Supporting information

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The repeating unit of polymer xanthan



- 1. P.E. Jansson, L. Kenne, B. Lindberg, Carb. Res, 1975, 45, 275-282
- 2. J. Lecourtier, G. Chauveteau, G. Muller, Int. J. Biol Macromol., 1986, 8, 306-310.

Materials and Methods:

Xanthan polymer purchased from Sigma Aldrich [catalog No. G1253, molecular weight not known]. The liquid crystalline phase of xanthan was prepared according to the known procedure [T. Sato, T. Norisuye, H. Fujita, *Polymer J.*, **1984**, 16, 341-350.], by initially dissolving 700 mg of xanthan obtained from Sigma-Aldrich, in 35 ml of D_2O and exposing this aqueous solution to 20 kHz sonic irradiation for about six hours (sonication done with 20 sec MW pulse and 10 sec interval). Sonication was done under ice bath to avoid sample heating. The sonicated xanthan solution was centrifuged at 8000 rpm for 30 minutes and the supernatant was precipitated using large quantity of acetone and subsequently vacuum dried for about 1-2 hours. The liquid crystalline phase was prepared by adding 120 mg of this aqueous xanthan solution and 380 µl of 0.1M NaCl to NMR tube and centrifuging back and forth to get visibly homogeneous phase. The alignment of the phase was confirmed by detecting the quadrupole split doublet of the solvent D_2O .

SElective ReFocusing Experimental pulse Sequence



All the pulses are selective. Phases are, $\Phi_1=X$, $\Phi_2=\Phi_R=X$, -X, -X, X

Experimental Details

1. ²H NMR of solvent D_2O :

The solution was prepared with 70 mg of xanthan in 300 μ l of 0.1 M NaCl solution. The one dimensional spectrum was recorded using Bruker DRX-500 NMR spectrometer. The 16 transients were accumulated with a relaxation delay of 1 s. The 90⁰ pulse width is 9.8 μ s. 2 k time domain data were collected and Fourier transformed without using any window function.

2. ¹H NMR of (R/S)- β -butyrolactone

The solution was prepared with 120 mg of xanthan, 10 μ l of (*R/S*)- β -butyrolactone in 380 μ l of 0.1 M NaCl solution. The one dimensional spectrum was recorded using Bruker DRX-500 NMR spectrometer. The single transient was collected. The 90⁰ pulse width is 8.25 μ s. 3 k time domain data were collected and Fourier transformed without using any window function.

3. SERF Experiment on (*R/S*)-β-butyrolactone

The solution was prepared with 120 mg of xanthan, 10 μ l of (*R/S*)- β -butyrolactone in 380 μ l of 0.1 M NaCl solution. The 2D SERF spectra were recorded using Bruker DRX-500 NMR spectrometer. 90⁰ pulse width is 8.25 μ s. Two transients were accumulated with 256 t1 increments. The size of the 2D data matrix is 300 X 1024. The data was Fourier transformed using unshifted sine bell window function in both the dimensions. 3 k time domain data were collected and Fourier transformed without using any window function.

4. HSQC experiment on a tri peptide in xanthan medium

The solution of tripeptide Gly-Glu-Cys was prepared taking 5 mg of the solute in 50 mg of xanthan and 380 μ l of NaCl solution. The size of the 2D data matrix was 512 X 1536. Sixteen free induction decays were accumulated and Fourier transformed using sinebell window functions in both the dimensions.

Indication of mesophase formation:

The ²H quadrupole doublet of solvent D_2O , indicating the alignment of guest molecule. 120 mg of xanthan and 380 µl of 0.1M NaCl solution was taken. The one dimensionl ²H spectrum was recorded in Bruke DRX 500 NMR spectrometer. The doublet separation corresponds to 140 Hz indicating the formation of the mesophase.



The ¹H NMR spectrum of (R/S)- β -butyrolactone



Temperature dependence of quadrupole split doublet

The temperature dependence of quadrupole split doublet separation of solvent D_2O aligned in the mesophase of xanthan.



Transition from isotropic to anisotropic phase

The ²H-NMR spectrum of solvent D_20 as a function of concentration of Xanthan in 380 ul of 0.1 M NaCl indicating the minimum quantity of xanthan required to obtain mesophase. The spectra are recorded at 61.4 MHz on AVIII 400- Bruker NMR spectrometer.



Variation of quadrupole split doublet separation as a function of quantity of xanthan

The variation of deuterium quadrupole splitting of solvent D_2O as a function of the quantity of xanthan, in 380 µl of 0.1M NaCl.



Sustainability of anisotropic phase even at 370 K

The ²H NMR spectra of solvent D_2O at different temperatures between 280- 370 K, varied in steps of 10 degrees. Spectra recorded at 76.73 MHz frequency on Bruker DRX-500 NMR spectrometer. It is apparent that the anisotropic medium is sustained over a wide range of temperature, even when the boiling point of water is approached. Sample prepared using 70 mg of Xanthan in 300ul of 0.1 M NaCl. The measured quadrupolar splitting varied from 138 (at 280 k) to 89 Hz (at 370 K).



Prochiral discrimination of Glycine-D₂

Additional example of prochiral discrimination in Glycine-D₂. The 10 mg of glycine-D₂ was dissolved in 70 mg of xanthan in300 μ l of 0.1 M NaCl solution. The ²H NMR spectra were recorded at different temperatures. The pro-chiral discrimination was observed from 300 -370 k. The peaks marked * are the doublets from natural abundant ²H of solvent water.



The temperature reversibility of the mesophase:

The quadrupole doublet separation was monitored for the solvent D_2O from 280-370 K and 370-280 K, in steps of five degrees. The process of measurement was repeated twice and each temeprature sufficient time was given to attain temperature stability. The plots confirm not only the sustainability of the mesophase over a wide temperature range, but also reversibility of the mesophase.



The ²H NMR spectrum of a scalemic mixture of D/L-alanine-d₃

The scalemic mixture of D/L-alanine-d₃ with 33.3 % enrichment of L enantiomer was prepared to measure the enantiomeric excess. The ²H NMR spectrum was recorded at ambient temperature at 76.73 MHz. The ratiometric analysis of the intensities provided the enantiomeric excess of 32.78 %.

