo benzyl)-4-phenyl-1*H*-1,2,3-triazole (3d)⁴



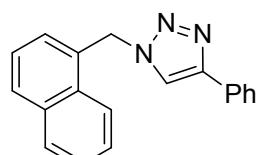
White solid; m.p.: 120–122 °C (Lit. m.p.: 122–124 °C)⁴; **1H NMR (400 MHz, CDCl₃)**: δ 5.50 (s, 2H), 7.10–7.12 (m, 1H), 7.24 (d, *J* = 8 Hz, 2H), 7.32 (d, *J* = 7.2 Hz, 1H), 7.40 (t, *J* = 7.2 Hz, 2H), 7.66–7.70 (m, 3H), 7.79–7.81 (m, 2H); **13C NMR (100 MHz, CDCl₃)**: δ 53.3, 94.8, 119.6, 125.8, 127.2, 128.3, 128.9, 130.4, 130.8, 136.8, 136.9, 137.9, 148.4.

1-Cinnamyl-4-phenyl-1*H*-1,2,3-triazole (3e)⁵



White solid; m.p.: 136–138 °C (Lit. m.p.: 132–134 °C)⁵; **1H NMR (400 MHz, CDCl₃)**: δ 5.15 (dd, *J* = 7.6, 1.2 Hz, 2H), 6.35–6.41 (m, 1H), 6.69 (d, *J* = 16 Hz, 1H), 7.29–7.43 (m, 8H), 7.80–7.84 (m, 3H); **13C NMR (100 MHz, CDCl₃)**: δ 52.4, 119.5, 121.9, 125.7, 126.8, 128.2, 128.6, 128.8, 128.9, 130.6, 135.4, 135.5, 148.1.

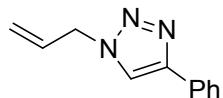
1-(Naphthalen-1-ylmethyl)-4-phenyl-1*H*-1,2,3-triazole (3f)⁶



White solid; m.p.: 133–134 °C (Lit. m.p.: 135 °C)⁶; **1H NMR (400 MHz, CDCl₃)**: δ 5.99 (s, 2H), 7.26–7.34 (m, 3H), 7.46–7.72 (m, 5H), 7.90 (s, 2H), 7.99 (s, 2H), 8.0 (s, 1H); **13C NMR**

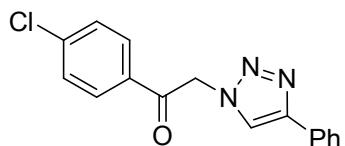
(**100 MHz, CDCl₃**): δ 52.4, 119.6, 122.9, 125.4, 125.7, 126.5, 127.4, 127.9, 128.1, 128.8, 129.0, 129.9, 130.1, 130.5, 131.2, 134.0, 148.0.

1–Allyl–4–phenyl–1*H*–1,2,3–triazole (3g)¹



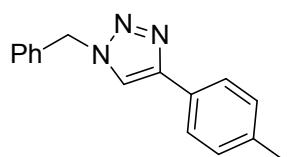
White crystalline solid; m.p.: 62–64 °C (Lit. m.p.: 57–58 °C)¹; **¹H NMR (400 MHz, CDCl₃)**: δ 4.96 (d, *J* = 6 Hz, 2H), 5.26–5.34 (m, 2H), 5.99–6.01 (m, 1H), 7.29–7.31 (m, 1H), 7.36–7.40 (m, 2H), 7.44–7.80 (m, 3H); **¹³C NMR (100 MHz, CDCl₃)**: δ 52.7, 119.6, 120.1, 125.7, 128.1, 128.8, 130.6, 131.3, 147.9.

1–(4–Chlorophenyl)–2–(4–phenyl–1*H*–1,2,3–triazol–1–yl)ethanone (3h)⁷



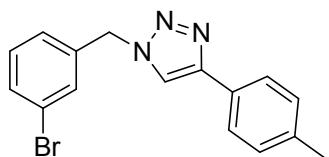
White solid; m.p.: 104–106 °C (Lit. m.p.: 106–109 °C)⁷; **¹H NMR (400 MHz, DMSO-d₆)**: δ 6.24 (s, 2H), 7.33 (s, 1H), 7.44 (s, 2H), 7.68–7.69 (m, 2H), 7.85 (s, 2H), 8.08–8.09 (m, 2H), 8.50 (s, 1H); **¹³C NMR (100 MHz, DMSO-d₆)**: δ 56.5, 123.5, 125.6, 128.4, 129.5, 129.6, 130.6, 131.2, 133.3, 139.7, 146.8, 191.8.

1–Benzyl–4–(p–tolyl)–1*H*–1,2,3–triazole (3i)¹



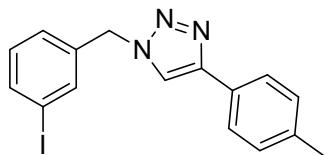
White crystalline solid; m.p.: 152–154 °C (Lit. m.p.: 154–155 °C)¹; **¹H NMR (400 MHz, CDCl₃)**: δ 2.35 (s, 3H), 5.52 (s, 2H), 7.19 (d, *J* = 8 Hz, 2H), 7.27–7.36 (m, 5H), 7.62 (s, 1H), 7.68 (d, *J* = 8 Hz, 2H); **¹³C NMR (100 MHz, CDCl₃)**: δ 21.3, 54.1, 119.3, 125.6, 127.7, 128.0, 128.7, 129.1, 129.5, 134.8, 138.09, 148.3.

1–(3–Bromo benzyl)–4–(p–tolyl)–1*H*–1,2,3–triazole (3j)⁶



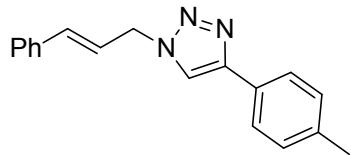
Light yellow solid; m.p.: 132–134 °C (Lit. m.p.: 132–134 °C)⁶; **1H NMR (400 MHz, CDCl₃)**: δ 2.37 (s, 3H), 5.53 (s, 2H), 7.23–7.26 (m, 5H), 7.46–7.49 (m, 2H), 7.65–7.70 (m, 3H); **13C NMR (100 MHz, CDCl₃)**: δ 21.3, 53.4, 119.1, 123.1, 125.7, 126.5, 127.5, 129.5, 130.7, 131.0, 131.9, 137.0, 138.2, 148.6.

1-(3-Iodo benzyl)-4-(p-tolyl)-1H-1,2,3-triazole (3k)⁶



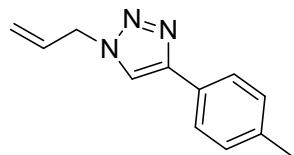
White solid; m.p.: 130–132 °C (Lit. m.p.: 130 °C)⁶; **1H NMR (400 MHz, CDCl₃)**: δ 2.37 (s, 3H), 5.50 (s, 2H), 7.1 (m, 1H), 7.20–7.26 (m, 3H), 7.64–7.71 (m, 5H); **13C NMR (100 MHz, CDCl₃)**: δ 21.3, 53.3, 94.8, 119.1, 125.7, 127.2, 127.6, 129.5, 130.8, 136.9, 137.0, 137.9, 138.2, 148.5.

1-Cinnamyl-4-(p-tolyl)-1H-1,2,3-triazole (3l)⁶



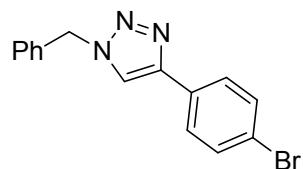
White solid; m.p.: 145–147 °C (Lit. m.p.: 145–147 °C)⁶; **1H NMR (400 MHz, CDCl₃)**: δ 2.33 (s, 3H), 5.13 (dd, *J* = 8, 1.2 Hz, 2H), 6.34–6.39 (m, 1H), 6.68 (d, *J* = 16 Hz, 1H), 7.21 (d, *J* = 8 Hz, 2H), 7.25–7.40 (m, 5H), 7.71 (d, *J* = 8 Hz, 2H), 7.76 (s, 1H); **13C NMR (100 MHz, CDCl₃)**: δ 21.3, 52.4, 119.1, 122.0, 125.6, 126.7, 127.8, 128.6, 128.8, 129.5, 135.3, 135.5, 138.0, 148.2.

1-Allyl-4-(p-tolyl)-1H-1,2,3-triazole (3m)⁶



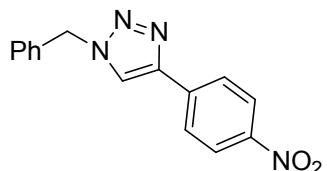
Pale yellow solid; m.p.: 88–90 °C (Lit. m.p.: 88–90 °C)⁶; **1H NMR (400 MHz, CDCl₃)**: δ 2.34 (s, 3H), 4.95 (d, *J* = 6 Hz, 2H), 5.27–5.34 (m, 2H), 5.97–6.01 (m, 1H), 7.19 (d, *J* = 8 Hz, 2H), 7.69 (d, *J* = 9.2 Hz, 3H); **13C NMR (100 MHz, CDCl₃)**: δ 21.3, 52.7, 119.2, 120.1, 125.6, 127.8, 129.5, 131.4, 138.0, 148.1.

1-Benzyl-4-(4-bromophenyl)-1*H*-1,2,3-triazole (3n)¹



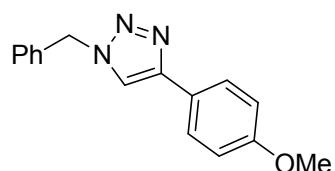
White solid; m.p.: 142–143 °C (Lit. m.p.: 144 °C)¹; **1H NMR (400 MHz, CDCl₃)**: δ 5.54 (s, 2H), 7.29 (s, 2H), 7.36 (s, 3H), 7.49 (d, *J* = 1.6 Hz, 2H), 7.64 (d, *J* = 8 Hz, 3H); **13C NMR (100 MHz, CDCl₃)**: δ 54.3, 119.7, 122.0, 127.2, 128.1, 128.9, 129.2, 129.5, 131.9, 134.5, 147.1.

1-Benzyl-4-(4-nitrophenyl)-1*H*-1,2,3-triazole (3o)⁸



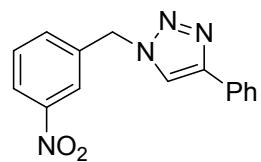
Yellow solid; m.p.: 166–168 °C (Lit. m.p.: 171–172 °C)⁸; **1H NMR (400 MHz, DMSO-d₆)**: δ 5.66 (s, 2H), 7.34 (d, *J* = 6.8 Hz, 5H), 8.09–8.10 (m, 2H), 8.26–8.28 (m, 2H), 8.88 (s, 1H); **13C NMR (100 MHz, DMSO-d₆)**: δ 53.7, 124.1, 124.8, 126.4, 128.5, 128.7, 129.3, 136.1, 137.5, 145.2, 147.0.

1-Benzyl-4-(4-methoxyphenyl)-1*H*-1,2,3-triazole (3p)¹



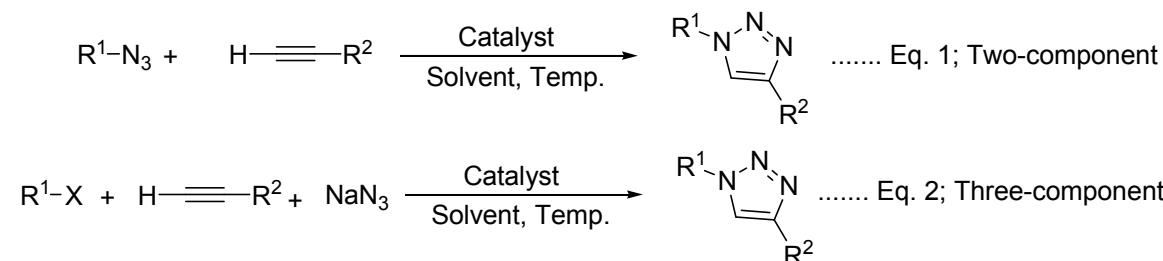
White solid; m.p.: 139–140 °C (Lit. m.p.: 139–141 °C)¹; **1H NMR (400 MHz, CDCl₃)**: δ 3.79 (s, 1H), 5.51 (s, 2H), 6.90 (dd, *J* = 7.2, 2 Hz, 2H), 7.25–7.28 (m, 2H), 7.32–7.35 (m, 3H), 7.58 (s, 1H), 7.70 (dd, *J* = 6.8, 1.6 Hz, 2H); **13C NMR (100 MHz, CDCl₃)**: δ 54.1, 55.3, 114.2, 118.8, 123.3, 127.0, 128.0, 128.7, 129.1, 134.8, 148.1, 159.6.

1-(3-Nitrobenzyl)-4-phenyl-1H-1,2,3-triazole (3q)⁹



Yellow solid; m.p.: 150–152 °C (Lit. m.p.: 150–153 °C)⁹; **1H NMR (400 MHz, CDCl₃)**: δ 5.66 (s, 2H), 7.31–7.33 (m, 1H), 7.37–7.41 (m, 2H), 7.52–7.56 (m, 1H), 7.60 (d, *J* = 7.6 Hz, 1H), 7.78–7.98 (m, 3H), 8.17–8.19 (m, 2H); **13C NMR (100 MHz, CDCl₃)**: δ 53.2, 119.8, 122.8, 123.7, 125.7, 128.5, 128.9, 130.1, 130.4, 133.9, 136.8, 148.5, 148.6.

Table S1 Comparison of catalytic performances of various Ni, Cu and other transition metal catalysts in AAC

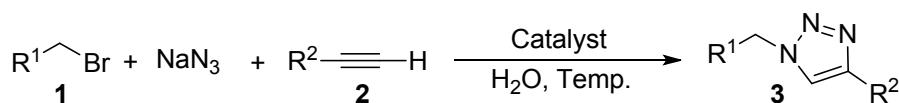


Entry	Catalyst	Reaction conditions	Merits	Limitations	Yield (%)	Ref
1	Raney Ni, Catalyst load: 20 mol%	Toluene, 45 °C, 14 h, N ₂ (Two-component)	Mechanism proposed on the basis of isotope labelling experiment	Mixture of regioisomers, limited substrate scope, instability of Raney Ni	89–93	10
2	Cp ₂ Ni–Xantphos, Catalyst load: 10 mol%	Cs ₂ CO ₃ , H ₂ O, r.t., 1.5–17 h, (Two-component)	Reaction at r.t. & on-water	Mixture of regioisomers, Requires base and ligand Xantphos	42–95	11
3	Ni–TLOP, Catalyst load: 5 mg per mmol (Ni content not given)	hv, 4M NaCl, r.t., 2–5 h, (Two-component)	Regioselective and high yield of triazoles	Catalyst preparation requires expensive chemicals and catalyst becomes active only under visible light irradiation	95.5–99.9	12
4	Ni–rGO–zeolite, Catalyst load: ≤ 2.6 mol%	H ₂ O, 70–90 °C, 4–6 h, (Three-component)	Easy preparation from cheap chemicals, robust, high efficiency on-water reaction, regioselective synthesis and recyclable	Microstructures of the heterogeneous Ni catalyst not unequivocally established, though major information obtained	77–94	This work

5	Cu ₂ O@ARF Catalyst load: 25 mg per mmol	H ₂ O, SDS, r.t., 1–8 h, (Three–component)	Heterogeneous polymeric Cu catalyst, on–water reaction, multi–component approach	Requirement of phase transfer catalyst (SDS)	68–92	6
6	Cu(II) poly–5– vinyltetrazolate Catalyst load: 0.5 mol%	H ₂ O, r.t., 0.5–6 h, (Three–component)	Heterogeneous polymeric Cu catalyst, on–water reaction, catalyst recyclable for 8 runs with 10% drop in conversion	Limited substrate scope examined	64–98	13
7	Cu@β–CD–PEG– mesoGO Catalyst load: 5 mol%	H ₂ O, r.t., 1–2 h, (Three–component)	On–water reaction at ambient temperature, catalyst recyclable for 10 runs	Catalyst preparation requires several steps	82–91	14
8	[Rh(CO) ₂ Cl] ₂ Catalyst load: 2 mol%	MeCN, r.t., 0.5–24 h, (Two–component)	Reaction at r.t. to 100 °C, Reaction mechanism proposed on the basis of control experiment and DFT studies	Expensive catalyst and not recyclable	22–98	15
9	Metalloprotein inspired Ru– polymeric complex Catalyst load: 0.4 mg per mmol	H ₂ O, r.t., 0.16–3 h, (Both two–component & Three–component)	On–water, Gram scale synthesis, Recyclability checked for 6 runs	Two steps catalyst preparation, expensive chemicals and metal salt	82–98	16
10	[Ir(cod)Cl] ₂ Catalyst load: 2 mol%	CH ₂ Cl ₂ , r.t., 8 h, (Two–component)	Synthesis of bioactive scaffold, biocompatible synthesis explored	Expensive catalyst, DCM solvent, catalyst not recyclable	61–95	17

11	Ag-Al ₂ O ₃ @Fe ₂ O ₃ Catalyst load: 20 mg per mmol	Conc. HCl:H ₂ O (1:1), r.t., 6 h, (Three-component)	Reaction at r.t., Recyclability checked for 5 runs	Limited substrate scope	86–96	18
12	Nanoporous TiO ₂ supported Au NPs Catalyst load: 20 mg per mmol	CH ₂ Cl ₂ , ~100 °C., 0.5 h, (Two-component)	Reaction at r.t. and on-water	Two-component approach, catalyst not recyclable	86–97	19
13	Zn(OAc) ₂ Catalyst load: 10 mol%	Ascorbic acid, H ₂ O, MW (75–130 °C, 80–150 W), 6–9 h, (Two-component)	MW-assisted and on-water reaction	Requirement of reducing agent, catalyst not recyclable	47–97	20
14	Sm[N(SiMe ₃) ₂] ₃ Catalyst load: 5 mol%	BuNH ₂ , toluene, 50 °C, 24 h, (Two-component)	Mechanism proposed on the basis of trapping experiment	Requirement of base, catalyst not recyclable	30–97	21

Table S2 Comparative catalytic performance between Cu₂O@ARF⁶ and Ni–rGO–zeolite



Entry	R ¹	R ²	Cu ₂ O@ARF ^a	Ni–rGO–zeolite ^b
1	C ₆ H ₅	C ₆ H ₅	92%	94%
2	1-Naphthyl	C ₆ H ₅	79%	85%
3	CH ₂ =CH	4-MeC ₆ H ₄	82%	94%
4	3-IC ₆ H ₄	C ₆ H ₅	76%	91%
5	3-BrC ₆ H ₄	C ₆ H ₄	78%	90%

Reaction conditions: ^a**1** (1 mmol), **2** (1.2 mmol), NaN₃ (1.5 mmol), Cu₂O@ARF (25 mg), sodium dodecyl sulfate (0.1 mmol) and H₂O were stirred at r.t. for 1–8 h. ^b**1** (1 mmol), **2** (1 mmol), NaN₃ (1.5 mmol), Ni–rGO–zeolite (30 mg) and H₂O (2 mL) were stirred at 70–90 °C for 4–6 h.

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SI-5. Scanned copies of ^1H and ^{13}C NMR spectra of various 1,2,3-triazole derivatives

