15A/2007G002

A small-angle X-ray scattering study of dialysis-induced Curdlan gel

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Introduction

Curdlan dissolved in alkaline solution forms a unique gel consisting of anisotropic gel and amorphous gel in alternating layers by a dialysis into aqueous calcium chloride [1]. In this study we have measured the small-angle X-ray scattering (SAXS) intensity profiles observed from different directions to clarify the anisotropy of the gel structure.

Experimental

Materials

Curdlan (Mw=5.9×10^5) was purchased from Wako Pure Chemical Co. Ltd. and used without further purification. A desired amount of Curdlan was dissolved at 5 wt% in 0.3 M NaOH. 30 ml of the Curdlan solution was poured into a seamless cellulose tube with the diameter of 28.6 mm (UC-36-32, Sanko Junyaku, Japan), immersed into 600 ml of 8 g/dl aqueous calcium chloride bath and dialyzed for 72 h to make a gel at 25°C. A round slice of the gel with the desired thickness and its two cross sections were excised out, as shown in Fig. 1.

Measurements

Small-angle X-ray scattering (SAXS) was measured with a focusing diffractometer at beamline 15A1, Photon Factory (PF), using an X-ray wavelength (λ) of 0.15 nm at the room temperature. The beam size was adjusted to 1.5 mm at the specimen. The disk-like sample with the thickness of 1 mm prepared above was cut into a series of thin sheet, each was set into a sample holder for the SAXS measurement and X-rays were irradiated from the indicated direction in Fig. 1. The scattering data were detected with an image intensifier coupled to a CCD camera.

Results and Discussion

Near the center of the gel no anisotropy was observed in the scattering profiles. In the outermost layer, the scattering intensity in the horizontal direction was larger than that in the vertical direction in both z- and x-directions, whereas no difference was observed between horizontal and vertical ones (isotropic entirely) in the y-direction. These results clearly indicate that the degree of inhomogeneities in the radial direction and that in the circumferential direction are different, and that the larger intensity in the horizontal direction than in the vertical direction reflects the larger degree of inhomogeneities in the radial direction.

Such a profile resembles the one for the abnormal butterfly pattern observed for swollen elongated gels, i.e., isointensity curves with the major axis parallel to the elongation direction are observed [2]. The abnormal butterfly pattern has been explained by a larger increase in the degree of quasi-static frozen inhomogeneities in the elongation direction than in the parallel direction. If the anisotropy of Curdlan gel induced by dialysis is attributed to the shear stress by ionic flow normal to the dialysis tube, it is not surprising to find a structure similar to the swollen elongated gel. The larger degree of inhomogeneities in the radial direction is consistent with the visual observation of the gel under crossed nicols for the samples cut at different directions [1].

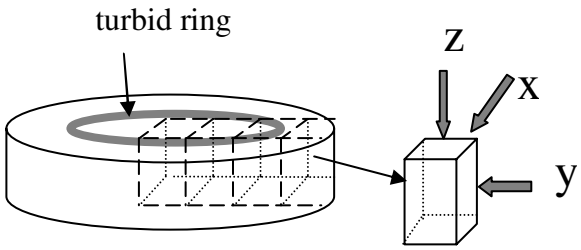

Figure 1. Illustration of the sample geometry. The scattering profiles were observed by irradiating X-rays from the indicated directions.

References

[1] Dobashi, T., Nobe, M., Yoshihara, H., Yamamoto, T., Konno, A., *Langmuir*, **2004**, *20*, 6530-6534.
[2] Mendes, E., Lindner, P., Buzier, M., Boue, F., Bastide, J., *Phys. Rev. Lett.*, **1991**, 66, 1595-1598.

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