

Dynamic Measurement of Starch Granule Swelling During Gelatinization

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ABSTRACT

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Swelling of corn starch granules during gelatinization was monitored by a new method based on particle size analysis (PSA). Using a modified PSA chamber, for which temperature could be increased at a controlled rate, a series of particle size distributions was collected throughout the gelatinization process. Swelling behavior of common corn starch, waxy maize starch, and cross-linked waxy maize starch was shown to be consistent for a given starch, yet different among the three starches. Maxi-

mum swelling power (maximum swollen granule volume per initial hydrated granule volume) was 11.2, 16.4, and 10.3 for common corn, waxy maize, and cross-linked waxy maize, respectively. Two other dynamic methods of monitoring gelatinization, differential scanning calorimetry and viscometry (Brabender Viscoamylograph) were employed for comparison to the dynamic swelling data.

The term gelatinization can be confusing because it has historically meant different things to different people. As recently as 1988, a concerted effort was made to define the term (Atwell et al 1988). The proposed definition was "... the collapse . . . of molecular orders within the starch granule manifested in irreversible changes in properties such as granular swelling, native crystallite melting, loss of birefringence, and starch solubilization." Starch gelatinization is monitored by observing the changes in these properties. Changes in viscosity, commonly determined using the Brabender Viscoamylograph, result from a combination of granule swelling and solubilization (Doublier et al 1987). Changes in heat uptake, as monitored by thermal analysis, are at least partially due to crystallite melting (Liu et al 1991). The loss of granule birefringence, as determined by polarized light microscopy, may be due to a randomization of crystallite orientation induced by the initial stages of swelling or to crystallite melting (Liu et al 1991). The increase in granule volume upon swelling may be observed directly by light (Ghiasi et al 1982) or scanning electron microscopy (Hoseney et al 1977). It may also be evaluated on a macroscopic scale after centrifugation, either by direct measurement (Hansen et al 1991) or by dye exclusion methods (Tester and Morrison 1990). Changes in viscosity, heat uptake, birefringence, and granule volume reflect different aspects of the molecular changes occurring during the gelatinization process. Consequently, the temperature range over which gelatinization is observed depends on the property being determined.

Gelatinization has been shown to be a kinetically controlled process (Slade and Levine 1988). Therefore, regardless of how gelatinization is monitored, any quantitative description of the process will be affected by changes in conditions such as the heating rate and extent of agitation. Some properties (viscosity, heat uptake, and loss of birefringence) may be monitored continuously during the process, while others (volume) are most conveniently monitored after interrupting the process (Leach et al 1959). Because gelatinization is a kinetically controlled process, this interruption, and the conditions that prevail until a measurement is made, confound the meaning of volume measured by these techniques.

Because the purpose for using starch in various applications is often related to an increase in viscosity on gelatinization, a thorough understanding of the basis for the increased viscosity is in order. An improvement in the ability to determine volume changes and solubilization during the gelatinization process would lead to improved understanding of viscosity behavior during the process.

Laser light scattering is a rapid, nonintrusive, and precise technique for particle size analysis (PSA). Measurements are based on fundamental physical properties and no calibration is required. The angle of diffraction is inversely proportional to particle size,

and the intensity of the diffracted light at any angle is a measure of the particle volume (Allen 1981). Of significance to the present study, sizing of particles suspended in water (or other liquid medium) is possible, and particle size distribution may be obtained from an analysis as brief as 1-2 sec. One significant obstacle to the measurement of granule size during heating was the lack of a technique to accurately and reproducibly control the temperature and rate of temperature increase.

The objective of this work was to develop a dynamic method to observe starch swelling behavior by monitoring changes in the particle size distribution of starch granules at intervals during the gelatinization process. For comparison, thermal behavior and changes in viscosity were also monitored dynamically during gelatinization.

MATERIALS AND METHODS

Starches

Three corn starch samples were obtained from the National Starch and Chemical Co. (Bridgewater, NJ): waxy maize (Amioca, Lot J.H.-4406), common corn starch (Melojel, Lot M.D.-8660), and cross-linked waxy maize (Clearjel A, Lot F.D.-5924).

Particle Size Analysis

PSA was conducted using a Malvern MasterSizer (Malvern Instruments Ltd., Malvern, England) laser diffraction particle size analyzer with a 300-mm range lens, to which a PS1AS small volume (15 ml), water-jacketed cell and PS3 cell support and magnetic stirrer had been adapted.

The temperature of the sample cell was regulated using a water bath circulator (Haake D8, Fisons Instruments, Paramus, NJ) externally controlled by computer (Apple Mac II, Apple Computer, Inc., Cupertino, CA; using LabView 2 software, Version 1.0, National Instruments, Austin, TX) equipped with a data acquisition and control board (NB-MIO-16L, National Instruments, Austin, TX). The temperature of the circulator was programmed by supplying a signal equivalent to 10 mV/°C (referenced to 0 mV at 0°C) to the external control input. Cell temperature was monitored with a type K thermocouple, centrally located within the cell just above the laser path.

Stock solutions of starch were prepared immediately before use by dispersing 0.9 g of starch in 1 L of vacuum-deaerated deionized water. For common corn starch and cross-linked waxy starch, 1 ml of the stock solution was diluted to 12 ml to yield a final concentration of $7.5 \times 10^{-3}\%$ (w/v). For waxy starch, 0.6 ml was diluted to 12 ml to yield $4.5 \times 10^{-3}\%$ (w/v).

For laser light scattering, the fundamental instrumental measurement is volume, and the result of an analysis is a volume distribution. Standard derived mean diameters from $D[4,3]$ to $D[1,0]$ can be computed. These derived outputs are numerical transformations assuming spherical particles (Malvern Instruments Ltd. 1990). In addition, the median diameter, $D[v,0.5]$ (i.e., the size at which 50% of the particles by volume are smaller and 50% larger), is also calculated. In this study, $D[v,0.5]$ was chosen, *vide infra*, as representative of the starch granule size.

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Thermal Analysis by Differential Scanning Calorimetry

Thermal analysis was performed using a differential scanning calorimeter (model DSC-4, Perkin-Elmer, Norwalk, CT). Starch powder samples (~3 mg) were placed in preweighed coated aluminum hermetic pans (90001-201, DuPont, Wilmington, DE), and the precise weight was determined (Autobalance, Perkin-Elmer). Deionized water was added to give a dry starch content of 30%, and the pan was sealed. The reference pan contained deionized water. Samples were scanned from 15–115°C at a rate of 5°C/min. Temperature was calibrated using indium. As specified by the manufacturer, the scanning rate resolution was 0.1°C/min. Endotherms were analyzed (Thermal Analysis Data Station, Perkin-Elmer) to calculate peak onset temperature (T_o) and peak maximum temperature (T_{max}).

Viscoamylograph Viscosity

Starch samples were prepared at a concentration of 7% (w/w) in deionized water. Slurries were heated from 45 to 90°C at a rate of 1.5°C/min, held for 30 min at 90°C, cooled to 45°C at 1.5°C/min, and held at 45°C for 30 min in a viscoamylograph (model VA-1A, C. W. Brabender Instruments, Inc., South Hackensack, NJ) fitted with a 700-cm³ cartridge. Brabender units versus time were plotted.

Statistical Procedures

Third-order polynomial regression of temperature-particle size data and linear regression of time-temperature data was performed (Cricket Graph 1.3 for the Apple Macintosh, Computer Associates, San Diego, CA). A two-tailed Student's *t* test for independent samples with unequal variance was used for comparison of mean transition temperatures. Analysis of variance of initial and maximum median particle diameters was performed (Minitab Statistical Software, Macintosh Ver. 8.2, Minitab Inc., State College, PA).

RESULTS AND DISCUSSION

Success of the method depended on the ability to accurately and reproducibly control the temperature of the sample cell for PSA. This was accomplished using a water-jacketed measurement cell and a circulating water bath controlled externally by computer. Figure 1 is a typical time-temperature profile of a starch sample during heating. After an initial lag, the rate of temperature increase was linear between 40 and 80°C ($R^2 \geq 0.99$, $n = 12$). Two heating rates were selected. When set at 5.0°C/min, for comparison to thermal analysis data obtained by differential scanning calorimetry (DSC), the measured heating rate within the PSA cell was $4.76 \pm 0.10^\circ\text{C}/\text{min}$. When set at 1.5°C/min, for comparison to viscosity changes determined using the

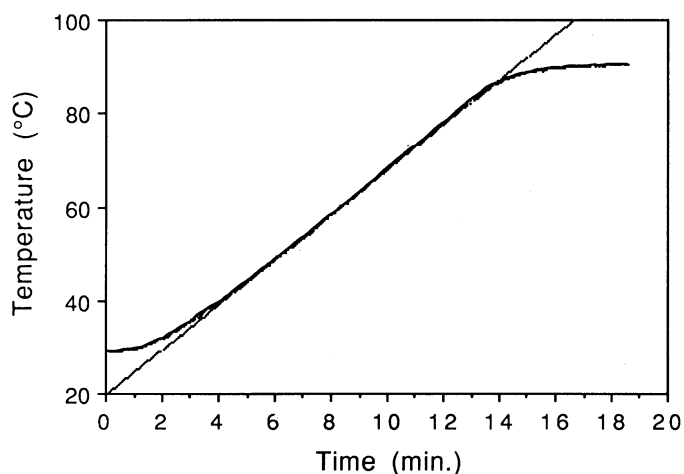


Fig. 1. Heating curve from direct measurement of temperature within the particle size analyzer cell. Straight line = linear regression of data at 40–80°C.

Brabender Viscoamylograph, the measured heating rate was $1.436 \pm 0.004^\circ\text{C}/\text{min}$.

Laser scattering PSA requires that the suspending medium and the particle to be measured be optically distinct, i.e., that they have different refractive indices, and that the suspending medium be transparent to the laser light. The fraction of light scattered away from the main beam by the sample is referred to as the obscuration. Ideally, the obscuration ranges from 0.1 to 0.3, but the usable range typically extends to 0.5 (Malvern Instruments Ltd. 1990). Temperature gradients may cause large-scale fluctuations in the refractive index of the suspending medium. Such variations in the refractive index scatter light at low angles and appear as very large particles, a phenomenon known as “beam steering.” Depending on the particle size distribution of interest, measurements in the presence of such fluctuations can still be made (Malvern Instruments Ltd. 1990).

The dynamic heating regime used in these experiments resulted in temperature gradients within the measurement cell and, therefore, some degree of beam steering. The problem increased as the heating rate was increased. Beam steering was excessive at heating rates above 5°C/min. Stirring the sample helped reduce temperature gradients, but high stirring speeds caused vortices that interfered with size measurement. Therefore, a stirrer speed was selected that minimized spurious low-angle scattering. Experiments were conducted with an initial obscuration of approximately 0.2, corresponding to an initial volume fraction of starch granules equal to $0.0134 \pm 0.0020\%$ ($n = 12$).

Using the software provided with the particle size analyzer, data resulting from spurious low-angle scattering can be suppressed before calculating the particle size distribution. Accurate sizing can be accomplished, provided the suppressed data does not overlap the desired data. To eliminate the measurement error contributed by beam steering, data from the lowest angles (the first 10 rings of the 32-ring detector) were suppressed before size calculation. Lower angles correspond to larger particles; suppressing the low-angle data would have the greatest potential effect on the measurement of swollen granules. To quantify the effect of data suppression on the measured starch granule size, several measurements were made with and without suppression once the temperature had reached its maximum (~90°C) and temperature

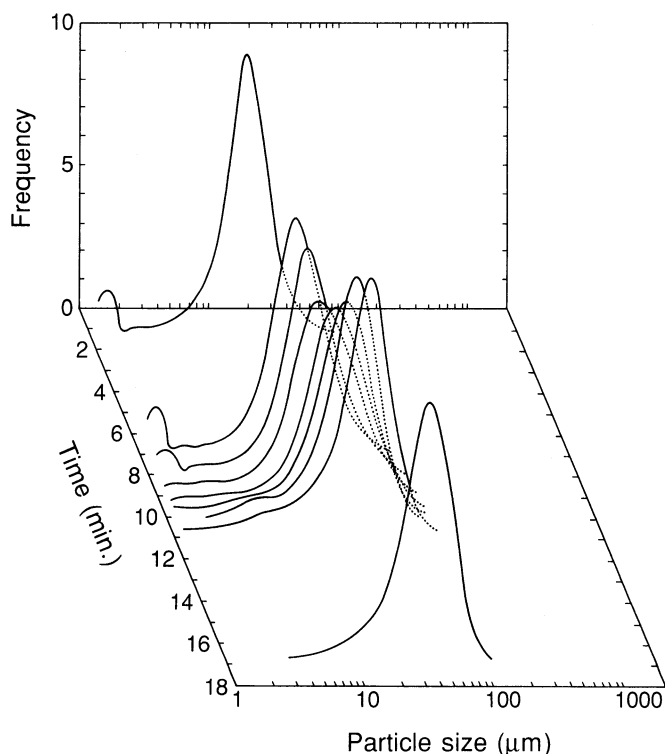


Fig. 2. Particle size frequency distributions for a sample of common corn starch during heating at 4.8°C/min.

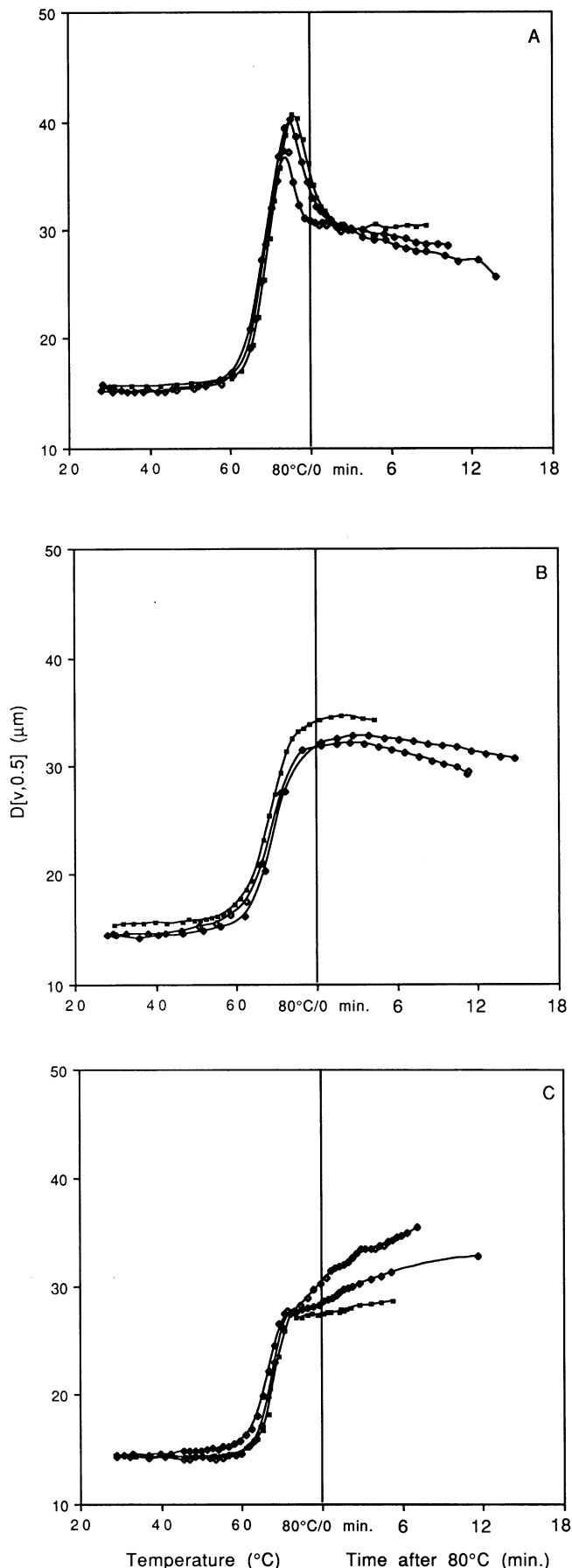


Fig. 3. Median particle size, $D[v,0.5]$, as a function of temperature and time. A, Waxy corn starch. B, Common corn starch. C, Cross-linked waxy corn starch. Average heating rate $4.76 \pm 0.10^\circ\text{C}/\text{min}$ at $40\text{--}80^\circ\text{C}$. Data are for three replicate runs.

gradients and beam steering were no longer a problem. Suppression of the data from the first 10 rings did not substantially compromise accuracy: the calculated median granule diameter ($D[v,0.5]$) differed by $<1\%$ when the selected data were suppressed. Therefore, spurious data caused by temperature gradients could be eliminated without affecting the diffraction data resulting from starch granules.

When measuring granule size during gelatinization, the obscuration peaked between 0.6 and 0.8. This occurred when temperature gradients were the greatest. Roughly 50% of this obscuration was attributable to beam steering, i.e., $\sim 50\%$ of the scattered light struck the first 10 rings of the detector. Therefore, the obscuration resulting from the starch granules was between 0.3 and 0.4, within the usable range.

An example of the particle size frequency distributions obtained during the dynamic measurement of starch granule swelling is presented in Figure 2. The change in the peak shape as swelling proceeds clearly illustrates the diverse swelling behavior of the population of granules. Comparing the first and last distributions, we noticed the disappearance of a small fraction of particles in the size range below $2\ \mu\text{m}$. The disappearance of this fraction is consistent with the interpretation that it comprised granule fragments that disintegrated upon heating. Plots of mean ($D[4,3]$) granule diameter versus temperature were similar to those of median ($D[v,0.5]$) granule diameter versus temperature. However, the scatter about the regression line was less for $D[v,0.5]$, which led to a more accurate calculation of transition temperatures.

Particle size-temperature curves for waxy maize, common corn, and cross-linked waxy maize starches are presented in Figure 3A, B, and C, respectively. Different swelling behaviors were observed for each starch type, and each behavior was reproducible within a type. $D[v,0.5]$ for waxy maize starch (Fig. 3A) increased sharply at $\sim 60^\circ\text{C}$, reached a maximum at $\sim 75^\circ\text{C}$, and then decreased dramatically. For common corn starch (Fig. 3B) $D[v,0.5]$ increased slightly at $\sim 50^\circ\text{C}$, increased sharply at 60°C , and reached a maximum around 90°C ; $D[v,0.5]$ then decreased, but at a much slower rate and to a lesser extent than that of waxy maize starch. For cross-linked waxy maize starch (Fig. 3C) the increase in $D[v,0.5]$ resembled that of waxy maize starch up to 75°C ; in contrast to waxy maize starch, $D[v,0.5]$ for cross-linked waxy maize continued to increase above 75°C , albeit more slowly.

$D[v,0.5]$ is a characteristic of a population of granules and may not reflect the specific behavior of an individual granule. Consequently, at least two different interpretations of a decrease in $D[v,0.5]$ at high temperatures are possible: granules of all sizes could be losing granular integrity, resulting in a mixture of soluble starch and granule fragments; or, larger granules could be preferentially broken down, leaving a population of smaller intact granules.

Leach et al (1959) reported that starch swelling occurs in two stages. Kinetic models have even been proposed to describe such behavior (Kokini et al 1992). However, they were based on the

TABLE I
Initial and Maximum Median Particle Diameters and Maximum Swelling Power of Waxy Maize, Common Corn, and Cross-Linked Waxy Maize Starches

Starch	Particle Size		Maximum Swelling Power
	D_i (μm) ^a	D_{max} (μm) ^b	$(D_{\text{max}})^3/(D_i)^3$ ^c
Waxy maize	15.61 ± 0.24 a ^d	39.63 ± 1.62 a	16.4
Common corn	14.87 ± 0.38 b	33.32 ± 1.20 b	11.2
Cross-linked waxy	14.46 ± 0.07 b	31.49 ± 3.20 b	10.3

^aInitial diameter.

^bMaximum diameter.

^cMaximum swelling power is the ratio of the maximum volume to the initial volume, calculated as the ratio of the maximum diameter cubed to the initial diameter cubed.

^dComparisons of D_i and D_{max} between starch types. Means followed by the same letter are not significantly different, $P < 0.05$.

measurement of swelling power that was determined by holding the sample at a series of discrete temperatures. Two-stage swelling of starch during the dynamic heating regime was not observed in this study.

Both the initial $D[v,0.5]$ of waxy maize starch dispersed in cold water and the maximum $D[v,0.5]$ upon heating were greater ($P \leq 0.001$) than those of common corn starch or cross-linked waxy maize starch (Table I). The maximum swelling power, calculated as the ratio $(D_{\max})^3/(D_i)^3$, of waxy maize starch was 16.4, compared to 11.2 and 10.3 for common corn starch and cross-linked waxy maize, respectively. Equal volume fractions ($0.0134 \pm 0.0020\%$) of starch were obtained when either $7.5 \times 10^{-3}\%$ (w/v) of common corn starch or cross-linked waxy maize starch or $4.5 \times 10^{-3}\%$ (w/v) of waxy maize starch were added to cold water for PSA, implying that the granule density in water (the weight of dry starch per starch granule volume in water) was less for waxy maize starch. The density referred to here is not the buoyant density obtained from displacement of a non-swelling organic liquid nor the apparent density calculated as the inverse of the specific volume (the increase in suspension volume per g of added starch) (French 1984). Instead, it is analogous to the density of a polymer network in the total volume of a gel and could be referred to as a network density. The observed difference in network density may have resulted from a greater initial uptake of water when waxy maize was dispersed in cold water, which may also explain the greater initial granule diameter.

To compare these results with those obtained from DSC, it was necessary to calculate some characteristic transition temperature for the swelling data. The temperature at which the maximum rate of change in granule size was observed, which we term T_{\max} , is mathematically analogous to the peak maximum of the endotherm obtained from DSC. Swelling T_{\max} was calculated from the size-temperature data of Figure 3 by first fitting the data to a third-order polynomial: $D[v,0.5] = b_0 + b_1T + b_2T^2 + b_3T^3$, then calculating the temperature at which the second derivative of the polynomial was equal to zero: $T_{\max} = -1/3(b_2/b_3)$. Figure 4 is an example of the calculation of T_{\max} for one sample of common corn starch. For the polynomial regression of $D[v,0.5]$ and temperature, R^2 was ≥ 0.99 in eight of the 10 samples. For the remaining two samples, R^2 was ≥ 0.98 .

Temperature maxima obtained from PSA and DSC are compared in Table II. These data were generated at the same nominal heating rate ($5^\circ\text{C}/\text{min}$). Although starch concentrations were very different, Yuan et al (1993) did not observe differences in DSC temperature parameters for the same starch at 10 and 30% concen-

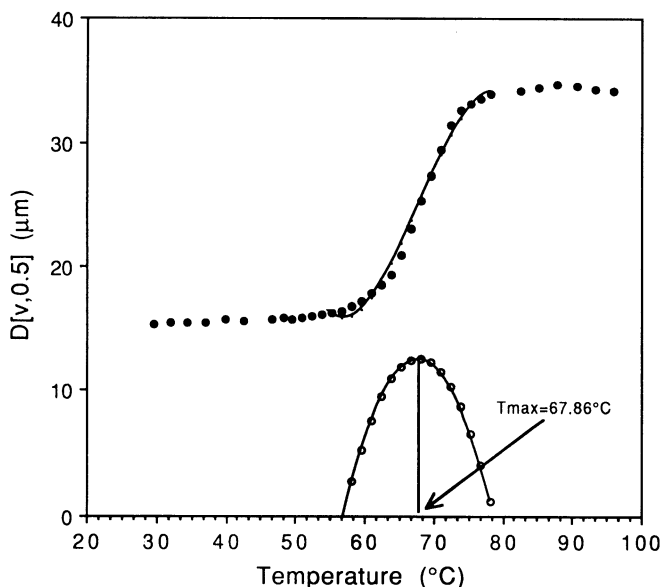


Fig. 4. Determination of T_{\max} for starch swelling from diameter-temperature data (\bullet) for a sample of common corn starch heated at $4.8^\circ\text{C}/\text{min}$. R^2 for the 3rd order polynomial regression of $D[v,0.5]$ vs. temperature was 0.993. \circ = 1st derivative of the polynomial.

tration. Apparently the changes monitored by DSC are not influenced by starch concentration at or below 30%, probably because water is present in excess of that needed to accomplish the transition under these conditions. For all three starches, the DSC T_{\max} was numerically greater than the PSA T_{\max} ; the difference was significant only for the waxy and cross-linked waxy starches. These data suggest that swelling events at least partially precede thermal events. For amylose-lipid complexes, Biliaderis (1991, 1992) showed that DSC monitors both crystallite melting and conformational disordering of helices. The latter appears to make the greater contribution to ΔH for the transition. Similarly, Cooke and Gidley (1992) suggested that enthalpy of gelatinization results more from loss of double helix than from loss of crystallinity. It is possible that the loss of crystallite structure could be associated with initial stages of swelling, and that loss of double helical character could be associated with the latter stages of swelling. On the other hand, it is possible that heat transfer was not as efficient to the contents of the DSC pan as it was to the individual granules dispersed in water. The resulting time lag might contribute to the lower swelling T_{\max} . In this respect, the DSC onset temperature, T_o , might provide insight because it is less dependent on heating rate than T_{\max} is. For the two unmodified starches, DSC T_o values were numerically lower than the PSA T_{\max} values. Assuming a homogeneous population of granules, we infer that melting events at least partially precede swelling for these starches. The behavior of the cross-linked starch may differ in this respect (Table II).

A comparison between swelling data and viscoamylograph viscosity data is shown in Figure 5A-C for the three starch samples. For this comparison, the nominal heating rate was fixed at $1.5^\circ\text{C}/\text{min}$. The viscosity profile from the viscoamylograph is strongly influenced by concentration (Rasper 1982). We chose a concentration of 7% (w/w) because this concentration was the highest that would allow the waxy starch sample to be on scale. The shearing forces are assumed to be very different in the PSA and the viscoamylograph. Consequently, comparison of the data from these two techniques has no absolute meaning for a particular starch sample. Nevertheless, a comparison of the PSA and viscoamylograph data between the three starch samples, treated identically, may shed light on the behavior of these starches.

The increase in viscoamylograph viscosity followed the increase in swelling within $\sim 10^\circ\text{C}$ for both the waxy and the cross-linked waxy starches. On the other hand, the lag for common corn starch was much greater, $\sim 25^\circ\text{C}$. This difference would be consistent with the concept that leached amylose makes an important contribution to viscosity for common corn starch.

For waxy maize starch, the size decrease at $\sim 75^\circ\text{C}$ is paralleled by a viscosity decrease soon after. Loss of granule integrity could account for both observations. For cross-linked waxy maize, no size decrease was observed, even on holding at 90°C . Viscosity continued to increase slightly when holding at that temperature. Maintenance of granule integrity would account for these observations. Steeneken (1989) suggested that a simple relationship between swelling and viscosity for waxy maize and cross-linked waxy maize may be confounded by a concentration dependence that differs according to the rigidity of the two types of swollen granules. Thus, we must be cautious in interpreting viscosity data based on a single concentration.

TABLE II
Comparison of Transition Temperatures from Particle Size Analysis (PSA) and Differential Scanning Calorimetry (DSC)^a

Starch	PSA ^b		DSC ^c	
	T_{\max}		T_{\max}	T_{onset}
Waxy corn	69.01 ± 0.88 a		72.7 ± 0.19 b	67.9 ± 0.19
Common corn	69.51 ± 1.44 a		70.4 ± 0.09 a	65.8 ± 0.13
Cross-linked waxy	67.28 ± 1.09 a		73.2 ± 0.17 b	68.4 ± 0.15

^aComparisons of T_{\max} within each starch type. Means followed by the same letter are not significantly different, $P < 0.05$ ($n = 3$, except for PSA of common corn starch, where $n = 4$).

^bHeating rate $4.76 \pm 0.10^\circ\text{C}/\text{min}$.

^cHeating rate $5.0^\circ\text{C}/\text{min}$.

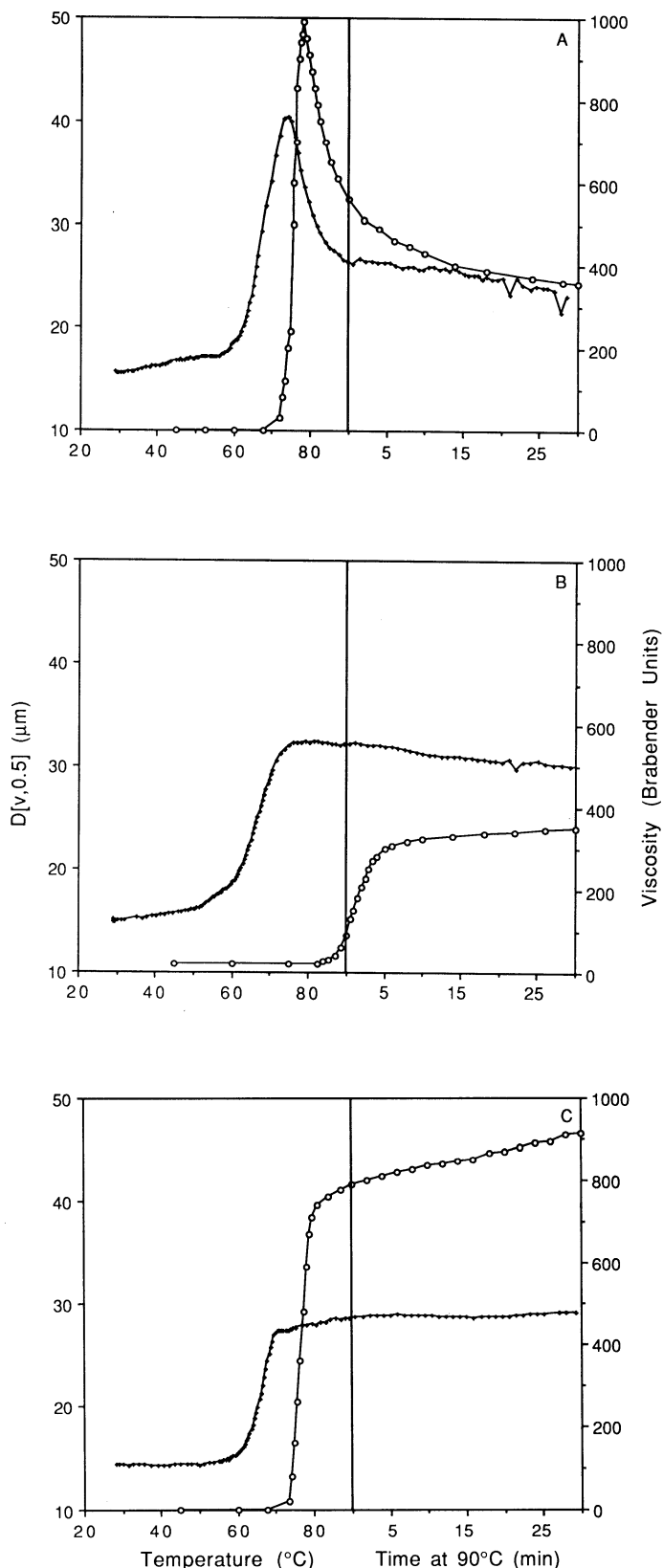


Fig. 5. Granule swelling relative to viscosity. A, Waxy corn starch. B, Common corn starch. C, Cross-linked waxy corn starch. \circ = Brabender units.

CONCLUSIONS

The PSA method described here allows starch granule swelling to be monitored dynamically during gelatinization. When combined with dynamic methods that monitor physical changes other than swelling, this method may lead to increased understanding of the nature of the gelatinization process.

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