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THE EFFECTS OF MICROWAVE ENERGY AND CONVECTION HEATING ON WHEAT STARCH GRANULE TRANSFORMATIONS

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Abstract

Wheat starch-water dispersions at 1:1, 1:2, 1:4, and *5* 95 (w/w) starch:water ratios, representing systems with varying levels of water availability, were heated under static conditions to 75°C by microwave energy at two power settings and by convection heating. Starch granule swelling was evaluated by light and scanning electron microscopy. Six stages in swelling were identified on the basis of swelling of small and large granules and development of an extragranular matrix. The range of swelling stages found in different locations within a sample decreased as water became more available and less limiting. At each starch:water ratio, the range of stages of swelling and matrix development was smaller in convection-heated samples than in microwave-heated samples, but the convection-heated samples were at more advanced stages of gelatinization than were the comparable microwave-heated samples. Each starch : water ratio and heating combination resulted in characteristic patterns of gelled and non-gelled regions that could be observed visually. The microscopic and macroscopic characteristics are explained on the basis of differences in interaction with microwave radiation and subsequent heat and mass transfer.

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KEY WORDS: Wheat starch, Microwave heating, Starch granule swelling, Gel formation, Heat and water transport-starch: water dispersions.

Introduction

Use of microwave energy for processing and cooking foods has increased greatly in recent years. Heating that occurs as a result of microwave irradiation is caused by molecular friction resulting from the dielectric coupling of molecules as they orient themselves with frequencies in the microwave region of the radiation spectrum and as molecules change their orientation with the oscillating microwave field in order to maintain their dielectric coupling. The heating of an object depends on its dielectric constant which determines how a material couples with microwaves, and on the dielectric loss factor which expresses the ability of the material to absorb microwave energy and transform it into heat. Moisture content is an important factor in determining each of these parameters.

Although many acceptable food products are produced by microwave heating, less satisfactory results are found in some starch-based foods. Development of acceptable structures in batter and dough systems is one example of such problems. The reasons for these differences may be related to fast heating rates, differences in heat and mass transfer mechanisms, or specific interaction of the components of the formulation with microwave radiation.

Starch is one component of formulated food systems for which the sequence of transitions when it is heated in the presence of water is known to be affected by the level of available water and the rate of heating. Collison and Chilton (1974) found that microwave-heated samples of potato starch were damaged more rapidly than forced air convection-heated samples, but the starch :water ratio was more important than heating rate in determining extent of damage.

In the present study, starch granule swelling was investigated over a range of water levels commonly found in starch-based food systems. Two heating rates were used in the microwave mode, one in the convection mode. A reference system sequencing granule swelling was developed for purposes of classifying the stages of granule swelling present at each combination of heating rate and starch:water ratios.

Materials and Methods

Wheat starch (Aytex-P, General Mills) and distilled water dispersions with four starch:water ratios, 1:1, 1:2, 1:4, and 5:95 (w/w basis), were prepared by mixing the appropriate weights of starch and water. Fifty gram samples of each ratio were weighed into 100 ml beakers (Teflon *PFA, diameter 50 mm; height 68 mm). Each sample was heated to 75°C in a household microwaveconvection oven (Sharp Carousel Model R-8310) using either the convection mode at l77°C or the microwave mode at Low (20% power) or Medium (50% power) settings. The microwave oven operated at 2450 MHz, 650 watts nominal, and 400 watts effective power (method of Van Zante, 1973) . Each beaker containing a sample was placed at the center of the carousel. Temperature at the center of each sample was measured with a fiber optic probe (Luxtron temperature probe, Model lOOOA). The probe was placed at the center axial position of the beake< and 10 mm from the bottom of the beaker. Temperature was recorded every 10 seconds during the first minute and then every 30 seconds until 75°C was reached. Samples were cooled immediately to 22°C in an ice bath to minimize further temperature increases after removal from oven.

Total weight loss and change in height were recorded for all samples. The 1:1 and 1:2 starch:water samples, which formed firm gels, were removed from beakers and cut in half. Tracings were made of the gelled and chalky appearing areas. Total areas of the cross-sections were measured with a planimeter. Proportions of gelled and chalky regions were determined by weighing total tracings and the cut-out sections of the gelled regions. Cross-sections of the 1:4 and 5:95 starch: water samples were prepared by cutting and examined, but the extent of the gelled and chalky areas was not quantified.

Five replications of each starch :water ratio heating rate series were prepared. The order of preparation was randomized within each series. Microscopy:

Light and polarized light microscopy Gelled and chalky areas were sampled for microscopic evaluation. A single layer of granules was observed with *a* polarizing microscope (Unitron, Model MPS-2) using both ordinary or polarizing light after some distilled water had been added to the microscope slide. Scanning electron microscopy (SEM)

Samples were taken from the same sample areas as were used for light microscopy. No water was added, however. A thin layer approximately 1.0 mm thick of the starch preparation was placed on a cover slip which had been cut to fit the stub. The cover slip was attached to the stub with silver conducting paint. The stubs were dried over calcium sulfate in a desiccator overnight and then coated with gold/palladium. Samples were examined in *a* Philips Model 500 SEM at 6 kV.

Results

Temperature Profiles

The time-temperature profiles for the microwave heated samples are shown in Fig. 1. As would be expected, shorter times were needed to reach 75°C at the medium power setting (200 watts, effective) than at the low power setting (80 watts, effective). At both power settings, the times to reach 75°C increased as the amount of water present in the samples was increased. The time-temperature profiles for equivalent amounts of water heated in the absence of starch (Fig, 2) show the same trend of longer times with increasing amounts of water. The actual times, however, were not necessarily equivalent (Table 1). At the medium setting, the times for starch :water and the equivalent amounts ot water were quite similar , but at the low setting, the times for the starch:water samples were shorter than those for the equivalent amount of water in the absence of starch. In addition, the temperature at the center of the starch : water samples heated at the higher setting continued to rise above 75°C, by as much as l5°C, before cooling was sufficient to begin to reduce the center temperature. This temperature rise was less than 5°C in starch :water samples heated at the lower wattage or in water heated in the absence of starch. These results

Table 1. Starch heating time measurements.

 a Means (X) and standard deviations $(s.d.)$ based on 5 measurements.

Microwave Wheat Starch Granule

Fig. 2 Time-temperature relationships using microwave heating at low and medium settings for weight of water equivalent to water present in the starch:water systems (25 g equivalent to 1:1; 33.3 g to 1:2; 40 g to 1:4; 47.5 g to 5:95. 50 g equivalent to total sample volumes).

Table 2. Height and weight losses and distribution of gelled and chalky areas in starch:water samples heated by microwave irradiation and convection.

suggest that water is important in heating samples by microwave irradiation, but it is not the sole molecular species that interacts with microwave irradiation,

The time-temperature heating profiles for convection-heated samples are shown in Fig. 3 . The times to reach 75 $^{\circ}$ C ranged from 15.9 min for 1:1 ratio to 17.8 min for 1:4 ratios with the times for $1:2$ and $5:95$, 16.7 and 16.2 respectively, falling within this range. They were closely related to water content than was the for microwave-heated samples. Macrostructural Characteristics

Both weight loss and shrinkage as measured by the change in height were greater in samples

heated by convection mode than in samples heated by either power setting using the microwave mode (Table 2). Weight loss and shrinkage for samples heated at the two microwave power settings were similar to each other although heating rates were not the same .

The locations of the gelled and chalky areas (1:1 and 1:2 starch:water ratios), the various gel and paste areas (1:4 starch:water ratios) and watery gel and gel areas (5:95 starch:water ratios) were determined by the heating mode as shown in Fig. 4. The gelled regions were the inner regions of the microwave samples and the outer regions of the convection samples.

Microwaye Wheat Starch Granule

The 1:1 and 1:2 starch :water ratios showed two distinct regions, one gelled, the other chalky. Voids were found at the center of some of the 1:2 ratio samples, The proportions of gelled to chalky areas increased as the starch water ratio was increased from 1:1 to 1:2 and more water was available. At the lower ratio, approximately 60% of the microwave sample and 50% of the convection-heated sample were gelled. At the 1:2 level, 85% of the sample was gelled under either microwave or convection heating conditions .

Patterns for the 1:4 ratio samples heated by microwave energy were more complex than those for the lower ratios. A soft gel region was at the top of the outer region, and a paste region was at the bottom of the outer region. The central gel region varied from an opaque gel at the top to a clear gel at the bottom. The patterns for the convection-heated samples consisted of a soft paste at the top and a gel region at the bottom.

The 5:95 samples, whether heated by microwave or convection, consisted almost entirely of a watery gel area with a small amount of a gel region (possibly 5% of the total) located at the center bottom of microwave samples and the outer bottom side of the convection-heated samples. Microstructural Characteristics

Six stages in the swelling patterns were identified on the basis of changes in small and large granules and the development of an amorphous-appearing matrix. Representative areas from each stage are shown in Fig. 5 and the characteristics of each stage summarized in Table 3. In the first three stages, (Figs. 5a-c) the small granules which are approximately 2 to $10 \mu m$ in diameter, are seen to progressively clump and then swell at the edges to form a swollen, dimpled granule. The large granules, which ranged in diameter from approximately 10 to 40 μ m, swell

radially to disc shapes and then fold. Development of matrix material which is defined as any material other than discrete granular structures, cannot be observed. Birefringence is retained through the first three stages. In the last three stages (Figs. 5d-f), matrix development, which is first observed at stage 4, advances until it reaches a stage where small amounts of the sample material also contain a fibrous state in stage 6 as shown in Fig. 5f. The small and large granules can be clearly identified in stage 4, but begin to flow together by stage 5. Small granules are less frequently seen as discrete entities; presumably they have become part of the matrix by stage 5. Some granule structure is retained, in stage 5 and 6, however, as can be seen by examination of washed samples by light microscopy (Inserts 5d-f) .

The range in microstructural characteristics within a sample decreased as the starch:water ratio was increased and water became less limiting (Table 4). For each starch:water ratio, structural differences were also associated with the heating mode. In the 1:1, 1:2, and 1:4 starch:water systems, the inner chalky or soft gel regions of the convection mode were more advanced than their counterparts in samples heated by the microwave mode (the outer chalky or soft gel regions). For example, at the 1:1 starch:water ratio and at either the medium or low microwave power, the outer regions were essentially unchanged in appearance from that of the unheated starch. The inner regions had reached stages 5 and 4, respectively, with small granules swollen and dimpled and the large granules disc-shaped and folded. The matrix had developed, and in the case of medium power, the small and large granules had flowed together. The convection-heated samples at this ratio of starch:water had reached a similar degree of swelling in the outer gelled regions, but degree of granule swelling in the inner chalky region was more advanced (stage 2) than the chalky area of the microwave samples (stage 1).

A similar contrast between microwave and convection-heated samples was present at the 1:2 starch:water ratio, except that for each heating mode the chalky area had structures that were one stage more advanced than the comparable regions of the 1:1 starch:water ratio samples. The same contrast between microwave and convection-heated samples was found at the 1:4 starch:water ratio, but the structures in the gelled region of the medium power samples were also at a more advanced stage than the gelled regions in the low power samples and showed a fibrous rather than amorphous matrix (stage 6). This fibrous matrix was also found in the gelled region of convection-heated samples.

In the 5:95 starch :water systems, the difference between the microwave and convectionheated samples was not as great as for the other starch :water systems, The watery gel areas in which starch apparently was not concentrated e nough to form a gel had structures that were similar to the gelled area, except that fibrous development in the matrix was not as extensive in the watery gel area. These watery gel areas may or may not contain small amounts of the chalky, limited water areas discussed earlier. Although

Table 3. Stages in swelling of starch granules,

 $C + 200$

1 Granules - essentially unchanged.

Sire fr ingent,

2 Small granules -clumped.

Large granules swelling to disc shapes, appear plastic prior to folding.

Bire fringent.

3 Small granules-cluster swell, dimple.

> Large granules - disc shaped, folding beginning.

Birefringent.

4 Small granules - swollen, dimpled.

Large granules - disc shaped, folded.

Matrix present,

Some birefringent granules.

5 Small granules no longer visible.

Flowing together of small and large granules.

No birefringence.

6 Granules as in stage 5 .

Matrix - fibrous,

the watery gel area appeared to be quite advanced on the basis of the SEM examinations, it retained a small amount of birefringence, and some granule retention could be seen in washed samples examined by light microscopy .

In summary, microscopic observations showed that the convection-heated samples were structurally more uniform in the sense that a narrower range and, therefore, fewer stages of starch granule swelling and matrix development were observed for samples at each starch:water ratio . This was attributed to the chalky areas in the convection-heated samples being further along in the gelatinization process than the comparable areas in the microwave mode, yet the gelled areas were at similar or less advanced stages in the gelatinization process. Within the microwave mode, differences in the rate of heating (medium vs low power) did not result in major differences in swelling patterns .

Fig. 5 Stages in granule swelling. Larger photos are SEM micrographs; upper lefthand inserts are light
micrographs; upper righthand inserts are polarized light micrographs (when birefringence was observed in
(a), (b), and

Table 4. Stages of swelling in various regions in starch:water samples heated by microwave irradiation and convection.

afor locations of areas, see Fig. 4. bFor descriptions of stages, see Table 3 and Fig. 5.

Discussion

The specific roles that the water content and mode of heating play in the range of granule swelling observed microscopically are difficult to separate from one another, since they are interre-

lated. A number of researchers (Burt and Russell, 1983; Donovan, 1979; Eliasson, 1980; Eliasson et al. 1981; Ghiasi et al. 1982; Wootton and Bamunuarachchi, 1979; Lund, 1983; Biliaderis et al, 1980) have shown, in differential scanning calorimetry experiments, that the effect of decreasing water content is to suppress the initial gelatinization endotherm (as shown by a decrease in enthalpy) until it disappears at limited water contents. The water content at which this occurs varies somewhat with the experimental conditions but is usually placed at about 30%. When water content is above this critical level and the gelatinization endotherm is present, the onset temperature of the endotherm for wheat starch is relatively constant. Since the water contents in our experiment were above this critical level, the advancement in the stage of swelling with increasing water content which we observed microscopically is not likely to be due to a change in onset temperature of starch phase transitions, The enthalpy may be different and this could contribute to changes in the swelling as observed microscopically. The starch:water ratio may also affect other properties of the system that are more nearly related to the manner in which microwave energy couples with localized areas within the sample. In microwave-heated samples, the time to reach the predetermined final temperature increased as the amount of water was increased. Thus, the more advanced stages of starch gelatinization as water became less limiting in microwaveheated samples might be partly due to longer heating times once a certain critical stage of swelling had taken place. Also, in contrast to the gelled region where gelatinization was more advanced, there were chalky regions that were unaffected by exposure to microwave radiation as seen in the 1:1 and 1:2 ratios. Since the dielectric loss factor is much less in the chalky region because there are rigid, more crystalline structures within the unchanged granules, less heating will result in these regions as long as less ^c rystalline or amorphous structures also exist in other parts of the samples. This is analogous to the boiling of water when ice is present, as is seen in heating frozen materials by microwave energy. Callaghan et al. (1983) concluded from nuclear magnetic resonance measurements that about 60 % of the starch molecules in extensively heated pastes are in a "mobile liquid-like" polymer form and 40% in an "immobile crystalline" state. Exchange of water occurs between the free water and mobile polymer form and not with the crystalline form. If this is also true under the heating conditions of the present study, interaction with microwave radiation in the crystalline form would be restricted as compared with interactions in the liquid mobile polymer fraction. In the convection mode in the absence of interactions between microwave irradiation and crystal structures, the advancing stages, as water content was increased, might be more closely related to the differences seen on the microscale by differential scanning calorimetry studies as summarized above.

The total sample size of 50 g, although smaller than often used in microwave applications, was large enough so that some heat trans fer by convection within the sample would be expected .

This could lead to localized regions of high and low water content with free water being immobilized as starch transformations occur. The patterns of the gelled and chalky areas for the 1:1 and 1:2 ratios were reproducible, suggesting that heat transfer within the sample vessel also followed consistent patterns .

The patterns in stages of starch trans formations observed at both the macrostructural and microstructural level within a given sample probably contribute to the non-uniformity in texture and other properties seen in microwave-heated starch-based products. For example, Evans et al. (1984) found that the cracking patterns and surface appearance of cakes were related to heating modes (microwave vs convection) and the time during baking at which the heating mode was interchanged .

Excess ive and rapid hardening is also often observed in microwave-heated starch-based products. If development of staling depends on recrystallization of starch, and recrystallization of partially melted crystallites is faster than that of completely melted crystallites, uneven staling and moisture redistribution could occur.

The stages of swelling that were identified suggest that the same overall sequence of swelling was occurring in the various samples. The stages for the large granules were similar to those identified by Bowler et al. (1980) for radial swelling and the initial stages of tangential swelling. When samples differed as to stage, it was not possible to isolate a single cause such as water being limit ing, a heating rate effect, or a microwave radiation coupling effect. Each of these factors interacted, so that a direct effect on starch transformation could not be identified. Further research with smaller samples and more rapid heating rates in microwave and convection/ conduction modes are in progress in an attempt to isolate these heating rate effects from effects inherent in microwave interactions.

- Implications for Microwave Heating
1. Water in part controls the rate of heating in microwave heating, but external temperature controls the rate of sample heating in the convection mode within the water content normally found in food products. Therefore, overall water content as well as localized movement of water will affect rate or kinds of starch granule transitions seen in microwave heating. Greater water content means that there are fewer localized differences in water content and fewer structural differences will be observed .
- 2. When a material such as starch absorbs more water as in gel formation, increased heating rates will occur in that location due to the interaction of microwave radiation with water. Therefore, phase transitions can continue to advance. When a component is more crystalline or latticed, as might be expected in the less advanced stages of starch granule transformations, microwave interaction is smaller and fewer changes occur, especially when the water is more limited.
- 3. When non-uniformity in the stage of granular transformation develops within a sample, nonuniformity in other characteristics that

depend on the stage of gelatinization will be present also.

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Discussion with Reviewers

E.M. Snyder: Since it takes 12-14 minutes for the starch-water mixtures to reach 75° C when heated by convection (Figure 3), does the starch settle out in the higher moisture systems, i.e., 1:4 and 5:95, or at 80% and 90% moisture? Was any effort made to keep water levels constant by covering beakers? D.D. Christianson: Did the granules settle during convection heating in the 5:95 starch:water system? Thermal histories in Figure 3 would indicate this.

Authors: Settling could occur, and might be responsible for the small gel regions at the bottom of the 5:95 samples whether heated by convection or microwave. It could also occur in the 1 :4, but the positions of the swollen and less swollen granules are also consistent with the differences in heat transfer, so the two effects. settling and specifics of heat transfer may be superimposed. The beakers were not covered because the relative water losses during heating by different methods were of interest in the experiments.

E.M. Snyder: In your polarized light microscopy work, were samples suspended in a non-aqueous media as well as water for comparative purposes? (Partially hydrated starch and granules damaged by excessive heat may swell in water at room temperature .)

Authors: No, we did not do this.

E.M. Snyder: Does the amorphous appearing matrix stain blue to iodine? If not, what could it be due to?

Authors: We did not try this because the matrix was not obvious in the light micrographs and was differentiated only by SEM and we do not stain for SEM. However, it would be useful to try some staining with iodine in the future.

E.M. Snyder: Sample preparation of high moisture specimen for SEM has plagued those of us in the food industry for years. We are all too familiar with the various artifacts that are introduced during dehydration procedures. Do you think the fibrous material in stage 6 from preparations originally at 80% and 95% moisture are due to shrinking and shrivelling of starch material as it dries to a tight film? If it is not an artifact, do you have any theory or ideas as to the cause? Was any other dehydration method attempted? Authors: We have been concerned also that the fibrous material may be an artifact arising during drying. However, the artifact is different in the variously heated samples. In freeze-etch studies (Cloke et al. 1982. Freeze-etch of emulsified cake batters during baking. Food Microstructure 1: 17 7-187) we have seen some "beading" close to the granule surface that might be related to the fibrous matrix. We are extending our freezefracture studies, although we recognize that the technique also has potential for development of artifacts (Davis, E.A. and Gordon, J. 1984.

Microstructure analyses in gelling systems. Food Technol. (In press); Davis, E.A. and Gordon, J. 1983. Freeze-fracture, freeze-etch techniques. In New Frontiers in Food Microstructure. Ed. D.B. Bechtel. American Association of Cereal Chemists, Inc., St. Paul, Minnesota pp. 241-268).

D.O. Christianson: It is interesting to speculate that at a higher microwave temperature even more solubilization would occur. Microscopically, the gel should show some correlation with Callaghan 's NMR work.

Authors: We have not attempted to go beyond the upper limits of the gelatinization ranges (approximately 85°C). but we are trying, in experiments currently underway in our laboratory, to increase the rates of heating as compared to those used in this study. To do this, we have used smaller samples and placed the samples directly in the waveguide of a specially designed hybrid convection:microwave oven (Wei, C.K., Davis, H.T., Davis, E.A., and Gordon, J. 1984. Heat and mass transfer in water-laden sandstone: microwave heating. AIChE J. (Submitted.)) We are comparing these samples with samples heated to the same temperature in the same time by conduction in an oil bath. Callaghan et al (1983) reported that they heated their samples for 1 hr at 95°C and then held them at 30°C for 1 hr before making measurements. These conditions are much more drastic than those that we used.

P. Resmini: Have you attempted to support your observations on starch structure by light and scanning electron microscopy with chemical and physical analysis? Authors: We have used differential scanning

calorimetry to determine whether endotherms are found in the various regions to confirm whether starch phase transitions have occurred.

P. Resmini: Are the starch ultrastructure modifications comparable to those found by other authors?

Authors: Relatively few studies of the effects of microwave irradiation on starch granule transformations have been made. The overall sequence of ultrastructure modifications appear similar to those reported by other authors working with a variety of heating methods. No structures that resulted uniquely from microwave heating appeared to be present,

D. Bechtel: In the "Results" section, you mention that "...small granules...progressively clump
and then swell at the edges..." Do the granules and then swell at the edges...' clump and then swell to form a "Swollen, dimpled granule" or does the center collapes? Author: This is a possibility.