

Supplementary Information

Comparative Study of the Inverse versus Normal Bicontinuous Cubic Phases of the β -D-glucopyranoside Water-Driven Self-Assemblies Using Fluorescent Probes

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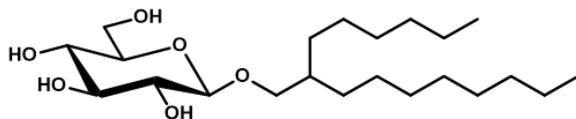
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^1H NMR and ^{13}C NMR for the 2-hexyl-decyl- β -D-glucopyranoside, β -Glc-OC₁₀C₆



^1H NMR (400 MHz, CD₃OD): δ (ppm) = 4.22 (d, 1H, H-1), 3.86 (dd, 1H, H- α a), 3.81 (dd, 1H, H-6a), 3.68 (dd, 1H, H-6b), 3.25-3.41 (m, 4H, H- α b, H-3, H-4, H-5), 3.18 (dd~t, 1H, H-2), 1.60 (m_c, 1H, β -CH₂), 1.29 (m_c, 24H, CH₂), 0.90 (t, 6H, CH₃).

$^3J_{1,2} = 8.0$ Hz, $^2J_{6a,6b} = 12.0$ Hz, $^2J_{\alpha a,\alpha b} = 12.0$ Hz, $^3J_{\alpha a,\beta\text{-CH}_2} = 2.5$ Hz.

PENDANT ^{13}C NMR (400 MHz, CD₃OD): δ (ppm) = 103.41 (C-1), 76.81 (C-3), 76.49 (C-4), 73.79 (C-2), 72.68 (C- α), 70.31 (C-5), 61.43 (C-6), 38.22 (CH), 22.46-31.76 (CH₂), 13.23 (CH₃).

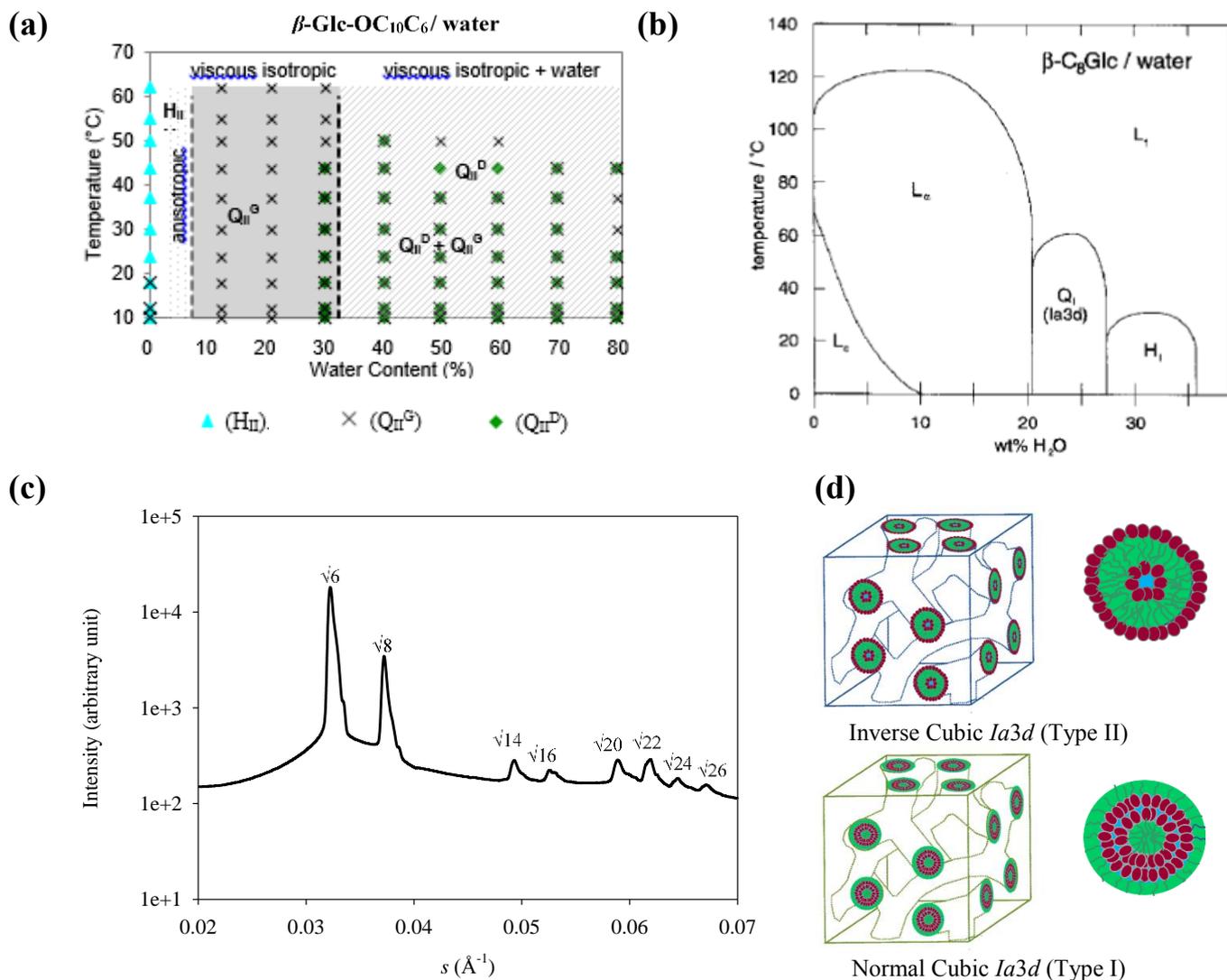


Fig. S1 X-ray investigation of 2-hexyl-decyl- β -D-glucopyranoside (β -Glc-OC₁₀C₆) and octyl- β -D-glucopyranoside (β -Glc-OC₈): (a) partial binary phase diagram of β -Glc-OC₁₀C₆, (reprinted with permission from Zahid et al. [1] Copyright (2013) American Chemical Society); (b) binary phase diagram of β -Glc-OC₈, (reprinted with permission from Sakya et al. [2] Copyright (1997) Taylor and Francis); (c) small-angle X-ray scattering pattern at 20 % (w/w) water content of β -Glc-OC₁₀C₆/water system (unpublished result from reference [1]) and (d) schematic cartoon representations of inverse (Type II) and normal (Type I) *Ia3d* bicontinuous cubic phases. In the inverse phase the bicontinuous network region are filled with water while the lipid chains occupy the matrix region. Conversely, in Type I, the network region is hydrophobic, filled with the lipid tails, while the intermediate space is filled with the hydrophilic sugar heads and water.

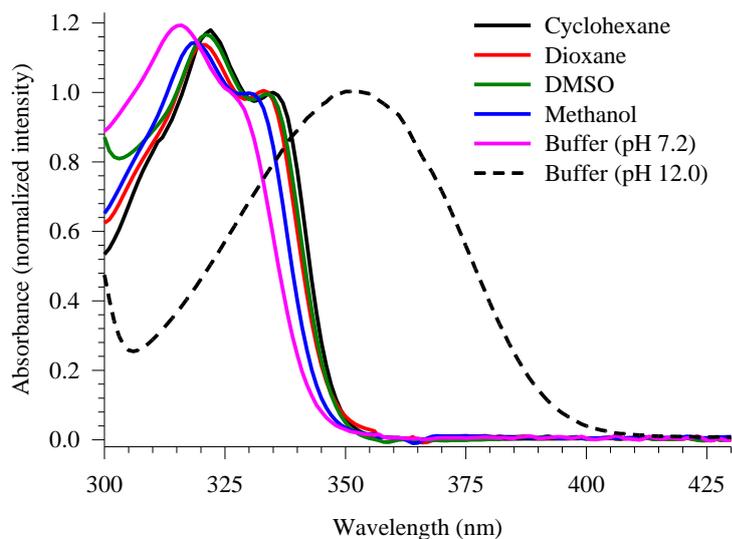


Fig. S2 Absorption spectra of HBO in different solvents, showing the lowest-energy band. Taken from reference [3].

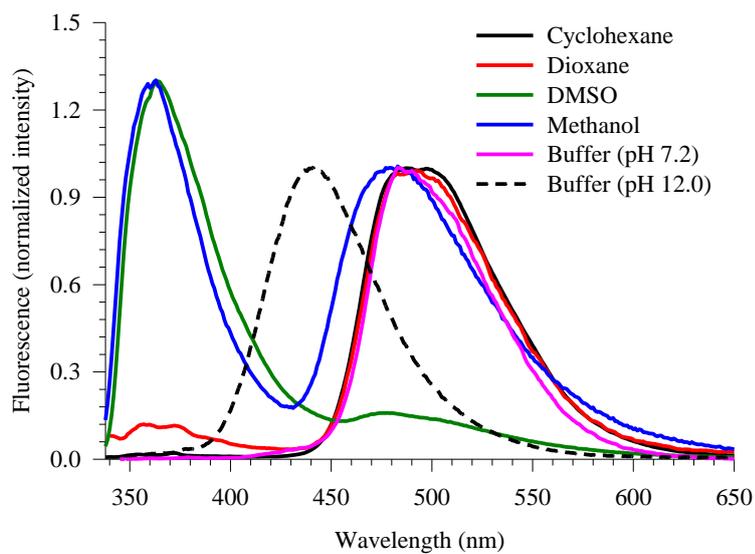


Fig. S3 Fluorescence spectra of HBO in different solvents. $\lambda_{\text{ex}} = 330$ nm. Taken from reference [3].

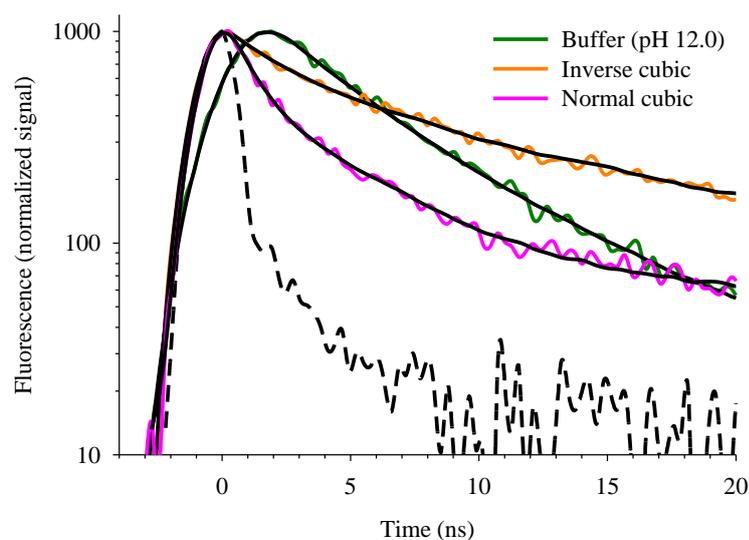


Fig. S4 Fluorescence decay transients of HBO in inverse cubic phase (β -Glc-OC₁₀C₆) and normal cubic phase (β -Glc-OC₈). The decay transient of HBO in buffer of pH 12.0 is included for comparison. $\lambda_{\text{ex}} = 380$ nm. Signal was measured using a 400 nm long-path filter. IRF is shown in a dashed line. Black solid lines represent the best fits.

Table S1. Fluorescence lifetime measurements of HBO and its derivatives in inverse cubic phase (β -Glc-OC₁₀C₆) and normal cubic phase (β -Glc-OC₈)

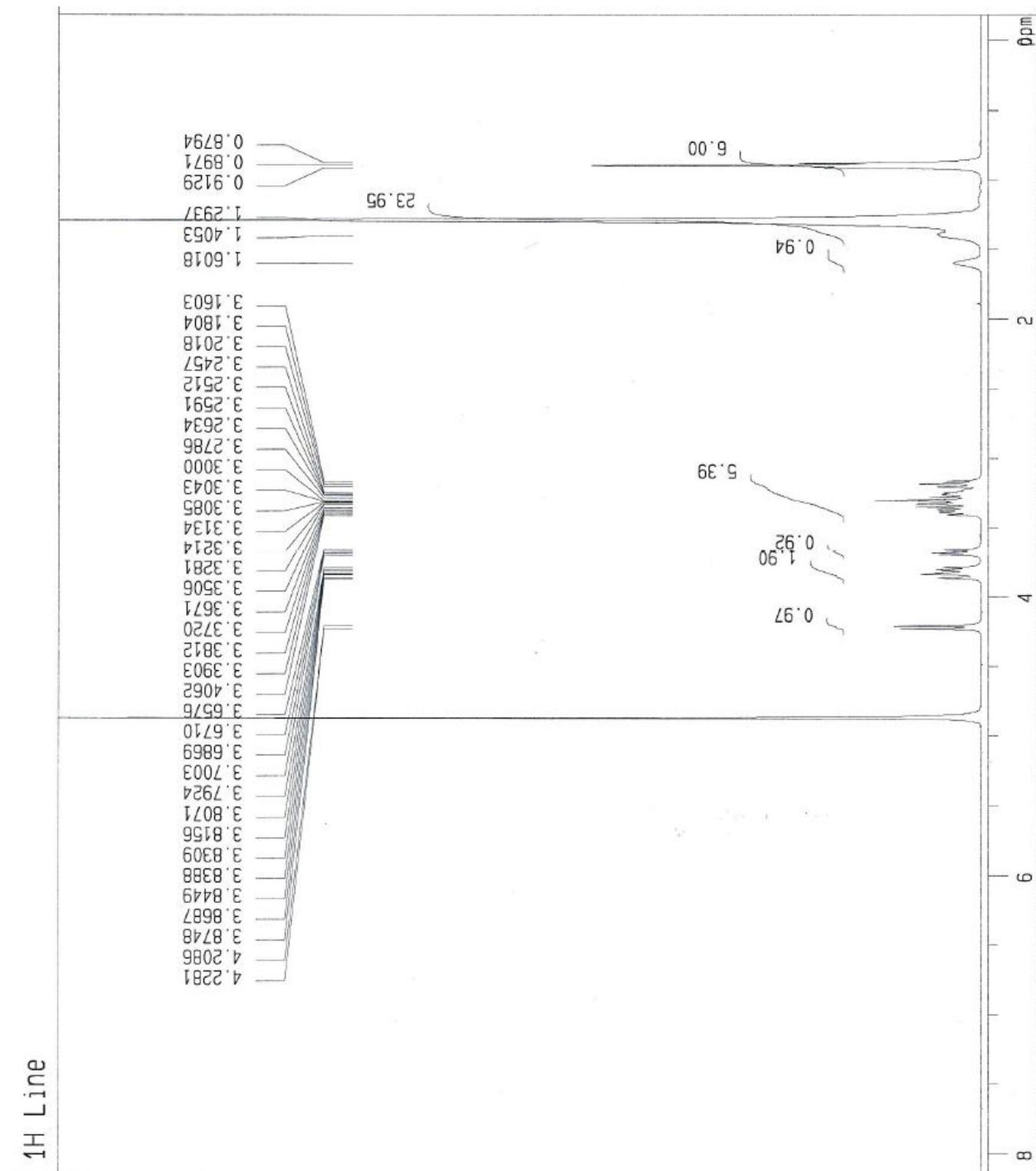
Probe	Lifetime, τ		
	τ_1^a Keto	τ_2^b Anion	τ_3^c Solvated <i>syn</i> - enol
Inverse cubic			
HBO		1.6 (0.19)	9.4 (0.81)
HBO-C ₄		0.9 (0.30)	6.5 (0.70)
HBO-C ₈		1.4 (0.21)	7.2 (0.79)
Normal cubic			
HBO	5.3 (0.58)	0.9 (0.42)	
HBO-C ₄	5.2 (0.52)	1.1 (0.48)	
HBO-C ₈	5.0 (0.52)	1.0 (0.48)	

Uncertainty in measurements is ^a ± 0.2 ns; ^b ± 0.1 ns; ^c ± 0.2 ns. Relative contributions are listed in parentheses. $\lambda_{\text{ex}} = 380$ nm. Emission was detected using a 400 nm long-path filter.

References

- [1] N. I. Zahid, C. E. Conn, N. J. Brooks, N. Ahmad, J. M. Seddon and R. Hashim, *Langmuir*, 2013, **29**, 15794–15804.
- [2] P. Sakya and J. Seddon, *Liq. Cryst.*, 1997, **23**, 409–424.
- [3] O. K. Abou-Zied, N. I. Zahid, M. F. Khyasudeen, D. S. Giera, J. C. Thimm and R. Hashim, *Sci. Rep.*, 2015, **5**, 8699.

¹H NMR



PENDANT ^{13}C NMR

