## Preliminary communication

## A granular cold water-soluble starch gives a V-type X-ray diffraction pattern\*

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Eastman and Moore<sup>1</sup> recently developed a new process for preparing granular, cold-water-soluble (g.c.w.s.) starch by high-temperature treatment of native starch with aqueous alcohol. G.c.w.s. starch has the desirable property of pasting without heating. This property provokes questions regarding the change that has occurred within the granule. The present communication reports data to help answer that question.

Scanning electron micrographs showed that g.c.w.s. corn and wheat starches retained their granular structure, but the granules were slightly misshapen. When viewed with a light-microscope under crossed Nicol prisms, g.c.w.s. starch displayed a strong birefringence without any discernable pattern. This was different from the birefringence of native starch, which exhibited the characteristic Maltese cross. The loss of the Maltese cross indicated destruction of the ordering of crystallites in the native granules.

An X-ray diffraction pattern of g.c.w.s. corn starch was taken with Cu, Ni-foilfiltered, Ka radiation (see Fig. 1A). Operation was at 35 kV and 18 mA. Peaks appeared at  $2\theta = 13.0$  and  $20.1^{\circ}$ , which were equivalent to d-spacings of 6.80 and 4.42 Å, respectively. This diffraction pattern is similar to that obtained for the hydrated, V-amylose structure with hexagonally close-packed helices spaced 13.6 Å apart. The peaks at  $2\theta$ ~16 and 23°, typical of native corn starch<sup>3</sup>, were not present.

To quantitate the V structure in g.c.w.s. corn starch, a series of mixtures containing different proportions of amylose-ethanol complex (see Fig. 1B) and amorphous, waxy corn starch (see Fig. 1C) were prepared. The amylose-ethanol complex was prepared by adding ethanol (20% by volume) to an aqueous solution of amylose (1 mg/mL) at 95°. The mixture was slowly cooled during 16 h, and the complex was isolated by centrifugation, rinsed with ethanol, and dried in a vacuum oven<sup>4</sup>. The amorphous, waxy corn starch was obtained by vacuum-oven drying of a boiled (1 h) mixture consisting of 1% of starch in water. The X-ray diffraction patterns of the mixtures were recorded, and the total areas under the peaks at  $2\theta = 13.0^{\circ}$  and  $20.1^{\circ}$  of each mixture (corrected to equivalent weight) were used to construct a standard curve. Based on the standard curve, we estimated that ~50% of V-structure existed in the g.c.w.s. corn starch.

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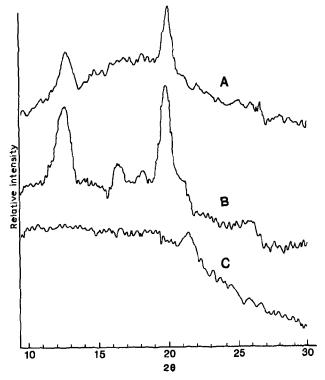


Fig. 1. X-Ray diffraction patterns of (A) g.c.w.s. corn starch, (B) amylose—ethanol complex, and (C) amorphous, waxy corn starch.  $CuK\alpha$  radiation was used, at 35 kV and 18 mA.

The g.c.w.s. corn starch contained 27% of amylose, as determined by the blue value method<sup>5</sup>. It follows that amylopectin, as well as amylose, formed a V complex with the aliphatic alcohol during formation of g.c.w.s. starch. V-Amylose complexes consist of single helical chains and are known to be water-soluble<sup>6</sup>. On the other hand, the double-helix structure of native amylopectin or retrograded amylose is insoluble.

Thus, the cold-water-solubility of g.c.w.s. starch can be explained by conversion of the native double helix into a V (single helix) structure.

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