METHODS IN CARBOHYDRATE CHEMISTRY

Edited by
ROY L. WHISTLER

IV *Starch*

METHODS IN
Carbohydrate Chemistry

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Preface

Continuing the broad aims of this series as stated in Volume I, the present volume concentrates the specialized methods needed in work with starch and starch fractions. Many highly specific methods are required in work with starch, but there are needed also a host of methods that are of broad applicability in the carbohydrate field. These more general methods may be found elsewhere in the series, but especially in Volume V, which provides methods more generally applicable to polysaccharides.

As in each volume of the series, a glossary of proprietary substances and equipment is given at the end, thus obviating repetitious listings.

Mr. Robert J. Smith handled correspondence with authors, did the initial copy editing, and demonstrated high editorial ability. His excellent work and especially his fine cooperation, helpful advice, and wise counsel are greatly appreciated.

December, 1963 ROY L. WHISTLER

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Section I. Preparation of Starch and Starch Fractions

WHOLE STARCH

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WHOLE STARCH

[1] Corn Starch

Isolation

By Stanley A. Watson

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Introduction

All recoverable starch in the corn kernel is located in the endosperm. The endosperm comprises 82–86% of the kernel (*1*) and is composed of two regions: the horny or hard endosperm and the floury or soft endosperm. Individual starch granules are encased in a protein matrix of variable thickness. In the horny endosperm, starch granules are small, and the protein matrix is relatively thick; in the floury endosperm, granules are large, and the protein matrix is thin and weak (*2*). Simple water soaking and grinding of corn kernels liberate most of the starch from the floury endosperm region, but more drastic measures must be taken to obtain maximum starch yield. A laboratory batch steeping and milling procedure designed to simulate the conditions and results of commercial corn wet milling have been described (*3*). However, isolation of starch in good yield for carbohydrate or biochemical research may be adequately accomplished by a simpler steeping procedure using a hydrogen sulfite solution with no pH adjustment.

Procedure

A solution of 1.22 g. of sodium hydrogen sulfite[1] in 750 ml. of water is prepared; this solution will analyze about 0.10% of sulfur dioxide by iodimetric titration. About 300 g. of clean, sound, whole corn (10–20% moisture) is added to the hydrogen sulfite solution, and the mixture is maintained at 50° (±2°) for about 20–24 hr. with either continuous or intermittent gentle agitation or circulation of the liquid. After 24 hr., the steepwater will analyze about 0.05% of sulfur dioxide, and the pH will be about 5.2. The steepwater is drained off, and the corn is ground in a suitable mill such as a Quaker City Laboratory mill, Labconco mill, coffee mill, or Waring Blendor. If a Waring Blendor is found to be most convenient, 250 ml. of steeped corn are ground with 250 ml. of water in an

[1] An equivalent amount of sodium or potassium disulfite (pyrosulfite, metabisulfite) may also be used.

800-ml. blender bowl for 3 min. at full speed.[2] The ground slurry is screened through an 80- or 100-mesh wire screen, and the residue is washed free of starch. The filtrate is passed successively over 200-mesh and 325- or 400-mesh screens. Screening is performed much more conveniently if screening materials are stretched over a frame attached to a shaking machine (*4*). In this case, nylon bolting fabric will be most useful, and the initial screening on 80-mesh wire may be eliminated.

The starch–gluten slurry that passes the finest screen must next be separated into its components. Starch readily separates from gluten by gravity because of the greater density of starch granules (1.5 g./cc.) compared to the density of the gluten particles (1.1 g./cc.). The mixture is allowed to stand 1 hr., whereupon the water and gluten layers may be removed by suction. The starch layer should then be resuspended and resettled or centrifuged in wide-mouthed cups. The residual protein may be removed by scraping off all non-white material.

The best and most convenient starch separation procedure employs the "starch table" (*4*). The "starch table" is merely a trough inclined sufficiently to allow starch to settle, but allows gluten and water to run off. This method will be preferred if frequent starch preparations are to be made. To construct a handy laboratory starch table, obtain a straight 10-ft. (3 meter) piece of 3-in. (7.6 cm.) extruded aluminum "I" beam and polish one concave surface with fine steel wool. Close one end of one channel with a piece of aluminum or plastic glued in place with epoxy cement. Support the beam above the working surface so as to accommodate an effluent receptacle under the open end, and raise the closed end about 1 in. (2.5 cm.) higher than the open end. The starch-protein mixture is adjusted to a concentration of about 15 g. of solids per 100 ml. (sp. gr. 1.058, 15.56°/15.56°) and is dribbled in a steady stream onto the upper end of the table at a rate sufficient to permit the starch to settle and the protein and water to flow off the open end of the channel. After all starch has been deposited on the table, adhering protein is flushed from the starch surface by passing 500 ml. of water over the table at a rate about 4 times faster than the flow rate used for the starch–protein slurry. It may be necessary to remove additional adhering protein by washing the starch surface with a fine stream of water from a wash bottle. The starch is removed from the channel, slurried in distilled water, filtered, and dried at 30–40° (see also Vol. IV [3]).

If adequate separation of starch cannot be obtained by the above procedures, a method employing pentanol (amyl alcohol) may be useful. To

[2] Oil liberated from grinding the germ is absorbed by the gluten and not by the starch. It does not interfere with the starch isolation. The leading edges of the blender blades may be ground to a flat edge so more impact and less cutting action is obtained.