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# Impact of microwave heating on the physico-chemical properties of a starch–water model system

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#### **Abstract**

The objective of this work was to understand the physico-chemical changes induced in a wheat starch model system as a result of microwave heating. Wheat starch dispersions in water, with final solids content of 33%, 40% or 50%, were heated in a microwave oven. Following heating the samples were stored at 25 °C for up to 120 h and analyzed periodically. Microwave heated gels were significantly different from conduction heated gels in all parameters measured. The differences in properties are a reflection of the differences in the heat and mass transfer of the different modes of heating. The lack of granule swelling and the resulting soft gel are two key observations. The results of this study suggest a different mechanism of starch gelatinization compared to conduction heating. The vibrational motion and the rapid increase in temperature also result in granule rupture and formation of film polymers coating the granule surface.

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#### 1. Introduction

Microwave heating of foods is a thermal treatment that offers many advantages in processing including less start-up time, faster heating, energy efficiency, space savings, precise process control, selective heating and final products with improved nutritive quality (Sumnu, 2001). However, baking using microwave energy has been limited due to poor final product quality compared to products baked by using conventional energy sources. The causes for these differences in the quality are not fully understood as yet but the specific changes to starch granules are generally thought to contribute to the poor baked product texture. The quality differences are a reflection of the differences in the mechanism of heat and mass transfer (Sakonidou, Karapantsios, & Raphaelides, 2003) or due to the interac-

tions between microwave energy and the individual polar molecules (Goebel, Grider, Davis, & Gordon, 1984).

The published literature with respect to microwave heating of starch suspensions is limited and focuses primarily on the differences in the swelling of starch granule during conduction and microwave heating. However, a comprehensive investigation addressing the changes occurring in starch-water systems during microwave heating at molecular and macroscopic level is missing. Goebel et al. (1984) and Zylema, Grider, Gordon, and Davis (1985) compared the properties of starch-water systems at granular level following microwave heating. Zylema et al. (1985) found no differences in the swelling of granules when heated using microwave or conduction at the same heating rate. Umbach, Davis, and Gordon (1990) reported differences in bagel structure by using microscopy following conventional or microwave baking. Ndife, Sumnu, and Bayindirli (1998) reported the rates of gelatinization for different starches as influenced by microwave heating and also developed a quantitative model describing the relationship between water content and rate of gelatinization for corn, wheat and rice starches during microwave heating. They

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expressed the rate of gelatinization was as a ratio of the gelatinized starch to total starch at a specific time. Sakonidou et al. (2003) postulated that the appearance of chalkiness in starch suspensions following heating in a microwave oven was a result of poor mass transfer of water molecules.

The aim of this work was to investigate and comprehensively characterize the changes in a wheat starch model system at a molecular, granular and macroscopic level at higher starch concentrations.

#### 2. Materials and methods

#### 2.1. Preparation of microwave treated samples

Dispersions of wheat starch (Midsol 50, Midwest Grain Products, Inc., Atchinson, KS, USA) in distilled water were prepared to get final solids concentration of 33%, 40% or 50% on a dry weight basis.

The starch dispersions were mixed by using a Kitchen Aid mixer (Classic Model, St. Joseph, MI, USA) for 10 min at slow speed. The starch dispersions were then heated in a Radarange RC/20SE (Amana Refrigeration Inc., IA, USA) microwave oven (frequency 2450 MHz, power 4300 W) for 10, 20 or 30 s in Pyrex cylinders open at both ends (50  $\varnothing \times$  40 mm). The cylinders were covered with Saranwrap® to minimize moisture loss during heating. The amount of sample heated was 76  $\pm$  0.5 g. Care was taken to place the sample at the same place in the microwave for each treatment.

Before the experiment, the heating time for the microwave treatments was determined by trial and error. Following 5 s of heating the starch was ungelatinized, while after 40 s, the resultant sample was a hard gel due to excessive moisture loss. The temperature at the center of each sample was measured by using a T-type thermocouple periodically.

### 2.2. Sample preparation by using conduction heating

For the preparation of conduction samples, 25 g of water was heated in a 100 ml beaker on a boiling water bath to a temperature of 60 °C. A 50 g starch slurry (1:1: starch:water) was added to this beaker to achieve a final starch concentration of 33%. The sample was stirred gently and heated until a final temperature of 95 °C was reached. The sample was then cooled to room temperature and used for various analyses. The sample took 10 min to attain a temperature of 95 °C. The entire experiment was repeated three times and each set of sample was analyzed by using different techniques in duplicate or triplicate as described below.

# 2.3. Preparation of samples for analyses

Following different treatments, samples were cooled to room temperature (25 °C) and then cored into  $30 \times 30$  mm cylinders. These cylinders were covered with Saranwrap® and stored at room temperature until further

analysis. The samples were analyzed following 2, 24, 72 and 120 h of storage. A second set of samples was freeze dried immediately following respective storage times and milled by using a coffee grinder, sieved through a 250 µm sieve and stored in tightly sealed vials. Freeze-dried samples were used for different analyses as detailed below.

#### 2.4. Moisture and water activity

Sample moisture content was determined by using the standard AACC procedure (American Association of Cereal Chemistry; method 44-19, 2000). Water activity was measured using the AQUALAB CX-2 instrument (Decagon Devices Inc., WA, USA). All analyses were conducted at least in triplicates and the average values are reported.

#### 2.5. Textural properties

Starch gels, stored at 25 °C for the respective storage time, were analyzed using a TA.XT2 instrument (Texture Technologies Inc., NY, USA) with a 1.5 in. diameter cylindrical probe at test speed of 1 mm/s. The gel was compressed by 5 mm. The probe was held at 5 mm for 60 s and then subsequently removed from the gel at a speed of 1 mm/s. From the force–deformation curve, the peak height at 5 mm compression was termed as firmness ( $F_{\rm f}$ ). A minimum of three measurements on different gel samples were taken and the average values are reported.

# 2.6. Thermal properties

Thermal properties of samples were evaluated by using a Differential Scanning Calorimeter (PYRIS 1 DSC, Perkin–Elmer, CT, USA). Approximately 20 mg of freeze-dried sample was weighed into the stainless steel pans and twice the amount of water was added. The pans were sealed and allowed to equilibrate for 20 min before the run. The samples were then scanned from -20 to 160 °C at 10 °C/min and then cooled to -20 °C at 10 °C/min and then rescanned from -20 to 160 °C at 10 °C/min. An empty stainless steel pan was used as a reference. All analyses were conducted in duplicates and the average values are reported.

## 2.7. Enzyme susceptibility

Enzyme susceptibility of the freeze-dried samples was estimated following the procedure described by Cui and Oates (1997). Briefly, 200 mg (dry basis) of lyophilized sample was hydrolyzed with porcine α-amylase (Sigma Chemicals, St. Louis, MO, USA) in 0.1 M phosphate buffer (pH 7.1) for 2 h at 37 °C with continuous shaking. Following hydrolysis, the enzyme was inactivated by adding 1 ml of 0.4 mM mercuric chloride and incubated at 90 °C for 15 min. The amount of soluble carbohydrates in supernatant was estimated by using the phenol sulphuric acid procedure as described by Dubois, Gilles, Hamilton, Rebers, and Smith (1956).

#### 2.8. Microscopy

The lyophilized samples (not ground) were sprinkled on double-sided adhesive allowance tape mounted on aluminum stubs. The samples were then coated with AuPd sputter at the rate of 10 nm/10 s at 10 mA using a Bal-Tec SCD 050 Sputter coater. Following sputtering, the samples were examined with a JSM5400 SEM (JEOL Inc., MA, USA) working at 20 keV accelerating voltage. Granule dimensions from the digital image were calculated using the SPOT 3.5.6 for windows (Diagnostic Instruments Inc., Sterling Heights, MI, USA) software.

#### 2.9. Pasting properties

The pasting properties were determined by using a Rapid Visco-Analyzer IV (RVA; Newport Scientific, Australia) instrument with Thermocline 3.1 software. Twenty eight grams of sample with 14% solids was analyzed by using RVA. The sample was heated from 25 °C to 95 °C in 7 min with a mixing speed of 160 rpm. The sample was held at this temperature for 3.30 min and then it was cooled to 25 °C. Maximum viscosity between 7 and 12 min was reported as peak viscosity, minimum viscosity between 12 and 14 min was reported as trough, and viscosity at the end was reported as final viscosity. Difference between peak viscosity and trough was reported as breakdown and between trough and final viscosity as setback. Each sample was analyzed at least twice and average results are reported.

#### 2.10. Statistical analysis

Statistical analysis was performed using Minitab 14<sup>®</sup> (State College, PA, USA) and SAS (ver 9.1, SAS Institute, Cary, NC, USA). General linear model procedure was used for the statistical analysis. The means were compared using Tukey comparisons at 5% level of significance.

#### 3. Results

The first part of the experiment compared microwave heated samples to samples prepared by conduction heating. In the second part of the study, the microwave treated samples were characterized by using various analytical techniques.

# 3.1. Comparison of microwave- and conduction-heated samples

Table 1 summarizes all the data obtained for starch samples heated by using conduction or microwave energy sources. Following storage, the decrease in the moisture content of the conduction-heated samples was 38% compared to 21% in the microwave-heated samples. The lower initial moisture content 2 h following microwave treatment, as compared to conduction-heated sample, is likely due to flash evaporation as has been suggested previously (Sumnu, Sahin, & Sevimli, 2005; Zylema et al., 1985).

The firmness of the microwave-heated sample was significantly lower than that for conduction-heated samples, despite the lower moisture content in the microwave-heated sample. This observation is significant because it reflects a fundamental difference in the make up of the gel following the two modes of heating. The firmness of the conduction-heated sample increased 4× following 120 h of storage, while that of the microwave-heated sample increased 3× following storage.

Amylopectin recrystallization was observed within 2 h of heating in the microwave-heated samples and the extent of crystallization increased following storage. Amylopectin recrystallization was minimal in samples following 2 h of conduction heating. However, the increase in amylopectin recrystallization following 120 h of storage was lower in the microwave-heated samples when compared to that observed for conduction-heated samples (Table 1). Previous studies have also reported the absence of amylopectin recrystallization in conduction heated samples when measured immediately following heating (Colonna, Leloup, & Buleon, 1992; Cui & Oates, 1997; Eerlingen, Jacobs, & Delcour, 1994). However, Seetharaman, Chinnapha, Waniska, and White (2002) reported the presence of amylopectin crystals in tortillas within 20 min following baking due to the limited granular disruption during tortilla baking. The higher initial amylopectin recrystallization in microwave-heated samples suggests a closer proximity of these polymers in the gel, leading to a more rapid and greater amount of crystal formation.

Table 1
Properties of 50% solids concentration conduction-heated and microwave-heated samples following 2 and 120 h of storage at 25 °C after the respective heating treatments

Property measured	Conduction-heated	l sample	Microwave-heated sample		
	2 h	120 h	2 h	120 h	
Moisture content (%)	42.9ax <sup>a</sup>	27.0by*	36.5ay	28.7bx	
Force of firmness (g)	1873.25bx	7433.1ax	148.10by	457.8ay	
Enthalpy of amylopectin recrystallization (J/g)	0.03by	0.97ax	0.23bx	0.64ay	
Enzyme susceptibility (g/g of sample)	0.93ax	0.73bx	0.67ay	0.27by	

<sup>&</sup>lt;sup>a</sup> Values followed by the letters x and y compare data for conduction and microwave treatment at the same storage time.

<sup>\*</sup> Values followed by the same letter not significantly different (P < 0.05).

The amount of reducing sugars produced by the action of  $\alpha$ -amylase was lower for the microwave-heated samples when compared to the conduction-heated samples suggesting greater granular disruption following conduction heating. However following storage, the decrease in enzyme susceptibility was lower for the conduction-heated sample suggesting greater reassociation of leached polymers following conduction heating.

The differences in the pasting properties of conduction and microwave-heated samples are summarized in Table 2. The pasting temperature of the microwave-heated sample was significantly lower than that observed for conduction-heated samples suggesting the initiation of viscosity development at temperatures a little above room temperature. The peak viscosity was higher in the microwave-heated samples while the final viscosity was lower when compared to conduction-heated samples following 2 or 120 h of storage. The higher peak viscosity of microwaveheated samples suggests that the granular integrity was not completely destroyed during microwave heating while the granules were more pasted following conduction heating as has been reported earlier (Hoseney, 1994). Furthermore, the lower final viscosity of microwave heated samples when compared to conduction heated samples, likely suggest the differences the extent of amylose leaching and its contribution to the setback phenomenon.

All of the above observations indicate that the properties of gels formed as a result of microwave heating are different from those observed for conduction-heated gels. The basis for these differences is likely to the mode of heating when heated using microwave energy; i.e., very high heating rate. The rapid heating rate during microwave heating likely results in lesser amount of leached amylose into the

extragranular matrix and also a weaker amylose network formation. This is reflected in the softer gel texture and rapid rehydration observed RVA analysis. These observations lay the basis for the basic differences in starch gel properties following conduction and microwave heating. However, there is a paucity of data in the literature that comprehensively informs on the different attributes of a gel following microwave heating. The next section, therefore, delves further into characterizing the microwave-heated gels and the properties of starch in the gel matrix.

#### 3.2. Characterization of microwave-heated samples

All microwave treatments were conducted in a microwave oven with a stated power of 4300 W. However, the microwave power absorbed was calculated to be 1140 W. All samples were of the same weight, same dimension and were placed at the same spot within the microwave oven. This was done to minimize the changes in power absorbed by the sample during heating due to the sample geometry, mass or the cavity in the microwave oven. The final temperatures attained (Table 3) indicate that all samples absorbed the same amount of power, irrespective of the solids content. The final temperatures, however, increased with increasing heating time (P < 0.00, where P is the power of the test).

#### 3.2.1. Moisture and water activity

Moisture content decreased significantly as a function of heating time and storage time for a given solids concentration (Table 4). No significant differences were observed in the sample water activity (data not shown) following various treatments.

Table 2 Comparison of pasting properties of raw starch, 50% solids concentrations samples after microwave and conduction heating following 2 and  $120\,\mathrm{h}$  of storage at  $25\,^\circ\mathrm{C}$ 

	Raw starch	Conduction-hea	ted sample	Microwave-heated sample	
		2 h	120 h	2 h	120 h
Peak viscosity (cP)	13,725 (±1.29) <sup>a</sup>	4931ay*	4040by**	6881ax	7765bx
Trough (cP)	$3097 (\pm 0.56)$	3834ay	3639by	4052ax	3566bx
Breakdown (cP)	$10,628~(\pm 2.0)$	1097ay	401by	2487ax	4199bx
Final viscosity (cP)	all viscosity (cP) 9545 $(\pm 2.4)$		12,887bx	11,823ax	10,956by
Setback (cP)	back (cP) 6448 (±1.8)		12,486bx	8235ax	7390by
Pasting temperature (°C)	$63.2~(\pm 0.4)$	51.4ax	54.9bx	34.6ay	38.3by

<sup>&</sup>lt;sup>a</sup> Values in parenthesis denote the standard deviation among measurements.

Table 3
Final temperatures attained by the samples following microwave treatment

Heating time (s)	50% solids	40% solids	33% solids
10	94.4 °C (±0.3) <sup>a</sup>	94.7 °C (±0.4)	94.8 °C (±0.1)
20	97.5 °C (±0.2)	97.6 °C (±0.3)	97.8 °C (±0.4)
30	98.3 °C (±0.2)	98.4 °C (±0.1)	98.6 °C (±0.0)

<sup>&</sup>lt;sup>a</sup> Values in parenthesis denote the standard deviations across measurements.

<sup>\*</sup> Values followed by the letters x and y compare data for conduction and microwave treatment at the same storage time. Values followed by the same letter not significantly different (P < 0.05).

<sup>\*\*</sup> Values followed by the letters a and b compare data for 2 and 120 h within each mode of heating. Values followed by the same letter are not significantly different (P < 0.05).

Table 4
Moisture content of microwave-heated samples following different heating time and stored for different time at 25 °C

Time (s)	50% solids		40% solids			33% solids			
	10	20	30	10	20	30	10	20	30
Storage (h)									
2	36.49ax*	31.81ay**	29.65az	43.54ax	39.67ay	37.28az	52.93ax	49.03ay	45.86az
24	36.27ax	31.01by	29.01az	43.23ax	38.85by	36.00bz	51.58bx	47.77by	43.78bz
72	32.17bx	29.92cy	27.88bz	41.99bx	36.39cy	34.76cz	49.99cx	45.75cy	41.66cz
120	28.67cx	28.26dx	25.62cy	36.97cx	33.72dy	30.71dz	42.39dx	38.55dy	37.19dz

<sup>\*</sup> Values followed by the same letter (x-z) in the same row within the same solids content are not significantly different (P < 0.05).

# 3.2.2. Microscopic observations

The average size of the native A- and B-type starch granules was 19  $\mu$ m and 9  $\mu$ m, respectively (Fig. 1A). Following microwave treatment, the granule size across all heating times ranged from 10 to 28  $\mu$ m (Fig. 1C and D).

These observations suggest that the granules did not swell following microwave heating. Following 30 s of heating the granules appeared to be covered by a film of molten polymers (Fig. 1D). Fig. 1E shows granule rupture following 10 s of microwave heating and at the end of 30 s a film of

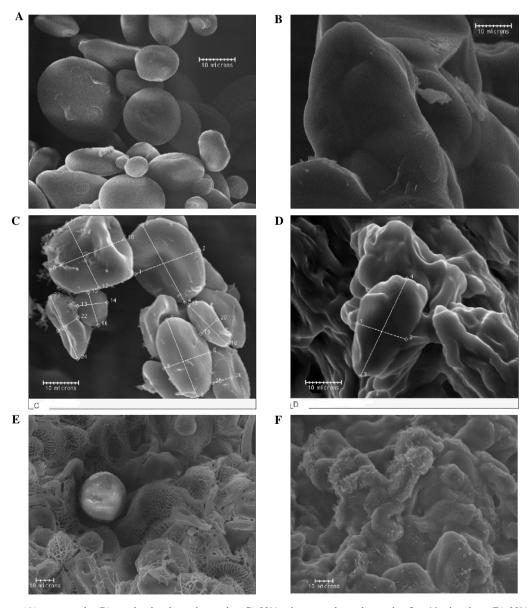


Fig. 1. SEM pictures (A) raw starch, (B) conduction-heated sample, (C) 33% microwave-heated sample after 10 s heating, (D) 33% microwave-heated sample after 30 s heating, (E) 50% microwave-heated sample after 30 s heating. The length of the scale bar is equivalent to 50 microws.

<sup>\*\*</sup> Values followed by the same letter (a-d) in the same column are not significantly different (P < 0.05).

molten polymers covered all granules (Fig. 1F). Observations using light microscopy of microwave-heated samples revealed ruptured granules and extensive leaching of polymers in the background (data not shown). These images demonstrate that microwave treatment restricts granule swelling and disrupts granules such that the polymers are spilled into the medium rather than leaching. Granule swelling in concentrated systems is dependent on the space available (Keetels, van Vliet, & Walstra, 1996a). However, the spatial limitation did not restrict granule swelling during conduction heating (Fig. 1B). In microwave-heated samples, it is likely that kinetic limitation and not the spatial restriction that is the reason for the lack of swelling.

# 3.2.3. Textural properties

The firmness values of the gels increased with increasing heating time, solids content and storage time (Fig. 2). The increase in the firmness value of 50% solids sample was greater than that observed in the 33% or 40% starch samples stored for 120 h. The higher rate of increase in the firmness of the 50% starch gel is likely due to the greater decrease in moisture and possibly the greater proximity of the polymers thus increasing the extent of complex formation between them. This is also corroborated by the higher increase in amylopectin recrystallization observed in these samples as will be discussed below. As stated earlier, the firmness of these samples and the increase in firmness following storage is still significantly lower that observed for conduction-heated samples.

Previous research has shown that the firmness of starch gels heated by conduction heat, increases upon storage (Keetels, van Vliet, & Walstra, 1996b; Miles, Morris, Orford, & Ring, 1985a; Miles, Morris, Orford, & Rings, 1985b; Vandeputte, Derycke, Geeroms, & Delcour, 2003). Eliasson and Bohlin (1982) reported that starch gels have

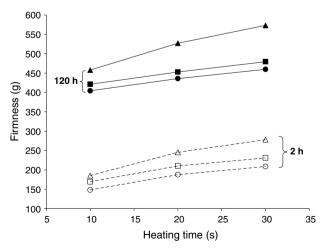


Fig. 2. Firmness of samples as a function of heating time (s). Symbols  $\triangle$ ,  $\square$  and  $\bigcirc$  stand for 50%, 40% and 33%, respectively. The filled symbols and solid lines represent measurements after 120 h of storage whereas the empty symbols and dashed lines represent measurements after 2 h of storage.

a continuous phase of polysaccharides in water with the granules acting as filler materials and the leached amylose decreased the modulus of elasticity and thus increased gel rigidity. Miles et al. (1987a) reported that the properties of the gel partly depend on the swollen gelatinized granules or granular fragments. Ring (1985) reported that for concentrated gels, swollen granules increased the modulus of amylose gels. It is likely that the lack of granule swelling was the cause for weaker gels following microwave treatment. Therefore, in the absence of significant granule swelling, formation of gel is likely due to the concentration of the gel, i.e., loss of moisture, and potentially due to the

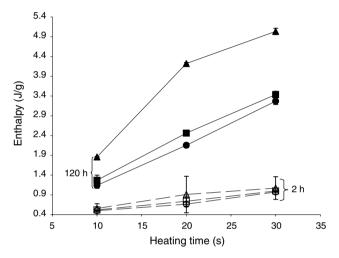


Fig. 3. Enthalpy of amylopectin recrystallization of freeze-dried samples (1:2 : starch:water) by DSC as a function of heating time (s). Symbols  $\triangle$ ,  $\square$  and  $\bigcirc$  stand for 50%, 40% and 33%, respectively. The filled symbols and solid lines represent measurements after 120 h of storage whereas the empty symbols and dashed lines represent measurements after 2 h of storage. The error bars show the standard deviation among the readings.

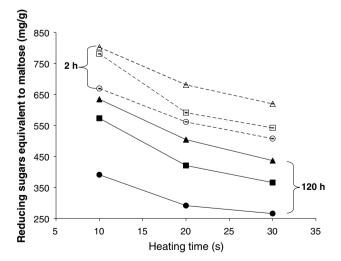


Fig. 4. Amount of reducing sugars per gram of sample (d.b.) produced by the action of  $\alpha$ -amylase. Symbols for  $\Delta$ ,  $\square$  and  $\bigcirc$  stand for 50%, 40% and 33%, respectively. The filled symbols and solid lines represent measurements after 120 h of storage whereas the empty symbols and dashed lines represent measurements after 2 h of storage. The error bars represent the standard deviations among the readings but the error bars are too small to be seen.

polymers leaching from the granules. Weaker gels also suggest the absence of a continuous network formation through the leaching of amylose. Waxy corn starch gels, which are devoid of amylose, are softer gels compared to gels prepared from common corn starch (Czuchajowska, Klamczynski, Paszczynska, & Baik, 1998).

#### 3.2.4. Thermal properties

The microwave treated samples were completely gelatinized based on the absence of enthalpy of gelatinization when measured by using a DSC immediately after the microwave treatment (data not shown). The thermal profile

of freeze-dried samples in excess water exhibited an amylopectin recrystallization melting endotherm following 2 h of storage (Fig. 3). The enthalpy of amylopectin recrystallization increased significantly following storage, as has also been reported by other researchers (Miles et al., 1985a; Miles et al., 1985b), and the increase was highest for 50% solids sample. Following storage for 120 h, the amylopectin recrystallization melting enthalpy increased and the increase was significantly higher for the 50% starch sample. This is likely due to an increase in the proximity of the polymers in the gel resulting in greater recrystallization and is also reflected in the increased firmness of these samples.

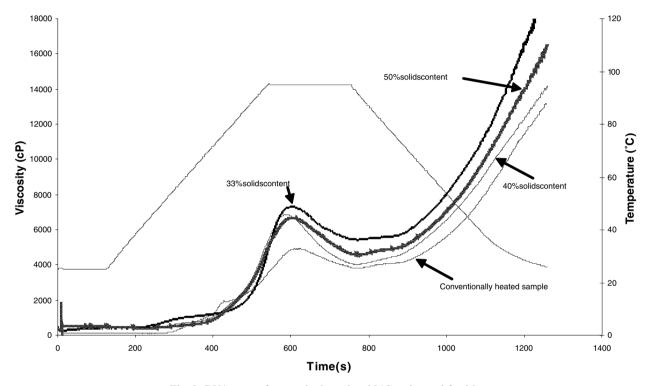


Fig. 5. RVA curves for samples heated to 95  $^{\circ}\text{C}$  and stored for 2 h.

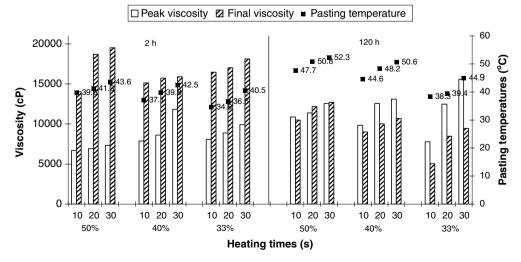


Fig. 6. Pasting properties of microwave heated samples after 2 h and 120 h of storage measured by using RVA.

#### 3.2.5. Enzyme susceptibility

The enzyme susceptibility of the samples decreased with increasing heating time and with decreasing solids concentration (Fig. 4). Furthermore, enzyme susceptibility decreased following storage for 120 h. Similar results have also been reported by other researchers (Cui & Oates, 1997; Eerlingen et al., 1994; Fredriksson et al., 2000; Ring, Gee, Whittam, Orford, & Johnson, 1988). The decreasing enzyme susceptibility is indicative of increasing polymer re-association.

#### 3.2.6. Pasting properties

The initial viscosity, at room temperature, of the microwave-heated samples was significantly higher than that observed for raw starch (Fig. 5). The pasting temperature and the peak viscosity of the microwave-heated samples were lower than that observed for raw starch. The peak viscosity, final viscosity and pasting temperature increased with increasing time of heating (Fig. 6). The pasting temperature increased following storage while the final viscosity decreased following storage. The differences in the pasting properties, especially when compared to conduction-heated samples, highlight the differences in the structural aspects of the starch granule following microwave heating.

# 3.3. Mechanism of heating and gelatinization during microwave heating

Based on the above data, it is evident that heating by using microwave energy results in starch with different properties compared to that heated by conduction heat. The differences can be attributed to aspects related to rate of heating, which is very high when using microwave energy, and possibly due to the mechanism of heating during microwave heating. Heat produced during microwave heating, is the result of friction generated from the rotational forces on the polar molecules present in the sample as they attempt to orient themselves in the direction of the oscillating microwave field (Buffler, 1993). The dielectric properties of the starch-water system affect the heat generated or the temperature increase in the system. In this study, water molecules were the primary polar molecules present in the system. It is likely that the microwave energy also vibrates the water molecules present in the crystalline regions of the starch granules thereby destroying the lamellar arrangement of the amylopectin crystals, even before the system reaches gelatinization temperature and possibly fragment the granule at higher temperatures.

At slower heating rates, as is typical during conduction heating, the temperature increase is gradual and there is adequate time available for starch granules to undergo all the steps involved in gelatinization; i.e., granule swelling, loss of birefringence, amylose leaching and granule folding. However, during microwave heating, due to the rapid heating rates and vibrational motion of the water molecules,

the granules are subjected to a rapid increase in temperature. The rapid rise in temperature and mechanism of heating possibly results in restriction of granule swelling and the rupture of granules. An alternative theory could be that there is a rapid generation of pressure within the granule during microwave heating resulting in the rapid expansion of granules. However, granule hydration fails to keep pace with granule expansion and the resultant stress generated causes the collapse of granules and in some cases rupture of the granules. The SEM pictures that show restricted swelling of the granules provide the strongest evidence for this theory. The presence of amylopectin crystals within 2 h of heating, softer gel and the reduced enzyme susceptibility further support this theory.

#### 4. Conclusions

The results presented in this study show that resultant gels formed by heating starch slurries by using microwave energy had significantly different properties than those heated by using conduction heat. These differences in the properties are a reflection of the differences in the heat and mass transfer due to the different modes of heating. The lack of granule swelling and the resulting soft gel are two key observations that highlight the differences in the two modes of heating. The significant differences in the other molecular properties, including enzyme susceptibility and amylopectin recrystallization, suggest a different mechanism of gelatinization during microwave heating. We propose that starch granules during microwave heating lose their birefringence much earlier than gelatinization temperature due to the vibrational motion of the polar water molecules. The vibrational motion and the rapid increase in temperature also result in granule rupture and formation of film polymers coating the granule surface. This results in a soft gel even in the absence of a continuous network of amylose chains.

#### References

American Association of Cereal Chemists (2000). Approved methods of the AACC 10th Ed. Methods 44-19. The Association of Cereal Chemists: St. Paul, MN.

Buffler, C. R. (1993). *Microwave cooking and processing*. Van Nostrand: Reinhold.

Colonna, P., Leloup, V., & Buleon, A. (1992). Limiting factors of starch hydrolysis. European Journal of Clinical Nutrition, 466, 17–32.

Cui, R., & Oates, C. G. (1997). The effect of retrogradation on enzyme susceptibility of sago starch. *Carbohydrate polymers*, 32, 65–72.

Czuchajowska, Z., Klamczynski, A., Paszczynska, B., & Baik, B. K. (1998). Structure and functionality of barley starches. *Cereal Chemistry*, 75(5), 747–754.

Dubois, M., Gilles, K. A., Hamilton, J. K., Rebers, P. A., & Smith, F. (1956). Colorimetric method for determination of sugars and related substances. *Analytical Chemistry*, 28, 350–356.

Eerlingen, R. C., Jacobs, H., & Delcour, J. A. (1994). Enzyme-resistant starch. V. Effect of retrogradation of waxy maize starch on enzyme susceptability. *Cereal Chemistry*, 71,3(4), 351–355.

Eliasson, A. C., & Bohlin, L. (1982). Rheological properties of concentrated wheat starch gels. Starch/Starke, 34, 267–271.

- Fredriksson, H., Bjorck, I., Andersson, R., Liljeberg, H., Silverio, J., Eliasson, A. C., et al. (2000). Studies on α-amylase degradation of retrograded starch gels from waxy maize and high-amylopectin potato. *Carbohydrate Polymers*, 43(1), 81–87.
- Goebel, N. K., Grider, J., Davis, E. A., & Gordon, J. (1984). The effects of microwave energy and convection heating on wheat starch granule. *Food Microstructure*, 3(1), 73–82.
- Hoseney, R. C. (1994). *Principles of cereal science and technology* (2nd ed.). St. Paul: American Association of Cereal Chemists.
- Keetels, C. J. A. M., van Vliet, T., & Walstra, P. (1996a). Gelation and retrogradation of concentrated starch systems: 1. Gelation. Food Hydrocolloids, 10, 343–353.
- Keetels, C. J. A. M., van Vliet, T., & Walstra, P. (1996b). Gelation and retrogradation of concentrated starch systems: 2. Retrogradation. Food Hydrocolloids, 10, 355–362.
- Miles, M. J., Morris, V. J., Orford, P. D., & Ring, S. G. (1985a). Recent observations on starch retrogradation. In R. D. Hill & L. Munck (Eds.), New approaches to research on cereal carbohydrates. Amsterdam: Elsevier, pp. 109–114.
- Miles, M. J., Morris, V. J., Orford, P. D., & Rings, S. G. (1985b). The roles of amylose and amylopectin in the gelation and retrogradation of starch. *Carbohydrate Research*, 135(10), 271–281.
- Ndife, M., Sumnu, G., & Bayindirli, L. (1998). Differential scanning calorimetry determination of gelatinization rates in different starches due to microwave heating. *Lebensmittel-Wissenschaft-und-Technologie*, 31, 484–488.

- Ring, S. G. (1985). Some studies on starch gelation. *Starch/Starke*, 37(3), 80–83.
- Ring, S. G., Gee, J. M., Whittam, M., Orford, P., & Johnson, I. T. (1988).
  Resistant starch: Its chemical form in foodstuffs and effect on digestibility in vitro. *Food Chemistry*, 28, 97–109.
- Sakonidou, E. P., Karapantsios, T. D., & Raphaelides, S. N. (2003). Mass transfer limitations during starch gelatinization. *Carbohydrate Polymers*, 53, 53–61.
- Seetharaman, K., Chinnapha, N., Waniska, R. D., & White, P. (2002). Changes in the textural, pasting and thermal properties of wheat buns and tortillas during storage. *Journal of Cereal Science*, 35, 215–223.
- Sumnu, G. (2001). A review on microwave baking of foods. *International Journal of Food Science and Technology*, 36, 117–127.
- Sumnu, G., Sahin, S., & Sevimli, M. (2005). Microwave, infrared and infrared-microwave combination baking of cakes. *Journal of Food Engineering*, 71(2), 150–155.
- Umbach, S. L., Davis, E. A., & Gordon, J. (1990). Effects of heat and water transport on the Bagel-Making process: Conventional and microwave baking. *Cereal Chemistry*, 67, 355–360.
- Vandeputte, G. E., Derycke, V., Geeroms, J., & Delcour, J. A. (2003).
  Rice starches. II. Structural aspects provide insight into swelling and pasting properties. *Journal of Cereal Science*, 38, 53–59.
- Zylema, B. J., Grider, J. A., Gordon, J., & Davis, E. A. (1985). Model wheat starch systems heated by microwave irradiation and conduction with equalized heating times. *Cereal Chemistry*, 62, 447–453.