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SHORT COMMUNICATIONS

Scattering Studies on Fingerprint Texture of Cyanoethyl Chitosan†

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KEY WORDS Small-Angle Light Scattering / Chitosan / Cyanoethyl Chitosan / Lyotropic Liquid Crystal / Fingerprint Texture / Pitch / Cholesteric Phase /

Small-angle light scattering (SALS) was first introduced to polymeric research by Stein[1] in 1960. Since then, it has been widely used to investigate the submicrostructures of polymers, especially spherulites. However relatively few works have concentrated on liquid crystals using SALS. In this paper, a new method to measure the pitch of fingerprint texture of liquid crystalline phase was reported.

EXPERIMENTAL

Material

O-Cyanoethyl chitosan was synthesized at this lab by mixing 0.5 g chitosan (degree of deacetylation, 78%; size of powder, 100 mesh), 20 mL acrylonitrile and 1 mL 5% aqueous sodium hydroxide, and stirring for 12 h at room temperature.

Preparation of Liquid Crystalline Box

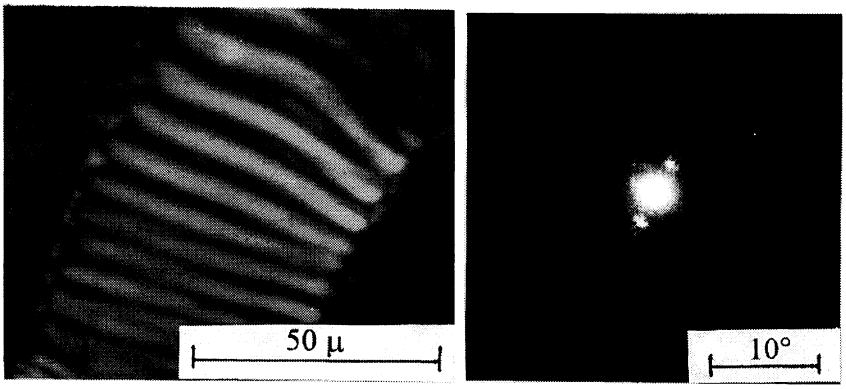
Cyanoethyl chitosan solutions of given concentration (well above critical concentration) were prepared by mixing cyanoethyl chitosan and several solvents in vials, then sealed for 7 days before use it. A liquid crystalline box used for polarized light microscopy (PLM) and SALS observation was made by putting solution between two slide glasses.

RESULTS AND DISCUSSION

Chitosan (β-1-4-linked 2-amino-2-deoxy-D-glucopyranose structural repeat units) and its derivatives show lyotropic liquid crystalline phases when dissolved in suitable solvents.[2-5] In this study, *O*-cyanoethyl chitosan exhibited lyotropic liquid crystalline behavior. Fingerprint texture can be observed when *O*-cyanoethyl chitosan is dissolved in trifluoroacetic acid, *m*-cresol, formamide, *N*,*N*-dimethyl formamide, *N*,*N*-dimethyl acetamide, and dimethyl sulfoxide, showing cholesteric phase. The fingerprint texture of *O*-cyanoethyl chitosan was quite regular and divided into different pieces which usually differed in direction. Big pieces were few and usually isolated. This provided the possibility for SALS to measure the pitch of a single big piece of fingerprint.

The SALS H_v pattern of a single big piece of fingerprint (size 60 × 120 μm) prepared from 20% trifluoroacetic acid solution is shown in Figure 1(b). The whole piece was within the spot of the laser beam. It was not difficult to find the corresponding fingerprint (Figure 1(a)) from a few big pieces in PLM according to the special orientation.

In a single piece, the periodic repeat structure behaved as a grid. So diffraction spots appeared in the direction vertical to layer planes of fingerprints in SALS pattern. Normally only the first order diffraction can be seen. From the distance *d* between center and diffraction spot,



(a) (b)

Figure 1. A single big piece of fingerprint texture of cyanoethyl chitosan in 20% trifluoroacetic acid solution. (a) PLM photograph (650×); (b) SALS pattern (H_v scattering, r=80 mm).

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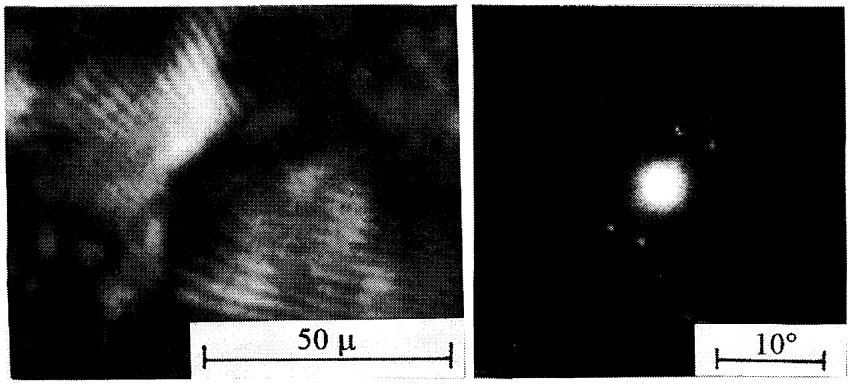
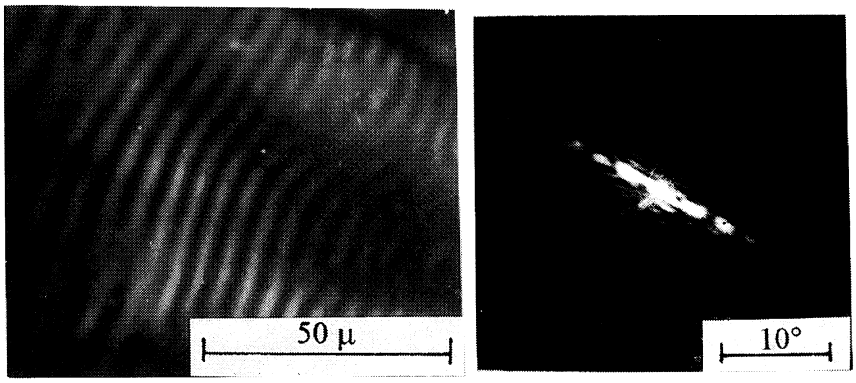


(a) (b)

Figure 2. Two big pieces of fingerprint texture of cyanoethyl chitosan in 20% trifluoroacetic acid solution. (a) PLM photograph (650×); (b) SALM pattern (H_v scattering, $r=80$ mm).



(a) (b)

Figure 3. A very big piece of fingerprint texture of cyanoethyl chitosan in 20% dichloroacetic acid solution. (a) PLM photograph (650×); (b) SALS pattern (H_v scattering, $r=80$ mm).

the average pitch P of the piece was determined as,

$$n\lambda = P \cdot \sin\theta_{max}$$

$$\theta_{max} = t_g^{-1} d/r$$

Where n is interference order; λ wavelength of the laser (632.8 nm); θ_{max} scattering angle in maximum intensity; and r distance between sample and film. From Figure 1(b), $P=10\,\mu$m was obtained. This result accorded with that of PLM.

When two big pieces with different orientation were within the area of laser beam simultaneously (Figure 2(a)), an interesting four spot diffraction pattern appeared (Figure 2(b)). Each pair of spots corresponded to one piece.

When a fingerprint was big and regular (Figure 3(a)), multi-order diffraction was documented (Figure 3(b)). Figure 3(b) shows four order diffraction spots.

This is a quick and simple method for determining the pitch of the cholesteric phase. An average value of the pitch can be obtained directly. However, the method is only suitable for isolated, regular and big pieces of fingerprints.

REFERENCES

1. R. S. Stein and M. B. Rhodes, *J. Appl. Phys.*, **31**, 1873 (1960).
2. K. Ogura, T. Kanamoto, T. Sanan, K. Tanaka, and Y. Iwakura, Proc. 2nd Int. Conf. Chitin/Chitosan, Tottori, Japan, 1982, p 39.
3. D. K. Rout, S. P. Pulapura, and R. A. Gross, *Macromolecules*, **26**, 5999 (1993).
4. D. K. Rout, S. P. Pulapura, and R. A. Gross, *Macromolecules*, **26**, 6007 (1993).
5. D. K. Rout, S. P. Barman, and S. K. Pulapura, *Macromolecules*, **27**, 2945 (1994).