

Model Wheat Starch Systems Heated by Microwave Irradiation and Conduction with Equalized Heating Times

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ABSTRACT

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A structural comparison of starch gels prepared by either microwave irradiation or by convection heating in an oil bath was made. The heating time in each paired comparison was kept constant to minimize any difference caused by the rate of heating. Starch to water ratios ranged from 1:1 to 1:8. Wheat starch granule swelling was evaluated by scanning electron microscopy and polarizing light microscopy, and residual crystallinity was determined by differential scanning calorimetry. Time-temperature profiles for the two heating methods differed even though the

total times to reach 65 or 85°C for each starch-water ratio were essentially equal. The distribution of variously swollen granules and the range in degree of swelling within the samples depended on the heating method and subsequent heat transfer. No structures unique to a given heating method were found. Residual crystallinity was found in swollen but birefringent granules. Onset temperatures of endotherms for these granules did not differ from those of the unheated starch granules.

Times for heating most cereal-based formulated foods are generally much shorter using microwave irradiation under usual home microwaving conditions than those for conductively or convection-heated foods. Differences in quality characteristics are also seen (Martin and Tsen 1981, Evans et al 1984). Whether these quality differences are caused by differences in heat and mass transfer, or in reaction mechanisms as microwave irradiation interacts with individual molecules, is not known.

In this study, starch-water samples were heated in a test tube coupler placed in the waveguide of a microwave generating system. The results were compared to those from similar samples heated in an oil bath. To eliminate differences in heating rates, oil bath temperatures were adjusted so the time to reach a particular temperature in the sample was the same as for a similar sample heated by microwave irradiation. Gel patterns within the test tubes were observed visually. Starch granule structures were examined by polarizing microscopy and scanning electron microscopy (SEM). Residual starch crystallinity was measured by differential scanning calorimetry (DSC).

MATERIALS AND METHODS

Sample Preparation and Heating Conditions

Dispersions of wheat starch (Aytex-P, General Mills) and distilled water of 1.4 ml each were prepared in ratios (w/w) of 1:1, 1:2, 1:4, and 1:8. Nonmicrowave-absorbing Teflon test tubes (76 mm high, 8 mm inside diameter) were used to ensure that microwave absorption was by the sample only.

Samples heated by microwave irradiation were placed directly in a test tube coupler in the waveguide of a specially constructed hybrid microwave/convection environmental oven (Hung 1980). A diagram of the oven is given in Figure 1. The 2,450-MHz oven was operated with an effective 75 W transmitted power (110 W transmitted and 35 W reflected), as measured with an empty Teflon test tube in the waveguide.

Samples heated by conduction were placed in an oil bath (either mineral oil or corn oil depending on the heating rate needed). Oil bath temperatures (Table I) were adjusted to give heating times equal to those used for microwave irradiation.

Sample temperatures were measured with a fiber optic probe (Luxtron Fluoroptic Thermometer, model 1000 A) at the center of

the 1.4-ml dispersion. Temperature was recorded at 5-sec intervals during heating, and the highest temperature reached after removal from the heat source was also recorded.

The targeted final temperatures were 65°C and 85°C. The maximum temperatures recorded, based on three to seven replications, are summarized in Table II.

Sample Appearance After Heating

Samples were removed from the tubes in their entirety. The appearance and relative proportion of each gel region that could be identified visually were noted. The regions were designated as gelled, watery, or chalky. Gelled regions were opaque and gel-like in consistency. Watery regions were liquid and easily pourable. Chalky regions were white and slightly dry. These regions were sampled for microscopy and DSC evaluation.

Microstructure Evaluation

Light and polarizing light microscopy. Each region in the samples was examined by ordinary and polarized light microscopy (Unitron, model MPS-2). Chalky regions from limited water systems (1:1) heated by microwave irradiation required added water to facilitate examination; the remainder were examined without adding water.

SEM. Samples from the same regions examined by light and polarizing microscopy were also examined by SEM, as described by Goebel et al (1984). A thin layer (approximately 1-mm thick) of each sample was placed on a cover slip. The cover slip was then placed on a SEM stub coated with silver paint, dried overnight in a desiccator containing calcium sulfate, and then coated with gold/palladium. Samples were examined in a Philips model 500 SEM at 6 KV.

DSC. Samples from each region were examined by DSC to determine the presence of "residual crystallinity." If an endotherm was found, it was considered to be evidence for the presence of residual crystallinity. The following sampling procedure was used.

Each region found in samples heated to 65°C was examined. If no endotherm in the range 20–92°C was found, but microscopy showed that the granules were birefringent and minimally swollen, the DSC measurements were repeated after adding water to the sample. If an endotherm was found (either with or without the addition of water), samples heated to 85°C were similarly tested.

Measurements were made in triplicate using a Perkin-Elmer differential scanning calorimeter (model DSC-2) standardized with indium and K₂CrO₄, using heating rates of 5 or 10°C/min and sensitivity of 1–10 mcal/sec. Aluminum pans were used with an empty pan as reference. Measurements were made over a temperature range of 20–92°C. Onset temperatures of endotherms were recorded.

The Wilcoxon-Mann-Whitney two-sample test and Wilcoxon two-sample test (Steel and Torrie 1980, Sokal and Rohlf 1969)

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were used to test the significance of the differences between temperatures at 5-sec intervals for each pair of microwave and conductively heated samples, and of the differences in maximum temperature between each pair after removal from heat. Analysis of variance was used to examine total heating times for each treatment pair.

RESULTS

Temperature Experiments

Representative time-temperature profiles for each starch-water ratio are shown in Figure 2 for samples heated to 85°C. The times for each treatment pair were not significantly different in most cases ($P > 0.05$). Exceptions were 1:1 and 1:2 samples heated to 85°C. In the 1:1 samples, the oil bath temperatures could not be raised sufficiently to match the microwave times. Time differences were small, 4.5 sec in the 1:2 samples. Thus, the goal of equalizing heating times was achieved, but as can be seen in the figures, some differences in temperatures were found within the heating periods. For example, in samples with ratios of 1:2 and 1:8, the temperatures in conductively heated samples were higher than those of the microwaved samples in the intermediate portions of the heating period. The maximum temperatures reached after removing samples from heat (Table II), like the times, were not significantly different for each pair of treatments ($P > 0.05$) and averaged 70°C for samples removed at 65°C and 90°C for samples removed at 85°C.

TABLE I
Oil Bath Temperatures for Wheat Starch-Water Systems Heated by Conduction

Starch:Water Ratio	Oil Bath Temperature to Heat Sample (°C)	
	To 65°C	To 85°C
1:1	192 ^a	192 ^a
1:2	125	170 ^a
1:4	119	137
1:8	106	135

^a Corn oil was used for these samples. Mineral oil was used for all other samples.

Heated Sample Appearance

The relative proportions of the gelled, chalky, and watery regions changed as water became less limiting (Fig. 3). In samples heated by microwave irradiation, the chalky region, which was present along with the gelled region in the most limited water system (1:1), disappeared when the starch to water ratio was increased to 1:2, with the result that the samples appeared to be entirely gelled. When the starch-water ratio was increased further, a watery region was present in addition to the gelled region. In samples heated by conduction, the chalky and gelled regions were present in both 1:1 and 1:2 samples as well as in 1:4 samples heated to 65°C, although the amount of chalky region decreased progressively as the amount of water increased. The 1:4 samples heated to 85°C were the only conductively heated samples that contained only a gelled region. The watery region did not appear in conductively heated samples until the starch-water ratio reached 1:8, and it occupied a greater portion of the sample than in the microwave-heated sample.

TABLE II
Maximum Temperatures Reached by Starch-Water Systems Heated by Microwave Irradiation and Conduction

Starch:Water Ratio	Heating Method	Targeted Temperature					
		65°C			85°C		
		Mean ^a	Range	n ^b	Mean ^a	Range	n ^b
1:1	Microwave	68.5	67.8-68.9	3	90.0	89.0-91.6	5
	Conduction	72.6	69.5-78.1	7	90.6	87.3-92.4	6
1:2	Microwave	69.2	67.8-71.4	4	89.7	89.5-90.0	3
	Conduction	69.0	68.1-70.8	6	89.7	88.7-90.7	5
1:4	Microwave	68.4	66.6-69.8	5	92.0	89.9-93.9	4
	Conduction	71.2	68.0-74.1	5	89.8	87.9-91.4	3
1:8	Microwave	68.3	67.7-69.2	3	90.5	88.8-92.3	4
	Conduction	67.5	66.1-71.3	6	89.5	87.5-92.4	7

^a Differences between temperatures for microwave and conductively heated samples were not significantly different ($P > 0.05$) for each pair of samples.

^b Number of replications.

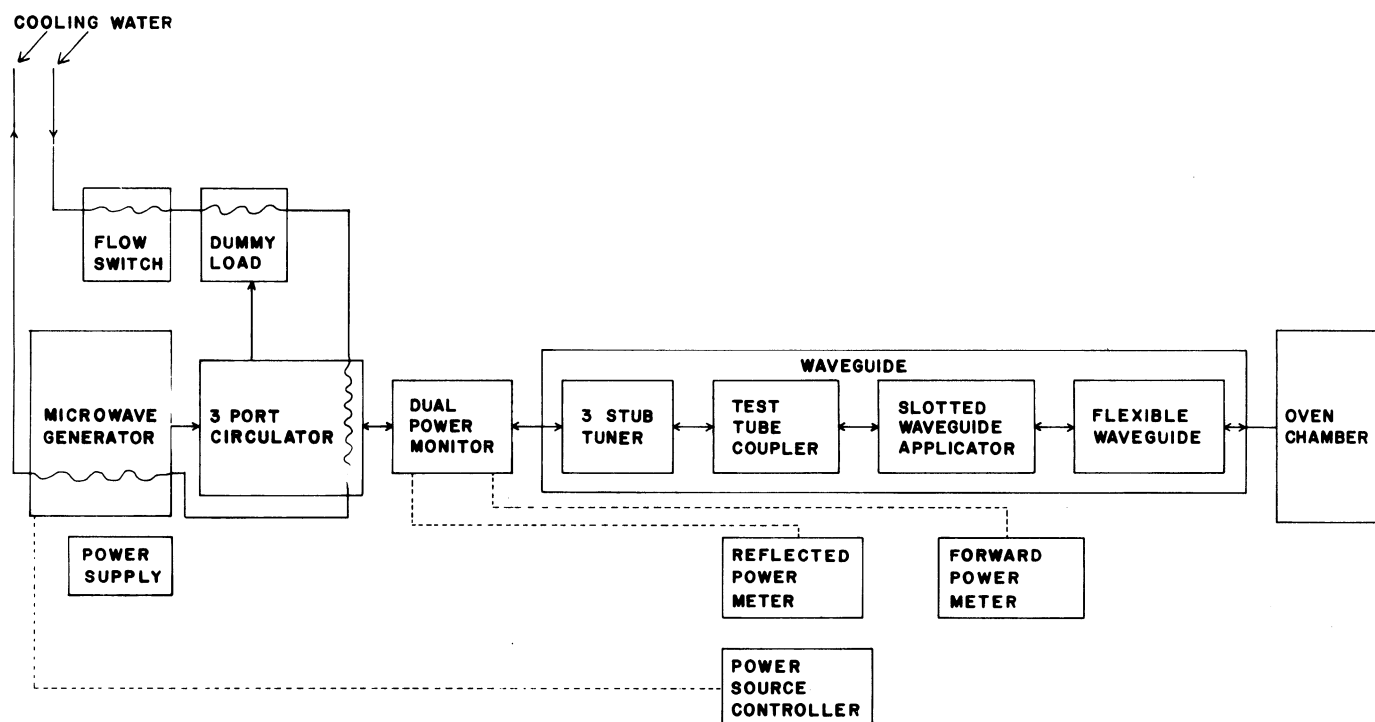


Fig. 1. Microwave transmission system. Dashed lines = electric wires connecting the units at both ends of the lines. Arrows = direction of microwave transmission.

In microwave-heated samples, chalky regions were located along the outer edge on the side closest to the source of the microwave beam, but in conductively heated samples they were located at the center and surrounded by the gelled region. Watery

regions, when present, were always located at the top of the gelled regions. The gelling patterns in each heating method demonstrate that settling of starch was not a major factor during the short time required for heating.

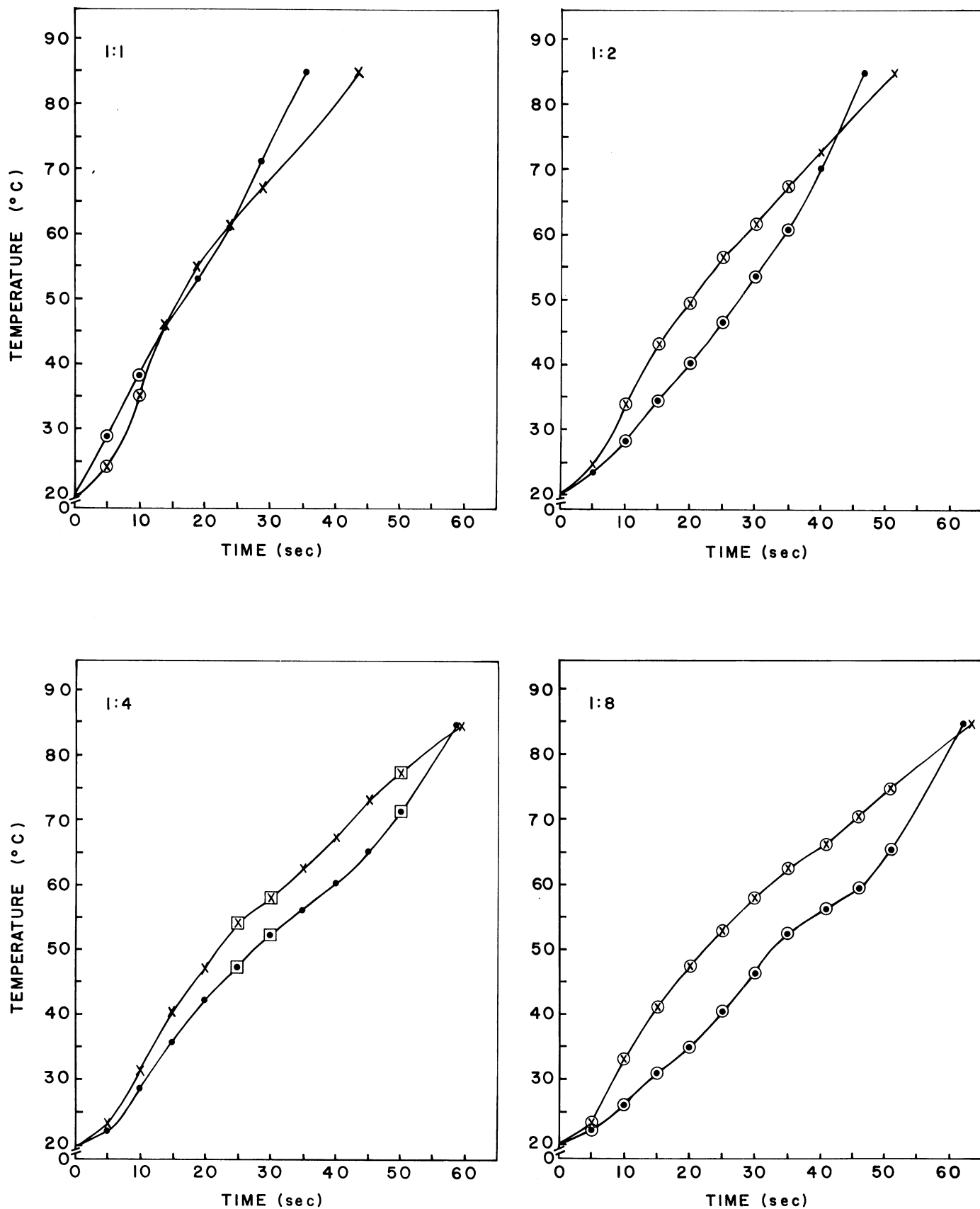


Fig. 2. Time-temperature curves for wheat starch-water systems heated to 85°C by microwave irradiation (●) and by conduction (x). Data points circled are significantly different ($P \leq 0.05$) for that time interval. For data points enclosed in squares, $P \leq 0.10$.

Microstructure Evaluation

Overall, five stages in granule swelling were identified by SEM, using changes in large and small granules and the development of matrix material as criteria (Fig. 4, Table III). Small granules, similar to native starch granules in stage 1, became clumped and dimpled (stages 2 and 3) and then began to swell (as illustrated for stages 4 and 5). Large granules swelled into disc shapes and then folded into saddle shapes (stages 2–4). This sequence for large granules is similar to that described by Bowler et al (1980) for the early stages of swelling. This swelling was accompanied by a flowing together of large granules into a matrix (stages 3–5), so that granules were less easily distinguishable by stage 5. Many of the granules in stages 1 and 2 showed birefringence when viewed under polarized light; whereas only a few birefringent granules were found in stage 3, and none in stages 4 and 5.

The stages of granule swelling found in each region of the starch-water samples are summarized in Table IV. Granules from the gelled regions of microwave-heated samples heated to 65°C were classified as stage 4. Large granules were swollen and folded;

small granules were clumped and dimpled; and matrix development was present. Granules from conductively heated samples were swollen to the same extent as those from microwave-irradiated samples in the limited-water systems (1:1 and 1:2) but were less swollen in the higher ratio systems (1:4 and 1:8). As would be expected, the granules from the gelled region of samples heated to 85°C were more swollen than comparable granules heated to 65°C.

Granules in the chalky regions of the limited-water systems were essentially unchanged (stage 1) or just beginning to swell (stage 2). Granules from the chalky region of the 1:4 ratio sample heated by conduction (the only higher ratio water system in which the chalky region was found) were somewhat more swollen (stage 3) than in the limited water systems.

Granules from the watery regions tended to resemble those from the gelled regions with which they were associated. The watery instead of gelled appearance was probably caused by an increase in localized water availability rather than by differences in the extent of swelling.

The range of swelling within a given sample can also be seen in Table IV. In limited-water systems, whether heated by microwave

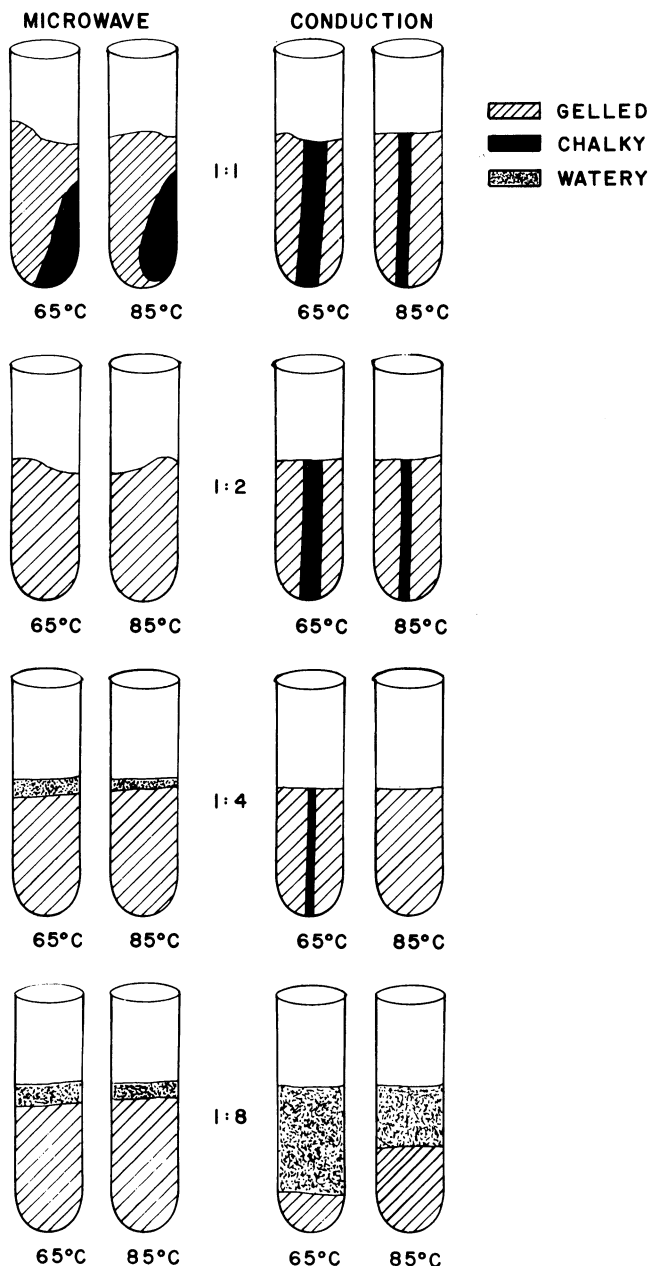


Fig. 3. Distribution of gelled, chalky, and watery regions in starch-water systems heated by microwave irradiation and by conduction. Microwave beam traveling from right to left.

Table III
Stages in Starch Granule Swelling

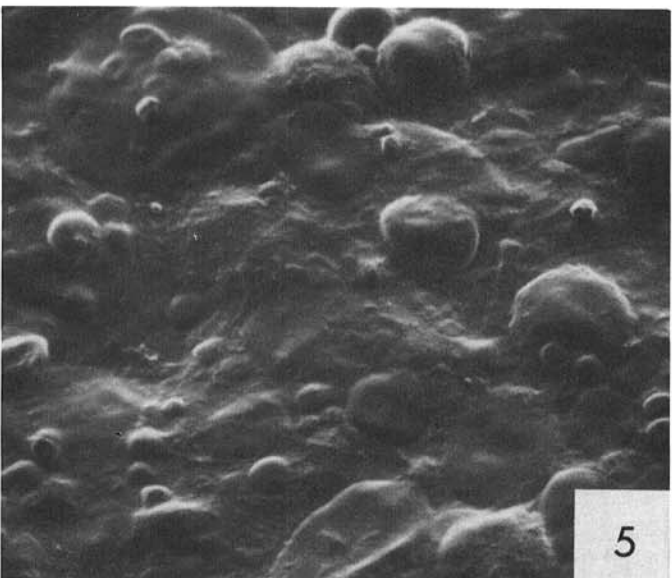
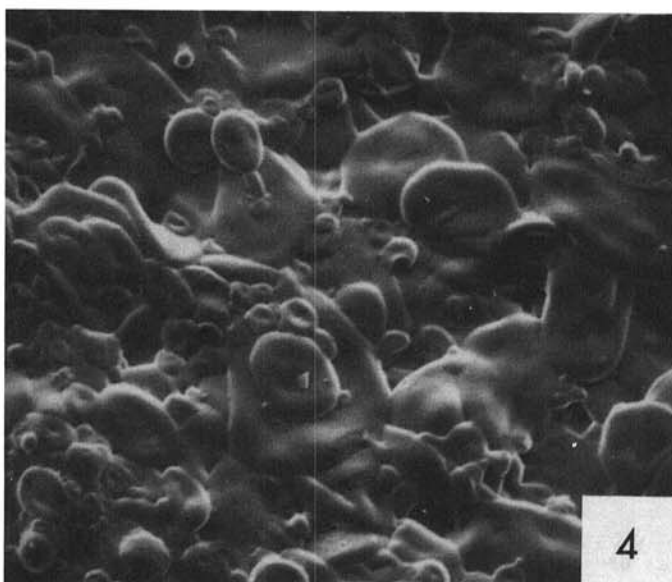
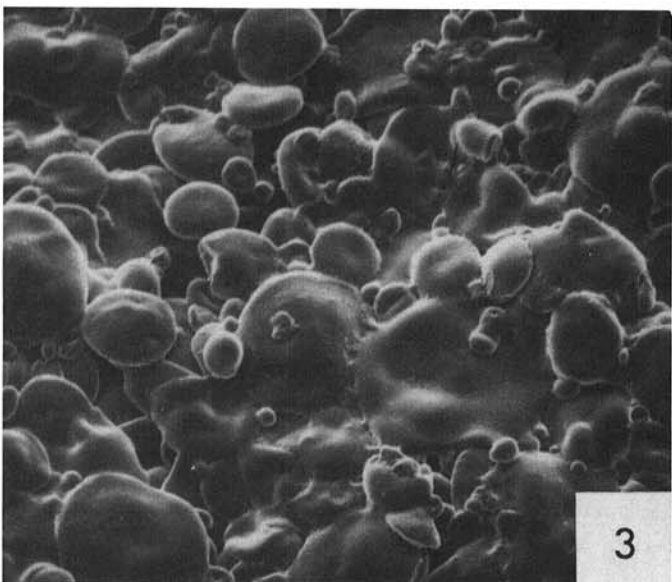
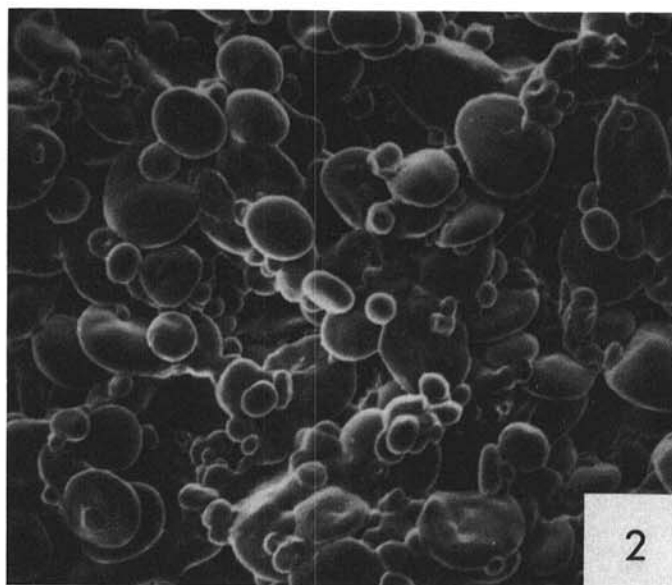
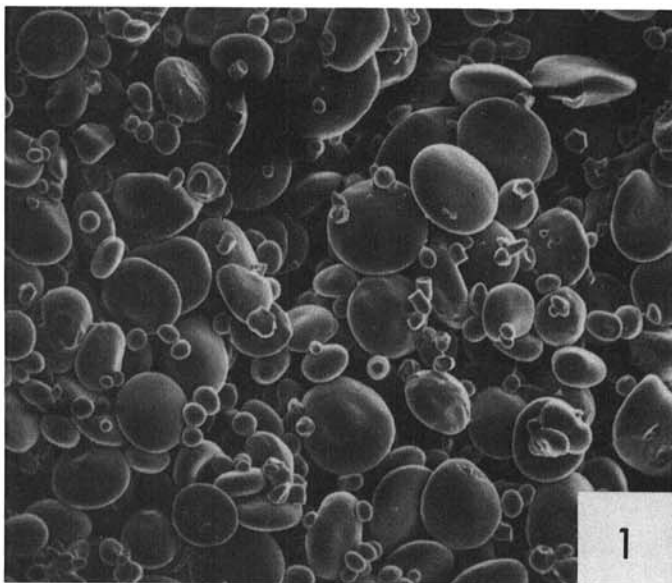
Stage	Description
1	Granules—essentially unchanged. Birefringent.
2	Small granules—some clustered and dimpled. Large granules—swelling to disc shapes. No matrix present. Birefringent.
3	Small granules—many clustered and dimpled. Large granules—disc shaped, folding beginning. Matrix beginning to develop. Some birefringent granules.
4	Small granules—clustered, dimpled, slightly swollen. Large granules—disc shaped, folded. Matrix present. No birefringence.
5	Small granules—clustered, dimpled, swollen. Large granules—extremely swollen. Most flowing together. Matrix present. No birefringence.

TABLE IV
Stages of Granule Swelling as Determined by Scanning Electron Microscopy (SEM) and Residual Crystallinity Determined by Differential Scanning Calorimetry in the Regions Found in Wheat Starch-Water Systems Heated by Microwave Irradiation and Conduction

Starch:Water Ratio	Heating Method	Final Temperature					
		65°C			85°C		
		Gelled	Watery	Chalky	Gelled	Watery	Chalky
1:1	Microwave	4 ^a	...	2 E(W) ^b	5	...	2 E(W)
	Conduction	4	...	1 E(W)	5	...	2 E(W)
1:2	Microwave	4	5
	Conduction	4	...	2 E	5	...	2 E(W)
1:4	Microwave	4	4	...	5	4	...
	Conduction	3 NE	...	3 E	4
1:8	Microwave	4	3 NE	...	5	4	...
	Conduction	3 E	3 NE	...	4	5	...

^aNumbers represent stages of granule swelling based on SEM.

^bResidual gelatinization: E, endotherm without addition of water; E(W), endotherm with addition of water; NE, no endotherm with or without addition of water. Data given for stages 1–3. No endotherms were found in samples at stages 4 and 5.



20 μm

Fig. 4. Stages in granule swelling.

irradiation or by conduction, regions of swollen granules and developed matrix coexisted with regions in which the granules were relatively unchanged. As water became less limiting, granule swelling became more uniform throughout the sample.

Residual Crystallinity by DSC

Measurements of residual crystallinity by DSC supported microscopic evaluations of granule swelling. Endotherms were observed in all of the regions in which granules were in either of the first two stages of swelling (chalky regions from 1:1 and 1:2 ratios, Table IV). Additional water was needed for all of the 1:1 samples and for the 1:2 sample heated to 85°C by conduction.

Endotherms were also observed in regions in which the swelling was more advanced (stage 3) but which still retained some birefringent granules (chalky regions from 1:4 samples conductively heated to 65°C, and gelled regions from 1:8 samples conductively heated to 65°C, Table IV). Endotherms were not observed in regions that had a similar degree of swelling but retained fewer birefringent granules (gelled, 1:4 conductively heated to 65°C, and watery, 1:8 microwave heated and conductively heated to 65°C).

Endotherms were also absent in all regions in which granule swelling had advanced to stage 4 after heating to 65°C. Accordingly, additional testing of these regions from samples heated to 85°C was not needed.

Onset temperatures ranged from 57 to 60°C and averaged 58°C; which is slightly higher than the mean value of 57°C for control starch-water systems examined by DSC without heating. These values are similar to those reported previously for wheat starch (Donovan and Mapes 1980, Eliasson 1980, Kugimiya et al 1980, Ghiasi et al 1982).

DISCUSSION

Heating Effects

These experiments showed that, with temperature adjustments, it would be possible to compare the course of starch gelatinization in samples heated for the same length of time by conduction and by microwave irradiation. The time-temperature histories of conductively heated and microwave-heated samples were not the same, however, even though it was possible to equalize the overall times. Heat transfer in conductively heated samples is driven by the differences between the low internal and high external temperatures. Heat transfer in samples heated by microwave irradiation differs in that the internal generation of heat as a consequence of absorption of microwave energy, coupled with low external temperature, reverses the temperature gradient that drives heat transfer. Absorption of microwave energy and its conversion to heat are related to the dielectric properties of the materials being heated. Because starch gelatinization involves a phase change from crystalline to less crystalline material, the dielectric properties would be expected to change also. The modeling studies of Wei et al (1985a,b) of a porous medium saturated with water, and without phase changes in the matrix, show the complex interactions that occur to produce typical temperature profiles. Without detailed analysis, it is difficult to isolate the effects in a two-component system undergoing phase change as in the starch-water system. Nevertheless, some consequences of these changes can be seen in the studies reported here.

Except for the most limited water system (1:1), temperatures in the early stages of heating in conductively heated samples were higher than those in microwave-heated samples. After 60°C, which is near the onset temperature of the endotherm as measured by DSC, the rate of temperature increase was greater in the microwave-heated samples than in those heated by conduction. This is probably why it was possible to match the times for microwaved and conductively heated samples, because the final time marks the intersection of the slowing curve of the conductively heated sample with the rising curve of the microwave-heated sample.

The increasing rate of temperature rise in microwave-heated samples could be caused by increased efficiency of energy conversion in starch granules after the changes in crystallinity accompanying the phase transition. It would be accompanied by a loss in

efficiency in the water component because a portion of the water is immobilized through coupling with the transforming starch.

Because temperatures were measured at the center, the temperature might begin to increase rapidly if the region of maximum temperature were initially some distance away from the center of the tube, and the center regions heated by conductive and convective heat transfer, even though the source of energy is microwave irradiation. The location of this maximum temperature region depends on a number of factors. If the wave is unidirectional, the intensity of a microwave beam as it is transmitted through the sample will decay as a function of distance and the dielectric properties of the system. The temperature profile rarely parallels this because of heat requirements for surface evaporation and heat transfer by conduction and convection to the environment and to the interior of samples. Although we used small samples and placed them in the waveguide, reflected power as well as transmitted power was present. The reflecting surfaces that would contribute to the measured reflected power could be at the point of the beam incident on the tube, the tube surface at the point where the transmitted beam exits the tube, oven surfaces, and the tuners. Some of this reflected power that returns through the sample can contribute to heating the sample.

Starch Effects

In the most limited-water system (1:1) the gelled region, which is the most swollen, is farthest from the microwave source, and the chalky region, which is least swollen, is closest to the microwave source. This pattern could represent a temperature gradient, with the higher temperature away from the microwave source. Or the dry chalky region could result from flash heating and vaporization that depletes water in this region. DSC demonstrated that the granules here retain the potential to undergo phase transitions when more water is present. This observation eliminates the possibility that the entire chalky region undergoes irreversible high-temperature cross-polymer linking between the molecules present in the starch granule analogous to cross-linking observed in other polymers.

The status of starch that has been exposed to high temperatures and presumably low levels of water is a matter of some controversy. Abboud and Hosney (1984) found that baked cookies showed typical gelatinization endotherms in the presence of excess water. Burt and Ross (1983) concluded that at the extremely low moisture content typical of biscuits, loss of birefringence is associated with the melting endotherm rather than the gelatinization endotherm. Varriano-Marston et al (1980) found that starch from cookies showed the A-type X-ray diffraction pattern. In bread crust, the granules showed the A-type pattern superimposed on the V-type pattern, and Varriano-Marston et al (1980) attributed this result to insufficient water being present at the surface to bring about a phase transition. However, in potatoes, a high-moisture system, the granules in the outer crust (a region about 400 μm wide) remained unswollen throughout baking even though substantial amounts of water, measured by water loss data, passed through (Galletti et al 1980). In this case, the initial exposure to high temperatures might have resulted in transformations that were not reversible in the presence of water and heat. Granules in the chalky regions of the low-ratio starch-water samples in the present experiment appear, on the basis of the DSC results, to resemble more closely the low-moisture cookie/biscuit system than the high-moisture potato system, although the possibility that some portion of the molecules undergoes irreversible transformations also exists.

As the water became less limiting, the degree of granule swelling became more uniform throughout the sample. The additional water present in the system could contribute to the uniformity in a number of ways. It could increase efficiency of microwave coupling throughout the heating process, aid in convective and conductive heat transfer, and bring the water concentration throughout the sample above the minimum required for the phase transitions that began at 58°C.

The relative position of chalky and gelled regions in the conductively heated samples is what would be expected if the

temperature gradient were from the outer regions to the inner regions. The dry, chalky regions were present not only in the 1:1 and 1:2 limited-water systems but also up to a 1:4 ratio. The chalky region at higher water levels was not accompanied by more extensive swelling in the gelled regions. The degree of swelling in gelled regions of these conductively heated samples was similar or even less than in the microwave-heated samples in which the chalky region was not present. Thus, more extensive binding of water did not occur via more extensive swelling in the gelled region. Instead, it occurred by successive reductions in the amount of the chalky region as the water or temperature was increased.

When the stages of granule swelling of both series were compared, no structures were found that were unique to one of the heating methods. A similar result was found by Goebel et al (1984), who used larger samples and higher microwave powers than were used in this study.

The stages of swelling are also similar to those reported by Goebel et al (1984) except that the initiation of swelling of small granules paralleled more closely the beginning of swelling of large granules into disc shapes; the small granules, albeit swollen, were visible in all stages, and a fibrous matrix did not develop. In each case, the swelling sequence for large granules was similar to that described by Bowler et al (1980) for the early stages of swelling. Rockland et al (1977) also developed a sequence of changes for lima bean starch based on swelling and dimpling of granules. However, they choose to select certain granules within a field while we consider the concomitant appearance of large and small granules and matrix to define a stage.

Both large and small granules had undergone some swelling before birefringence was lost and the endotherm disappeared. This observation supports the hypothesis that minimal swelling can occur within granules before the ordered structure is disrupted to the point where birefringence is lost and the endotherm disappears. SEM does not provide information on sequences in which regions within the granules (crystalline or amorphous) are affected.

Because no structures unique to either heating method were found, the differences between microwave and conductive heating appear to arise primarily from differences in heat and mass transfer within the samples. These result from the properties of each heating method and cause the characteristic distributions of swollen granules within a sample. These distributions of swollen granules then can be expected to cause differences in the properties of starch-based products that depend on the underlying swelling characteristics of the granules. These properties, which also relate to the quality of the final product, range from structural development in batters and doughs to viscosity of starch pastes and viscoelastic properties of starch gels. Understanding these changes

in starch granules under various conditions of heating and formulation can optimize the quality of these products.

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