

Electrical Conductance Study of Fluid Motion and Heat Transport During Starch Gelatinization

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ABSTRACT: An electrical conductance technique was employed with remarkable success in investigating the water dispersion during the gelatinization of starch suspensions. Water dispersion was realized by *instantaneous* conductance time records of the electrically accessible water in the samples. Different pasting procedures were examined and it was shown that problems with fluid motion and heat transport could strongly influence the water dispersion upon gelatinization. Furthermore, it was found that, under well mixed conditions at low shear, the water distribution at the end of gelatinization did not depend either on the employed heating rates or temperatures, thus implying that conductance measurements may have the potential to accurately quantify the extent of gelatinization.

Key Words: starch gelatinization, electrical conductivity, water dispersion, pasting, granule swelling

Introduction

THE GELATINIZATION OF STARCH GRANULES IS AN IMPORTANT phenomenon that occurs in many food processing operations, such as the baking of bread and cakes, extrusion cooking of cereal based products, production of soups, etc. During gelatinization, starch granules absorb water upon heating and form either a viscous solution or a gel depending on the amount of water present. Gelatinization in its broadest terms is attended by 2 principal phenomena. First, a loss of crystalline order; second, a swelling of the amorphous granules many times their original size. Although many aspects of starch gelatinization have been extensively studied, comparatively little work has been devoted to give a coherent account on the time-dependent role of water in granule swelling at a fundamental physicochemical level (Slade and Levine 1991).

Several methods have been employed in the past to register gelatinization phenomena. (Lund 1984). A major part of this literature was devoted to methods for studying *rheological* properties during gelatinization since changes in viscosity are of paramount importance for practical applications. However, the review of instruments shows that there is a dependence of rheological readings from specific instrumentation (Dolan and Steffe 1990). Besides, several authors have pointed out the limited knowledge of the types of rheological changes which occur in aqueous starch suspensions when heated (Lund 1989).

Another extensively exploited method is the Differential Scanning Calorimetry (DSC), which has been used to study the enthalpy required to overcome intermolecular bonds on gelatinization, e.g., Biliaderis and others (1980) and Russel (1987). Although the information obtained by DSC studies is very significant, mainly due to the well-controlled temperature conditions, several drawbacks are recognized. The method is not online in the sense that it involves batch samples that are treated progressively. Also, due to fast sedimentation of starch granules in cold water (Schierbaum and others 1962), it is very difficult to avoid stratification inside the DSC crucibles when working with suspensions at low temperatures. On the other hand, if a slurry or a gel is placed inside the crucibles, it is often hard to achieve reproducible contact with the solid walls and to avoid air pockets in the bulk of the sample. Errors from these sources may be appreciable considering the very small size of the samples.

Thus, the need for an alternative technique able to isolate the significant contribution of water on gelatinization and measure it

explicitly and accurately is obvious. Measurement of the effective electrical conductivity of starch suspensions appears to be a tempting option. The dielectric properties of a polymer-water system depend upon the proportion and state of the water component as well as on the polarization and conductivity of other substances present in the system (Hasted 1973; Hedvig 1977). In food polymers, such as starch, water can exist as strongly bound water characterized by strong molecular interactions with the matrix and as weakly bound (free) water present in capillaries and microcavities within the polymer (Hardman 1985). In order to study the dispersion of free water during gelatinization, a modified version of a conductance technique, originally developed by Tsochatzidis and others (1992) to study flow characteristics in pipes and packed beds, was employed here. This is a non-intrusive technique characterized by satisfactory accuracy and stability and it is particularly sensitive to liquid volume fraction. Another advantage of the technique is that it is so simple and robust that may be employed for online measurements in processing equipments.

The only other study that was found regarding quantitative electrical conductance measurements during starch gelatinization is the recent work by Wang and Sastry (1997), concerning ohmic heating of starch suspensions. Ohmic heating is a food processing operation in which heat is internally generated within foods by the passage of high voltage alternating electric current (100 to 200 V, 50 to 60Hz; Halden and others 1990). Wang and Sastry (1997) observed that during ohmic heating, electrical conductivity changes are accompanied by structure changes due to starch gelatinization.

A primary objective of this work was to assess a novel conductance technique as a potential diagnostic tool for *continuous* monitoring of starch gelatinization phenomena. Another objective was to use this technique to investigate problems related to fluid motion (agitation) and heat transport during pasting. Among the several parameters that were suspected to be important in gelatinization, here only the influence of different pasting procedures and mixing/heating patterns were examined.

Results and Discussion

TO PRESENT AND COMPARE THE EXPERIMENTAL DATA FROM various experiments, it is advisable to normalize the apparent conductance of the mixture $-K_{app}(T)-$ measurements with the conductance of the test section full of water $-K_{max}(T)-$, in order to

eliminate errors in liquid conductivity measurements. This also allows differentiation between actual gelatinization structural changes and contribution of temperature to the effective electrical conductivity of the mixture. The effective electrical conductivity γ_e , of the mixture is a function of the continuous conducting phase (liquid) conductivity γ , and the liquid volume fraction β . Starch granules are the dispersed substance here. As electrical potential field theory shows, i.e., (Coney 1973), for uniformly distributed systems the ratio γ_e/γ is equal to the ratio K_{app}/K_{max} . For natural starch with a density around 1.5 to 1.53 g/cm³ (Pearson 1976), a 10% w/w mixture corresponds to a liquid volume fraction of approx. 0.92. Working with a three phase system and liquid volume fractions close to 0.30, Karapantsios and others (1993) showed that K_{app}/K_{max} falls between predictions of Maxwell (1954), Eq (1), and Begovich & Watson (1978), Eq (2):

$$\gamma_e/\gamma = 2\beta/(3 - \beta) \quad (1)$$

$$\gamma_e/\gamma = \beta \quad (2)$$

with a tendency towards the latter as β increases. Thus, for $K_{app}/K_{max} \approx 0.92$ (mixture at ambient temperature) the liquid volume fractions are $\beta_{[1]} = 0.95$ and $\beta_{[2]} = 0.92$, respectively. Similar considerations were reported in recent studies aiming to determine the *thermal conductivity* of starch-water gels (Sakiyama and others 1993; Morley and Milles 1997). The work of Sakiyama and others (1993) provided strong support for the series model (Eq (2)). On the other hand, Morley and Miles (1997) showed that measurements lie roughly between curves based on the Maxwell model and the series model whose predictions are, in fact, quite closely spaced apart especially at high liquid volume fractions. It should be added here that Eq (2) was originally developed for three-phase fluidized beds where the nonconducting (gas+solid) medium did not have rigid boundaries, a case which also holds for the swollen starch granules (Christianson and Bagley 1982). Furthermore, it is interesting to compare estimations of liquid volume fraction by Eq (1) and Eq (2) with those calculated by centrifuging the suspension, customary designated as 1-cQ (Bagley and Christianson 1982) where c is the concentration of starch and Q is the weight of swollen starch granules divided by the dry weight of starch prior to cooking. It is hoped that elec-

trical measurements can sense liquid dispersion down to a smaller scale (in the inter- and intra-particle interstices) involving also the water that is connected by tiny capillary liquid bridges and may, therefore, be difficult to remove by centrifugation. Moreover, one can not exclude the possibility that swollen particles may deform and compress under centrifugation (Evans and Haisman 1979) and discharge sizable amount of originally imbibed water. In the present study, the short heating times (< 15 min) employed to bring the cold suspension to gelatinization allows for very limited initial cold water sorption. Therefore, a rather constant value of K_{app}/K_{max} is expected at temperatures below gelatinization.

Flash gelatinization

Fig. 1 presents reduced conductance data, K_{app}/K_{max} , obtained at a target mixture temperature of 80 °C, but from 2 different experiments: one involves instant mixing of a 40 °C suspension with hot water at 97 °C and the other instant mixing of a 54 °C suspension with hot water at 92 °C. In both curves, a steep increase is observed immediately at the moment of mixing, followed by a gradual decrease. However, both the peaks and the subsequent declines have different conductance (and therefore liquid volume fraction) values, indicating a strong influence of initial mixing conditions on pasting. A similar behavior was also observed with other target temperatures and mixing combinations.

Mixer configuration (A), 500 rpm

This agitation scheme simulates to a good extent the often-employed stirring procedure by either a magnetic spin bar or a single bottom impeller, operated at a *constant* high rotation speed, e.g., Wong and Lelievre (1981), Okenchukwu and Rao (1996), and Zamora (1995). Fig. 2 depicts a typical apparent conductance trace with a 150 Watts heating power together with the corresponding thermal history of the sample. After climbing to about 70 °C, the temperature shows no remarkable change. As the temperature initially rises, the conductance increases almost linearly with time up to about 62 °C which appears to be the onset temperature of gelatinization. After this point, conductance continues to rise at a lower rate and attains a maximum value at about 70 °C, where after it presents a gradually decreasing trend. The conductance signal is quite smooth and its reproducibility is

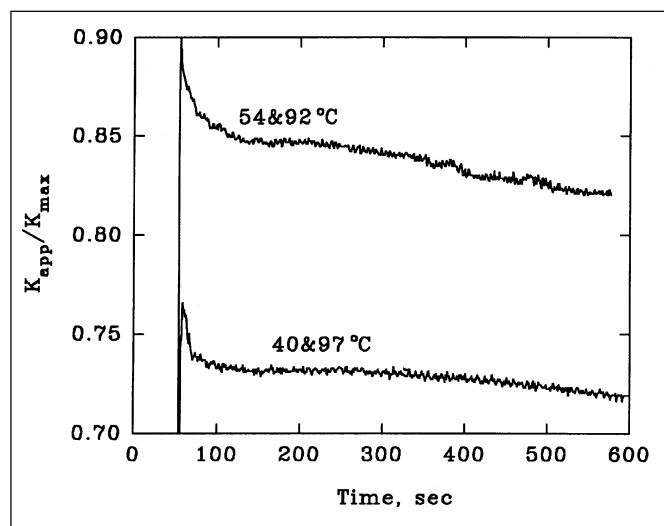


Fig. 1—Reduced apparent conductance signals, K_{app}/K_{max} , vs time taken with the flash gelatinization procedure using 2 different blending combinations that both lead to a 80 °C final mixture. One involves mixing of a 40 °C suspension with 97 °C hot water and the other mixing of a 54 °C suspension with 92 °C hot water.

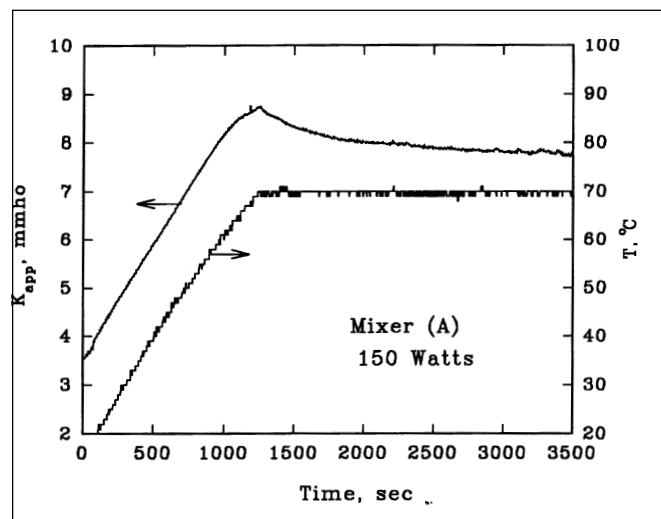


Fig. 2—Apparent conductance signal, K_{app} , vs time and the corresponding temperature profile taken with mixer configuration A and power 150 Watts. Mixer speed at 500 rpm throughout.

excellent; some low noise level may be attributed to inherent features of the fluid motion during agitation rather than to imperfections of the experimental system.

Figure 3 illustrates the reduced apparent conductance, K_{app}/K_{max} , trace of Fig. 2 as well as traces obtained at 300 and 600 Watts heating powers. The respective temperature profiles, not shown due to space limitations, are the same as that in Fig. 2, that is, the temperature after reaching about 70 °C remains essentially constant. It is apparent that the observed decay in the reduced conductance curves depends strongly on heating rate. This is rather unexpected for such dilute starch/water systems and may indicate that just by reaching the temperature where gelatinization is expected to conclude (about 70 °C for maize starch) might not be enough to complete gelatinization. This might be so if the necessary amount of energy is not supplied due to inadequate heat transport. The initial conductance fast decay, following the onset of gelatinization, turns always to a rather gradual decrease. Examination of samples taken at various times during heating, showed that even at the end of these runs, gelatinization was not completed. Unfortunately, due to increasing evaporation losses, it was not possible to extend the duration of these experiments to much longer times. The K_{app}/K_{max} values around 0.80, at the end of the experiments, indicate that the electrically accessible liquid volume fraction $-\beta_{[1]} = 0.86$, $\beta_{[2]} = 0.80$ - was still quite appreciable. Slow water diffusion phenomena from the bulk to the starch granules might be responsible for such a behavior; a more detailed discussion is presented in a subsequent section.

Two distinct texture regions were identified in the samples. Around the impellers, where the fluid was kept always in motion, the paste developed a semitransparent appearance with considerable fluidity (low consistency). It is possible that the intense centrifugal action of the impellers might be capable of segregating the solids by expelling them towards the vessel walls while leaving only exuded water at the region of agitation. Analogous hydro-lubrication phenomena are frequently met in mixing particulate solids with liquids, i.e., ceramics, concrete mixing, powder metallurgy (Beddow 1980). Furthermore, it is recognized that starch gels under the action of external forces exhibit excessive syneresis phenomena (Stainsby and others 1984). It might be also possible that at least a part of this low consistency texture is due to a substantial degradation of starch granules by the local high shear action.

Away from the impellers, between the measuring rings, the

fluid hardly moved and the paste was opaque, bright white, and considerably firm (high consistency). It is probable that with this particular mixing scheme (mixer A, 500 rpm), complete gelatinization throughout the sample mass is retarded due to poor agitation. Thus, serious concerns might be expressed about all experiments that involve high mixing rates applied locally, e.g., Doublier (1981).

Mixer configuration (B), 330 40 rpm

In an effort to improve circulation inside the test section, a third impeller was incorporated. This created excessive vortexing at low temperatures, so initial agitation was reduced to 330 rpm. As soon as the conductance signal reached a maximum value, the rotation rate was adjusted to 40 rpm to maintain sufficient mixing without damaging starch granules (Xu and Raphaelides 1998). Fig. 4 shows reduced apparent conductance data obtained at 150 Watts. The respective thermal profile (not shown) was the same as in Fig. 2 and 3. Interestingly, the major part of the conductance curve looks qualitatively similar to curves of Fig. 3. Yet, after the initial sharp decay, a spurious signal is detected marked by large amplitude undulations. This peculiar form may be ascribed to poor homogeneity in the mixture which results in lumps of moving material with varying concentrations and electrical properties. Nevertheless, these deviations are a proof that the present mixer configuration causes some limited circulation inside the vessel, contrary to the rather immobile samples of mixer configuration (A).

Mixer configuration (C), 260 40, 160, 260 rpm

The motion pattern of this mixer's beams covers almost the entire cross-section of the vessel. Therefore it is expected to maintain well-mixed conditions throughout the mass of the sample. Due to vortexing problems the initial mixing rate was further reduced to 260 rpm. Fig. 5 presents reduced conductance curves, K_{app}/K_{max} , along with the original conductance traces, K_{app} . These data were obtained from three experiments all at the same heating power (150 Watts) but different final mixing rates (40, 160, 260 rpm). The insets in the graphs display the corresponding temperature profiles. In all these runs, the temperature of the samples rises gradually beyond 70 °C. The higher the final mixing rate the higher the temperature rise. This may be due to a progressively better fluid agitation and better heat transport from the heater to the bulk; much better than with any of the

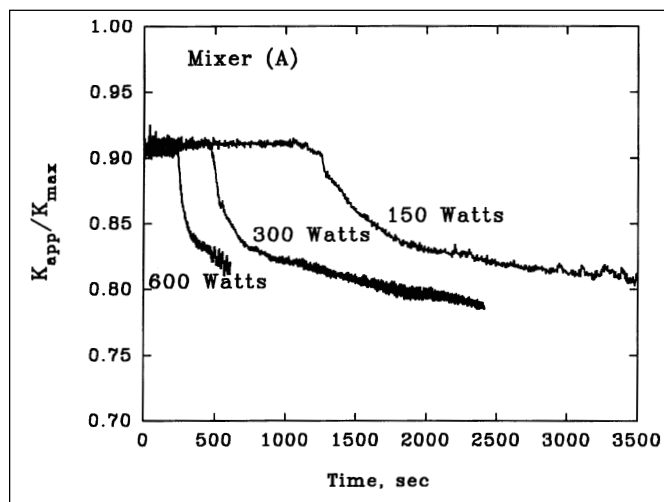


Fig. 3—Reduced apparent conductance signals, K_{app}/K_{max} , vs time taken with mixer configuration A and power 150, 300, and 600 Watts. Mixer speed at 500 rpm throughout. The temperature profiles of all runs are the same with that in Fig. 2.

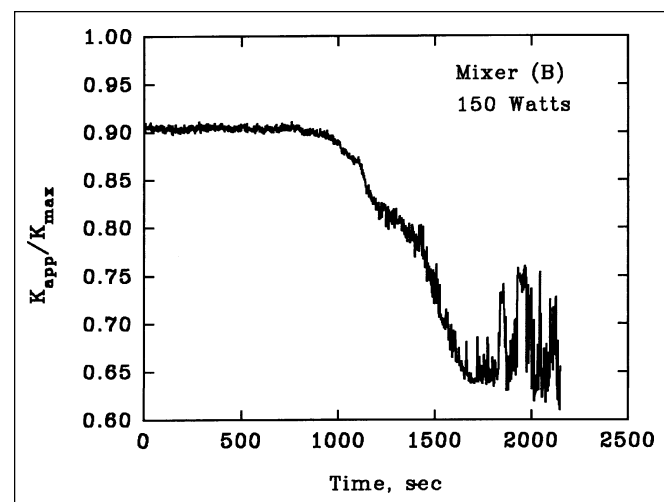


Fig. 4—Reduced apparent conductance signal, K_{app}/K_{max} , vs time taken with mixer configuration B and power 150 Watts. Mixer speed initially at 330 rpm and at 70 °C adjusted to 40 rpm. The temperature profile is the same with that in Fig. 2.

previous mixer configurations.

The conductance curves in Fig. 5 are quite smooth having the same overall appearance as those in Fig. 2 and 3. However, unlike in previous runs, with the present mixer configuration (C), a constant value (plateau) was ultimately reached in longer times. The conductance plateaus are located at $K_{app}/K_{max} = 0.70$, roughly the same value for all mixing rates (s.d. = 0.01). Examination of samples taken during the experiments showed that when the plateau is reached, gelatinization is complete. The different mixing rates produce fairly different textures in the mixture. At 40 rpm the starch paste appears to be as opaque, white and firm as in the samples of mixer configuration (A) - away from the impellers. At 160 and 260 rpm, the mixture is still opaque and white but with a somewhat less firmness.

Fig. 6 presents results from experiments similar to those depicted in Fig. 5 but with 300 Watts heating power. The qualitative resemblance between curves of Figs. 5 and 6 is remarkable if one notices the different temperature profiles of the samples. This is

more striking at final mixing rates of 160 and 260 rpm, since then not only the times to reach the conductance plateau but also the temperature profiles are significantly different from runs with 150 Watts. At the stage of the plateau, gelatinization was determined once more to be completed. What is perhaps of greater significance is the value of this plateau, which again reads about 0.70.

Inspection of curves in Figs. 5 and 6 reveals that the time it takes to arrive at the plateau after the onset of gelatinization varies: the longer times correspond to lower heating rates. However, the decay pattern in these curves is essentially constant, a fact which might indicate a common kinetic mechanism of water dispersion during gelatinization, an issue which will be dealt with in a subsequent publication.

Employing Eq (1) and (2), the following liquid volume fractions are estimated at the stage of the plateau, $\beta_{[1]} = 0.78$, $\beta_{[2]} = 0.70$. These values are lower than the final values obtained in experiments with mixer configuration (A), indicating that less water is electrically accessible now in the gelatinized samples. One

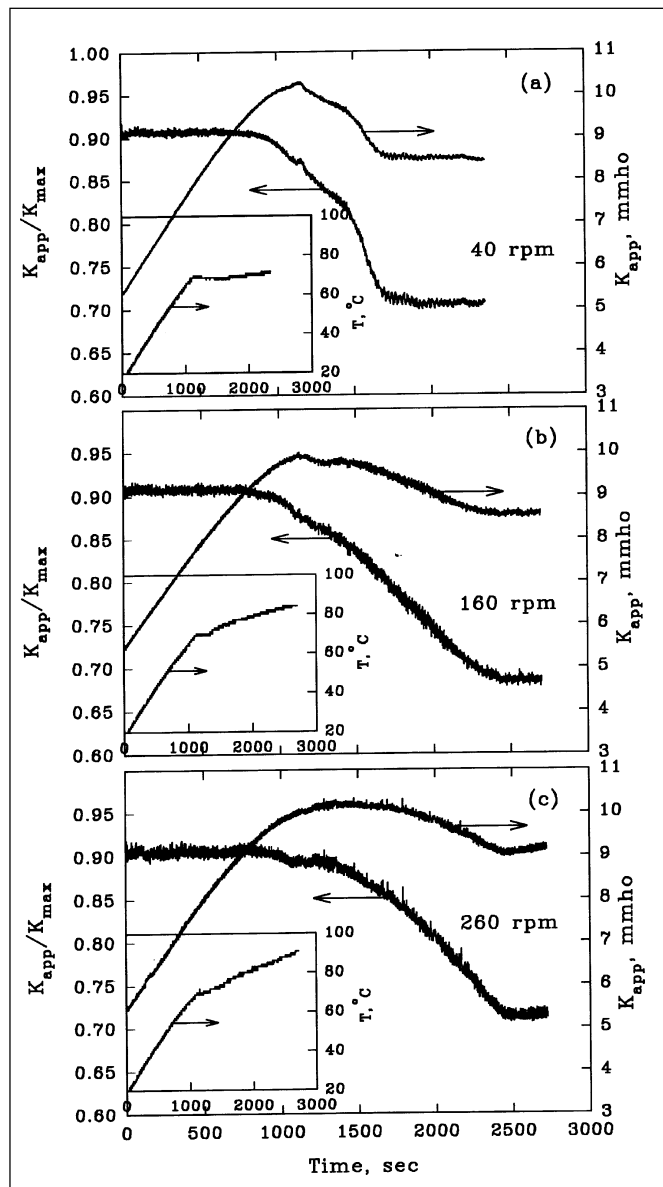


Fig. 5—Reduced apparent conductance, K_{app}/K_{max} , and apparent conductance signals, K_{app} , taken with mixer configuration C, final mixer speeds of 40, 160, 260 rpm and power 150 Watts. Mixer speed initially at 260 rpm and at 70 °C adjusted to final values. The insets display the respective temperature profiles.

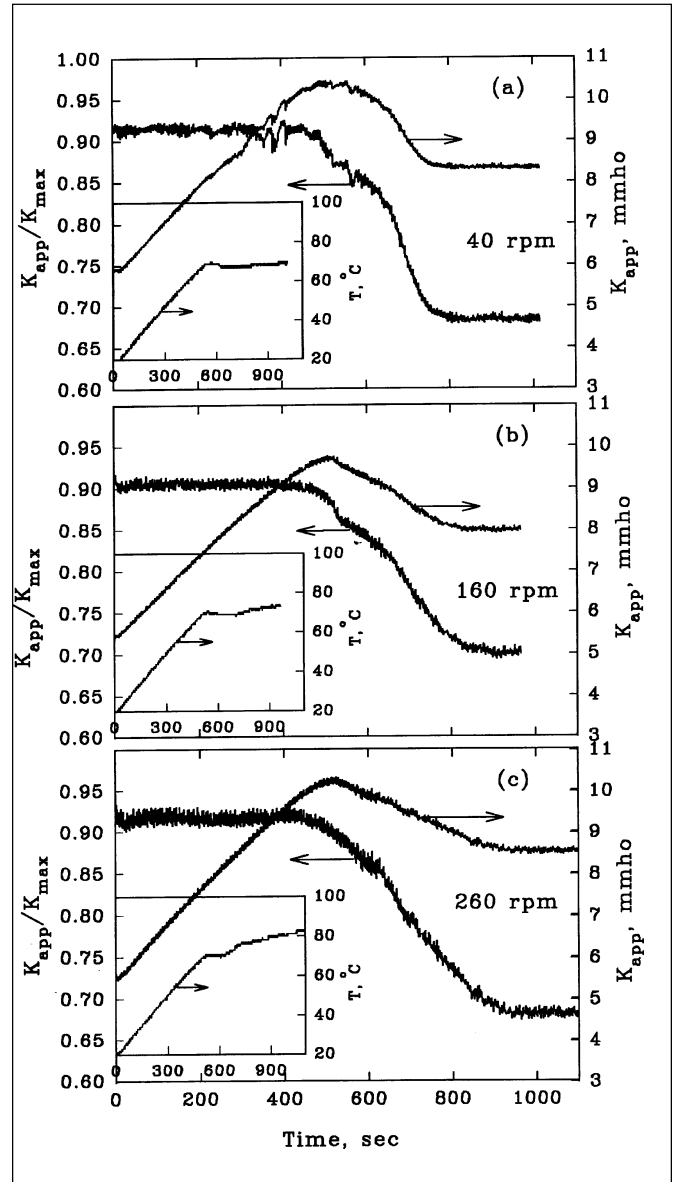


Fig. 6—Reduced apparent conductance, K_{app}/K_{max} , and apparent conductance signals, K_{app} , taken with mixer configuration C, final mixer speeds of 40, 160, 260 rpm and power 300 Watts. Mixer speed initially at 260 rpm and at 70 °C adjusted to final values. The insets display the respective temperature profiles.

might argue that higher liquid volume fractions are characteristic of incomplete gelatinization in the samples of case (A), perhaps due to the absence of any eventual mixing there. The picture might be as follows. During gelatinization the granules first imbibe rapidly the surrounding water as they swell and increase their chances of coming into contact with each other, forcing at the same time free water to leave the space between granules and accumulate into larger interconnecting local pools. Without an external means of agitation to move granules past each other and disperse water between granules, further ingress of free water to the granules may occur only by a very slow diffusion process from these pools and, therefore, the granules subsequent expansion is deteriorated. The gradual final decline of the conductance signal in Figs. 2 and 3 might reflect such diffusion phenomena. The above description is in line with the recent work by Stapley and others (1998), who studied water penetration into whole wheat grains. These authors have identified two pools of water with differing propagation characteristics, which was attributed to the presence of restricting barriers such as cell walls.

One should be cautious in comparing present measurements with swelling information from previous studies not only because of the different determination procedures but also because swelling is strongly dependent on temperature, concentration, and processing conditions (Lund 1984). Okechukwu and Rao (1996) used a laser diffraction particle size analyzer, to provide evidence that the extent of granule swelling is a rapid phenomenon. In particular, they found that after one minute of heating, swelling at 70 °C is about 70% complete but it regularly takes much longer than an hour to reach a final conclusion. By comparison with the time scales of the present measurements, one could argue that granule swelling may not be directly analogous to the electrically accessible free water in the mixture. Miller and others (1973) and Christianson and others (1982) communicated a similar divergence between time scales of granule swelling and another macroscopic property, the viscosity of the mixture. They found that although swelling equilibrium is reached within a short cooking period, viscosity may still develop.

Despite the different processing conditions, a fair agreement exists between the present plateau β -values and the respective long term 1-cQ data reported by Okechukwu and Rao (1995) for 2.6% cornstarch suspensions and temperatures below 80 °C. On the contrary, the 1-cQ values communicated by Christianson and Bagley (1983), for a 10% suspension heated for 75 minutes at 70 °C or 75 °C, are considerably lower, around 0.25. A possible explanation for this discrepancy may be found in the words of Okechukwu and Rao (1995) that the scatter in cQ data "reflect the difficulty and subjectivity involved in siphoning off the supernatant liquid after centrifugation."

Regarding the shape and the roughly constant plateau value of the conductance curves in Figs. 5 and 6, one is tempted to ar-

gue that once the supplied energy suffices to trigger and conclude gelatinization ($T_{\text{final}} >$ approximately 70 °C for maize starch), water disperses inside the granules according to a specific macroscopic pattern inasmuch as adequate mixing and no substantial granule degradation occurs. In this case, the extent of gelatinization — expressed as apparent electrical conductance of the mixture — does not appear to depend on heating rate or temperature. An analogous situation is observed in DSC thermograms regarding the enthalpy of gelatinization. This information may be extremely useful for kinetic studies in the field in that it seems to offer a new method for quantifying the extent of gelatinization in a thermally independent way.

To the best of the authors knowledge, only the work by Wang and Sastry (1997) provides some data for comparison with the conductance measurements of this study. In fact, that study differs because it involved direct passage of electric current through the sample (17% w/w), which resulted in much higher heating rates (all runs lasted less than a minute). Nevertheless, their conductance curves bear a very important common feature with the present ones: a substantial local variation (peak) during gelatinization. The area of the peak was used further to determine the gelatinization progress in a manner similar to DSC peak analysis. Unfortunately, these researchers did not differentiate between the pure temperature contribution and the structural contribution in their measurements. Besides, one must be particularly careful when employing ohmic heating to systems which include biological cells since an earlier work by Palaniappan and Sastry (1991) reported much higher electrical conductivity values during ohmic heating than during conventional heating. This result was primarily attributed to electro-osmotic effects when electric current passes through biological tissues.

Conclusions

ELECTRICAL CONDUCTANCE FOOTPRINTS OF INSTANTANEOUS fluid dispersion patterns were used to assess the influence of fluid motion (agitation) and heat transport on the gelatinization of starch/water suspensions. From a series of different mixer configurations, only a specially designed grid paddle assembly did not seem to warrant consideration due to its excellent mixing action across the entire sample volume at low shear rates. Perhaps the most important observation in this work was that the reduced conductance signals at the end of gelatinization attained roughly the same value (a plateau) regardless the thermal history of the sample if adequate energy (and mixing) was provided to promote gelatinization. This conclusion will permit subsequent analysis of the obtained conductance data to investigate the water dispersion kinetics during gelatinization. As regards practical applications, the conductance probe employed in this study offers an inexpensive, sensitive and easy to install and operate tool in thermal processing equipment, for *online* monitoring of starch gelatinization.

Materials and Methods

Materials

Commercial maize starch was purchased from Group Amylum S.A., Greece, with a moisture content of 13.5%. The total amylose content was $26.0 \pm 0.3\%$, determined by the method of Morrison and Laignelet (1983). The volumetric granule size distribution, as determined in starch suspensions at 20 °C using a Malvern MasterSizer (Malvern Instruments Ltd., U.K.) laser diffraction particle size analyzer, was essentially unimodal over the range 6 to 30 μm with only a very small fraction of particles below 3 μm . The granules' mean diameter was 14.95

μm with a standard deviation of 5.8 μm . Only starch/water mixtures with concentration of 10% w/w were investigated in this study. The liquid used in this work was tap water, filtered mechanically to remove suspended particles larger than 2 μm . Its specific conductivity varied between approx. 600 and 700 $\mu\text{S/cm}$.

Apparatus

A schematic of the set-up is shown in Fig. 7. The test section used in the experiments was a cylindrical column made of transparent Plexiglas with i.d. = 6.8 cm and height = 23 cm. A circular plexiglas plate was flanged tight to the bottom side of

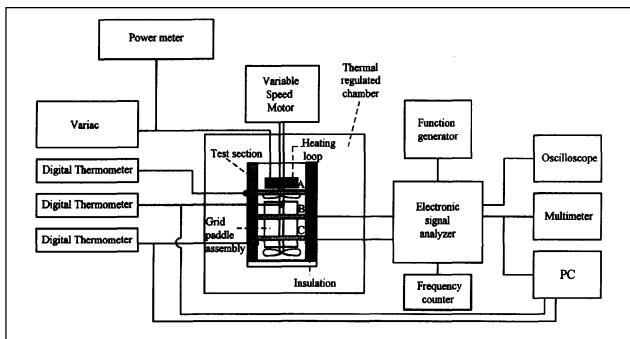


Fig. 7—Experimental set-up with cylindrical test section and signal analyzer connections.

the cylinder, while the top side was left open for thermal/mixing stimuli and temperature diagnostics to put in. A submersible heating coil with its heating loop (5 cm dia) running circumferentially along the inner plexiglas surface was used to produce heat at the top of the vessel. The shape and immersion depth of the heater (about 3 cm) were chosen to avoid obscuring the conductance measurements (being away from the measuring volume) and the access to the inside of the vessel. The selected configuration proved unique in diminishing evaporation losses from the top of the test section by creating, rapidly upon gelatinization, a few millimeters thick crust over the free surface that was difficult for vapor to penetrate. A firm parafilm foil stretched over the rims of the vessel was used as a provisional means of additional seal. Moreover, the choice of heating rates, nominally set at 150 and 300 Watts ($\Delta T_{liq}/\Delta t_{20^{\circ}-60^{\circ}} \approx 3$ and $6^{\circ}\text{C}/\text{min}$, respectively), also conformed with the evaporation concern, since for lower values the experiments were substantially prolonged whereas for higher ones the increased heat transfer rates created local boiling. A few tests with 600 Watts ($\Delta T_{liq}/\Delta t_{20^{\circ}-60^{\circ}} \approx 12^{\circ}\text{C}/\text{min}$) were performed only at very high mixing rates (500 rpm). Several exploratory runs showed that, for the range of our experimental parameters, sample weight losses during heating were always less than 2% of the total water content and occurred chiefly at the free surface of the mixture with negligible consequences to the bulk mixture.

To assure homogeneity of the liquid mixture, a center stirrer was employed with a varying combination of four impellers mounted along an electrically insulated shaft (o.d. = 0.35 cm). Two of the impellers, Fig. 8(A), were fastened on the shaft at all times. A small dual-blade paddle impeller was placed at the center of the heating coil in order to produce radial flow for enhancing heat dissipation to the bulk. In addition, an ordinary three-blade impeller was mounted at the far end of the shaft. For most runs, an additional ordinary 3-blade impeller, Fig. 8(B), was placed just below the heater. This was used alone or in combination with a custom made 3-beam grid (copper, Tyler mesh size 7) paddle assembly capable of spanning the entire cross-section of the vessel on rotation, Fig. 8(C). The ordinary impellers were meant to promote axial flow along the test section and also create moderate vortexing at low temperatures to speed up dispersion of the hard-to-wet starch granules. The grid assembly was designed to achieve mixing of every part of the gelatinized viscous material as a result of tumbling under low shear and also conduct heat to the mixture through its metallic body. Mixing rates were controlled by a variable-speed motor over the range 40 to 500 rpm. Temperature measurements were performed at 2 posi-

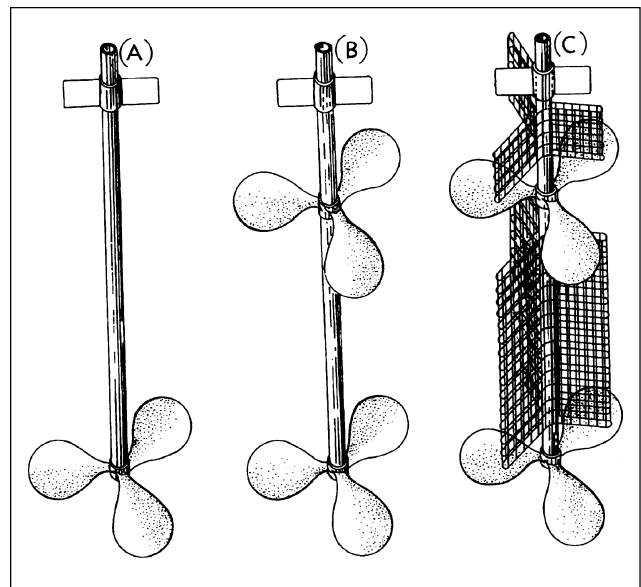


Fig. 8—Employed mixer configurations. A = a small dual-blade paddle impeller to fit at the center of the heating coil and a three-blade impeller at the end of the shaft. B = configuration A plus an additional three-blade impeller to lie just below the heating coil. C = configuration B plus a three-beam grid paddle assembly.

tions across the vessel: one at its center axis and another close to the wall. Sufficient external insulation was applied to minimize heat losses from the vessel walls. The whole set-up was put inside a thermal regulated air chamber. Even under the most unfavorable conditions — highest heating/lowest mixing rates — radial temperature differences were always below 1°C .

Conductance measurements were made by means of 3 parallel stainless steel ring electrodes (strips) with a width of 3 mm, located 3 cm apart. The electrodes were flush mounted onto the inner surface of the test section to avoid disturbing the cross section of the column. Two conductance probes were formed comprised of neighboring electrodes, AB and BC, but only results from the latter are presented here. Criteria for the selection of the separation distance between electrodes were based on the volume-averaging approach of modeling porous media (e.g. Carbonell and Whitaker 1984). The size of the probe averaging volume, i.e., the required minimum volume surrounding a probe, had to be large enough to average porosity undulations yet small enough to preserve the local character of the measurements. Copious preliminary tests have shown that a spacing of 3 cm was in accord with the above criteria and also in agreement with other evidence (Celmins 1988). Moreover, the ring electrodes were placed at a distance greater than 3 cm from the heater and the impellers, a distance that was considered sufficient to avoid end effects as shown elsewhere (Tsochatzidis and others 1992).

An alternating current (a.c.) carrier voltage of 0.5 volt peak-to-peak was applied across each probe pair at a frequency of 25 kHz in order to eliminate capacitive impedance. At this frequency, the measured phase angle shift between the applied voltage and the current across the material was virtually zero. High frequency electrical measurements are most representative of free water in the intergranular spaces and in the pores of the material since strongly bound water has a resonance at much lower frequencies (Hardman 1985). The re-

sponse of each pair of probes was fed to a special electronic analyzer, similar to that employed by Karabelas and others (1989, 1993, 1995). The analog direct current (d.c.) voltage output of the analyzer was converted to apparent conductance of the medium between the electrodes, K_{app} , using a calibration curve based on precision resistors. The signal from the electronic unit was monitored by a digital voltmeter and collected and stored in a microcomputer. Data were acquired with a 1 to 4 Hz sampling frequency that proved to be more than adequate for these experiments. At least 3 records were acquired at all experimental conditions and the reproducibility was excellent. Pearson correlation coefficients among sampled curves were always above 0.99, whereas average instantaneous signal deviations were around 1.5%, a value close to the measured signal's noise.

Pasting procedures

Two pasting procedures were employed. First, the technique described by Kubota and others (1979) and repeatedly used by Rao and co-workers (Okechukwu and others 1991; Okechukwu and Rao 1995, 1996a, 1996b) was tested. It calls

for flash gelatinization of a mixture at a predetermined target temperature by initial mixing of appropriate quantities of a cold aqueous concentrated starch dispersion with hot water inside an agitated thermal regulated vessel. Yet, in most runs, a second *standard* procedure was followed: approx. 600 g of starch/water suspension were added into the test section at room temperature and the experiment was started by energizing stimuli and diagnostics. Mixing was performed at a constant rate of 260 or 500 rpm throughout, or initially at 260 or 330 rpm until the conductance signal reached a maximum (always around 70 °C) where it was finally adjusted to a value of 40 or 160 rpm. This final adjustment was implemented while the sample still had appreciable fluidity. The degree of gelatinization was assessed by the classical methods of loss of birefringence using polarizing microscopy and application of Congo red dye to stain the swollen granules while DSC thermograms were employed occasionally for increasing the determination's confidence. Thermal measurements performed on maize starch/water samples using a Perkin-Elmer DSC-6 instrument showed that the onset of gelatinization was at 67.5 ± 0.6 °C and the peak of gelatinization was at 72.4 ± 0.4 °C.

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