

COMMUNICATION TO THE EDITOR

Physical Properties of Films from Dimethyl Sulfoxide-Pretreated Amylomaize Starches

DEAR SIR:

Investigation of certain physical properties of amylomaize (high-amylose-content) starch film required the preparation of aqueous dispersions of the undegraded starch. It is known that amylomaize starch, in contrast to ordinary corn starch, requires temperatures considerably in excess of 100°C. for complete aqueous dispersion (1). However, these conditions are conducive to hydrolytic degradation of the polysaccharide, and less strenuous methods of dispersion were therefore sought.

Various chemical pretreatments of amylomaize starch by Sloan *et al.* (1) have shown that its solubility characteristics can be modified. The

comparatively recent recognition of dimethyl sulfoxide (DMSO) as a nondegrading solvent for various granular starches, including high-amylose corn starch (2,3), prompted use of this medium for the pre-treatment of amylo maize starch in film studies. Accordingly, it was found that amylo maize starch containing 13% moisture can be dispersed in 99.9% DMSO in a ratio of 1 part of starch to about 3 parts of solvent (w/w) at temperatures ranging from 75° to 85°C. Starch was recovered quantitatively by precipitation upon addition of the dispersion in DMSO to twice its volume of methanol, ethanol, or isopropanol. Extraction with alcohol removes adhering DMSO. The alcohol-wet starch is soluble in water at temperatures well under 100°C. The recovered starch can be dried and subsequently dispersed in water after being rewetted with any of the named alcohols or with n-butanol.

A preferred method of film preparation is as follows: A 25-g. (d.b.) quantity of ethanol-wet product is stirred into 150–175 ml. of water at 70°C. Heating is continued to permit a 10-min. reflux period and distillation up to 92°C. pot temperature for removal of almost all the alcohol. Although filtration is usually unnecessary, the dispersion is filtered hot through a coarse-sintered glass funnel and into a warm flask. The flask is then stoppered and its contents deaerated under vacuum. The clear dispersion is cast on silicone-treated glass plates by means of a 16-in. doctor blade with 30-mil clearance.

Films prepared from starches of 59 and 73% apparent amylose (4) that were treated with DMSO had respective intrinsic viscosity values of 1.35 and 1.05 in normal potassium hydroxide at 25°C., the same as the parent high-amylose starches. This suggests that little depolymerization occurred, if any, during the treatment with DMSO and subsequent film preparation.

A summary of the tensile properties of the films prepared from amylo maize starches is presented in Table I. These values compare favorably with those of films prepared by Wolff *et al.* (5) from amylose fractionated from corn starch.

TABLE I
PROPERTIES OF AMYLO MAIZE STARCH FILMS

APPARENT AMYLOSE IN AMYLO MAIZE STARCH	DRY TENSILE STRENGTH (INSTRON)	FOLDS M.I.T.	MULLEN BURST POINT
%	lb./sq. in.		
59	10,821	265	29
73	11,483	421	30
71 ^a	11,084	461	20

^a Amylon VII.

The starches with 59 and 73% apparent amylose were prepared in the pilot plant of this Laboratory (6,7). Amylon VII¹ is a commercial product from National Starch and Chemical Corporation.

A. M. MARK
W. B. ROTH
C. L. MEHLTRETTER
C. E. RIST

November 26, 1963

Northern Regional Research Laboratory
Agricultural Research Service
U.S. Department of Agriculture
Peoria, Illinois

Literature Cited

1. SLOAN, J. W., MACMASTERS, MAJEL M., and SENTI, F. R. A note on methods of dispersing fifty-percent-amylose corn starch. *Cereal Chem.* **36**: 196-198 (1959).
2. KILLION, P. J., and FOSTER, J. F. Isolation of high molecular weight amylose by dimethyl sulfoxide dispersion. *J. Polymer Sci.* **46**: 65-73 (1960).
3. LEACH, H. W., and SCHOCH, T. J. Structure of the starch granule. III. Solubilities of granular starches in dimethyl sulfoxide. *Cereal Chem.* **39**: 318-327 (1962).
4. DEATHERAGE, W. L., MELVIN, E. H., GLASS, C. A., and DIMLER, R. J. Spectrophotometric determination of amylose in high-amylose corn. Presented at 47th annual meeting, American Association of Cereal Chemists, St. Louis, Mo., May 20-24, 1962.
5. WOLFF, I. A., DAVIS, H. A., CLUSKEY, J. E., GUNDRUM, L. J., and RIST, C. E. Preparation of films from amylose. *Ind. Eng. Chem.* **43**: 915-919 (1951).
6. ANDERSON, R. A., VOJNOVICH, C., and GRIFFIN, E. L., JR. Wet-milling high-amylose corn containing 49- and 57-percent-amylose starch. *Cereal Chem.* **37**: 334-342 (1960).
7. ANDERSON, R. A. A note on the wet-milling of high-amylose corn containing 75-percent-amylose starch. *Cereal Chem.* **39**: 406-408 (1962).

¹Mention of firm names or trade products does not imply that they are endorsed or recommended by the U.S. Department of Agriculture over other firms or similar products not mentioned.