Supporting Information

Preparation and UCST-type Phase Behavior of Glycopolypeptide in Alcoholic

Solvents

Xi Wang, Chenglong Ge, Ying Ling,* and Haoyu Tang*

Key Laboratory of Polymeric Materials and Application Technology of Hunan Province, Key Laboratory of Advanced Functional Polymer Materials of Colleges and Universities of Hunan Province, College of Chemistry, Xiangtan University, Xiangtan, Hunan, 411105, China

Correspondence to: Haoyu Tang (Email: <u>htang@xtu.edu.cn</u>) and Ying Ling (Email: <u>yingling0202@gmail.com</u>)

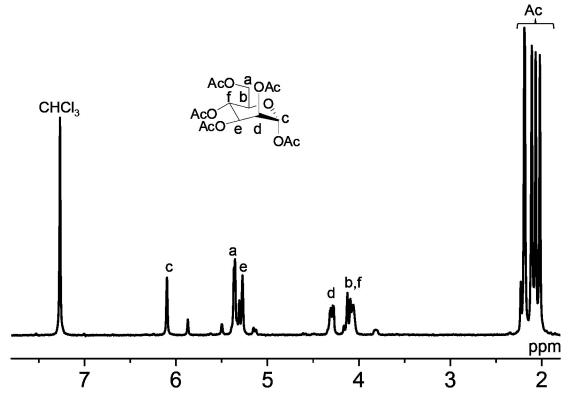


Figure S1. ¹H NMR spectrum of 2 in CDCl₃.

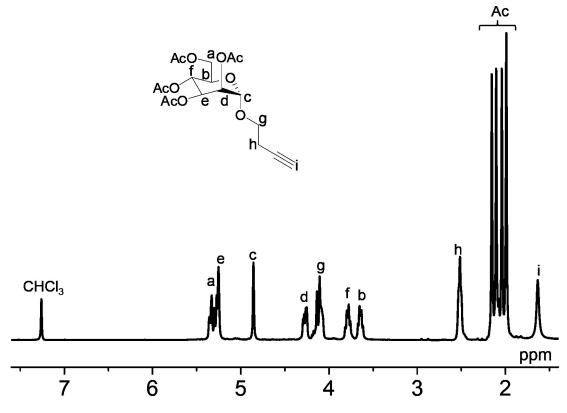


Figure S2. ¹H NMR spectrum of 3 in CDCl₃.

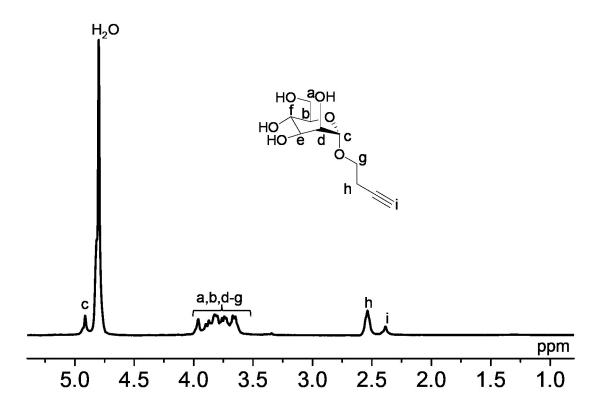


Figure S3. ¹H NMR spectrum of 4 in D_2O .

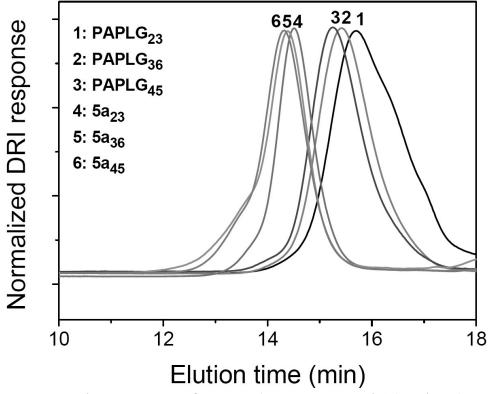


Figure S4. GPC chromatograms of PAPLG (DP = 23, 36, and 45) and **5a** (DP = 23, 36, and 45).

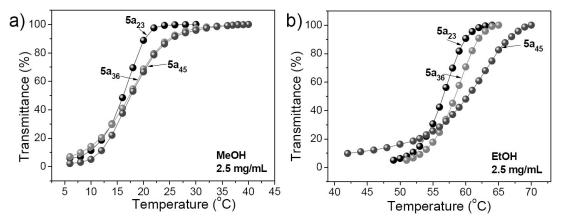


Figure S5. The plots of transmittance at $\lambda = 500$ nm versus temperature for the (a) MeOH solution and (b) EtOH solution of **5a** (DP = 23, 36, and 45) at 2.5 mg·mL⁻¹.

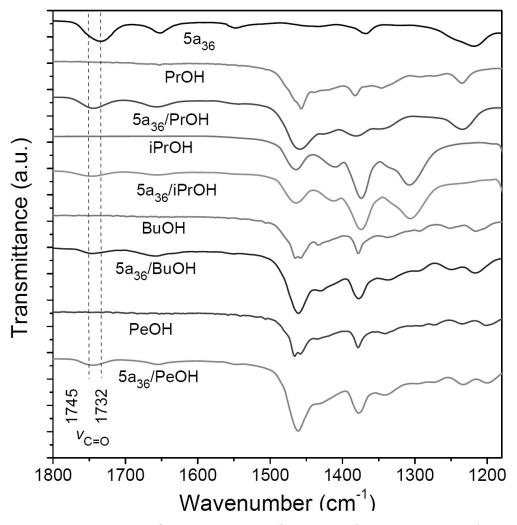


Figure S6. FTIR spectra of neat $5a_{36}$, ROH (i.e., PrOH, iPrOH, BuOH, and PeOH), and $5a_{36}$ /ROH mixtures.

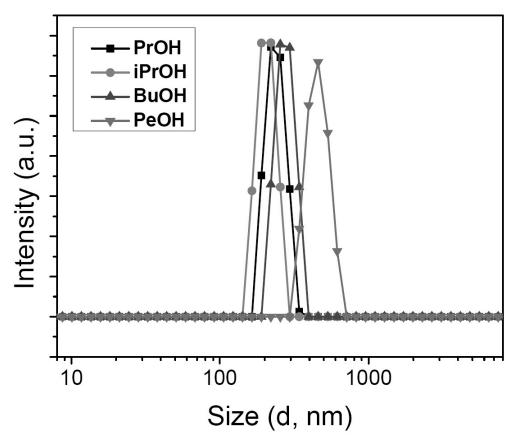


Figure S7. DLS size distribution plots of $5a_{36}$ in ROH (i.e., PrOH, iPrOH, BuOH, and PeOH) at 1 mg/mL (25 °C).

Table S1. UCST-type phase transition temperature of glycopolypeptides in MeOH at different concentrations.

Name	$T_{\rm pt}{}^{\rm a}({}^{\rm o}{\rm C})$				
	1 mg/mL	2.5 mg/mL	5 mg/mL		
5a ₃₆	7.4	17.4	27.4		
5c ₃₆	8.2	22.6	33.4		
5d ₃₆	b	21.5	35.0		

^a UCST-type phase transition temperature in alcoholic solvents determined by variable-temperature UV-vis spectroscopy.

^b The transmittance at 50% was below 5 °C.

	Diameter (nm), PDI ^a					
Name	МеОН	EtOH	PrOH	BuOH	РеОН	iPrOH
5a ₃₆	474.3, 0.262	348.5, 0.271	240.7, 0.104	276.8, 0.121	458.8, 0.170	206.8, 0.363
5c ₃₆	605.5, 0.213	361.2, 0.258	b			
5d ₃₆	750.3, 0.129	617.3, 0.197				

Table S2. DLS results of glycopolypeptides in various alcoholic solvents (1 mg·mL⁻¹, 25 °C).

^a Distribution of polymer aggregates in the solvents.

^b $5c_{36}$ and $5d_{36}$ were insoluble in PrOH, BuOH, PeOH and iPrOH at the concentration of 1 mg·mL⁻¹ (25 °C).

Table S3. UCST-type phase transition temperature of $5a_{36}$ in various alcoholic solvents.

Name	$T_{\rm pt}^{\rm a}(^{\rm o}{\rm C})$						
	MeOH ^b	EtOH	PrOH	BuOH	РеОН	iPrOH	
5a ₃₆	7.4	51.9	58.3	69.9	77.9	62.2	

^a UCST-type phase transition temperature in alcoholic solvents determined by variable-temperature UV-vis spectroscopy $(1 \text{ mg} \cdot \text{mL}^{-1})$.

^bMeOH = methanol, EtOH = ethanol, PrOH = 1-propanol, BuOH = 1-butanol, PeOH = 1-pentanol, iPrOH = isopropanol.