

Mechanism of starch gelatinization and polymer leaching during microwave heating

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Abstract

The objective of this study was to investigate the process of gelatinization, granule swelling, and polymer leaching following microwave heating of wheat starch suspensions. Polymer leaching was investigated as a function of heating rate, final temperature and starch concentration. Different heating rates were achieved by changing the mass of the starch slurry heated in the microwave oven and the temperature study was conducted on samples of same weight heated to different final temperatures. The total soluble carbohydrate and amylose content in the supernatant was measured. Granule swelling and leaching of polymers occurred only after complete loss of granule birefringence presenting evidence of an asynchronous process of gelatinization compared to conduction modes of heating. The amount of soluble carbohydrate and amylose in the supernatant increased with the increase in temperature and the amylose leached into supernatant decreased with increasing heating rates.

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1. Introduction

Microwaves have found application in various food processing operations such as tempering, thawing, cooking, pasteurization, drying, and expansion (Decareau & Peterson, 1986). Microwaves are electromagnetic waves in the frequency range of 300–300,000 MHz. Polar molecules, such as water or ions, absorb microwave energy and try to orient themselves with respect to the electric field. The rapid change in their orientation generates heat by the molecular friction caused by disruption of weak hydrogen bonds (Sumnu, 2001).

Although microwaves have many advantages, the quality of products baked using microwaves are inferior compared to those baked using conventional energy sources (Bell & Steinke, 1991; Ovadia & Walker, 1995). Previous

studies that compared microwave heating to conventional heating have been restricted to product quality attributes and do not focus on the changes to starch in the system (Icoz, Sumnu, & Sahni, 2004; Martin & Tsen, 1981; Sumnu, Sahin, & Sevimli, 2005; Umbach, Davis, & Gordon, 1990). Furthermore, studies related to changes in starch granules during microwave heating in model systems are limited, and focus only on the comparison of granule swelling patterns between microwave and convection heated starch suspensions or starch based foods (Goebel, Grider, Davis, & Gordon, 1984; Zylema, Grider, Gordon, & Davis, 1985). Studies related to starch gelatinization as affected by microwave heating are missing in the literature to the best of our knowledge. This study, therefore, elaborates on the particular differences in the mechanism of starch gelatinization by focusing on the individual events that comprise gelatinization when heated by using microwave energy.

The process of gelatinization has been extensively investigated over the past three or more decades. All of the current understandings of gelatinization are based on heating starch granules by using conduction heating modes. Under

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these conditions, gelatinization is understood as the cumulative irreversible changes that occur to a starch granule in the presence of moisture and heat. The changes include granule swelling due to absorption of moisture in the amorphous regions of the granule, leaching of small molecular weight polymers including amylose, loss of the crystalline order and the consequent loss of birefringence, leaching of larger molecular weight polymers from the granule including fragments of amylopectin and, finally, starch solubilization (Atwell, Hood, Lineback, Varriano-Marston, & Zobel, 1988; Leach, 1967; Olkku & Rha, 1978; Osman, 1965; Sakonidou, Karapantsios, & Raphaelides, 2003). The specific changes occur sequentially and synchronously. We hypothesized that the specific events of starch gelatinization during microwave heating will be different from that observed following conduction modes of heating due to the particular mechanism of heating by microwave energy. The objective of the study therefore was to investigate the mechanism of gelatinization as influenced by the starch concentration, heating rate and final temperature when heated by using microwave energy.

2. Materials and methods

2.1. Materials

The wheat starch used in the study was MIDSOL-50 (Midwest Grain Products, Inc., KS, USA).

2.2. Methods

2.2.1. Heating conditions

Starch samples with predetermined starch concentrations were made by using wheat starch and deionized water. The samples were stirred gently just before heating them in the microwave oven in order to ensure that the starch was fully suspended in the water. A General Electric turntable microwave (Louisville, KY, USA) oven with output power 1.3 kW was used for conducting the experiments in this study. In order to avoid any confounding effects due to change in power absorbed due to the position of the sample in the microwave, care was taken that the sample was always placed at the same place within the oven. Immediately following the heating treatment, sample temperatures were measured by using a T-type thermocouple. Multiple replications were conducted in the preliminary stages to determine the time required to reach specified temperatures for each treatment. The sample dimension and weight were chosen specifically to minimize the temperature gradient in the sample. The recorded temperatures are the average sample temperatures.

2.2.2. Effect of final temperature

A 30 g starch–water suspension at 1%, 5%, 7% or 8% starch concentration were heated in the microwave oven to achieve final temperatures between 60 and 95 °C. The details of the analysis of the supernatant are described below.

2.2.3. Effect of heating rate

The power of the microwave oven was calculated by using the following equation (Sumnu et al., 2005):

$$mC_p\Delta T/\Delta t = 14.32P, \quad (1)$$

where m is the mass of sample in grams, C_p is the specific heat capacity of the substance in cal/g °C, $\Delta T/\Delta t$ is the heating rate in °C/m, and P is the power in Watts.

Based on Eq. (1), the heating rate is inversely proportional to the mass of the sample when heated in a microwave oven. Therefore, to vary the heating rate, the amount of sample heated in the microwave was varied from 5 to 50 g. Different mass of samples of 1%, 5%, 7% or 8% starch concentration were heated in the microwave oven to a final temperature of 75 °C. The supernatant was then separated and analyzed as detailed below.

2.2.4. Sampling and analytical techniques

Following different treatments, the samples were cooled to 25 °C and an equal amount of water at 25 °C was added to the sample. The samples were vortexed at low speed for 5 s and centrifuged at 2000g for 5 min. The supernatant was then decanted and used for further analysis.

2.2.4.1. Soluble carbohydrate content. The supernatant was analyzed for soluble carbohydrate content by using the phenol-sulphuric acid method described by Dubois, Grilles, Hamilton, Rebers, and Smith (1956). All analyses were conducted in at least duplicates and the average values are reported.

2.2.4.2. Amylose determination. The amount of amylose leached was determined by treating supernatant equivalent to 1 mg of soluble carbohydrates with 0.2% KI and 0.02% I₂. The color developed was quantified after 20 min at 620 nm by using a Thermo Spectronic Heλios α spectrophotometer. A standard curve was obtained by using the same procedure for aliquot samples of pure amylose solutions of known concentrations. Amylose was fractionated from high amylose corn starch following the procedure described by Klucinec and Thompson (1998). All analyses were conducted in at least duplicates and the average values are reported.

2.2.4.3. Microscopy. Treated starch samples were observed by using light and polarized light microscope, observed under 40× magnification by using an Olympus BX41TF microscope (Olympus Optical Co. Ltd., Tokyo, Japan). The images were captured using SPOT Insight QE camera (Diagnostic Instruments Inc., MI, USA). Samples observed under light microscope were stained with vapors of Lugol's solution for 1–2 min based on the procedure described by Hug-Iten, Escher, and Conde-Petit (2001).

2.2.4.4. Thermal analyses. An 8% starch sample heated to different final temperatures was analyzed by using a Differential Scanning Calorimeter (PYRIS 1 DSC, Perkin-Elmer,

CT, USA). Approximately 20 mg of sample was weighed into a stainless steel pan and hermetically sealed. Each sample was scanned from 20 to 160 °C at 10 °C/min and then cooled to 20 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 duplicate and the average values are reported.

3. Results

3.1. Microscopy and thermal analyses

Fig. 1 shows the polarized light microscope images for raw starch and microwave-treated starches heated to final temperatures of 55, 60 or 65 °C. At 55 °C, about half of the starch granules exhibited birefringence, although the starch granule size did not appear to have changed. At 60 °C, only the B-type, small starch granules, exhibited birefringence, again with little granule swelling. At 65 °C, all starch granules lost their birefringence and the granules exhibited an increase in size. Fig. 2 shows the light microscopic images of granules heated to final temperatures of 65, 75, 85 or 95 °C. The granules are swollen and folded at 65 °C with little or no leachate in the suspension background. However, with increase in temperatures beyond 65 °C, there is significant amount of leachate in the suspension and the granules appear ruptured with granule remnants visible. At 95 °C, the granules appeared completely ruptured with dark clumps of staining visible.

The gradual loss of birefringence observed by using polarized microscopy is also reflected in the data obtained by using the DSC (Table 1). There is an almost 10-fold

decrease in enthalpy between raw starch and starch heated to 55 °C and a further decrease in enthalpy at 60 °C. Furthermore, the remnant crystals in samples heated to a final temperature of 60 °C melted at a higher temperature suggesting the melting of B-type starch granules.

3.2. Effect of final temperature and concentration

The amount of soluble carbohydrate in the supernatant and the proportion of amylose in the supernatant as a function of final temperature are shown in Figs. 3 and 4, respectively. The soluble carbohydrate content in the supernatant increased with increasing temperature at all starch concentrations and the soluble carbohydrate content was lower at the higher starch concentrations across all temperatures. The increase in soluble carbohydrate content in the 1% starch suspension beyond 80 °C suggests complete disintegration of the granules as is also evident in the microscopic images shown in Fig. 2c.

The proportion of amylose in the soluble carbohydrates present in the supernatant, on the other hand, was higher at the higher starch concentrations and increased with increasing temperature (Figs. 4a and b). The absolute amount of amylose in the supernatant was higher at the higher starch concentration at the lower temperature (Fig. 4a). As the temperature increased beyond 65 °C, the amount of amylose in the supernatant did not increase as rapidly for the 8% starch suspension. The absolute amount of amylose leached into the supernatant was highest for the 1% starch suspension heated to 95 °C. At 95 °C, almost 95% of the soluble carbohydrate in the supernatant was amylose in the 8% starch suspension (Fig. 4b).

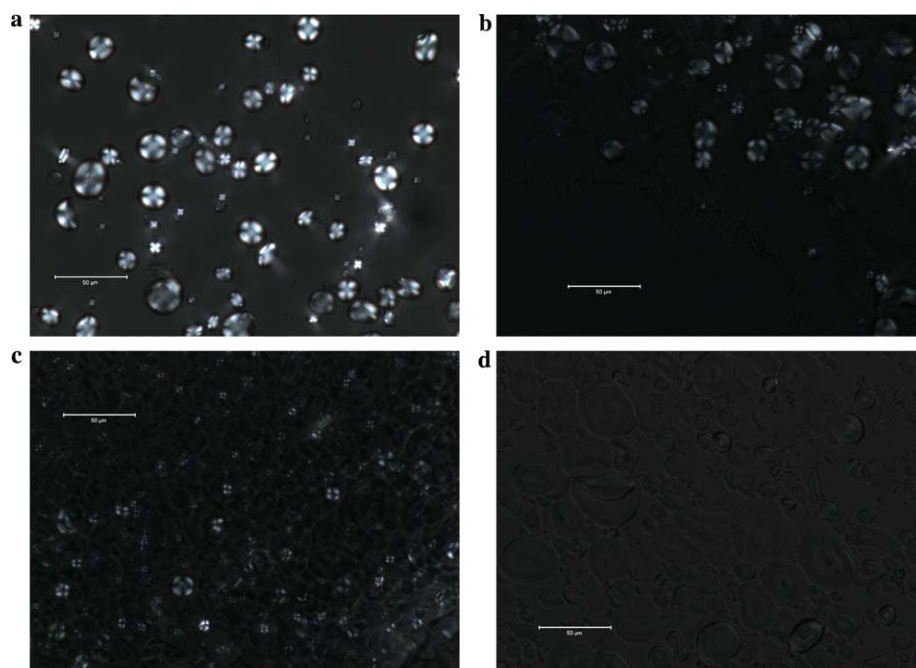


Fig. 1. Polarized light microscopic images of 8% starch samples heated in microwave oven to the stated temperatures (a) raw starch; (b) 55 °C; (c) 60 °C; (d) 65 °C. Scale bar corresponds to 50 μm.

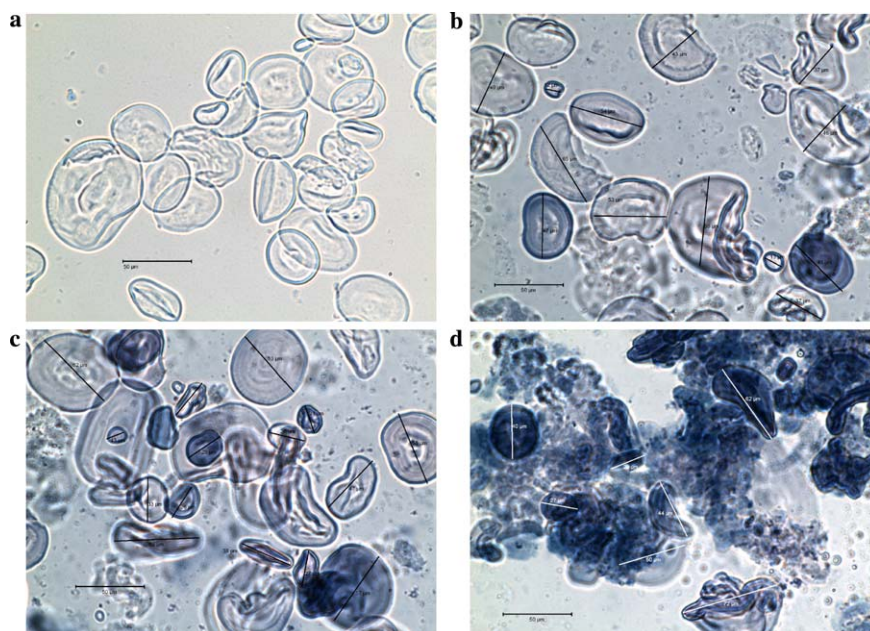


Fig. 2. Microscopic images of 5% starch sample heated in microwave oven to (a) 65 °C; (b) 75 °C; (c) 85 °C; (d) 95 °C. The starch samples have been exposed to iodine vapor and the blue color represents amylose. Scale bar corresponds to 50 μm .

Table 1

Thermal properties of 8% starch sample heated to different final temperatures as measured by using a differential scanning calorimeter

Temperature (°C)	T_0 (°C) ^a	T_e (°C) ^b	ΔH (J/g) ^c
Raw starch	63.5 (± 0.12) ^d	72.1 (± 0.08)	12 (± 0.14)
55	62.0 (± 0.2)	76.3 (± 0.1)	1.44 (± 0.05)
60	66.3 (± 0.09)	69.0 (± 0.06)	0.43 (± 0.02)
65	— ^e	—	—

^a Onset temperature of gelatinization.

^b End temperature of gelatinization.

^c Enthalpy of gelatinization.

^d Standard deviations are indicated in parenthesis.

^e No endotherm observed.

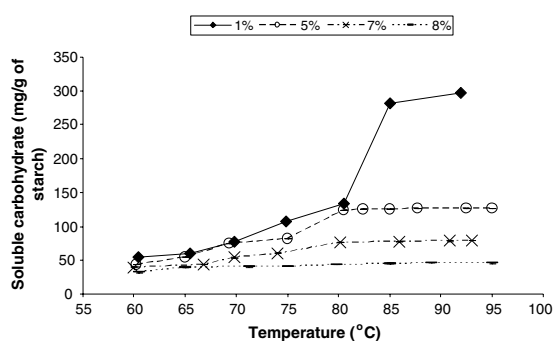


Fig. 3. Amount of soluble carbohydrates leached as a function of temperature. The error bars show the standard deviation across measurements, but are too small to be seen.

3.3. Effect of heating rate

The heating rates achieved in the experiment ranged from 90 to 350 °C/m (Fig. 5). A crossover trend was

observed for the leaching of soluble carbohydrates as a function of heating rate with increasing starch concentration. At the 1% and 5% starch concentrations, the amount of soluble carbohydrate leached increased with increasing heating rate and the magnitude of increase was greater for the 1% starch suspension compared to 5% starch suspension. At 7% starch concentration, soluble carbohydrate concentration in the supernatant increased up to about 125 °C/m heating rate after which the level of soluble carbohydrate leached remained fairly constant. In contrast, the amount of soluble carbohydrate leached from 8% starch suspension decreased with increasing heating rates. The proportion of amylose in the supernatant decreased with increasing heating rate at all starch concentrations. Furthermore, the proportion of amylose in the supernatant was higher at the higher starch concentration (Fig. 6).

4. Discussions

In a previous study in our laboratory, we observed that the mechanism of heating by using microwave energy resulted in different gel properties when compared to a conduction heated suspension (Palav & Seetharaman, 2006; manuscript in review). We proposed that the mechanism of gelatinization due to microwave heating was different compared to conduction heating due to both the higher heating rates achieved and the vibrational motion of the polar molecules due to microwave energy. This work further investigates the leaching of polymers and gelatinization during microwave heating as a function of starch concentration, final temperature, and heating rate.

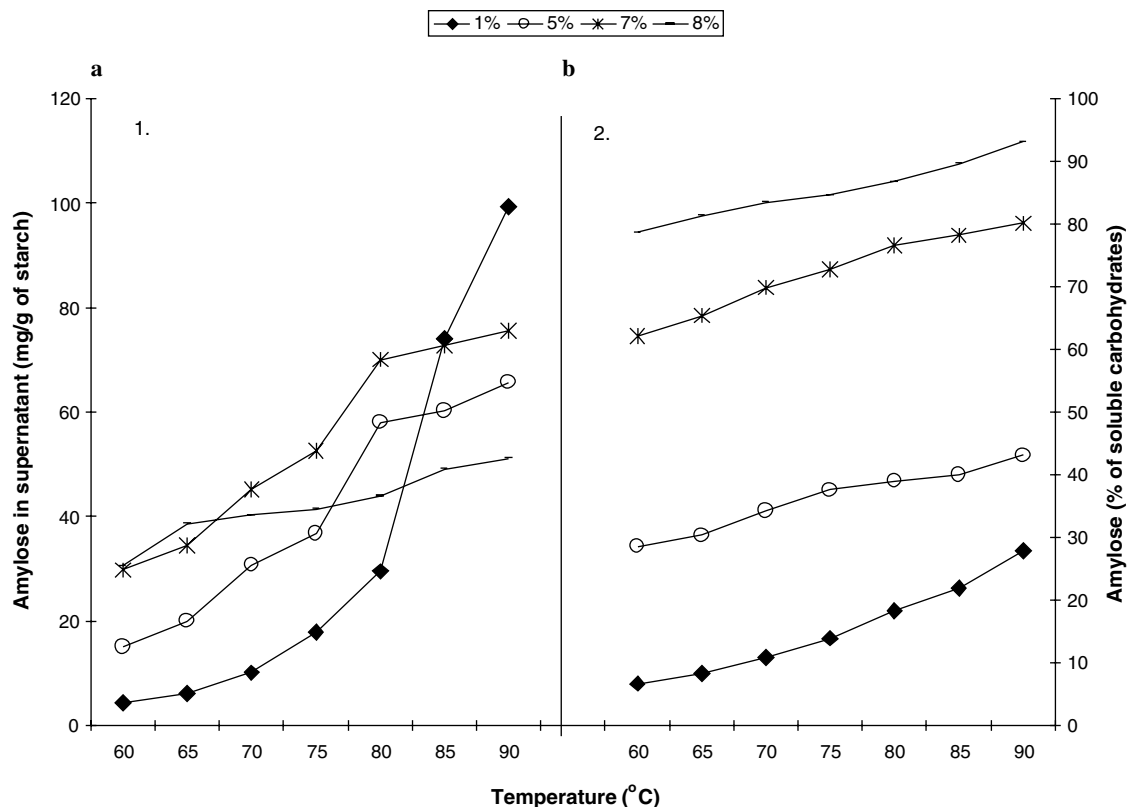


Fig. 4. Amylose in supernatant represented as mg/g of starch (a) as % of soluble carbohydrates (b) as a function of temperature. Error bars denote standard deviation of measurement but are too small.

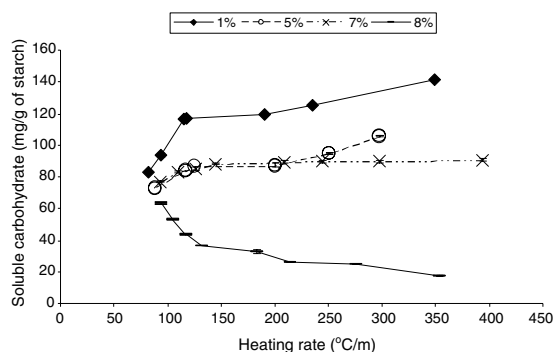


Fig. 5. Amount of soluble carbohydrates in the supernatant as a function of heating rate on the leaching of polymers during microwave heating. Standard deviations among measurements are denoted by error bars.

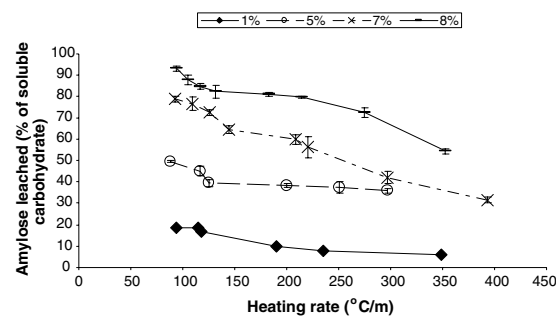


Fig. 6. Amount of amylose leached (% of soluble carbohydrate) as a function of heating rate. Error bars denote the standard deviation among the measurements.

4.1. Microscopy and thermal properties

Data from the microscopic observations suggest that granule swelling following microwave heating does not occur prior to the loss of birefringence (Fig. 1). This is the first evidence of loss of birefringence in the granules preceding granule swelling when heated by using microwave energy. This behavior is different in starch suspensions heated by using conduction heating wherein granule swelling and loss of birefringence occur almost simultaneously (Cameron & Donald, 1993; Donald, 2004; Donovan, 1979; French, 1984; Leszczynski, 1987; Tester &

Morrison, 1990). During conduction heating, the destruction of the radial arrangement of amylopectin chains is assisted by the swelling in the amorphous region of the granule (Donald, 2004; Donovan, 1979; Jenkins & Donald, 1998; Tester & Morrison, 1990; Waigh, Gidley, Komansek, & Donald, 2000). Furthermore, the thermal analyses data (Table 1) obtained by using a DSC suggest that the loss of crystalline arrangement in a microwave-heated sample occurs at a lower temperature compared to that observed for conduction-heated sample. It is likely that the vibrational motion of the polar molecules during microwave heating directly impact the crystalline lamella of the amylopectin thus disrupting the radial arrangement

of the amylopectin lamellae. Therefore, the crystalline arrangement is destroyed prior to the glass transition of the amorphous region of the granule, thus resulting in no swelling.

The loss of birefringence of B-type granules occurred at a higher temperature compared to A-type granules. The differences in the gelatinization temperature of A- and B-type wheat starch granules have been reported earlier (Karlsson & Eliasson, 1983) and it has also been reported that the crystalline region of the B-type wheat starch granules have greater stability compared to A-type wheat starch granules (Chiotelli & Le Meste, 2002; Wong & Lelievre, 1982).

At temperatures higher than 65 °C, granule swelling was observed in the microwave-heated samples (Fig. 2). Granule swelling was rapid following the loss of birefringence. In contrast during conduction heating, swelling of granules and the melting of crystallites are semi-cooperative processes (French, 1984). At higher temperatures following microwave heating, granular remnants, and leachate were observed in the background suggesting rupture of granules due to microwave heating. This observation is also different from that observed for starch samples heated by using conduction heating wherein granule maintains its integrity even at 90 °C (Bowler, Williams, & Angold, 1982; Doublier, 1987; Ghiasi, Hosene, & Varriano-Marston, 1982). The rupture of the granules is also likely a function of the vibrational motion of the polar molecules during microwave heating. These observations partially explain the differences in the granule and gel properties of microwave-heated starch suspensions compared to conduction-heated samples that we reported in an earlier research (Palav & Seetharaman, 2006; manuscript in review).

4.2. Effect of final temperature and concentration

The soluble carbohydrate content in the supernatant with increasing starch concentration is between 3.2 and 5.4% up to about 65 °C on the basis of the initial starch content (Fig. 3). However, the absolute amount of amylose in the supernatant at this temperature ranged from 0.4% to 2.5% (Fig. 4a) and the relative proportion of amylose as a function of the total carbohydrates in the supernatant ranged from 8% to 78% (Fig. 4b). This corresponds to the temperature at which the granule birefringence was lost completely (65 °C) and swelling was initiated (Figs. 1 and 2). Therefore, the leaching of carbohydrates occurs only following the initiation of swelling and a higher proportion of amylose leaches out at the higher starch concentrations.

At higher temperatures, the increase in the amount of soluble carbohydrates is dependent on the initial starch concentration in the suspension, with more carbohydrates leaching into the supernatant at lower starch concentrations compared that observed at higher starch concentrations. It has been reported that the dielectric loss factor of a starch suspension increases with increasing starch concentration (Piyasena, Ramaswamy, Awuah, & Defelice,

2003); i.e., the amount of microwave energy dissipated into heat increases with increasing starch concentration (Buffler, 1993). It is therefore likely that there is local increase in temperature around each granule at the higher starch concentrations that potentially results in the rupture of granules; thereby increasing the local viscosity and preventing leaching at higher concentrations. It is also likely that as the initial starch concentration increases, the potential for the sample to form a paste or a weak gel increases (Orford, Ring, Carroll, Miles, & Morris, 1987) thereby trapping the polymers and preventing leaching into the supernatant. Starch concentrations above 6% form a gel upon cooling (Miles, Morris, Orford, & Ring, 1985). However, the 8% starch suspension in this study only formed a paste and not a gel. Therefore, amylose does not appear to be contributing to the continuous network needed for gel formation following microwave heating.

4.3. Effect of heating rate solids concentration

The heating rates achieved in this study range from 90 to 350 °C/min and the time interval ranged from 9 to 37 s to reach a final temperature of 75 °C. These heating rates are significantly higher than what can be achieved by using conduction heating. The strong interaction between initial starch concentration and amount of soluble carbohydrate in the supernatant is a novel observation. A starch concentration of 7% appears to be a critical point. It is known that the dielectric loss factor increases with increasing starch concentration. It is likely that at 8% starch concentration, the amount of microwave energy converted to heat (dielectric loss factor) is significantly higher than that at lower concentrations. This would result in a very rapid increase in the local temperature in the suspension resulting in a rapid increase in local viscosity thus decreasing the amount of soluble carbohydrates leached into the supernatant. The proportion of amylose in the supernatant decreased with increasing heating rates probably due to kinetic limitations at all starch concentrations. However, at higher starch concentrations, the proportion of amylose in the supernatant was greater than that observed at the lower starch concentrations. This is likely because the suspension following the heat treatment has a higher viscosity in the 8% suspension compared to the 1% suspension and the granular remnants settle to the bottom leaving the amylose in the supernatant.

5. Conclusions

The primary observation in this study suggests that the gelatinization process during microwave heating is asynchronous compared to samples heated by using conduction heating. More specifically, loss of birefringence and granule swelling do not occur semi-cooperatively during microwave heating as is typical during conduction heating. The leaching of polymers occurred only following the initiation of swelling as has also been shown in samples heated by conduction. The particular kinetics of polymer leaching was

unique in microwave-heated samples and was strongly dependent on the initial starch concentration. Microscopic observations show that the granules, following initial swelling, are ruptured with granular remnants visible in the suspension. This is likely the result of the strong vibrational motion of the polar molecules due to the application of microwave energy. The nature of the remnants following microwave heating was different from that observed following conduction heating wherein granules retain their integrity even at 90 °C. These attributes will influence the gel or paste properties when using microwave energy as a source of heat. Further studies are required to better understand the concentration effect and the kinetics of polymer leaching. A better understanding of the specific changes occurring during microwave heating will aid in designing and improving the quality of microwave baked products.

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