

ESRF

Experiment title:

Supramolecular structure of wild and genetically modified starches

Experiment

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C Riekell

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Names and affiliations of applicants (* indicates experimentalists):

Dr AM Donald- Cavendish Laboratory, University of Cambridge, UK

*Mr TA Waigh - " "

*Dr MF Butler - " "

*Dr IM Hopkinson " "

Report:

In order to study starch granules in their native state it is important to ensure that they remain hydrated. For conventional experiments at a synchrotron source this poses no problem, as macroscopic cells may be used, containing bulk starch slurries. However in order to exploit the microfocuss facility, a new design of cell was required. We found it was possible to achieve this as follows. Starch was sieved and centrifuged to provide the largest available granules from each cultivar. Next, under a stereomicroscope, a bubble of water was placed on to the carbon support film (which needed to be less than 1mm in thickness), which had a copper locator grid glued on its other side. The locator grid had a 500µm hole punched in it. The presence of this hole permits the collection of diffraction data without the problem of copper peaks obscuring the data. Next a small number of granules were dropped into the bubble with an eyelash brush. Slowly, by carefully monitoring the build up in the microscope, a monolayer of starch could be produced on the carbon film. Natural evaporation was allowed to remove most of the excess water and the sample was immediately sealed with another (upper) grid mounted support film. Estimation of the water content of such minute samples is hazardous, but limiting water levels are of the order of 30% w/w (water/starch), and it is assumed these 'dry' samples have less water than this. High water content samples (c.a.45% w/w) were prepared by piercing the upper carbon film. Upon floating the cell in water and placing a bubble of water on top of the pierced hole, water was sucked through the into the cell by capillary action, hydrating the sample.

Potato starch was found to be the most satisfactory to work with, since it had both large granules and was comparatively resistant to beam damage. Wheat was much more susceptible. It was possible to compare the orientation from $2\mu\text{m}$ regions for dry and hydrated potato starch. An order parameter of 0.53 was obtained for a hydrated sample, and 0.46 for a dryer one, using the orientation of the (100) interhelix peak. However these results must be regarded as preliminary, since they were achieved through integration over azimuthal scans with significant levels of noise.

Data was also collected at 10mm steps across a potato granule, as shown in figure 1.

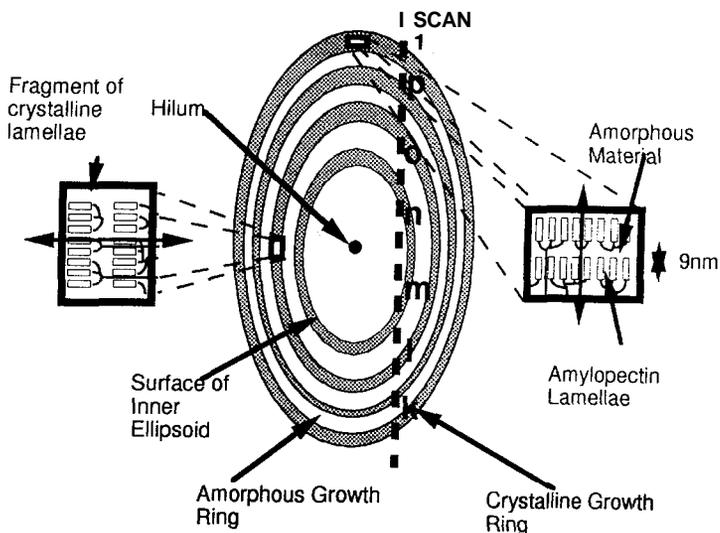


Figure 1 Granule in cross section showing how the orientation of amylopectin double helices. Scan 1 shows the location at which diffraction data were obtained.

By analysis of the direction of the (100) peaks it was possible to show that the orientation of the double helices did not point to a single focus (as seen in figure 1). With the limited number of data points obtained it is not possible to be exactly sure but the data is consistent with the helices pointing to the surface of an inner ellipsoid. Further work is required to confirm this hypothesis, and correlate this with the location of the hilum. The quality of the diffraction patterns obtained was good particularly at the edge of the granules, and demonstrated, as has long been assumed, that locally the packing is the same as in highly oriented amylose fibres. It is possible to compare the patterns with simulations based on the fibre unit cell, and good agreement is found.

Finally, data was obtained on genetically modified potato starch which showed interesting differences with wild type. This data has not yet been cleared for publication.