Electronic Supplementary Information for

A class of polytriazole metallogels *via* CuAAC polymerization: preparation and properties

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Preparation of metallogels

Synthesis of Gel1. To a solution of A1 (0.756 g, 6.0 mmol azide) and dipropargyl bisphenol A (0.912 g, 6.0 mmol alkyne) in DMF (7.1184 g), Cu(PPh₃)₃Br (0.1116 g, 0.12 mmol) was added. The resulting mixture was stirred at 70 °C until a dark brown gel, named as Gel1, was formed. The fresh Gel1 was immersed in distilled water for 5 d to remove DMF and then dehydrated by freeze-drying for 2 d to obtain brown xerogels, designated as Xgel1. HCl and EDTA aqueous solutions were used to remove the copper ions from the fresh gel to obtain pale beige polytriazoles, called as PTA1. IR (KBr, cm⁻¹) v: 3141 (C-H, triazloe ring), 1235 (C-O).

Synthesis of Gel2. To a solution of A2 (0.900 g, 6.0 mmol azide) and dipropargyl bisphenol A (0.912 g, 6.0 mmol alkyne) in DMF (7.6944 g), Cu(PPh₃)₃Br (0.1116 g, 0.12 mmol) was added. The resulting mixture was stirred at 70 °C until a dark brown gel, named as Gel2, was formed. The fresh Gel2 was immersed in distilled water for 5 d to remove DMF and then dehydrated by freeze-drying for 2 d to obtain dark green xerogels, designated as Xgel2. HCl and EDTA aqueous solutions were used to remove the copper ions from the fresh gel to obtain beige polytriazoles, called as PTA2. IR (KBr, cm⁻¹) v: 3145 (C-H, triazloe ring), 1155 (S=O), 1235 (C-O).

Synthesis of Gel3. To a solution of A3 (0.564 g, 6.0 mmol azide) and dipropargyl bisphenol A (0.912 g, 6.0 mmol alkyne) in DMF (6.3504 g), Cu(PPh₃)₃Br (0.1116 g, 0.12 mmol) was added. The resulting mixture was stirred at 70 °C until a dark brown gel, named as Gel3, was formed. The fresh Gel3 was immersed in distilled water for 5 d to remove DMF and then dehydrated by freeze-drying for 2 d to obtain yellow-brown xerogels, designated as Xgel3. HCl and EDTA aqueous solutions were used to remove the copper ions from the fresh gel to obtain light yellow polytriazoles, called as PTA3. IR (KBr, cm⁻¹) v: 3136 (C-H, triazloe ring), 1230 (C-O).

Synthesis of Gel4. To a solution of A4 (0.792 g, 6.0 mmol azide) and dipropargyl bisphenol A (0.912 g, 6.0 mmol alkyne) in DMF (7.2624 g), Cu(PPh₃)₃Br (0.1116 g, 0.12 mmol) was added. The resulting mixture was stirred at 70 °C until a dark brown

gel, named as Gel4, was formed. The fresh Gel4 was immersed in distilled water for 5 d to remove DMF and then dehydrated by freeze-drying for 2 d to obtain yellow-brown xerogels, designated as Xgel4. HCl and EDTA aqueous solutions were used to remove the copper ions from the fresh gel to obtain light yellow polytriazoles, called as PTA4. IR (KBr, cm⁻¹) v: 3136 (C-H, triazloe ring), 1230 (C-O).



Fig. S1 Self-sustaining nature of as-synthesized Gel1. (a) The Gel1 was tailored to disk shape. The disk (b) before (c) during and (d) after keeping a 25 mL water filled beaker atop of the disk for several hours showing negligible shrinking/compression of the gel matrix.



Fig. S2 Variation of G' and G'' of Gel1, Gel2, Gel3 and Gel4 against temperature. Solid concentration: 20 wt%. Solvent: DMF. Scanning frequency: 1 Hz. Strain: 10 %.





Fig. S3 ¹H-NMR spectra of PTAs (PTA1, PTA3 and PTA4) before and after the addition of 0.5×10^{-2} mmol mL⁻¹ of Cu⁺ ion in DMSO-*d*₆

	Proton on triazole (ppm)				
	PTA1	PTA2	PTA3	PTA4	
Blank	8.92	9.08	8.24	8.29	
After adding Cu ⁺	8.94	9.13	8.41	8.46	

Table S1 Chemical shift of proton on triazole of four PTAs before and after the addition of 0.5×10^{-2} mmol mL⁻¹ of Cu⁺ ion in DMSO-*d*₆.

Table S2 Diffraction angel (2 θ) of pristine PTAs and new diffraction shoulder peaks (2 θ) appeared in the PTA xerogels (Xgels).

Code	PTA1/Xgel1	PTA2/Xgel2	PTA3/Xgel3	PTA4/Xgel4
2θ (°)	19.25	19.02	19.78	20.01
2θ'(°)	21.78	21.93	21.55	23.57