ESRF	**Experiment title:** Microfocus mapping of starch ultrastructure	**Experiment number:** SC 345
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INTRODUCTION

In the preceding experiments, we performed a thorough mapping of both crystal orientation and natural polymorphism present in some native starches [1,2]. This beamtime was used for mapping of partially molten starch granules with pure B-type (potato starch) or mixed A+B type (pea starch - see [2]). The aim was to catch some intermediate structures present at different steps of melting, to detect any differences in the melting behavior of A and B crystalline types in native starch granules and to check if some local remnant structures were still present in ghosts obtained after melting of starch granules. Some diffraction experiments on spherolitic crystals of A and B types obtained from low polymerization degree amylose were also carried out.

EXPERIMENTAL

Partially molten starch granules were prepared by heating in a Differential Scanning Calorimeter in excess water (80%), the experiment was stopped at a temperature corresponding to the half height of the melting endotherm. All samples were deposited on special collodion coated electron microscope grids with a large central cavity and covered after sample rehydration by another collodion coated grid in order to prevent from water loss during the experiment. The grids were photographed in normal and polarized light with optical microscopy and mounted on the microfocus beam line ID13. The measurements were performed as described before with a 2 μm X-ray beam (λ = 0.78 Å), a 20μm aperture positioned between the capillary exit and the sample, scanning steps of 3 to 10 microns and recording times of 30 to 60 seconds using an image intensified MAR CCD detector (2048x2048 pixels of 63x63 μm).

RESULTS AND PERSPECTIVES

The diffraction signals obtained from partially melted pea starch granules are of pure A-type with a rather weak crystallinity and a complete disappearance of the B-type structure. It is consistent with the ring-like morphology of the granules (Fig. 1), the obvious hollow in the center of the granules could result from the melting of the B-type structure which has proved to be more present in the center of the granulesas shown in our previous work [2].

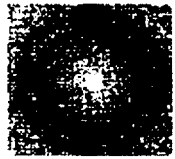

Fig. 1. Ring -like morphology of partially molten pea starch granules and corresponding A-type diffraction diagram

No intermediate diagrams between those characteristic from native and completely gelatinized states were recorded from gelatinized potato starch granules. No remnant crystalline or oriented structure were found, even at a local level, on the granule ghosts resulting from gelatinization(Fig. 2).

← Fig. 2: Granules ghosts resulting from potato starch gelatinization.

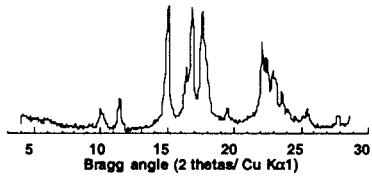

Fig. 3: Powder diagram of B type spheroltic crystals →

No available signal were recorded from both A and B-type spherolitic crystals of amylose in spite of their very high crystallinity as judged by classical powder X-ray diffraction (Fig.3). No explanation was found for this observation, the lifetime of the diagrams were very short. The amount of scattering matter was probably too small due to the dimensions of the crystal (< 10 microns in diameter) and the hydration decreased very rapidly, leading to a rapid decrease of the crystallinity.

These different results show that for small hydrated objects with poor scattering properties, it would be necessary to use a cooling system for both preventing the water loss and increasing the lifetime of the diffraction diagrams

[1] A. Buleon, B. Pontoire, C. Riekel, H. Chanzy, W. Helbert and R. Vuong (1997) Macromolecules, 30, 3952-3954.

[2] A. Buleon, C. Gerard, R. Vuong, C. Riekel and H. Chanzy (1998) Macromolecules, 31, 6605-6610.