WHEAT STARCH GELATINIZATION IN SUGAR SOLUTIONS. I. SUCROSE: MICROSCOPY AND VISCOSITY EFFECTS[1]

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Cereal Chem. 55(6): 936-944

ABSTRACT

A photomicrographic technique was used to study swelling behavior of individual wheat starch granules heated in water and various sugar solutions. With the polarizer rotated slightly off extinction, birefringent and gelatinized granules could be photographed simultaneously. Granule diameters were measured from enlargements of photographs taken during heating and were plotted as ratios of the granule diameters at 50° C. A rapid increase in diameter occurred during and immediately after loss of birefringence, and coincided with the first-stage gelatinization exhibited by amylograph viscosity curves. The rapid swelling stages occurred at progressively higher temperatures and at faster rates as sugar concentration was increased to 60%, the highest used. Amylograph viscosities emphasized the delaying effects of sucrose on gelatinization of starch and demonstrated that only first-stage swelling may occur in 50% sucrose solution before the medium boils. Such results are applicable to layer-cake systems in which the level of sugar often equals that of water in the batter.

Starch has been shown to be the principal ingredient determining the crumb structure of baked products. Sandstedt (1) showed glass beads could not substitute for starch in bread containing gluten as the only flour component. Conversely, others (2–6) have shown that various gums, cellulosic compounds, or emulsions of glycerol monostearate could be substituted for gluten to produce bread-type products from starch or nongluten flours. Howard et al (7) showed layer cakes could be produced with starch as the only flour component if certain

[1]Cooperative investigation of the Western and North Central Regions, Science and Education Administration, U.S. Department of Agriculture, and Department of Agronomy, Ohio Agricultural Research and Development Center, Wooster. Approved for publication as Journal Article 98-77 of the Ohio Agricultural Research and Development Center, Wooster, OH 44691.

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0009-0352/78/000105$03.00/0

ingredients were present to stabilize the batter emulsion during baking. Cauvain and Gough (8) baked cakes from starch and carboxymethylcellulose using a yellow cake formula.

Many ingredients affect in diverse ways the gelatinization properties of starch, which determine crumb structure of baked products. Osman (9,10) has published excellent reviews on the subject.

Sugars, especially sucrose, have been shown to have marked effects on starch gelatinization properties. Various sugars have been shown to decrease both hot paste viscosity and strength of aged gels (11,12). Microscopic observations on cooked slurries (13) and on starches extracted from baked products (14) have suggested limited swelling of granules in the presence of high levels of sucrose. Determination of gelatinization temperature by counting swollen granules (15), by loss of birefringence of granules (16,17), and by differential scanning calorimetry (18) has shown that initial gelatinization occurs at progressively higher temperatures as sucrose is increased. None of these studies, however, has indicated the extent to which starch has undergone the typical cereal-starch pattern of first-stage and second-stage gelatinization (19). Thus, determining the events occurring during starch gelatinization in the presence of sucrose seemed valuable.

This article describes a microscopic study of the effects of sucrose on the swelling behavior of wheat starch granules and relates these findings to viscosity effects noted in starch-sucrose systems.

MATERIALS AND METHODS

Starches used in this study included a soft white wheat starch washed from Avon variety flour and a commercial wheat starch (Aytex P, General Mills, Inc., Minneapolis). Carboxymethylcellulose (CMC) was the 7 HF type from Hercules Incorporated, Wilmington, DE.

Photomicrography

A Leitz Ortholux polarizing microscope was fitted with a 35-mm camera body and a Kofler heating stage. The field was viewed with the polarizing filter in place, but rotated slightly off extinction (about 7 degrees) to view gelatinized starch simultaneously with birefringent starch. Thus, loss of birefringence could be determined for individual granules during gelatinization while simultaneously noting granule size changes. Photomicrographs were made on high-contrast black and white film.

Slide Preparation and Heating

Starch (0.2–0.5%) was suspended in water or sucrose solutions. Sucrose concentrations are expressed as percent by weight of total solution (50% is equivalent to 50 g of sucrose in 50 g of water). A microscope slide was prepared for heating by forming a ring of mineral oil on the slide, placing a small drop of the appropriate slurry in the center of the ring, and positioning the cover glass to exclude air pockets. The mineral oil boundary prevented water evaporation during heating.

Ten to 20 large granules (10 μ or greater) could be viewed in the field to be photographed. The heating rate, controlled with a rheostat, was about 1°C/min throughout the gelatinization range. Exposures were made at selected temperatures during early heating, then at every degree from just before the loss of birefringence through the maximum swelling range, and then every few degrees until the granules collapsed or until the mounting medium boiled, causing field movement. Small-granule starch was not measured, because resolving power of the microscope with the hot-stage attachment did not allow sufficiently accurate measurement of these granules during the course of gelatinization.

Granule Measurements

Uniform enlargements were made of all exposures for direct measurement of granule diameters during gelatinization. A stage micrometer was photographed under the same optical conditions, and a transparency of the scale was used to measure granules (Fig. 1). Martin's diameter was used to follow the change in size

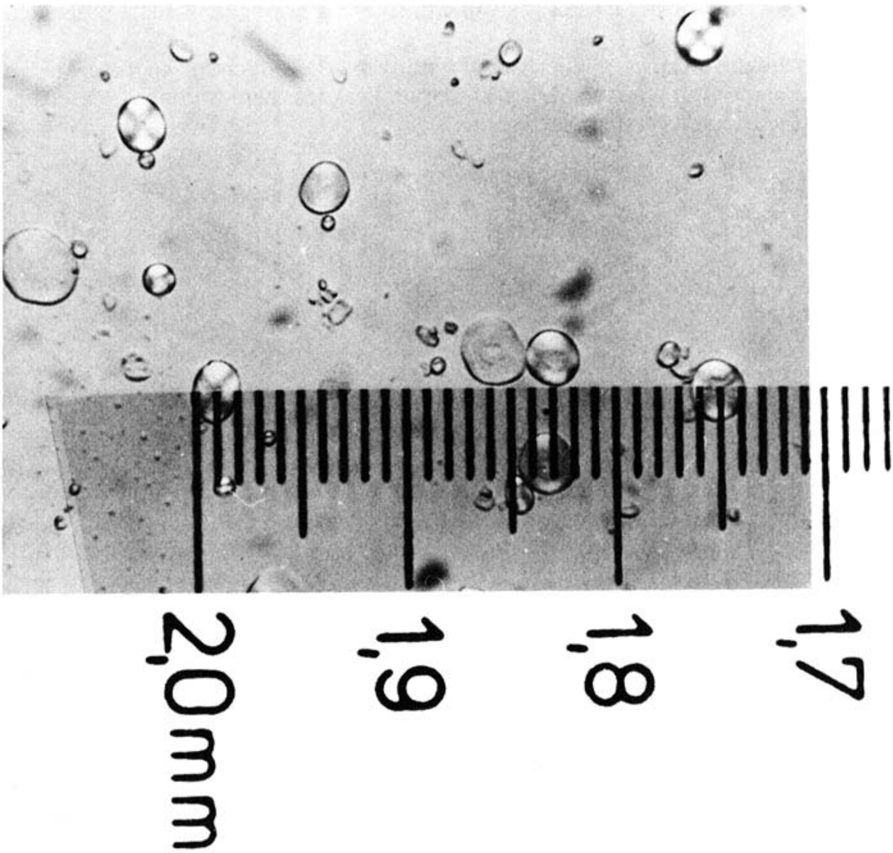

Fig. 1. Transparent scale superimposed on print of starch granules. Placed to measure horizontal bisect of one granule.

of granules during heating (20). With this method, diameter is measured horizontally on a line that bisects the granule. No correction was made if the granule rotated in the field during heating and concomitant photography. The new horizontal bisect was used. (This rotation seldom occurred, and when it did, the action was random.) Martin's method of measuring diameter is used widely for determining particle size by microscopic methods. It gives satisfactory results when compared with data using three actual dimensions of individual particles (20), and allows the convenience of making a single measurement for each granule at each temperature.

Measured diameters of several granules were averaged at each temperature and divided by the average diameter of the same granules at 50° C. The values thus obtained are the diameter ratios (DR) used in this article. Diameters at 50° C were used as the reference control after determining that diameters from room temperature to 50° C showed no measurable change by this method.

Viscosity Measurements

Viscosity results were obtained with a Brabender Amylograph equipped with a water-cooled cover and a heater programmed at 1.5° C/min. Unless otherwise noted, slurries were heated to 97° C (permitted by the sea level location of the senior author's laboratory). For each run, 450 ml of water or of sucrose solutions were used. Concentrated starch slurries contained 225 g of starch; dilute slurries contained 45 g of starch. These were calculated on 14% mb to facilitate applications to standard baking tests. Curves obtained with CMC were corrected for the viscosity of CMC when heated in water or sucrose solution without starch present (21).

RESULTS AND DISCUSSION

Figure 2 shows a DR curve average for 20 granules heated in water and compared with an amylograph viscosity curve obtained in the presence of CMC. The curve shows the two-stage viscosity increase that is typical for wheat starch.

The DR curve indicates that the most rapid starch swelling (diameter increase) occurred between 58 and 70° C, corresponding to the initiation of the first stage of viscosity increase noted in the amylograph curve. The rate of change in diameter slowed above 70° C and then increased above 80° C, corresponding to the onset of second-stage viscosity increase. Above 85° C, the granules viewed on the microscope hot stage became disorganized, collapsing and folding into distorted shapes, thus making diameter measurements meaningless beyond this point.

This granule disorganization agrees with results of Kulp (22), who determined the production of solubles for several wheat starches during heating. He showed major leaching of amylose from swollen granules to occur above 85° C. Expulsion of contents from swollen granules would account for the collapsing observed microscopically. During leaching, long chains of amylose would continue to be anchored in the granules, increasing the space occupied by each granule, and thus contribute to increased amylograph viscosity. Recent scanning electron microscopy by Miller et al (23) has clearly shown the fragmentation of starch granules during the second-stage viscosity increase.

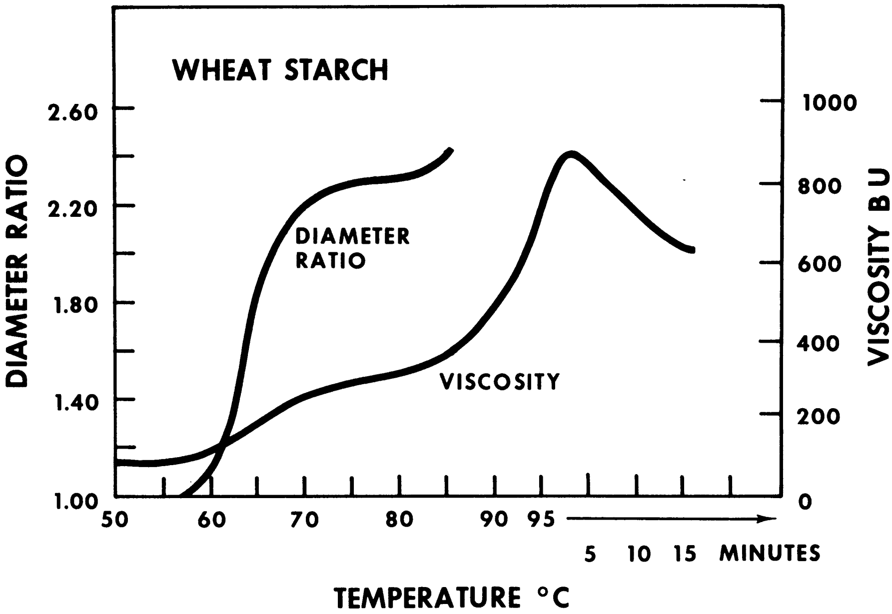

Fig. 2. Comparison of starch gelatinization in water by measurement of granule diameter change observed microscopically and amylograph viscosity obtained in presence of carboxymethylcellulose (CMC).

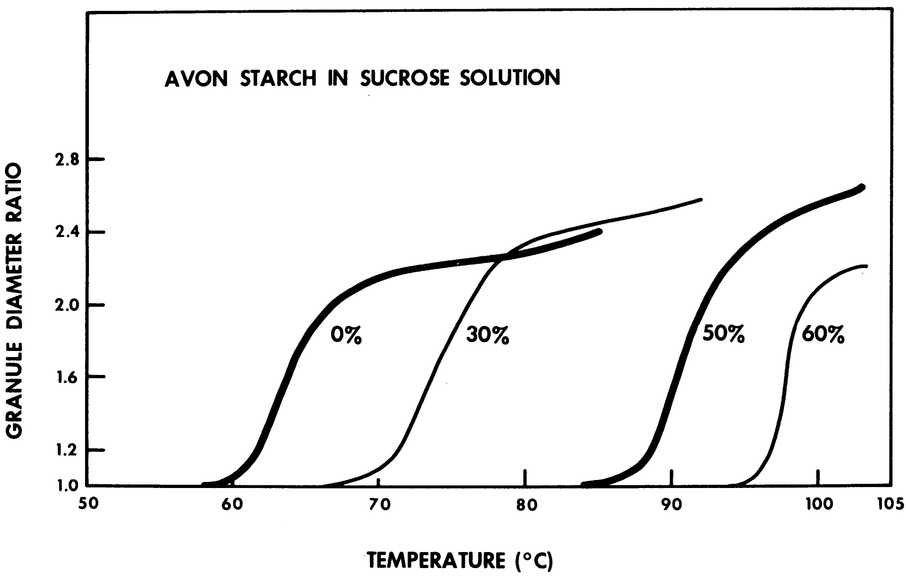

Fig. 3. Granule diameter changes in wheat starch measured on photomicrographs of starch heated in water and sucrose solutions.

Sucrose Effects on Swelling

Figure 3 shows the effects of sucrose concentration on starch swelling as measured microscopically by diameter changes. The major effect of increasing sucrose is a marked delay in the onset of swelling of the granules. In 50 and 60% sucrose solutions, it occurs above 85 and 95° C, respectively. Additionally, more apparent swelling of granules (larger DRs) occurs in 30 and 50% sucrose solutions than in water. While this increase was small, it was observed consistently throughout these studies when various starches were compared in water and in 50% sucrose.

Also, granule swelling was faster in sucrose solutions than in water. This is more clearly evident from comparison of temperature data at specific DRs as shown in Table I. From DR 1.0 to 1.2, the diameter increase was slow. From DR 1.2 to 2.0, the swelling rate increased, occurring over a narrower temperature range with increasing sucrose concentration. Apparently the energy built up in the unswollen granules at higher temperatures exerts an effect once swelling begins, contributing to the faster swelling rate and possibly to the higher swollen diameter observed before collapsing occurred.

Also shown in Table I are the temperatures for 50% loss of birefringence, determined from the photographs used for measuring DR. The loss of

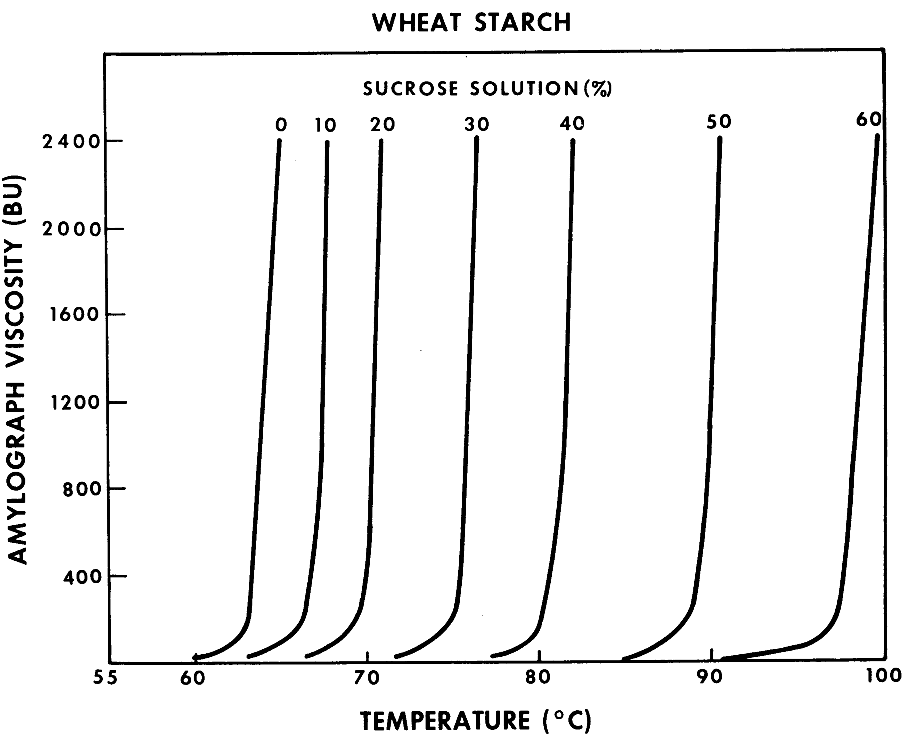

Fig. 4. Amylograph viscosity curves using 225 g of wheat starch in 450 ml of water or sucrose solution.

birefringence began when the diameter ratio indicated the first increase. Fifty percent loss occurred generally between DR 1.2 and 1.4. All birefringence was lost between DR 1.6 and 2.0.

TABLE I
Temperature History of Wheat Starch Granules Gelatinized in Sucrose Solutions[a]

Sucrose Level (%)	DR[b] 1.2 (°C)	DR 2.0 (°C)	Total Range (°C)	50% Loss of Birefringence (°C)
0	61.8	66.8	5.0	63
30	71.8	76.0	4.2	72
50	89.0	92.0	3.0	89.5
60	97.5	100.0	2.5	98

[a]From Fig. 3.
[b]DR = diameter ratio.

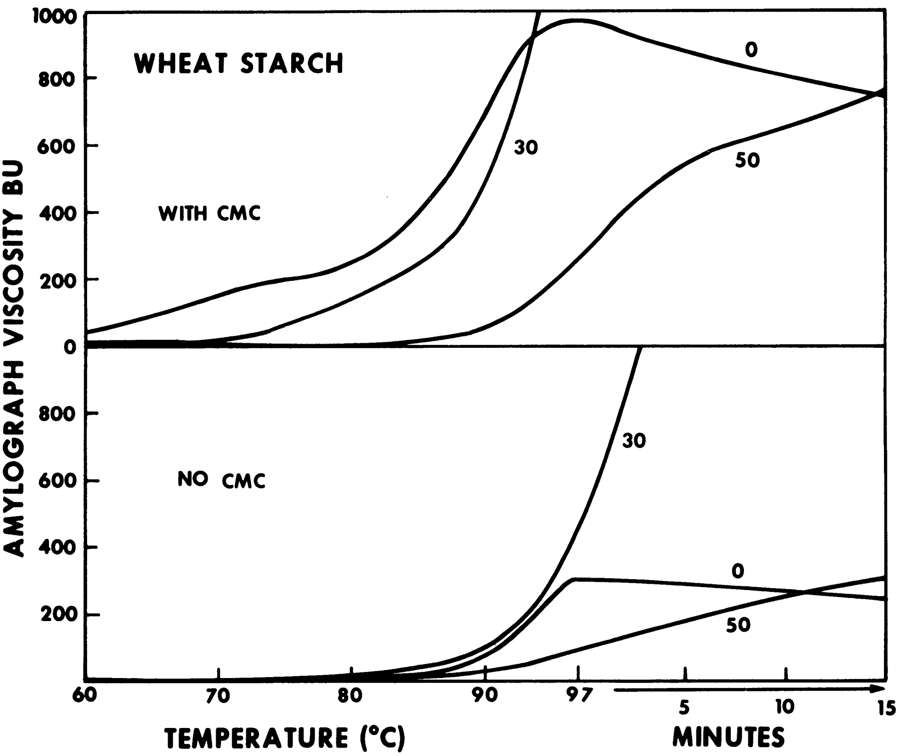

Fig. 5. Amylograph viscosity curves using 45 g of wheat starch in 450 ml water or sucrose solution. Upper set of curves obtained in presence of carboxymethylcellulose (CMC) to enhance first-stage gelatinization effect.

Sucrose and Viscosity

To relate viscosity effects to microscopic observations, viscosity increase must reflect the initial granule swelling at first-stage gelatinization. This can be achieved with high levels of starch, which create high viscosities on initiation of swelling. Figure 4 shows a series of such viscosities obtained in several sucrose solutions. The amylograph run was terminated when the viscosity exceeded the capacity of the machine. The rapid increase in viscosity occurred during the initial swelling of the granules before all birefringence was lost. The temperature range for viscosity increase corresponded with the range shown in Table I for the DR increases.

As noted earlier, the 50% sucrose solution represents a typical sugar-water mixture in layer cake formulas. Both DR and viscosity curves (Fig. 3 and 4) indicated that the onset of starch gelatinization is markedly delayed, from about 60°C in water until above 85°C in 50% sucrose, an important phenomenon in the formation of layer cake structure. These data are in agreement with reported values for the onset of gelatinization of wheat starch measured by various methods (17,18,24).

Previous reports (11–13) have suggested that sucrose inhibits starch gelatinization. From the above results, the effect would appear to be more of a delay than an inhibition. Figure 5 further develops this suggestion. Amylograph viscosity curves are shown for starch heated in water and in 30 and 50% sucrose solutions and also for the same compositions containing CMC to enhance first-stage gelatinization visualization. If viscosities obtained in water or sucrose are compared (lower set of curves), all three curves show an initial rise about the same time (within a narrow temperature range). The lower viscosity value obtained in 50% sucrose would suggest inhibition of swelling. Comparison of curves obtained in the presence of CMC (upper set) showing total gelatinization history, however, presents another view. The earlier onset of swelling in water is clearly evident by the appearance of the typical first-stage viscosity increase starting about 60°C. In the sucrose solutions, this first stage starts about 70°C in 30% sucrose and about 84°C in 50% sucrose. No clear transition point from first-stage to second-stage swelling is apparent in the sucrose curves. The rapid viscosity rise in 30% sucrose, however, suggests the beginning of second-stage swelling before the end of the heating cycle. In 50% sucrose solution, a second stage does not appear before 97°C is reached, but may account for some of the continued viscosity increase during the later part of the holding period. Extended holding curves (not shown) indicated further viscosity increases, which may be due in part to evaporation during a long holding period but also to further swelling or disruption of granules or both.

The data reported here appear to offer new insights into starch function in baked products, enhancing information on first-stage gelatinization important in layer cake systems. A subsequent article will report the relationship between effects of different sugars on starch and layer cake performance (25).

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[Received November 2, 1977. Accepted April 3, 1978]