

Effect of high-pressure on calorimetric, rheological and dielectric properties of selected starch dispersions



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ABSTRACT

Effects of high-pressure (HP) treatment on the rheological, thermal and dielectric properties of the four selected starch dispersions (two modified starches, one native and one resistant) were evaluated. Differential scanning calorimetry (DSC) and oscillatory rheometry were employed to assess the extent of starch gelatinization and the developed gel rigidity (G') of starch gels after HP treatment. It was observed that starch dispersions gelatinized completely at 500 MPa with a 30-min holding time. The HP-treated starch samples exhibited predominantly solid-like ($G' > G''$) behavior except for the resistant starch. Pressure-induced gel rigidity differed significantly among starch samples. The G' of starch gels increased with the pressure (400–600 MPa) in the studied frequency range (0.1–10 Hz) except for the native starch where a marginal decrease was recorded at similar condition. The holding time (15–30 min) and concentration (20–25% w/w) significantly attributed towards gel rigidity of starch samples. Measurement of dielectric properties of HP-treated samples over the frequency range 450–4450 MHz indicated differences in the dielectric constant (ϵ'), loss factor (ϵ'') and penetration depth among starch gels. Pressure did not show any effect on dielectric property of the resistant starch sample. Power penetration depth decreased significantly with frequency and with the pressure.

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1. Introduction

Starch is one of the most versatile biopolymers used in the food processing industry to impart viscosity, texture, and stability. Native starches have limited applications for their breakdown during prolonged heating, shearing, and pH modification. Furthermore, native starch has the tendency to retrograde and undergo syneresis. To overcome those limitations, the native starch is subjected to various treatments in order to modify its structure and thus to impart some functional properties suitable for specific industrial applications. The production of modified starch has increased many folds recently indicating that it is playing an increasingly important role in real-life applications (Wang, Li, Wang, Liu, & Adhikari, 2012). Till now, the thermal processing remains the best choice for food industry, and most starches (native and modified) are well defined for the process. Furthermore, rheological characterization of starch dispersions, similar to other food dispersions, are considerable important for applications associated with quality control, estimating textural

properties, sensory analysis, understanding the structure, handling and processing of starch-based foods (Kim & Yoo, 2009).

The concentration of macromolecules like starch plays an important role during self-diffusion in solution, and it is very important to understand the conditions whereby the solution behavior changes from dilute to semi-dilute and concentrated regimes. While increasing concentration in a good solvent, a threshold concentration is achieved at which macromolecular coils start to overlap in a solution, which is commonly known to as the overlap concentration, c^* (Rojas, Wahlund, Bergenstahl, & Nilsson, 2008). It is the transition point between the dilute and semi-dilute regime, and it governs further the domain for polymer random coils in solution whether it will be separated chains, overlapping chains, or/and a concentrated solution regime (Daoud et al., 1975).

Gelatinization,—which is known as an irreversible melting phase transition of starch granules from an ordered to a disordered state of dispersion (Douzals, Marechal, Coquille, & Gervais, 1996) and, gelatinization induced gels play important role in food product structure. Extreme conditions (temperature, pressure) can stimulate gelatinization of starch granules in starch–water suspensions (Bauer & Knorr, 2005). Furthermore, type of starch and processing conditions (pressure levels, temperature and

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holding time) strongly influence the extent of gelatinization and the rheological properties of starch suspensions (Stolt, Oinonen, & Autio, 2001). High-pressure (HP) treatment has been extensively used to gelatinize or modification of starch dispersions since HP-gelatinized starch shows different properties from heat-gelatinized one (Blaszcak, Valverde, & Fornal, 2005; Buckow, Heinz, & Knorr, 2007; Fukami, Kawai, Hatta, Taniguchi, & Yamamoto, 2010; Hayashi & Hayashida, 1989; Hibi, Matsumoto, & Hagiwara, 1993; Knorr, Heinz, & Buckow, 2006; Muhr & Blanshard, 1982). The number and types of starches (native, modified or resistant) are increasing in ingredients list everyday with a demand of the industry. Therefore, it is worth characterizing the effect of HP-treatment on functionality and structure/rheology of newly developed starches which can provide information to food processors about suitability of the process for starch-based products.

After processing, HP treated food products are stored at refrigerated temperature. Some of those products are subjected to further heating/processing or formulation before consumption. Dielectric heating is the most convenient way to reheat HP processed products. The dielectric properties of food materials describe how those materials interact with electromagnetic radiation (Ryyanen, 1995). Knowing the dielectric properties of a food is required to design a dielectric heating process since these parameters affect coupling and distribution of electromagnetic radiation during the heating process (Mudgett, 1986). The interaction between dielectric energy and food products at selected frequency range provides better understanding pertaining to processing either by microwave (MW) or radio-frequency (RF) heating. The dielectric properties of foods are often temperature dependent, thus, understanding the effect a wide range of temperatures has on the dielectric properties of biological materials is vitally important to properly predicting heating behavior. Data on dielectric properties of various foods have been reported in the literature and are available in several databases. However, to the best of our knowledge, information on dielectric properties of HP processed food is not available in the literature.

The objective of this research work was to determine the impact of high pressure on rheological, thermal and dielectric properties of various starch dispersions/gels so it can be translated to develop starch-based food products.

2. Materials and methods

2.1. Materials

The starches used in this study were all commercial grade samples (National Starch and Chemical Co, NJ): two modified starches [Fridgex (termed as MS1)—a chemically modified food starch with an average particle size of 15–40 μm refined from tapioca (moisture content $13.1 \pm 0.2\%$; ash content $<0.5\%$; fat $<0.15\%$; and protein 0.5%) and Thermtex (termed as MS2)—a chemically modified food starch with an average particle size of 15–40 μm derived from waxy maize (moisture content $12.4 \pm 0.3\%$; ash content $<0.6\%$; fat $<0.15\%$; and protein 0.5%), one native tapioca starch with average particle size 10–15 μm (Novation® 3300 termed as NS) (moisture content $12.8 \pm 0.2\%$; ash content $<0.5\%$; fat $<0.15\%$; and protein 0.5%) and one resistant starch with average particle size 10–15 μm (Hi Maize 260 corn starch termed as RS) (moisture content $10.8 \pm 0.1\%$; ash content $<0.4\%$; fat $<0.8\%$; and protein 0.8%). The starches were used as received and the proximate composition (dry basis) mentioned above for starch samples based on data provided by the manufacturer. The moisture content of starch samples was further verified by drying sample in an air-oven maintained at 130°C to constant weight. The drying experiments were carried out in triplicate. The observed moisture content data varied from the supplier data by less than 1%.

2.2. Preparation of starch dispersions

Dispersions of all the four starches were prepared at 20% (20 g starch and 80 mL water; 1:4 w/w) and 25% (25 g starch and 75 mL water; 1:3 w/w) concentrations by adding deionized water at room temperature (25°C). The dispersions were stirred on a magnetic stirrer for 30 min. Samples were deaerated by aspirating for 15 min to remove air bubbles.

2.3. High-pressure treatment

Starch dispersions were sealed (approx. 25 mL) in low-density polyethylene bags (Whirl-Pak®, USA) and pressure treated at 400, 500, and 600 MPa for 30 min using a 5 L static unit (ACIP 6500/5/12VB; ACB Pressure Systems, Nantes, France) equipped with temperature and pressure regulator devices. A starch concentration of 20% (w/w) was used for all pressure treatments except at 500 MPa where both 20 and 25% concentrations were used to evaluate the concentration effect. Similarly, one set of experiment was pressurized at 500 MPa for up to 15 min to study the time effect on starch samples. The pressurization rate was about 4.4 MPa/s and released at 26 MPa/s. The initial temperature of pressure medium was 18°C , which was sharply increased to 24.8 and 28.5°C due to the adiabatic effect during pressurization of 400 (minimum pressure used) and 600 MPa (maximum pressure used) however, temperature became steady at 22 and 25°C during holding period. Each experiment was conducted in duplicate.

2.4. Differential scanning calorimetric (DSC) measurement

A differential scanning calorimeter (DSC) (TA Q 2000, TA Instruments, New Castle, DE, USA) was employed to measure the gelatinization of the starch dispersions. The DSC was calibrated with indium and sapphire for temperature and heat capacity calibration. The samples (10–12 mg) were run at a $10^\circ\text{C}/\text{min}$ heating ramp from -10 to 150°C in a nitrogen atmosphere (flow rate 50 mL/min). An empty pan was used as a reference. Thermal transitions of starch dispersions were defined as T_0 (onset), T_p (peak gelatinization) and T_c (conclusion), and ΔH was referred to enthalpy of gelatinization. The gelatinization enthalpy was expressed in J/g of sample weight. The DSC measurements were done in triplicate.

2.5. Rheological measurement

Oscillatory rheological measurements of HP-treated starch dispersions were carried out using a controlled-stress rheometer (AR 2000, TA Instruments, New Castle, DE, USA). Samples were placed in a 1000- μm gap between two stainless steel parallel plates (plate diameter 40 mm). The sample temperature (25°C) was controlled by a peltier system and monitored by platinum resistance thermometer sensors (accuracy of $\pm 0.1^\circ\text{C}$) which are positioned at the upper and lower plates. The sample perimeter was covered with a thin layer of high-temperature-resistant silicone oil to prevent sample dehydration. Small-amplitude oscillatory strain sweep experiments (0.001–10%) were performed, and the elastic (G') and viscous (G'') shear moduli, at a constant frequency of 0.1 Hz were monitored to determine the limit of the linear visco-elastic region (LVR). The LVR was carried out for the entire studied pressure range (2–5 Pa depends on gel stiffness), and the measurement was carried out accordingly. Frequency sweep tests (0.01–10 Hz) were carried out in the linear regime, at constant strain (0.03%) at 25°C . All the rheological measurements were carried out in triplicate and rheological parameters were obtained directly from the

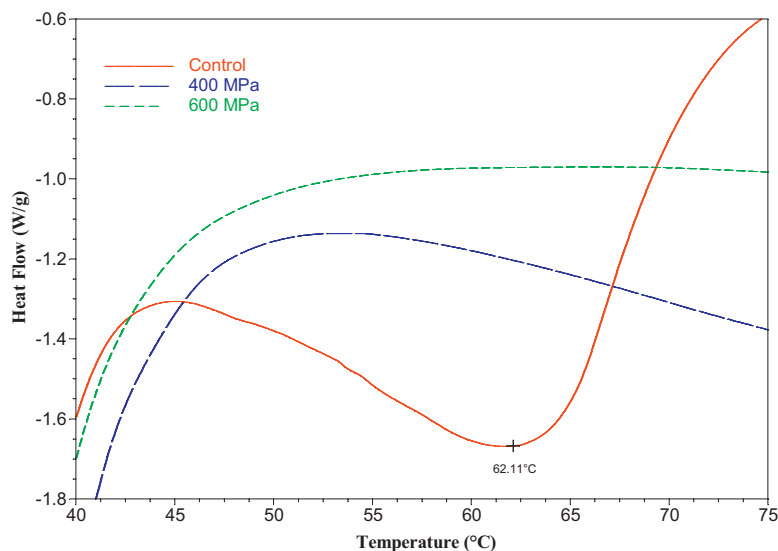


Fig. 1. DSC thermogram of modified starch (MS1) before and after high-pressure (HP) treatment.

manufacturer supplied computer software (Rheology Advantage software, TA Instruments, New Castle, DE, USA).

2.6. Dielectric properties measurement

A vector network analyzer (Model: Agilent 8722ES, Agilent Technology, Palo Alto, CA) with an open-ended coaxial cable (#8120-6192, Hewlett Packard) connected to a probe (85070C, Agilent Technology, Palo Alto, CA) in electronic calibration module (E-cal, 4693A) (which reduce operator's error and provide better precision) was used to measure dielectric properties of HP treated starch samples. The built-in S-parameter test set provided a full range of magnitude and phase measurements in both the forward and reverse directions. The instrument was calibrated by measuring the properties of air, short-circuit block, and water at 25 °C. To obtain uniform readings, the instrument was turned on at least 1 h before the calibration and measurements were made. The sample was placed in a small beaker so the open co-axial probe (probe diameter ≈ 2.4 mm) was set. The network analyser probe and the cable were fixed together so that there could be no movement during sample measurement and data acquisition. The dielectric properties were measured at 25 °C. A thermocouple temperature sensor (BK Precision 390A, Taiwan) was used to monitor the temperature during the study and the temperature variation within the sample was minimal, ± 0.5 °C. The dielectric properties of starch samples were measured in the frequency range of 450–4450 MHz. The dielectric spectra of the samples (dielectric constant ϵ' , and dielectric loss factor ϵ'') were automatically computed and recorded with the manufacturer supplied computer software (Version 85070 D1.00, Agilent Technologies, Palo Alto, CA). All the measurements were carried out in triplicate and were reproducible to $\pm 5\%$. Mean values and standard deviations were calculated from 3 replicates.

The penetration depth is an important parameter in characterizing temperature distribution in microwave heated foods. Penetration depth (D_p) is generally described in one of the two ways: (i) as the distance from the surface of a dielectric material where the incident power decreased to $1/e$ ($e=2.718$) or 36.8% of the incident power or (ii) as the distance from the surface of material where the power is one-half of the incident power at the surface

of the product (the half-power depth). The magnitude of D_p was calculated using the dielectric constant and loss factor at frequencies of 915 and 2450 MHz (Datta, 2001) as:

$$D_p = \frac{\lambda_o}{2\pi\sqrt{2\epsilon'(\sqrt{1+(\epsilon''/\epsilon')^2}-1)}} \quad (1)$$

where $\lambda_o = 0.328$ m at 915 MHz and 0.122 m at 2450 MHz.

2.7. Data analysis

Microsoft Excel software package (Microsoft Corporation, USA) was used to carry out the statistical analysis and linear regressions of experimental data. Trends were considered significant while means of compared sets differed at $P < 0.05$ (student's t -test).

3. Results and discussion

3.1. Gelatinization properties of high-pressure processed starches

The starch granule is a semi-crystalline macromolecule, and during thermal scanning of the control starch dispersion, an endothermic melting peak, associated with gelatinization, was detected. In order to examine the effect of high pressure on the starch gelatinization, samples were thermally scanned immediately after pressure treatment. Pressurized samples with incomplete gelatinization exhibited smaller peaks than the peak of the control sample. No peak was detected for the completely gelatinized starch samples. DSC thermograms of untreated and pressure treated starch (MS1) samples (400 MPa for 30 min) are presented in Fig. 1, and the gelatinization properties (gelatinization temperatures: onset, T_o ; peak, T_p ; conclusion, T_c ; and enthalpy of gelatinization, ΔH) of untreated and high-pressure treated starch samples are summarized in Table 1. The native starch sample had only one endothermic transition and the peak temperature for melting is about 95.3 °C, which is slightly higher than the reported value by the manufacturer (about 92 °C). The peak gelatinization temperatures (T_p) for modified starches, MS1 and MS2 were 62.4 and 63.6 °C, respectively which are also close to values provided by the supplier. For control resistant starch dispersion, two melting peaks were recorded at 69.2 and 92.8 °C, respectively. It is

Table 1

Calorimetric measurement of gelatinization as function of pressure for control and high pressure (HP) treated starch samples. No pressure treatment was performed on control samples. MS1, MS2, NS and RS samples were described in section 2.1.

Sample	T_o (°C)	T_p (°C)	T_c (°C)	ΔH (J/g)
MS1 control	44.7 ± 0.1	62.4 ± 0.3	79.2 ± 0.2	43.2 ± 0.2
MS1 400 MPa	ND ^a	ND	ND	ND
MS1 500 MPa	ND	ND	ND	ND
MS1 600 MPa	ND	ND	ND	ND
MS2 control	46.4 ± 0.2	63.6 ± 0.2	77.4 ± 0.3	37.2 ± 0.1
MS2 400 MPa	49.1 ± 0.3	65.5 ± 0.3	79.7 ± 0.2	5.6 ± 0.1
MS2 500 MPa	ND	ND	ND	ND
MS2 600 MPa	ND	ND	ND	ND
NS control	70.9 ± 0.3	95.3 ± 0.3	117.9 ± 0.2	43.2 ± 0.1
NS 400 MPa	ND	ND	ND	ND
NS 500 MPa	ND	ND	ND	ND
NS 600 MPa	ND	ND	ND	ND
RS control	43.6 ± 0.1; 81.9 ± 0.3	69.2 ± 0.2; 92.8 ± 0.3	80.5 ± 0.3; 104.7 ± 0.4	50.9 ± 0.2; 54.9 ± 0.3
RS 400 MPa	ND	ND	ND	ND
RS 500 MPa	ND	ND	ND	ND
RS 600 MPa	ND	ND	ND	ND

^a ND stands for not detected.

evident from Table 1 and Fig. 1 that a pressure level of 400 MPa and a holding time of 30 min were sufficient enough to gelatinize starch samples with the disappearance of the endothermic peak except for the sample MS2. At 400 MPa, the area of the gelatinization peak decreased significantly (about 85%) and the T_p increased by 2 °C for MS2. Vallons & Arendt (2009) observed an increase of the gelatinization temperature with more or less 5 °C when sorghum starch samples were pretreated with 500 MPa for 10 min. A progressive shift of the gelatinization endotherms to higher temperatures as well as a changing of the gelatinization endotherm inferred increased internal plasticization and destabilization of the amorphous regions of the granules (Stute, Klingler, & Boguslawski, 1996). The endothermic peak was completely disappeared (complete gelatinization) at a pressure level of 500 MPa for MS2 for a holding time of 30 min. It indicates that the crystallites of the endothermic peak of MS2 were more pressure resistant than the other samples. A similar observation was reported by Pei-Ling, Qing, Qun, Xiao-Song, Ji-Hong (2012) for 30% (w/w) waxy corn suspensions. Authors found an incomplete destruction of the crystalline structure by HP at 450 MPa with significant decrease of melting enthalpy. For tapioca starch, the complete gelatinization happened at 600 MPa (Vittadini, Carini, Chiavaro, Rovere, & Barbanti, 2008). The dependence of gelatinization pressure on starch content at tested temperatures was in agreement with that in the report on HP-treated starch dispersions at room temperature (Kasemwong, Ruktanonchai, Srinuanchai, Itthisoponkul, & Sriroth, 2011; Pei-Ling, Qing, Qun, Xiao-Song, & Ji-Hong, 2012; Vallons & Arendt, 2009).

Among the treated starch samples, the pressure sensitivity was different. The gelatinization behavior of HP-treated starch dispersion was highly dependent on the starch content, pressure, holding time and temperature. At a constant temperature, aqueous dispersion of most starches gelatinizes partially at a treatment pressure of above 300 MPa and completely at a treatment pressure of up to 600 MPa (Błaszczak, Fornal, Valverde, & Garrido, 2005; Stolt et al., 2001; Vallons & Arendt, 2009). In our previous study on isolated Basmati rice starch, the endothermic peak was disappeared at 550 MPa and a holding time of 15 min indicating complete gelatinization of rice starch (Ahmed, Ramaswamy, Ayad, Alli, & Alvarez, 2007). Furthermore, a combination of pressure and temperature significantly affect the gelatinization process. For example, the ΔH value of a mixture of 10 and 20% potato starches attained 0 J/g when the mixture was treated at 700 MPa and 20–40 °C, at 600 MPa and 50 °C, at 500 MPa and 60 °C, and 400 MPa at 70 °C (Kawaia, Fukamib, & Yamamoto, 2012).

It is worth mentioning that all control starch samples (without pressure-treatment) exhibited an additional melting peak in the higher temperature range. The T_p was 117, 121, 130 and 121 °C for MS1, MS2, NS and RS samples, respectively. The temperature range observed in this study is in agreement with the gelatinization temperatures found in literature. With applied pressure and time combination (400–600 MPa for 30 min), both T_p and ΔH values decreased substantially; however, the complete destruction of the crystalline structure did not happen (Fig. 2). The observed melting peaks are believed to be attributed by starch-lipid complex and the variation of temperature is associated with amount of lipid in the sample and the complexing behavior. A lower value of 105 °C has also been reported in the literature for waxy maize starch (Prokopowich & Biliaderis, 1995).

3.2. Rheological properties

Rheology has been often used to measure the gelatinization and retrogradation of starch and its polymeric constituents (Biliaderis & Zawistowski, 1990; Prokopowich & Biliaderis, 1995; Ring et al., 1987). However, rheological measurement requires a threshold concentration of starch for a defined network or to produce a meaningful data. It was observed that a starch concentration of 20% (w/w) without pressure treatment is too small to produce a repeatable rheogram using parallel plate geometry, and even rheogram of resistant starch (RS) dispersion was so scattered that was not fit for reporting. Two best available rheograms obtained for modified starch 1 (MS1) and native tapioca starch (NS) dispersions (control stress of 0.005 Pa) without high pressure treatment at 25 °C are presented in Fig. 3. The elastic (G') and viscous (G'') moduli of both starch dispersion remain parallel in the lower frequency range (0.1–1 Hz) with the opposite trend of moduli, and there is a cross-over between G' and G'' in the higher frequency range (1–10 Hz). For MS1, the G'' predominates over G' in the lower frequency range, and later on G' exceeded G'' at and above 1.26 Hz. The G' of the NS dispersion predominates over G'' between 0.1 and 1.59 Hz, and dropped marginally later on.

3.2.1. Effect of pressure

Pressure treatment of thin starch dispersions turned into gel, and the mechanical rigidity of gel (soft or hard) depends upon the applied pressure. The rate of structure formation increased with increasing pressure levels. An abrupt increase in gel rigidity, G' was observed for starch dispersions after pressurization except for resistant starch sample. For example, the G' increased about 5000

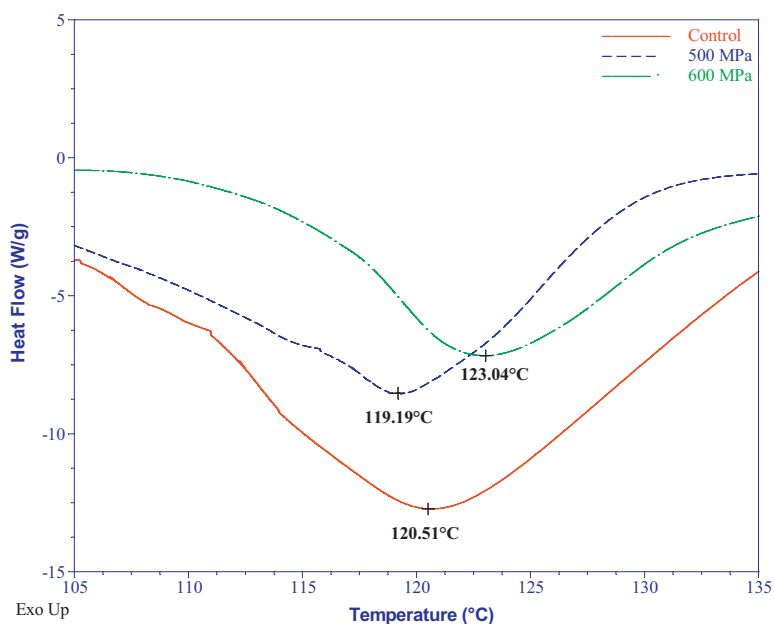


Fig. 2. Effect of high-pressure on high temperature melting curves of resistant starch (RS) dispersion.

times after pressurizing of MS2 dispersion at 400 MPa for 30 min, whereas the increase was about 30,000 times for NS at similar condition. The strongest gel was formed when the sample was pressurized at 600 MPa and a holding-time of 30 min. At similar condition, an increase of 600–1400 times in viscosity was displayed various starches (waxy rice, corn and tapioca) after pressurization at 600 MPa (Oh, Pinder, Hemar, Anema, & Wong, 2008). The mechanical strength of HP-induced gels has been compared among different starches (Fig. 4a and b). The G' was significantly higher for the MS1 than the MS2 except at 400 MPa (Fig. 4a). The difference in mechanical behavior under pressure of these two modified starches could be attributed to different factors such as: the level of amylopectin crystallinity, modification process and chain length. The mechanical behaviors of pressure-treated gel between native (NS) and resistant starch (RS) samples are illustrated in Fig. 4b. The mechanical strength of post-process NS increased as function of pressure, and the highest magnitude was recorded at 600 MPa and for a holding time of 30 min. Among starch samples, the NS

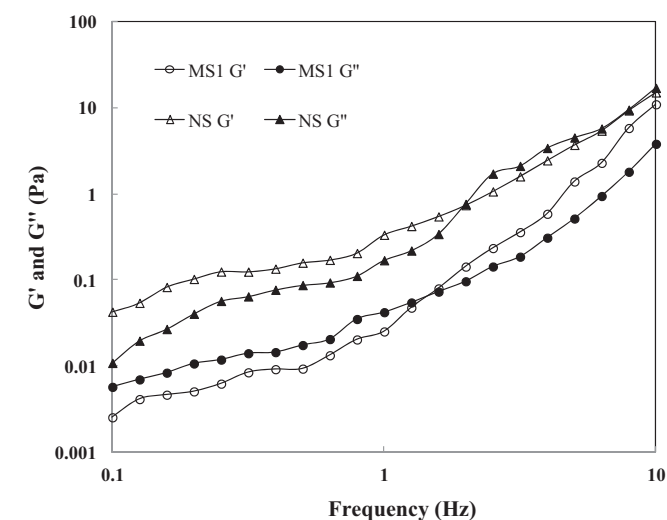
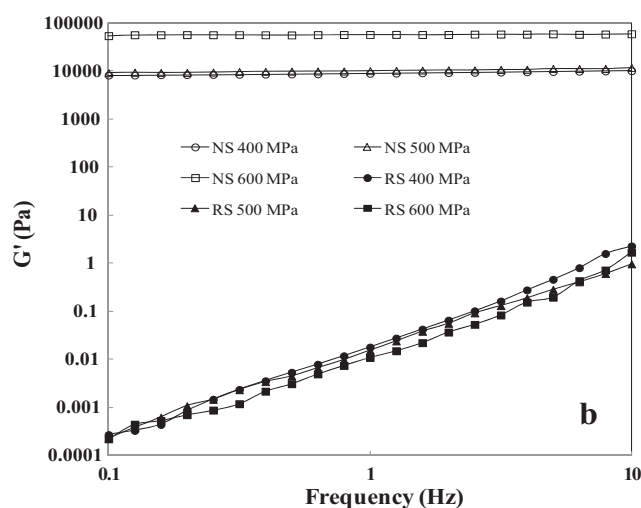
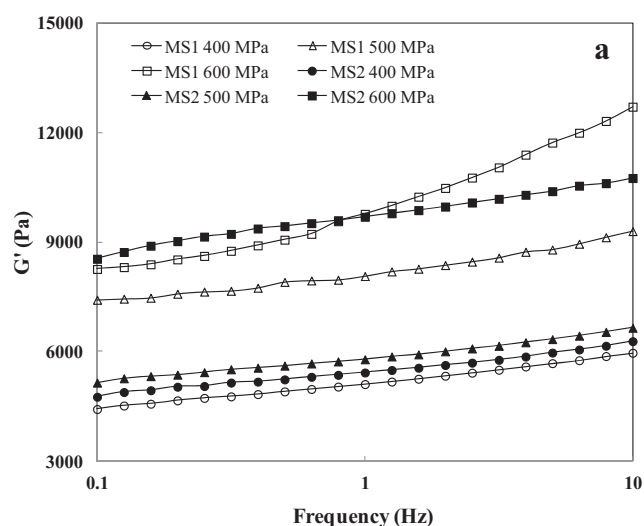


Fig. 3. Mechanical spectra of modified starch 1 (MS1) and native starch (NS) dispersions (20% w/w) at 25 °C without high-pressure treatment (control).

Fig. 4. Effect of high-pressure (HP) treatment on mechanical strength of (a) modified (MS1 and MS2) & (b) native (NS) and resistant starch (RS) dispersions at 25 °C.

produced the toughest gel. It can be hypothesized that the toughest texture of the HP-induced gel reflects stronger water–starch and/or starch–starch molecular interactions or a change in the conformational configuration and/or assembly of the starch components (Vittadini, Carini, Chiavaro, Rovere, & Barbanti, 2008). After HP treatment, RS samples displayed lowering the G' values with applied pressures. It indicates the pressure has a little effect on RS dispersion. Similar resistance of high amylose corn starch and potato starch to high pressure has been reported earlier (Michel & Autio, 2000; Oh et al., 2008). No viscosity increase was detected in corn starch at a pressure of 900 MPa, even after a 50-min holding time (Michel & Autio, 2000).

The differences in the gel rigidity of these starches under high pressure could be attributed to the difference in the composition of the starches, modification/manufacturing steps, swelling behavior of starch granules, water–starch interaction, amylose to amylopectin ratio, degree and length of branching, etc. Swelling of starch granules relates to the magnitude of the interaction between starch chains within the amorphous and crystalline domains (Oh et al., 2008). Furthermore, it is believed that amylose–lipid complexes could be another important factor for variation of gel rigidity for different starches since starches under pressure and that these complexes may restrict the swelling of the starch granules (Katopo, Song, & Jane, 2002). For RS, starch granules have a very compact condensed layer which seems to be more pressure resistant than the inner part of the granule and remains unchanged by high-pressure treatment (Blaszczyk et al., 2005).

Furthermore, the stiffness (solid-like property) of the HP-assisted gels can be obtained by calculated slopes of the linear regression of the power-type relationship of $\ln \omega$ vs $\ln G'$ (Eq. 2).

$$G' = A'\omega^n \quad (2)$$

The magnitude of slope for each treatment is summarized in Table 2. It is evident from the table that elastic modulus-frequency data fitted the power model adequately ($R^2 \geq 0.81$) and the G' values were found to be relatively independent of frequency as the slope varied between 0.04 and 0.09. It has been reported that a true gel is characterized by a zero slope of the power law model while viscous materials exhibit positive slopes (Ross-Murphy, 1984). The gel stiffness increased marginally with pressure level applied to starch dispersions. It is evident from the table that the stiffness of the gel made from modified starches and native starch are alike except for the gel at 600 MPa. The reason is not clear for soft gel characteristics of MS1. No firm measurable gel structure was induced in the RS dispersion for the pressure treatment. The slopes ranged for RS dispersion between 1.78 and 1.96.

3.2.2. Effect of holding time

A rise in the firmness in terms of complex viscosity, η^* was observed when the pressure-induced gels with a holding time increased from 15 to 30 min (data not shown). The starch dispersions mostly gelatinized when pressure exceeds 400 MPa. It was observed that the holding time of 15 min at 500 MPa was not adequate enough for a complete structure/gel network formation, which continues with additional holding time (30-min) to complete the process. During the pressure holding time, the microstructure of the gel is built up and stabilized. Our observation was supported by earlier reports on HP effect of various proteins including whey protein and ostrich-meat gel (Chattong & Apichartsrangkoon, 2009; Fertsch, Muller, & Hinrichs, 2003). A marginal drop in hardness was reported with a longer holding time (30 min) for HP treated tapioca starch gel (Vittadini et al., 2008). A combination of pressure and holding time essentially resulted an increase in network

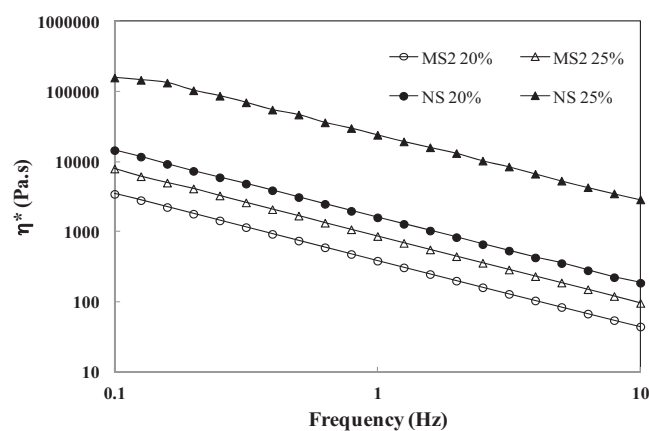


Fig. 5. Effect of sample concentration on mechanical strength of high pressure treated starch gels.

interactions. However, a pressure holding time (15–30 min) did not have any significant effect on solid-like property (Eq. 2) (Table 2).

3.2.3. Effect of starch concentration

Concentration contributes significantly on the rheology of starch and hydrocolloids, irrespective of the process is either induced by heat or pressure. A critical concentration (c^*) is required to form a gel network. A significant increase in G' and η^* values after pressure treatment confirmed the starch concentration level (c) used (20–25% w/w) in the work exceeded the critical concentration level ($c > c^*$) with a strong network. The η^* of modified starch dispersion (MS2) increased by 2 folds when the concentration increased from 20 and 25% (w/w) at a constant pressure level of 500 MPa and a 30 min holding-time, and the increase was almost one log cycle for the NS (Fig. 5). It indicates presence of stronger starch–starch molecular interactions by NS gel compared to MS gel. Above the c^* , the polymer coils begin to overlap and it increased with concentration. In fact, the critical concentration is a function of molecular weight ($c^* \sim M^{-4/5}$), (Rao, 2007) and also depends on the degree of polymerization and the radius of gyration. Furthermore, the concentration has a limit in aqueous medium when it reached equilibrium, there will be no increase in gel rigidity. Similar increase in yield stress and consistency index (which measures the gel strength) was noticed for HP-processed xanthan gum (Ahmed & Ramaswamy, 2004).

3.2.4. Time–pressure superposition of high-pressure treated starches

The basic method of superposition of time (frequency) and temperature (TTS) originating from rheological measurements of synthetic polymers have been employed to high-pressure treated starch gel (time-pressure-superposition, TPS) in order to broaden the observations horizon of viscoelastic properties. Following the TTS technique used earlier for thermo-rheological characterization of food materials (Ahmed, 2012, 2013; Ahmed, Varhney, Auras, & Hwang, 2010), log–log plots of the isobars of the elastic modulus, (G') can be superimposed by horizontal shifts $\log(a_p)$, along the $\log(\omega)$ axis, and vertical shifts given by $\log(b_p)$ such that:

$$b_p G'(a_p \omega, P_{\text{ref}}) = G'(\omega, P) \quad (3)$$

The linear visco-elastic master curves for the HP-treated starch samples are generated by the time-pressure superposition (TPS) principle and shifted to a reference pressure (P_{ref}) of 500 MPa with both the horizontal shift factor a_p and the vertical shift factor b_p . The reference pressure is selected based on the minimum pressure that gelatinized all starch samples completely. Since b_p (pressure expansion coefficient, $(P_{\text{ref}} \rho_{\text{ref}} / P \rho)$) has negligible effect on aqueous

Table 2Regression parameters from equation $\ln(G') = \ln(A) + n \ln(\omega)$ for pressure effect on starch gels.

Pressure (MPa)	MS1			MS2			NS			RS		
	Slope	R^2	S.E.	Slope	R^2	S.E.	Slope	R^2	S.E.	Slope	R^2	S.E.
400	0.06	0.99	0.00	0.06	0.99	0.00	0.05	0.99	0.00	1.96	0.99	0.15
500 ^a	0.05	0.98	0.00	0.05	0.99	0.00	0.05	0.98	0.01	1.78	0.99	0.08
500 ^b	ND ^d	ND	ND	0.05	0.99	0.00	0.05	0.99	0.00	ND	ND	ND
500 ^c	ND	ND	ND	0.04	0.98	0.00	0.12	0.81	0.10	ND	ND	ND
600	0.09	0.98	0.02	0.04	0.98	0.00	0.04	0.98	0.02	1.83	0.99	0.26

^a 30 min holding time; ^b 15 min and ^c 25% starch concentration.S.E. stands for standard error; ^d ND stands for not detected.

food systems (Rao, 2007), and, therefore, the vertical shift factor has been not considered for the TPS calculation. Fig. 6 illustrates the reduced angular frequency, $a_p\omega$ dependence of elastic modulus (G') for modified starch 2 (MS2) and resistant starch (RS) samples at three selected pressures (400, 500 and 600 MPa). It is observed that rheograms of MS2 are not superimposed using the horizontal shift factors (a_p) (Fig. 6a). Calculated values of the a_p coefficient for MS2 are not close to unity (0.0099 and 6.5 for 400 and 600 MPa), and therefore, scaling has significant effect on extending the frequency window. Similarly, rheograms of MS1 and NS samples were not superimposed within studied pressure range. However, the technique produced better superposition for RS as illustrated in Fig. 6b, and the calculated values of the a_p coefficient are close to unity (1.08 and 0.80 for 400 and 600 MPa). The master curves illustrate extension of the frequency window to 4–5 log cycles which is the

novelties of the technique. One of the possible reasons for non-applicability of TPS for starch samples could be the pressure range used in the experiment, which are extreme from the starch gelatinization especially a pressure level of 600 MPa that produced a stiff gel. Applicability of TPS for the pressure-treated RS dispersions cannot be considered as an ideal situation since the RS dispersion remained almost unaffected by pressure without forming a gel. It is worth mentioning that the HP-treated gelation, and the gel rigidity is a synergic effect of both pressure holding time and pressure level.

3.3. Dielectric properties

The dielectric properties of 20% (w/w) starch dispersions with and without pressure treatment were measured as a function of frequency and the pressure at 25 °C. Dielectric spectra of starch dispersions before pressure treatment are shown in Fig. 7. Starch

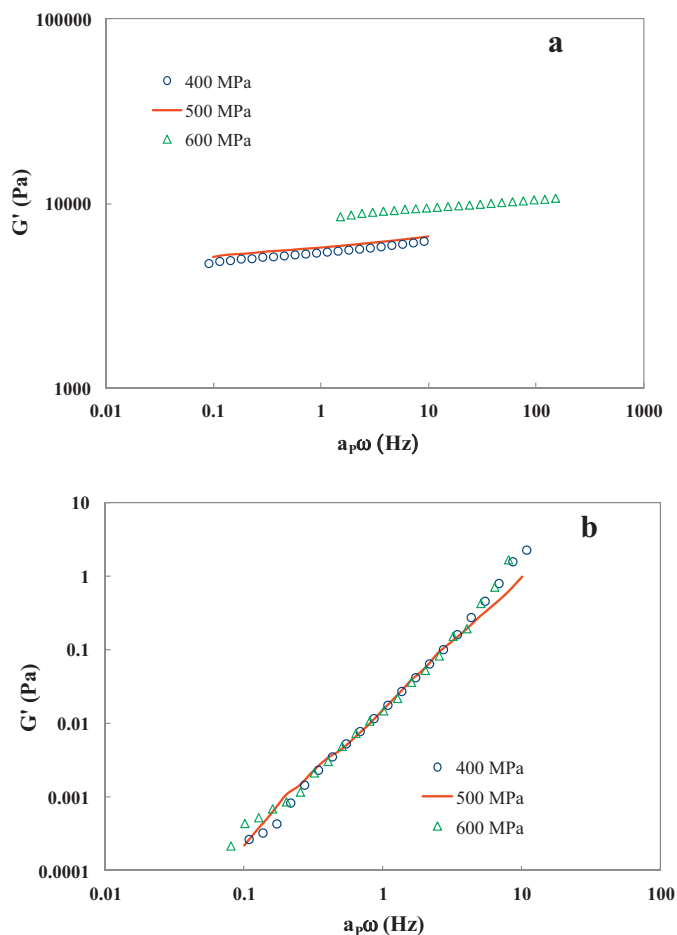


Fig. 6. Applicability of time–pressure superposition (TPS) technique for high-pressure (HP) treated (a) modified starch (MS2) and (b) resistant starch (RS) dispersion at 25 °C.

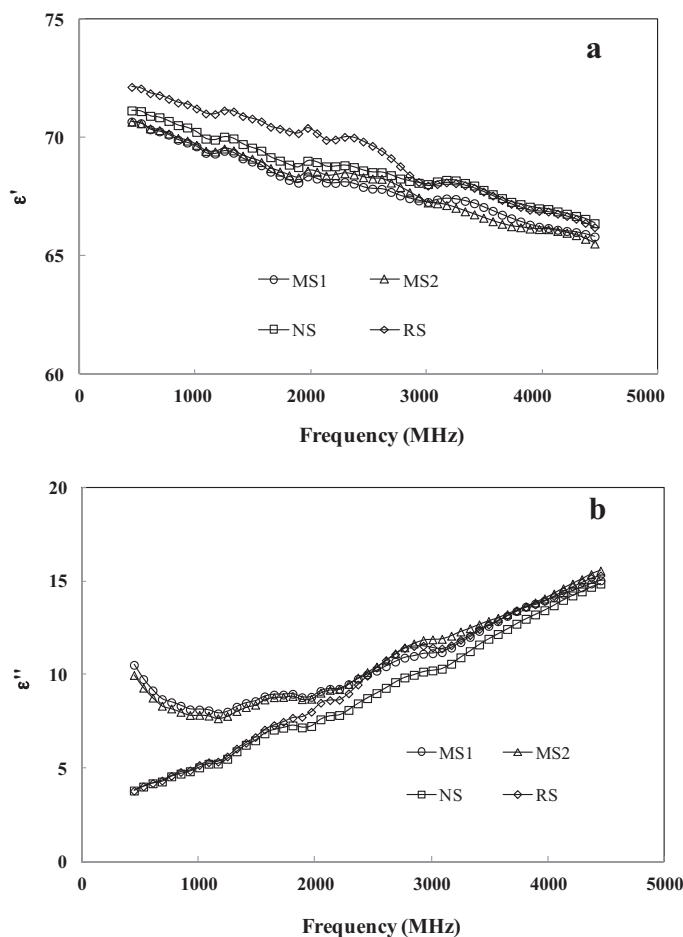


Fig. 7. Dielectric behavior of control starch dispersions, without high pressure treatment, at 25 °C: (a) dielectric constant, ϵ' and (b) dielectric loss factor, ϵ'' .

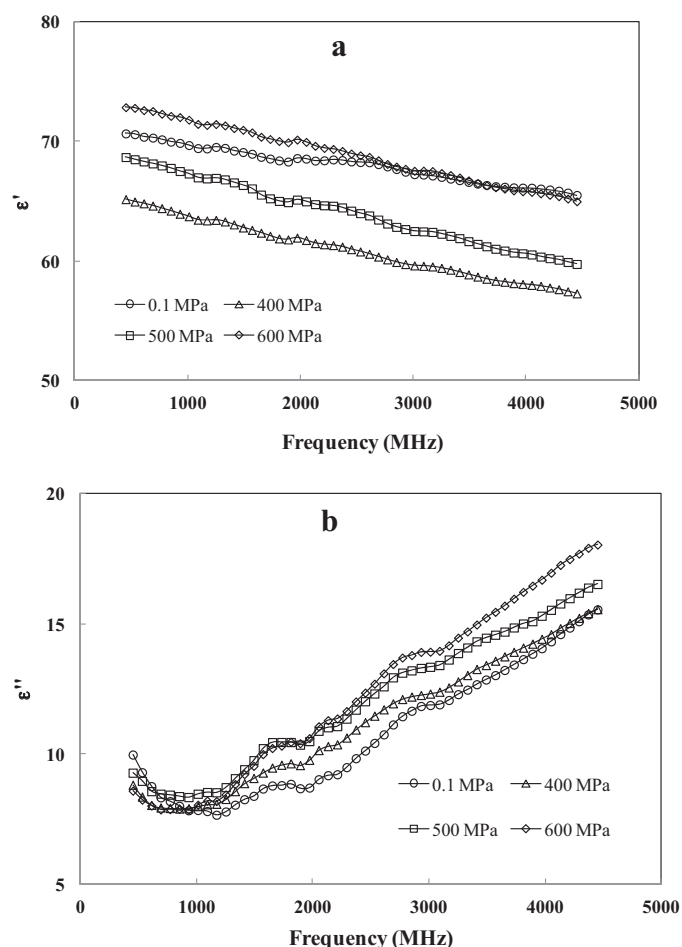


Fig. 8. Dielectric behavior of high pressure treated modified starch dispersions (MS2) at 25 °C: (a) dielectric constant, ϵ' and (b) dielectric loss factor, ϵ'' .

dispersions showed similar behavior for dielectric constant, ϵ' as function of frequency except for RS. The ϵ' values decreased as frequency increased from 450 to 4450 MHz and the values ranged between 66 and 72. These values are relatively higher than the reported values for various starch dispersion at higher concentration range (33.3–50%) as reported by Ndife, Sumnu, and Bayindirli (1998). Authors advocated that the higher ϵ' values was expected with presence of much free water available in the system and, electric field would see more polarizable dipole moments per unit volume of the sample. The ϵ' values of RS were relatively higher than other starches between 450 and 2690 MHz, and equaled later on. It indicates interaction between RS and water was minimum compared to other starches in the lower frequency range. The dielectric loss factor was the most influential parameter for microwave heating (Chen, Siochi, Ward, & Mcgrath, 1993), and the higher the loss factor the faster was the heating in microwave oven. The loss factor, ϵ'' values increased as function of frequency, and differed among starch samples although both modified starches overlap each other. The ϵ'' values obtained in our experiments are similar to those reported earlier by Ndife et al. (1998). Above 4000 MHz, ϵ'' values were superimposed with each other. The difference in ϵ'' values could be attributed by relative percentage of amylopectin which showed significantly more disordered structure than either amylose or retrograded amylase (Pethrick & Song, 2013).

3.3.1. Effect of pressure

A representative dielectric spectrum by pressure treated modified starch dispersion (MS2) at 25 °C is shown in Fig. 8, and dielectric

data (ϵ' and ϵ'') in selected microwave frequency ranges of 915 and 2450 MHz is summarized in Table 3. HP-treated starch dispersions followed a similar decreasing trend within the frequency range studied. Contrary to temperature effect, the ϵ' of pressure treated samples behaved differently. The ϵ' values decreased with increasing frequency, and the magnitude of this decrease varied with the pressure range used. A significant drop of ϵ' values was observed at 400 MPa and a 30-min holding time, and thereafter, increased gradually. The ϵ' values leveled off at 600 MPa with the control sample especially in the higher frequency range (Fig. 8a). At lower frequencies, the loss factor approaches a “plateau”, representing the dipolar relaxation region, and increased thereafter, with the frequency (Fig. 8b). It is observed that the variation of the ϵ'' with both frequency and pressure are similar. The ϵ'' values of modified starch samples increased with increasing pressure levels at 915 and 2450 MHz (except for 600 MPa MS1 at 915 MHz). An increasing trend of ϵ'' values was noticed for HP treated NS samples in the pressure range of 400–500 MPa, and suddenly dropped at 600 MPa, whereas RS samples showed no change in ϵ'' values with pressure treatment at 915 and 2450 MHz. The dielectric behavior of RS slurries is very close to pure water in the studied frequency range. Overall, the change in dielectric properties with high-pressure was predominantly dominated by the gelatinization phenomenon where a change in the mobility of water molecules occur in the system, and thus has the ability to influence the dielectric properties and rheological behavior of pressure-induced gel. It has been reported earlier that the ϵ'' value increased at and above gelatinization temperature (Ahmed, Ramaswamy, & Raghavan, 2007; Motwani, Seetharaman, & Anantheswaran, 2007), and in the present study gelatinized happened due to pressure effect, and therefore, the loss factor increased at and above gelatinization pressure level. Dielectric data of RS supported our postulates where no significant change observed in gel rigidity of post-gelatinized samples. At 600 MPa, the starch gelatinization had been completed with the peak value of the gel rigidity, and thus, the availability of water becomes limited resulting lowering of ϵ' value.

3.3.2. Effect of concentration

The ϵ' values of starch dispersions are strongly dependent on water content. For modified starch dispersion, the ϵ' values decreased with increasing starch concentration from 20 to 25% (w/w), however ϵ' values dropped markedly for NS dispersion (e.g. 74–57 at 915 MHz). A significant decrease of the ϵ' has been reported earlier by Motwani et al. (2007) with increase in starch concentration from 10% to 50%. The decrease in ϵ' values is expected with limited water availability in the dispersion (Ahmed, Ramaswamy, Ayad, et al., 2007; Ahmed, Ramaswamy, & Raghavan, 2007; Bircan & Barringer, 1998; Ndife et al., 1998). The ϵ'' values increased with increasing concentration of MS starch in the measured concentration range in the lower frequency range (>2000 MHz) which equaled in the higher frequency range. Between two NS concentrations, the ϵ'' did not differ significantly with frequency. Similar increase in ϵ'' values with concentration has been reported by Motwani et al. (2007), while other researchers opined that the increase in amount of starch concentration decreased the dielectric loss factor (Butler & Cameron, 2000; Ndife et al., 1998). It is believed that with presence of higher amount of starch in the system, the dielectric relaxation effects due to bound water in the molecular chains in the starch polymer start to interact with and possibly modify the dielectric influence of free water in the system Motwani et al. (2007).

3.3.3. Effect of holding time

The ϵ' values decreased insignificantly with the pressure holding time from 15 to 30 min. However, the ϵ'' values for all pressure-induced starch samples increased with holding period

Table 3
Dielectric parameters of high-pressure processed starch dispersions measured at 25 °C. The parameters ϵ' and ϵ'' are dielectric constant and dielectric loss factor; and D_p represents penetration depth as described in section 2.6.

Sample	915 MHz			2450 MHz		
	ϵ'	ϵ''	D_p (m)	ϵ'	ϵ''	D_p (m)
MS1 control	69.78 ± 1.09	8.14 ± 0.50	0.053 ± 0.00	67.91 ± 0.90	10.01 ± 0.30	0.016 ± 0.00
MS1 400 MPa	65.30 ± 0.88	8.57 ± 0.40	0.049 ± 0.00	61.86 ± 0.78	12.30 ± 0.23	0.012 ± 0.00
MS1500 MPa ^a	72.91 ± 1.20	8.63 ± 0.45	0.051 ± 0.00	70.09 ± 1.00	12.09 ± 0.43	0.013 ± 0.00
MS1500 MPa	72.57 ± 0.90	8.69 ± 0.33	0.051 ± 0.00	69.75 ± 0.80	12.32 ± 0.22	0.013 ± 0.00
MS1500 MPa ^b	71.90 ± 0.79	9.63 ± 0.60	0.046 ± 0.00	70.09 ± 0.68	12.28 ± 0.32	0.013 ± 0.00
MS1 600 MPa	72.63 ± 0.98	8.88 ± 0.50	0.050 ± 0.00	69.61 ± 0.87	12.67 ± 0.40	0.013 ± 0.00
MS2 control	69.86 ± 1.00	7.84 ± 0.33	0.056 ± 0.00	68.32 ± 0.70	10.13 ± 0.34	0.016 ± 0.00
MS2 400 MPa	63.94 ± 1.10	7.92 ± 0.43	0.053 ± 0.00	60.98 ± 0.56	11.23 ± 0.43	0.014 ± 0.00
MS2500 MPa ^a	71.68 ± 1.10	8.32 ± 0.66	0.053 ± 0.00	68.50 ± 0.77	12.07 ± 0.41	0.013 ± 0.00
MS2500 MPa	67.52 ± 0.89	8.34 ± 0.41	0.052 ± 0.00	64.20 ± 0.54	12.12 ± 0.50	0.013 ± 0.00
MS2 600 MPa	72.02 ± 0.80	7.86 ± 0.23	0.056 ± 0.00	68.98 ± 1.00	12.36 ± 0.70	0.013 ± 0.00
NS control	70.42 ± 0.94	4.83 ± 0.22	0.091 ± 0.05	68.61 ± 0.56	8.74 ± 0.30	0.018 ± 0.00
NS 400 MPa	74.21 ± 1.00	5.22 ± 0.32	0.086 ± 0.03	79.91 ± 1.30	10.45 ± 0.44	0.017 ± 0.00
NS 500 MPa ^a	76.23 ± 1.20	4.90 ± 0.30	0.093 ± 0.03	74.85 ± 0.70	9.73 ± 0.34	0.017 ± 0.00
NS 500 MPa	73.72 ± 0.80	5.53 ± 0.44	0.071 ± 0.00	71.56 ± 1.05	10.93 ± 0.80	0.014 ± 0.00
NS 500 MPa ^b	57.15 ± 0.76	5.54 ± 0.43	0.071 ± 0.00	54.60 ± 0.60	10.65 ± 0.58	0.014 ± 0.00
NS 600 MPa	74.51 ± 0.60	5.30 ± 0.33	0.085 ± 0.01	73.15 ± 1.25	10.75 ± 0.70	0.015 ± 0.00
RS control	71.40 ± 1.00	4.87 ± 0.26	0.090 ± 0.01	69.83 ± 1.03	9.94 ± 0.62	0.016 ± 0.00
RS 400 MPa	71.41 ± 1.01	4.88 ± 0.20	0.090 ± 0.02	69.85 ± 0.70	9.94 ± 0.76	0.016 ± 0.00
RS 500 MPa ^a	68.67 ± 0.60	5.16 ± 0.20	0.084 ± 0.00	66.68 ± 0.66	9.71 ± 0.42	0.016 ± 0.00
RS 500 MPa	69.51 ± 0.80	5.13 ± 0.32	0.085 ± 0.00	66.51 ± 0.78	9.91 ± 0.60	0.016 ± 0.00
RS 600 MPa	71.44 ± 1.30	4.86 ± 0.23	0.091 ± 0.02	69.87 ± 0.70	9.93 ± 0.54	0.016 ± 0.00

^a 15 min holding time.

^b 25% starch concentration.

except for RS sample at 915 MHz (Table 3). The reason for such decrease/increase of dielectric parameters is not clear. It could be associated with starch gelatinization and development of rigid gel at longer treatment period.

3.3.4. Penetration depth

Penetration depth (D_p) of food materials measures the depth to which heat is dissipated uniformly. The penetration depth decreased after HP-treatment as compared to control sample except RS dispersions. It indicates pressure-treated sample reduced the temperature uniformity during microwave heating. The D_p at 915 MHz was almost 5 times higher than that of 2450 MHz starch dispersions irrespective of treatment (Table 3). While working with various starches, Ndife et al. (1998) reported that there was no significant change in D_p as a function of temperature for rice starch while significantly higher values were observed for amylo maize and waxy maize starches.

4. Conclusions

The extent of high-pressure assisted gelatinization of various types of starch was monitored by DSC and it was observed that the starch gelatinization is pressure dependent. Most of the starch dispersions were gelatinized at 500 MPa for a holding time of 30 min. The resistant starch was more resistant to pressure than other starches, and, in general, gelatinization properties of starch dispersions depend on the types of starches or its modification process. The height of the starch-lipid melting curve reduced significantly by pressure treatment but it was not completely disappeared in the pressure range of 400–600 MPa. Oscillatory rheological measurements confirmed the viscoelastic nature of the HP-treated starch dispersions ($G' > G''$) which differed significantly in mechanical rigidity among starch samples. Native starch produced the strongest gel rigidity, and the resistant starch dispersions remained unaffected after pressure treatment. Increase in mechanical property of starch samples was contributed by pressure-induced gelatinization, starch concentration and amylose to amylopectin ratio. The study offers new information on the dielectric properties of high-pressure treated starch dispersions.

Dielectric properties of starch samples differed significantly within same moisture, pressure and frequency range. Noticeable changes of dielectric properties of starch samples were observed between 400 and 600 MPa which could be associated with nature of starch and gelatinization behavior of starch fractions. The penetration depth at 915 MHz was significantly higher than that of 2450 MHz and it decreased with pressure in the range of 400–500 MPa. Further study under various pressure and temperature conditions will be necessary for a better understanding of the mechanisms of HP-induced gelatinization and dielectric properties of various modified starches.

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