

THERMOTROPIC BEHAVIOR OF NEW CHOLESTERIC LIQUID-CRYSTAL POLYMERS

CRG BM16-SAXS

EXPERIMENT 16-02 63

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The experiment consisted in two parts:

- A). WAXS/SAXS measurement, were performed successfully for the eight powdered functionalized cholesteric liquid crystal polymers (CLCP), previously characterized by NMR and DSC.

Calibration was performed with Alumina Corundum in the 2θ range from 6 to 24 deg (Q between 9 to 35), with a monochromatized beam at $\lambda = 0,7514 \text{ \AA}$.

WAXS 2D data were recorded by a Princeton Instruments CCD (Charged Coupled Device) camera (1242x1152 pixels) on 2theta arm. The program Fit2D was employed to process images. Binary data were normalized and pixels radially averaged into 1D.

The effect of temperature on the polymers structure was studied by performing three controlled cycles of heating/cooling/ heating, for each polymer sample, at $10^\circ\text{C}/\text{min}$, between $T_{\text{room}} = 25^\circ\text{C}$ and temperatures bellow the decomposition temperature with a LINKAM THMS600 stage (temperature was not calibrated).

- B). SAXS measurements were carried out at room temperature, on complexes dispersed in TAE (0,001M EDTA), between two CLCP and eight different types of commercial DNA of increasing complexity: [Poly-A]; [Poly-C]; [Poly-T]; [Poly-G]; [Poly-C-Poly-G]; [Poly-A-dT]; Calf-Thymus-DNA. Plasmid series could not be measured.

Five proportions were studied for each complex:

DNA : Polymer

1 : 0

1 : 2

1 : 1

2 : 1

0 : 1

The CLC polymers were complexed with DNA by mixing the stoichiometric proportions, suspending in TAE and digesting for 12h in a swinging shaker.

A monochromatized beam at $\lambda = 0,7514 \text{ \AA}$ was used.

Binary two-dimensional data were registered with an MARCCD 165 detector, placed at 5975 cm from the sample.

The program Fit2D was employed to process 2D binary data. Raw data were normalized and pixels radially averaged into 1D.

Silver behenate ($d = 58.3 \text{ \AA}$) was used to calibrate the angular axis in a Q range between $[0,036 \text{ to } 1,64 \text{ nm}^{-1}]$ (600 \mu m to 27 nm).

The net scattering intensity $I(n)$ was attained through the standard equation.

$$I(n) = \frac{1}{c \text{Det}(n)} \left[\frac{I_s(n)}{I_{0s} T_s} - \frac{I_m(n)}{I_{0m} T_m} - \frac{I_e(n)}{I_{0e}} \left(\frac{1}{T_s} - \frac{1}{T_m} \right) \right],$$

Being s , m and e , respectively, for sample, matrix and empty cell; T transmission and c sample concentration.

References

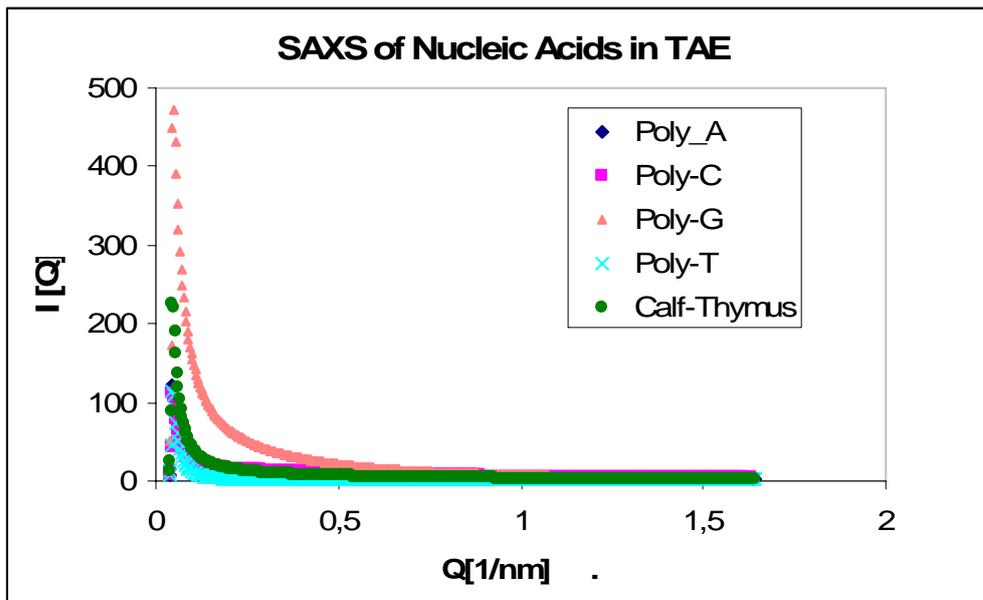
- [1] A. P. Hammersley (1995). ESRF Internal Report Exp/AH/95-01, Grenoble, FRANCE
- [2]. P. Strunz, J. Saroun, U. Keiderling, A. Wiedenmann and R. Przenioslo (2000), *J. Appl. Cryst.* **36**, 829-833.

CONCLUSIONS:

As we had expected all the synthesized liquid-crystal polymers behave as cholesteric, and thermotropic in the solid state, with phase transition under the effect of temperature.

The lyotropic behaviour of the complexes between CLCP-DNA is also proved by showing interaction with cholesteric DNA at the proportions studied.

The whole series of Plasmid-DNA could not be measured.



Similar results had been previously obtained.