Small-angle X-ray scattering studies on mechanism of particle formation of silica, and growth

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1. Introduction

It is well known that the formation of colloidal silica in an aqueous system proceeds through three stages: polymerization of monosilicic acid ($Si(OH)_4$) to form oligomers, the growth of particles and linking of particles, then three-dimensional networks, finally thickening it to a gel. The mechanism of silica particle formation and growth process was not been made clear sufficiently. We measured time-resolved small angle X-ray scattering (SAXS) of silica sol-gel process at SPring-8.

2. Experiment

The samples of silica sol were prepared from TEOS(tetraethoxsilane), ethanol and hydrochloric acid by volume ratio of 5:1:1.5. The measurements, which were carried out after addition of hydrochloric acid, were done at 3, 16, 59, 119, 164, and 175 minutes respectively. The sample was injected into the capillary made from 2mm ϕ quartz cell. As blank, solvent(ethanol) was put in and measured to the capillary. The SAXS measurements were preformed at BL-40B2. Two-dimensional SAXS pattern was recorded on an imaging plate using the R-AXIS (Rigaku Denki, Co. Ltd) and the wavelength of X-rays, λ, was tuned at λ=0.100nm.

3. Results

The result of SAXS measurement is shown in Figure 1. As time goes, the scattering intensities are increasing. The scattering patterns are changing to show fractal dimension of -2, finally. It may enhancing particle growth process of silica.

About this mechanism of particle formation, we are still working for further investigation.

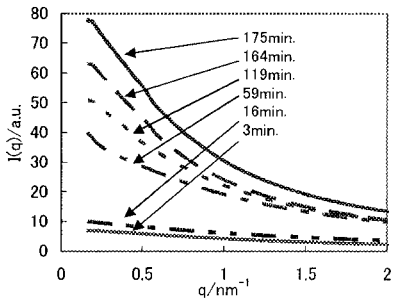

Figure 1. Time-resolved SAXS profiles from silica sol.

Structural analysis of a pectate lyase PL47 in complex with calcium

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Pectate lyases (PLs) catalyze the cleavage of α-1,4-glycosidic linkages in polygalacturonic acid chains, which are a major component of pectic substances of plant cell walls. The cleavage of the glycosidic linkage occurs through a β-elimination mechanism to generate products with a 4,5-unsaturated bond at the nonreducing end. Calcium is essential for the activity of PL. Pectolytic enzymes such as PLs play an important role in fruit ripening that is attributed to degradation and solubilization of the pectic substances responsible for cell cohesion. The enzymes are effectively used in some industrial processes including retting of flax, extraction and clarification of fruit juices, and maceration of vegetables. Thermostable PLs are of considerable interest for a biochemical process such as scouring of cotton fabrics.

We have previously solved the crystal structure of a thermostable pectate lyase PL47 from a thermophilic *Bacillus* sp. TS47 at 1.8 Å resolution. The next target is to solve the complexes of PL47 with substrate analogues. Information derived from the three-dimensional structures of the complexes might potentially lead to the design of new examples of PLs that have higher activity and stability.

Crystals of the complexes were prepared by the soaking and co-crystallization techniques. These crystals are trigonal, space group $P3_121$. However, due to a largely increased mosaicity, data collection of crystals of PL47 complexed with substrate analogues, except for a complex with calcium, was failed. A complete data set of PL47 in complex with calcium cation was collected on a CCD detector (ADSC Quantum 4R) at the BL40B2 station of the SPring-8. The wavelength was 1.0 Å and the crystal-to-detector distance was 200 mm. The oscillation range was 1° with exposure time of 60 sec for each image. The crystal diffracted to 1.9 Å resolution. The data set was processed using the program *MOSFLM* and scaled using the program *SCALA* from the *CCP*4 package. A total of 341,909 observed reflections were scaled and reduced to yield a data set containing 42,335 unique reflections with an R_{merge} of 5.7%. Structure analysis is now in progress.