ESRF

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Micro beam diffraction experiments on starch granules

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Christian RIEKEL

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Names and affiliations of applicants (*indicates experimentalists):

A. BULEON, INRA, NANTES *
B. PONTOIRE, INRA, NANTES *
H. CHANZY, CNRS/CERMAV, GRENOBLE *
R. VUONG, CNRS/CERMAV, GRENOBLE *

Report:

EXPERIMENTAL

Samples of starch granules were dispersed in an aqueous solution (0.15% w/w) of polyacrylic acid (ALDRICH). Drops of this suspension were deposited on collodion coated electron microscope 3 mm finder grids (200 mesh) and allowed to dry. As it was observed that some background scattering resulted either from the grid bar or grid lettering, squares corresponding to 25 grid bars were cut away m center of the grids prior to collodion coating. The grids holding the starch granules were photographed with optical microscopy (polarized light and Nomarski contrast) and mountedon the microfocus beam line ID13-BLl. A 2μm X-ray beam ($\lambda = 0.92$ Å) was produced by the combination of an ellipsoidal mirror with a tapered glass capillary. Important background scattering was cut with a 10μm aperture positioned between the capillary exit and the sample. The diffraction diagrams were recorded with a flux of 5.10 photons per second for 16 second. They were registered by an image intensified CCD camera with video frequency readout and on-line digitalization and frame accumulation. The camera was positioned at 5 cm from the sample. In order to localize the sample, we first investigated the areas where the grid bars of the sample holder had not been cut. In those areas, the lettering became apparent as background and therefore, the sample matrix could be identified with the help of the photomicrographs that. were shot ahead of the experiments. Having recognized the positioning of the grid, we could then move quickly to the areas of interest, located in the center of the grid where the grid bars were absent.

Individual granules were then scanned with steps of either 5 or 10 micron. Photomicrographs taken after the experiments showed clearly the areas that had been irradiated by the X-ray probe (Figure 1).

RESULTS AND PERSPECTIVES

Under the above experimental conditions, the granules had a life time of about 30 seconds. Using images intensified from the CCD readout, we were able to record a large number of meaningful X-ray diffraction patterns showing interesting orientation. Potato

starch had the best orientation (Figure 2) . The data showed clearly that the crystalline components were crystallized in the B starch allomorph and that the crystalline domains were oriented in such a way that their chain axes were aligned parallel with respect to the granule radii. Also, the central part of the granules were devoid of any orientation. The granules of wheat presented the A allomorph pattern (Figure 3). They showed a much poorer orientation than the potato granules. In addition, the patterns of wheat starch had line widths much smaller than those of potato starch. Thus, the crystalline domains in wheat starch appear much larger, but of poorer orientation as compared to those of potato.

These results are extremely encouraging as for the first time, they open a way to systematic ultrastructural studies on starch before and after processing. Numerous long standing problems of starch science and technology can now be addressed after the present successful experiments.

Figure 1: Typical sample of potato starch mounted on a sample holder after step irradiation with a 2 micron X-ray beam, each step consisting of a 16 second exposure. The grid was scanned with 5 micron steps : this is clearly visualized in the left part of the picture in an area where there was no granule. The imprint of the beam in the sample is associated with beam damage of the supporting collodion film.

Figure 2: A selection of a series of X-ray diffraction diagrams collected from a potato starch granule irradiated with a step irradiation with a 3 micron X-ray beam. The steps were separated with 10 microns and the irradiation time of each diagram was of 16 second.

Figure 3: A selection of a series of X-ray diffraction diagrams collected from a wheat starch granule irradiated with step irradiation with a 2 micron X-ray beam. The steps were separated with 5 microns and the irradiation time of each diagram was of 16 second.

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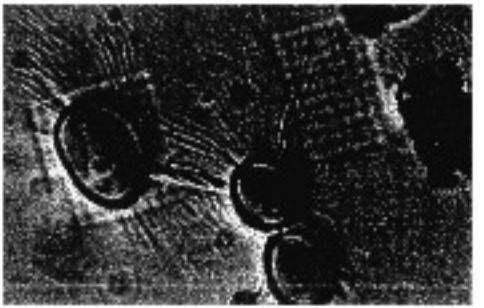

Figure 1

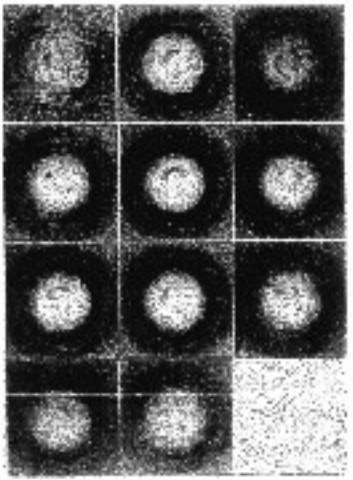

Figure 2

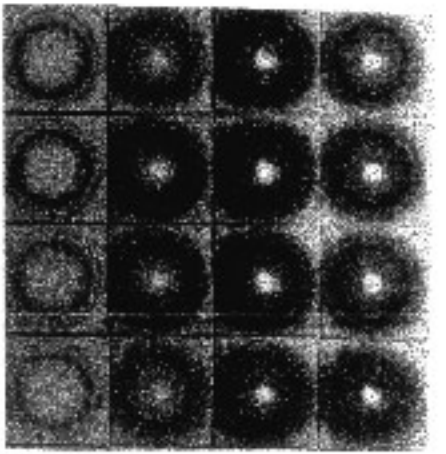

Figure 3