

Electronic Supplementary Information

Morphological transformation between three-dimensional gel network and spherical vesicles via sonication

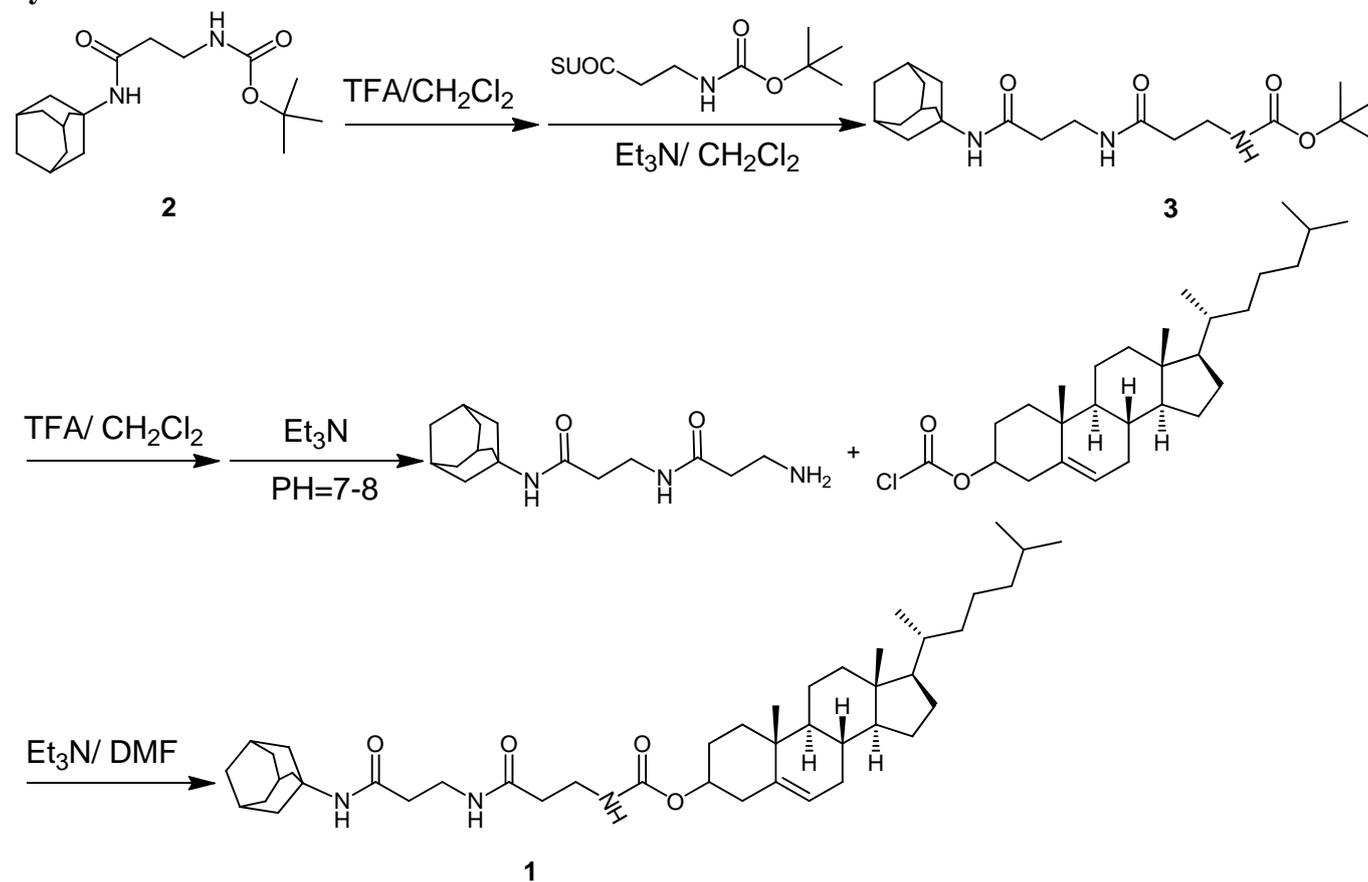
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Experimental details

Synthetic methods and characterizations:



Scheme S1 Synthesis of compound **1**

Compound **2** was synthesized according to our previous report [1]

Preparation of compound **3**

Trifluoroacetic acid (TFA, 8.2 mL) was added to a solution of **2** (2 g, 6 mmol) in CH_2Cl_2 (9.6 mL). The reaction mixture was stirred at room temperature for 2 h, and the solvent was evaporated in vacuum at 40°C . CH_2Cl_2 (3 mL), TEA (3 mL, 22 mmol) and Boc- β -Ala Osu (1.72 g, 6 mmol) were added. The reaction mixture was stirred overnight at room temperature and then evaporated to dryness. The solid was purified by column chromatography

(SiO₂, ethyl acetate/ petroleum ether = 1: 1) to give **3** as a white solid (2.1 g, 86%). Mp 183-186°C. ¹H NMR (400 MHz, CDCl₃) δ 6.56 (s, 1H), 5.35 (s, 1H), 5.23 (s, 1H) 3.49-3.45 (m, 2H), 3.40-3.36 (m, 2H), 2.37-2.34 (t, *J* = 6.0 Hz, 2H), 2.32-2.29 (t, *J* = 6.0 Hz, 2H), 2.06 (s, 3H), 1.97 (s, 6H), 1.66 (s, 6H), 1.42 (s, 9H).

Preparation of compound 1

Trifluoroacetic acid (TFA, 2.3 mL) was added to a solution of **3** (0.68 g, 1.73 mmol) in CH₂Cl₂ (2.7 mL). The reaction mixture was stirred at room temperature for 2 h, and the solvent was evaporated in vacuum at 40°C. CH₂Cl₂ (15 mL) was added and the solution was neutralized with TEA to pH 7–8. Then, a dichloromethane solution (10 mL) of cholesteryl chloroformate (0.78 g, 1.73 mmol) was added drop wise over 1.5 h at 0°C. The mixture was stirred for 12 h at room temperature and evaporated to dryness. The solid was purified by column chromatography (SiO₂, ethyl acetate) to give **1** as a white solid (0.8 g, 66%). Mp 220-223°C, ¹H NMR (400 MHz, CDCl₃) δ 6.50 (s, 1H), 5.36 (s, 1H), 5.30 (s, 2H), 4.47 (m, 1H), 3.49 (m, 2H), 3.44 (m, 2H), 2.38 (m, 2H), 2.33 (m, 2H), 2.08 (s, 3H), 1.98 (m, 6H), 1.85 (m, 4H), 1.68 (m, 6H), 1.66 – 0.85 (m, 33H), 0.67(s, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 171.7, 170.7, 156.2, 139.9, 125.6, 122.43, 77.3, 77.0, 76.8, 74.9, 74.3, 56.7, 56.2, 50.0, 42.4, 41.7, 39.8, 39.5, 38.6, 37.0, 36.3, 36.2, 35.8, 35.6, 31.93, 29.4, 28.2, 28.0, 24.3, 23.9, 22.8, 22.6, 21.0, 19.3, 18.7, 11.9; HR-Ms calculated for C₄₄H₇₁N₃NaO₄ [M+Na]⁺: 728.5342; found: 728.5325.

Table S1 Gelation property of compound **1**

Solvent	State (T-gel/ S-gel) ^a	<i>T_g</i> /°C (T-gel/ S-gel)	Stable Period
petroleum ether	I		
methylene chloride	S		
ethanol	G (37.5/ 14.2)	50.0/ 40.0	>1 month
ethyl acetate	P		
dioxane	P		
acetone	I		
acetonitrile	I		
methanol	P	83.0/ 80.0	>1 month
water	I		
1-hexanol	S		
1-pentanol	S		
chloroform	S		
DMF ^b	G (50)	53.0	>1 month
THF	I		
DMSO ^b	G (50)	69.0	>1 month
isopropanol ^b	G (50)	72.0	>1 month
cyclohexane	I		

^aState: G = gel; P = Precipitation; S = Solution; I = Insoluble. The critical gelation concentrations of T-gel/ S-gel were included in the parentheses (wt%). *T_g*: gel to sol transition temperature. ^b T-gel only.

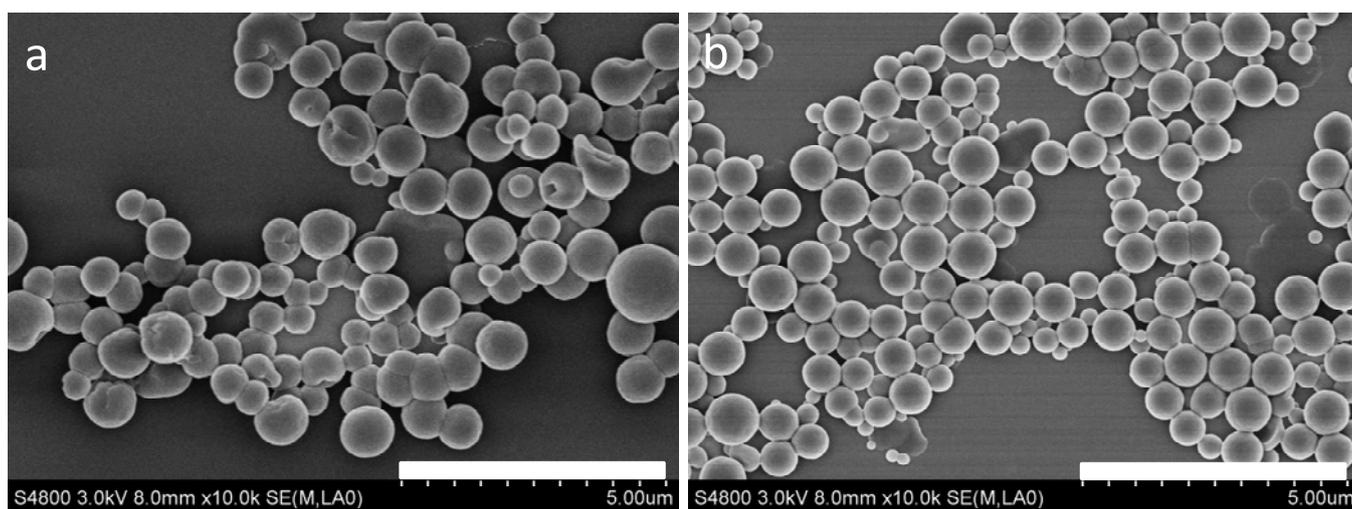


Fig. S1 SEM images of **1** from ethanol after a heating-cooling process at a concentration of (a) 10 mg/mL and (b) 30 mg/mL

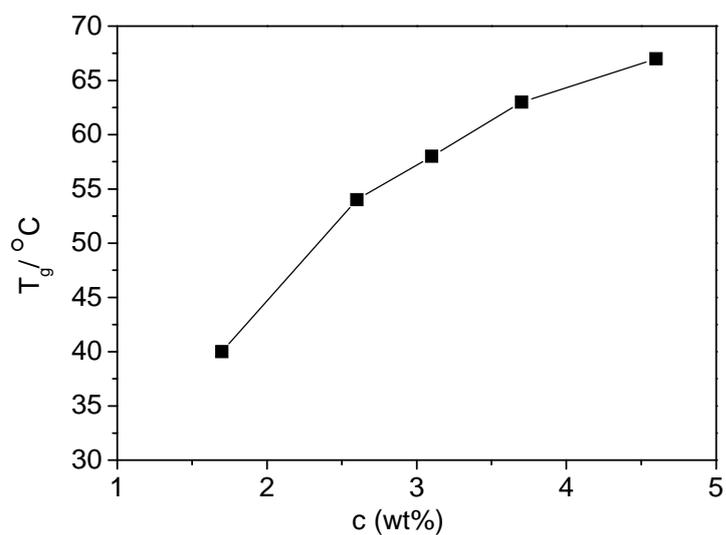


Fig. S2 The concentration dependence of T_g in S-gel of **1** in ethanol.

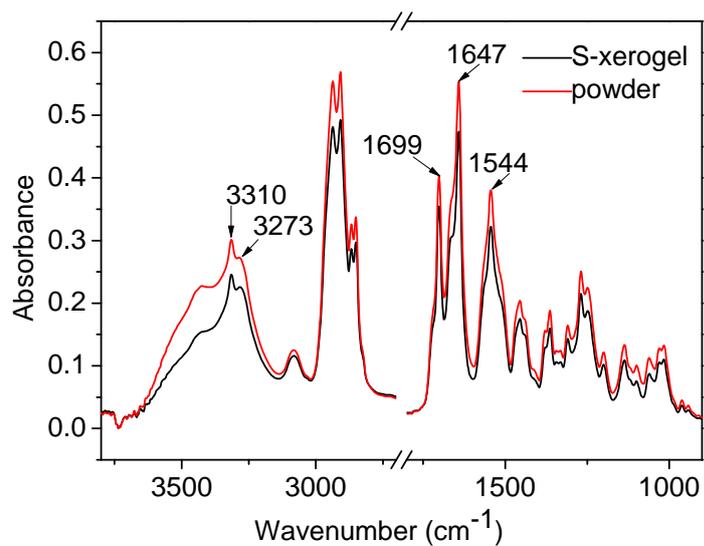


Fig. S3 IR spectra of the powder from heating-cooling process and the S-xerogel (20 mg/mL).

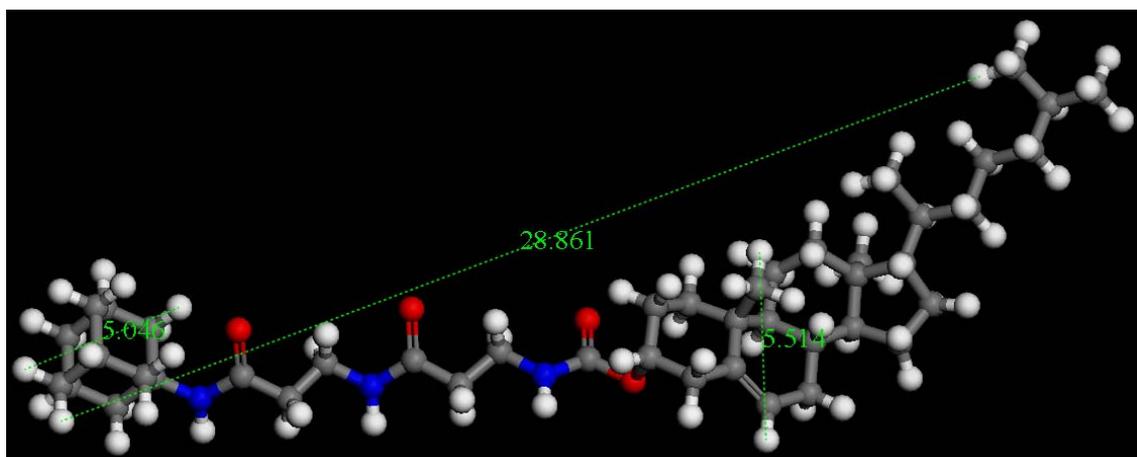


Fig. S4 The extended structure model of molecule 1

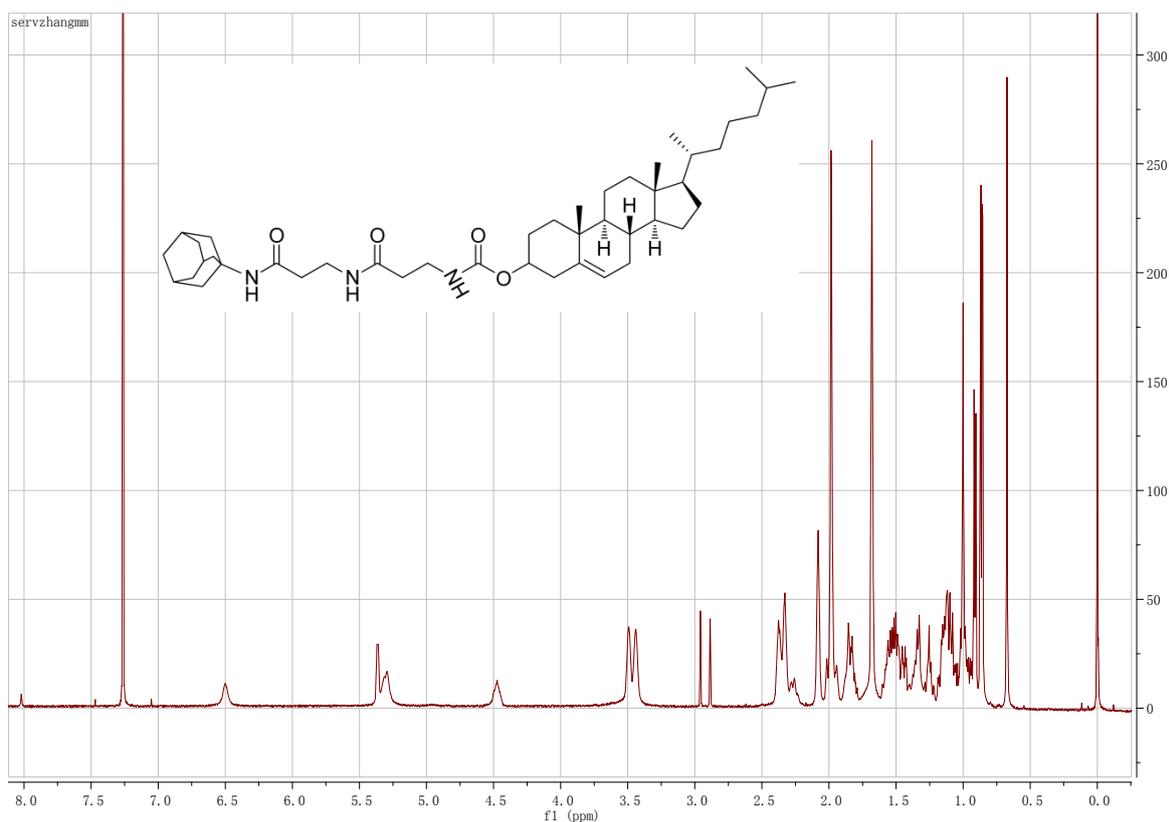


Fig. S5 ^1H NMR spectral of **1** in CDCl_3

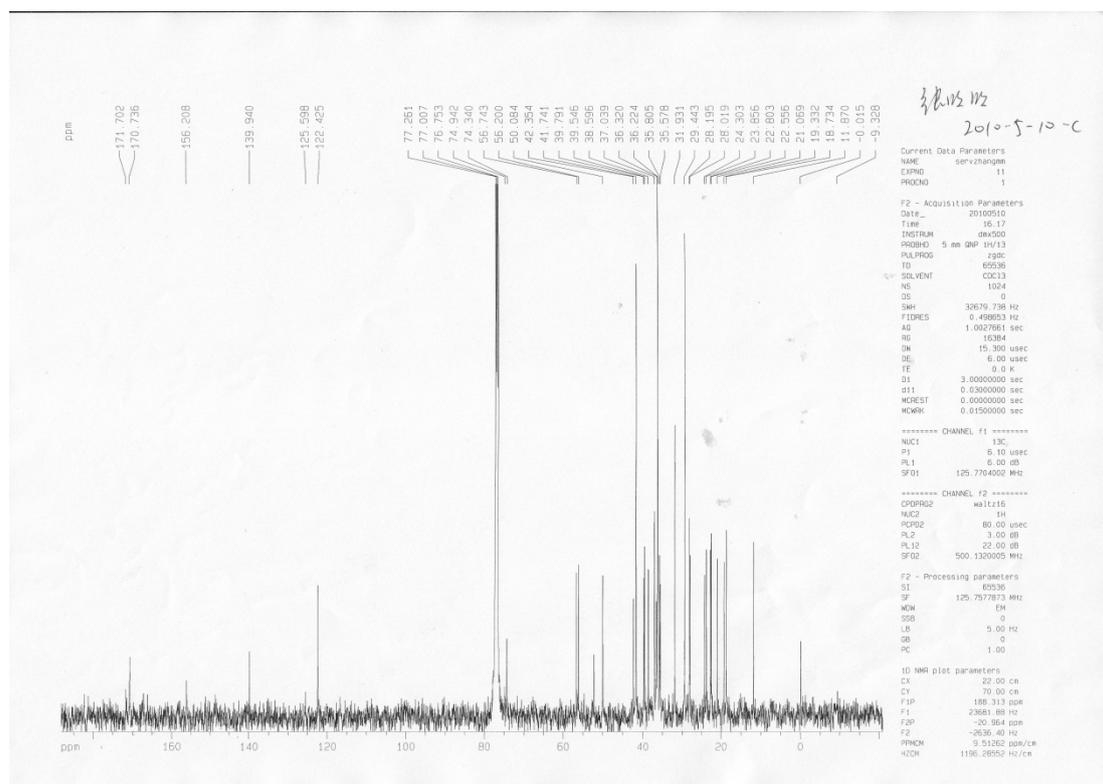
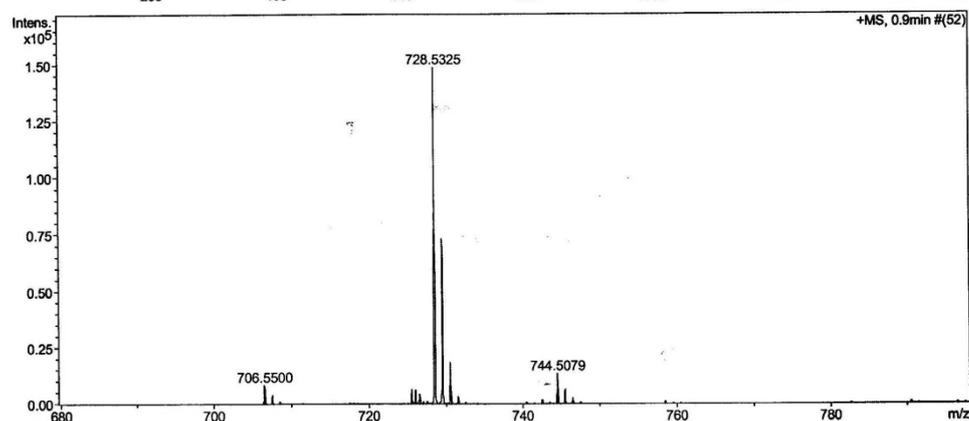
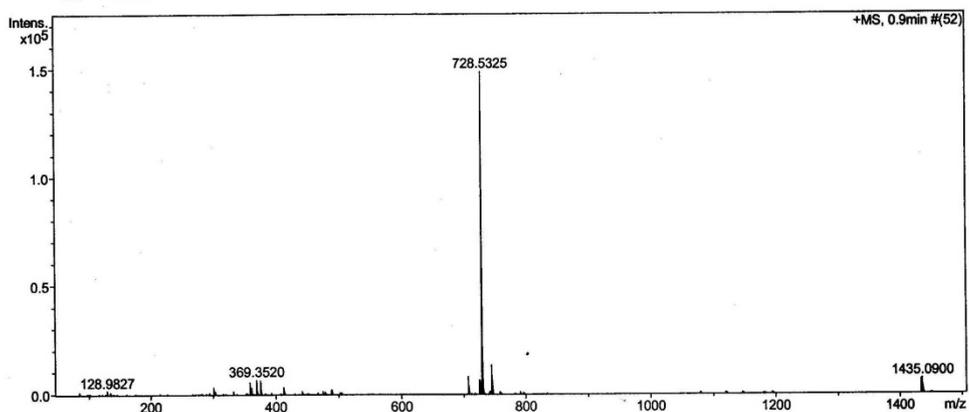


Fig. S6 ^{13}C NMR spectral of **1** in CDCl_3

Display Report

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Sample Name	aim	Comment	

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Focus	Not active	Set Dry Gas	4.0 l/min		
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Scan End	1500 m/z				



Bruker Compass DataAnalysis 4.0

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Fig. S7 HR-MS spectral of **1**

Reference:

[1] M. Zhang, S. Sun, X. Cao, X. Yu, Y. Zou and T. Yi, *Chem. Commun.*, 2010, **46**, 3553.