FIBER DIFFRACTION STUDIES ON POLYMERS USING SYNCHROTRON RADIATION SOURCE

K. Noguchi[1] K. Okuyama[2]
[1]Tokyo University of Agriculture and Technology Instrumentation Analysis Center 2-24-16 Naka-Cho KOGANEI TOKYO 184-8588 JAPAN [2]Faculty of Technology, Tokyo University of Agriculture and Technology

The advent of a high-flux and sharp X-ray beam with a small divergence makes it possible to record fiber diffraction patterns from thin filamentous specimens of muscle, collagen, silk and other systems and well-oriented sols of virus and bacterial flagellar filament. The advantage of synchrotron radiation is also useful for the data collection of the bundles of fibers that have been used in a laboratory. In this study, we collected fiber diffraction data of synthetic polyesters [poly(tetramethylene succinate)(PTMS) and poly(tetramethylene adipate)(PTMA)] and chitosan obtained from a crab tendon whose size were comparable to those in laboratory experiments (< 0.5 mm). Fiber diffraction patterns were collected at the BL40B2 of SPring-8 using an imaging plate area detector (Rigaku, R-AXIS IV++) with an X-ray wavelength of 1.0 Å. Synchrotron radiation experiments enabled us to obtain very good diffraction data with high S/N ratio and well-separated diffraction spots. In order to get sufficiently high S/N ratio we needed 3 minutes exposure. Crystal structures of PTMS, PTMA and chitosan were analyzed based on synchrotron radiation data using linked-atom least squares refinement technique. Although the resultant structures were essentially same as those analyzed with intensity data collected in our laboratory, R-values of PTMS(0.13), PTMA(0.11) and chitosan(0.11) were lower than those based on laboratory data (0.18, 0.12 and 0.16, respectively). It is suggested that exact and reliable intensities can be obtained from synchrotron radiation data even for weak diffraction spots.

Keywords: FIBER DIFFRACTION SYNCHROTRON RADIATION POLYMER

POSSIBLE PACKING OF TERMINAL *α*-HELICES IN THE INNER-CORE OF THE BACTERIAL FLAGELLAR FILAMENT

K. Hasegawa[1] K. Imada[1,2] S. Maki-Yonekura[1] K. Yonekura[1,2] F. Samatey[1] I. Yamashita[3] K. Namba[1,2,3]
Erato Protonic NanoMachine Project 3-4 Hikaridai SEIKA KYOTO 619-0237 JAPAN
[1]Protonic NanoMachine Project, ERATO, JST [2]Graduate School of Frontier Bioscience, Osaka University [3]Advanced Technology Research Laboratories, Matsushita Electric Industrial Co., Ltd

The bacterial flagellar filament is a helical assembly of a single protein flagellin. The filament is a supercoil acting as a helical propeller and switches between left- and right-handed supercoiled forms for swimming and tumbling. It is thought that flagellin molecules adopting two distinct conformations are arranged regularly in the supercoiled filament. To reveal the mechanism of polymorphism, we have been studying the structures of two types of straight filaments by the X-ray fiber diffraction method. We prepared highly well-oriented liquid crystalline sols of the filaments and recorded X-ray fiber diffraction patterns at SPring-8. To record intensity data including those near the meridian, diffraction patterns recorded with various tilt of the fiber axis were merged, and then layer-line intensities were extracted by the profile fitting procedure. Phases were derived from a filament model (Imada et al., unpublished data) constructed with the atomic model of a core fragment of flagellin (F41) placed and oriented using heavy atom positions as fiducials, which were identified in a difference Fourier map obtained with X-ray fiber diffraction amplitudes and phases from electron cryomicroscopy analysis. A difference Fourier map against the model phases ($2F_6$-F_c) at 5.5 Å resolution showed the N- and C-terminal chains lacked in the filament model. They form long alpha-helices and constitute the inner tube of the concentric double tubular structure of the filament core by intimate interactions with neighboring subunits as previously shown by electron cryomicroscopy analysis. These interactions in the filament core appear to stabilize the filament structure.

Keywords: **MACROMOLECULAR ASSEMBLY, FIBER DIFFRACTION, BACTERIAL FLAGELLAR FILAMENT**

THE THERMOSTABILITY AND ELASTICITY OF SPIDER DRAGLINE STUDIED BY SYNCHROTRON X-RAY DIFFRACTION

H.-S. Sheu[1] K.-W. Phyu[1] Y.-P. Chiang[1] Y.-C. Jean[1] J.-C. Yang[2] S.-L. Ferng[2] I.-M. Tso[3]
[1]Synchrotron Radiation Research Center Research Division No. 1, R&D Road VI, Hsinchu Science Based Park HSINCHU 30077 TAIWAN [2]Union Chemical Laboratories, Industrial Technology Research Institute, Hsinchu 300, Taiwan [3]Department of Biology, Tunghai University, Taichung 400, Taiwan

The thermostability and elasticity of Cyrtophora moluccensis (I) and Cyrtophora unicolor (II) spider silk have been studied by X-ray diffraction technique. Three motifs, elastic beta-spiral, crystalline β-sheet and helix, repeated many times in the same order along the silk direction form the spider dragline. The composition of beta-sheet is mainly constructed by polyalanine, which form nano size crystalline. The β-sheet may be the regions that bind the protein molecules together in the fiber and provide tensile strength. The thermostability of crystalline in spider (I) dragline is surprise high. X-ray diffraction under heating by dry air stream from room temperature up to 400C showed that the spider silk remained in its crystalline form until decomposed. It is interesting that the superlattice peak disappear at above 100C. In opposite, the reflections perpendicular to silk are much thermostable until above 350C. X-ray diffraction of spider silk (II) during stretching was also measured. The result showed that diffraction from superlattice and (0 0 2) reflections were shifted to lower angle indicted the interlayer spacing became enlarged, while the diffraction pattern perpendicular to silk remained the same position. The elongation at the length above 10 percent of the original length was reached saturation.

Keywords: **SPIDER SILK SYNCHROTRON RADIATION ELASTICITY**

CHARGE DENSITY STUDY OF HEXAAQUAZINC(II) DYIHYDROGEN-1,2,4,5-BENZENETETRACARBOXYLATE

N. G. Fernandes[1] R. Diniz[1] J. Ellena[2] T. Gustafsson[3]
Instituto De Cliencias Exatas-UFMG Quimica Department 6627 Av. Antonio Carlos BELO HORIZONTE MINAS GERAIS BRAZIL
[1]Department of Chemistry, Federal University of Minas Gerais, CP 702, 31270-901 Belo Horizonte, Brazil [2]Instituto de Fisica de Sao Carlos, Universidade de Sao Paulo, CP 369, 13560 Sao Carlos, Brazil [3]Department of Materials Chemistry, Angstrom Laboratory, Box 538, SE-751 21 Uppsala, Sweden

The structure of $Zn(H_2O)$ $C_{10}H_4O_8$, ZnH_2Bt, was analyzed by neutron and X-ray diffraction techniques at 100 K. The aim is to investigate the electron density distribution. ZnH_2Bt crystallizes in the monoclinic space group C2/c, Z = 4. The neutron data collection was performed using thermal neutrons with λ = 1.2055 Å. 1824 ($F > 0$) reflections used to refine 193 parameters. $R(F^2)$ = 0.0637, $wR(F^2)$ = 0.0696. The Zn^{2+} ion is coordinate to six water molecules in a octahedral arrangement. In the H_2Bt^{2-} ion there is a short asymmetric hydrogen bond. The O --- O distance is 2.411(3) Å, the O-H distances are 1.117(1) and 1.314(1) Å and the O-H-O angle is 168.91(2)°. The X-ray data were collected up to $\sin\theta/\lambda$ = 1.28Å^{-1} on a Nonius Kappa-CCD diffractometer, Mo Kα radiation, λ = 0.71073 Å. 10201 independent reflections used to refine 135 parameters. In the final refinement $R(F^2)$ = 0.0465 and $wR(F^2)$ = 0.0717. The hydrogen atom parameters were fixed at neutron values. However, their displacements have been modified according to Blessing [1]. Multipole refinements have been carried out using the XD package [2]. Chemical and symmetry constrainsts were applied. In Zn-atom, the 3d electron have an aspherical distribution. In the short hydrogen bond, deformation density map shows that hydrogen atom is bonded differently to the two oxygen atoms.

References

[1] R. H. Blessing, Acta Cryst. B51, 816-823, 1995.
[2] T. Koritsanszky, P. Mallinson, Z. Su, T. Richter & N. Hansen. A computer Program Package for Multipole Refinement, and Analysis of Electron Densities from Diffraction Data, 1995.

Keywords: **ELECTRON DENSITY HYDROGEN BOND MULTIPOLE REFINEMENT**