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Water Dynamics and Retrogradation of Ultrahigh Pressurized Wheat Starch

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The water dynamics and retrogradation kinetics behavior of gelatinized wheat starch by either ultrahigh pressure (UHP) processing or heat are investigated. Wheat starch completely gelatinized in the condition of 90000 psi at 25 °C for 30 min (pressurized gel) or 100 °C for 30 min (heated gel). The physical properties of the wheat starches were characterized in terms of proton relaxation times (T_2 times) measured using time-domain nuclear magnetic resonance spectroscopy and evaluated using commercially available continuous distribution modeling software. Different T_2 distributions in both micro- and millisecond ranges between pressurized and heated wheat starch gels suggest distinctively different water dynamics between pressurized and heated wheat starch gels. Smaller water self-diffusion coefficients were observed for pressurized wheat starch gels and are indicative of more restricted translational proton mobility than is observed with heated wheat starch gels. The physical characteristics associated with changes taking place during retrogradation were evaluated using melting curves obtained with differential scanning calorimetry. Less retrogradation was observed in pressurized wheat starch, and it may be related to a smaller quantity of freezable water in pressurized wheat starch. Starches comprise a major constituent of many foods proposed for commercial potential using UHP, and the present results furnish insight into the effect of UHP on starch gelatinization and the mechanism of retrogradation during storage.

KEYWORDS: Ultrahigh pressure processing; wheat starch; gelatinization; retrogradation; proton mobility; NMR; DSC

INTRODUCTION

A primary factor of food processing is to preserve the quality and microbiological safety of foods for extended periods by controlling or eliminating the factors that contribute to food deterioration, such as microbial growth and enzyme and chemical reactions. With the emergence of novel technologies for food preservation in recent years, food processing can be generally divided into two categories: thermal and nonthermal processing. Thermal processing involves the use of heat and is an excellent technique to control deteriorative processes and achieve shelf-stable foods by inactivating various degradative enzymes and foodborne spoilage or pathogenic microorganisms. Despite its ability to safely preserve foods, thermal processing can also significantly reduce food quality attributes, including losses in food texture, the production of off-flavors, or the degradation of nutrients.

Among the various nonthermal processing technologies currently available, high-pressure processing [HPP; also known as ultrahigh pressure (UHP) or high hydrostatic pressure] is

gaining increasing popularity for the production of value-added, more freshlike foods available in the commercial marketplace for the following reasons. In general, HPP significantly affects only large molecules, cell membranes, and enzymes. Under high pressure, large molecules, cell membranes, and enzymes tend to denature and lose their structures and functions. HPP can also inactivate microorganisms and enzymes that cause food spoilage without a contribution from heat to produce minimally processed food products. HPP generally has only minor effects on low molecular weight compounds that impact food quality, such as vitamins, pigments, and flavor substances or their precursors, all of which are highly susceptible to decomposition by heat during traditional thermal processing. Pressure also acts immediately and uniformly throughout the sample independent of the size, shape, or composition of the food product. In thermal processing, the heat-induced effects depend on temperature gradients in the food and on the rate of heat transfer to the center of the food. These characteristics make HPP an attractive nonthermal technique for improving the quality of foods available in the commercial marketplace and for the development of high quality, shelf-stable foods (*1*).

Inactivating pathogens or food spoilage microorganisms and enzymes are key priorities in the preservation of foods, and most

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research using HPP has focused on the behavior of microorganisms, inactivation of enzymes, and changes in functional properties of proteins in response to high-pressure conditions (2–11). In contradistinction, not much information is available on the influences of HPP on food biopolymers including starches (1, 12–26).

Most of the research efforts studying the effects of HPP on starch have concentrated on the gelatinization characteristics of starch, such as morphological features (1, 12–16), thermal characteristics (1, 13, 14, 17, 18), viscosity (15, 19), X-ray structure (14–16, 18), and enzyme susceptibility (20–22). These investigations determined the following characteristics relating to starch gelatinization: (i) All starch types can be gelatinized by HPP (even at subzero temperatures at sufficiently high pressures); (ii) generally, B type starches are more resistant to pressure than A and C type starches (with some exceptions); (iii) the high pressure required to gelatinize starches can be reduced, if heat is included in the high-pressure treatment; (iv) pressure-gelatinized starch exhibits less swelling than heat-gelatinized starch; and (v) pressure-gelatinized starch maintains intact granular structures, which is distinctive from the ruptured granules commonly observed with heat-gelatinized starch. These results provide useful information regarding the distinct gelatinization characteristics of various starches treated by HPP, and only limited information is available pertaining to the kinetics and mechanisms of retrogradation and the functional properties of pressurized starch.

Retrogradation is one of the most important physicochemical properties of starches, but only limited information is available on the retrogradation of pressurized starch (1, 14, 30). Cheon et al. (14) investigated the retrogradation of pressurized waxy and nonwaxy rice starches in terms of calorimetric analysis and X-ray structures of crystals. The retrogradation of pressurized starch is also quite different from the retrogradation characteristics commonly observed after heat gelatinization (1). Additionally, pressure can induce the formation of new crystal structures in starch not seen with heated starch gels (30). For example, pressurization converted rice starch from A type to a mixture of A, B, and V types, whereas heated rice starch converted to a mixture of B and V types (14). An understanding of retrogradation of pressurized starch is needed to elucidate the influences of high pressure on the properties and characteristics of starch. Presently, we investigate the retrogradation kinetics, water dynamics, and thermometric characteristics of pressurized wheat starches and furnish insight and fundamental information on the physicochemical properties of pressurized starch, which can serve as a basis for future industrial applications.

MATERIALS AND METHODS

Materials. Wheat starch (Gemstar 100 Plus, 11% moisture) was kindly donated from Manildra Co. (Hamburg, IA). Approximately 130 g of starch slurry (60% moisture, wet basis) was mixed and poured into a retortable pouch (3 Side Seal Pak, Kapak Corp., Minneapolis, MN) that was hermetically sealed using a heat sealer. Two methods were used to gelatinize starch slurry. For high-pressure gelatinization, starch slurry was put into a high-pressure unit (EPSI, Engineered Pressure Systems, Inc., Haverhill, MA) and pressurized from atmospheric pressure to 90000 psi (621 MPa) for 40 min. The vessel temperature increased from 25 to 28 °C during pressurization and returned to 25 °C after the pressure reached 90000 psi. The come-up time of pressurization was 30 s. In the case of thermal gelatinization, starch slurry was put into a boiled water bath for 30 min. After gelatinization, the starch gels were stored at 4 °C for 2 weeks.

Microscopy. The native, heated, and pressurized wheat starch suspensions (1 g/100 mL) were observed on a light microscope (Nikon,

Japan) using an objective lens $\times 40$ (Nikon). Images were observed directly through a binocular and recorded via a charge-coupled device camera. Special care was taken with focus and lightness adjustment. Image thresholding conditions were kept constant during all experiments.

Differential Scanning Calorimetry (DSC) Analysis. Approximately 20 mg of gelatinized sample was placed in a stainless steel sample pan (Perkin-Elmer, Somerset, NJ) and hermetically sealed. The sample was analyzed using a DSC instrument (DSC 220C, Seiko Instruments Inc., Horsham, PA) with an empty pan as a reference. These samples were cooled to -50 °C using liquid nitrogen and then heated to 120 °C at a rate of 5 °C/min. The temperatures and the enthalpies for the amylopectin and ice-melting transitions were determined as previously described (27, 28).

The amylopectin recrystallization rate was analyzed using the Avrami equation (29).

$$\theta = \frac{E_L - E_t}{E_L - E_0} = \exp(-kt^n)$$

$$\log \left[-\ln \frac{E_L - E_t}{E_L - E_0} \right] = \log k + n \log t$$

where θ is the noncrystallized fraction at time t , E_0 is the amylopectin recrystallization at time 0, E_t is the amylopectin recrystallization at time t , E_L is the maximum amylopectin recrystallization, k is the rate constant (time^{-1}), and n is the Avrami exponent, respectively.

NMR Analysis. A 20 MHz PCT 20/20 NMR Analyzer (Process Control Technology Corp., Ft. Collins, CO) was used to perform all of the NMR experiments at 30 °C. Approximately 10 g of gelatinized starch sample was transferred into a disposable glass test tube (13 mm o.d. \times 100 mm length, Fisher Scientific, Pittsburgh, PA), and the tube was sealed with Parafilm M (Fisher Scientific) to prevent moisture loss during measurement.

The 90° pulse sequence and the Carr–Purcell–Meiboom–Gill (CPMG) pulse sequence were used for acquisition of the free induction decay (FID) data for T_2 values. The experimental parameters were set appropriately to maximize the signal-to-noise ratio and to cover the entire relaxation range as completely as possible. The FID(s) obtained from the 90° pulse sequence and the CPMG pulse sequence were analyzed as a continuous distribution of exponentials using RI WinDXP software (version 1.2.2., Resonance Instruments Ltd., Oxfordshire, United Kingdom).

The pulsed-field gradient spin–echo pulse sequence was used to determine the water self-diffusion coefficient as described earlier (30). For single component diffusion, the ratio of the echo signal with and without the pulse gradient (i.e., the echo attenuation) can be expressed as follows:

$$\ln(A/A_0) = -\gamma^2 g^2 \delta^2 (\Delta - \delta/3) D$$

in which A = echo amplitude obtained with gradient, A_0 = echo amplitude obtained without gradient, γ = magnetogyric ratio, g = strength of the linear magnetic field gradient, δ = duration time, Δ = separation time, and D = self-diffusion coefficient.

In this experiment, the magnitude of the field gradient was varied from 0 to 0.405 T/m. δ and Δ of the linear magnetic field gradient pulses were 10.0 and 11.028 ms, respectively. The slope of the plot of $\ln(A/A_0)$ vs $\gamma^2 g^2 \delta^2 (\Delta - \delta/3)$ estimated the value for D .

RESULTS AND DISCUSSION

Microscopic Observation. Figure 1 shows the light micrographs of native, pressurized, and heated wheat starches. The wheat starch granules in both the pressurized and the heated wheat starch showed a similar degree of swelling, and the heated starch granules showed partial disintegration. It has been reported (12) that wheat starch samples partially gelatinize at high-pressure treatments of 300 MPa and completely gelatinize at high-pressure treatments above 500 MPa, as indicated by the

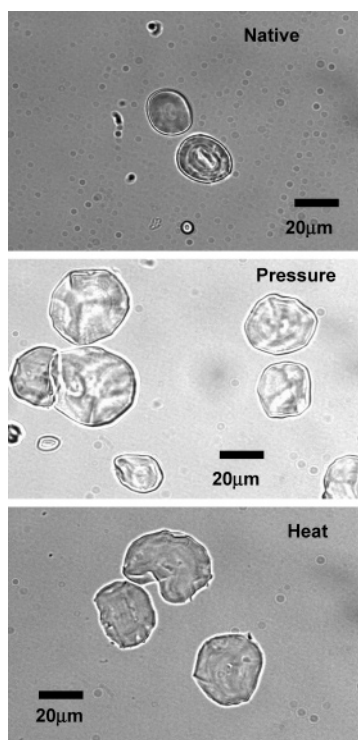


Figure 1. Light micrographs of native, pressurized, and heated wheat starches.

loss of birefringence (1, 13) and iodine discoloration (12). Calorimetric analysis confirmed these microscopic observations. Thermograms obtained with DSC for pressurized starch showed 15–88% of the melting peak relative to a native (unpressurized) starch sample (12). Differentiating between gelatinized and nongelatinized granules can be more difficult for a pressure-treated starch than for a heat-treated starch due to partial loss or fading of the birefringence. Less discernible swelling of the granules is observed after HPP treatment (in comparison to the significant swelling observed with heat gelatinization) that also makes differentiating gelatinized and nongelatinized granules more complicated (1). Although the heat-treated starch might be expected to show more swelling and possible rupturing of the granules that would help distinguish it from pressure-treated starch, we observed only minor differences microscopically in the starches after their respective treatments.

One plausible explanation for this discrepancy (or lack thereof) may be explained by the amount of water in the starch slurry. For example, most microscopic observations of starch gelatinization have involved a high water content ($\geq 80\%$ water and 1–20% starch) that readily allows the starch granules to swell during gelatinization. Presently, we used a starch slurry containing 40% starch and only 60% water for the respective pressure (90000 psi and 25 °C for 40 min) and heat (100 °C for 30 min) treatments. The decreased water content may have limited the uptake of water and swelling of the granules (especially in the heat-treated starch samples) and may account for the indiscernible differences between the pressure- and the heat-treated starches. Using more vigorous treatment conditions and increasing the water content would probably produce more obvious swelling and disintegration of the heat-treated starch granules.

DSC Analysis. Figure 2 shows profiles for the amylopectin recrystallization in pressurized and heated wheat starches during storage for 14 days at 4 °C. Pressurized wheat starch showed a relatively lower extent of amylopectin recrystallization than the

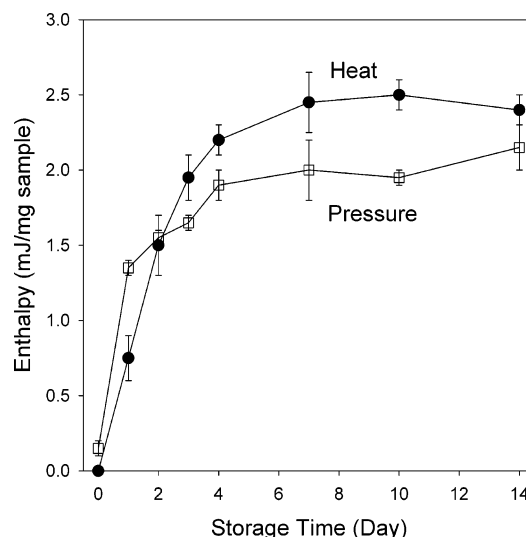


Figure 2. Change in amylopectin recrystallization of heated and pressurized wheat starch gels during storage at 4 °C.

heated wheat starch during storage, suggesting that less retrogradation is taking place in the pressurized starch than the heated starch.

The changes of amylopectin recrystallization during storage (3 days for heated starch and 7 days for pressurized starch, respectively) can be analyzed by the Avrami equation. The Avrami exponent (n) values for heated and pressurized wheat starch gels obtained by plotting $\log [-\ln \{(E_L - E_t)/(E_L - E_0)\}]$ against $\log t$ were 1.23 and 0.56, respectively. It is recognized that the Avrami exponent indicated the crystallization mode and its values can range from 1–4, in accordance with the nucleation and growth mode of crystal. Generally, the Avrami exponents of various starch gels are close to 1.0. This fact indicates that recrystallization of starch gel at a single temperature has instantaneous nucleation, followed by rodlike growth of crystals. It has been reported that wheat starch in bread (31) and both waxy and nonwaxy rice starch gels (32) showed typical retrogradation kinetics with instantaneous nucleation, followed by rodlike growth of crystals, which is similar to heated wheat starches. However, the Avrami exponent of pressurized wheat starch gel in this experiment was significantly less than 1.0. This result suggests that pressurized starch gel may have a different recrystallization mechanism that may be attributable to the presence of residual crystallinity after ultrahigh pressurization.

Figure 3 shows the ice-melting enthalpy change of pressurized and heated wheat starches during storage at 4 °C. Heated wheat starch showed a higher ice-melting enthalpy than pressurized wheat starch over the storage period indicating a relatively smaller amount of unfreezable water present in the pressurized wheat starch. In both pressurized and heated wheat starches, the ice-melting enthalpy gradually decreased with time suggesting that water incorporates into the crystalline structures of the starches during retrogradation and that some freezable water in both the pressurized and heated wheat starches becomes unfreezable water with retrogradation. During retrogradation, the calculated amount of the freezable water fraction changed from 44.6 to 41.9% for heated wheat starch, and from 41.5 to 40.3% for pressurized wheat starch.

Cheon et al. (14) investigated the retrogradation of pressurized waxy and nonwaxy rice starches in terms of calorimetric analysis and X-ray structure of crystals, and they reported that pressurized rice starch showed a higher amylopectin recrystallization than

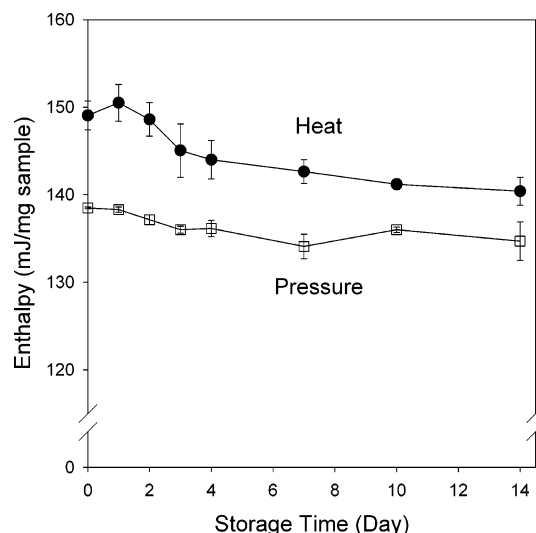


Figure 3. Change in ice melting enthalpy of heated and pressurized wheat starch gels during storage at 4 °C.

heated rice starch. This apparent inversion in recrystallization properties for the rice starch as compared to the wheat starch in the present study may be related to the nature of the starch type and its ability to gelatinize by high-pressure treatments. Specifically, the wheat starch shows complete gelatinization after being subjected to 90000 psi (621 MPa) at ambient temperature for 40 min. In contrast, rice starch did not completely gelatinize when treated at 600 MPa for 20–80 min (14). The presence of crystallinity remaining in the rice starch granules after pressurization could act as a source of nucleation that would increase the retrogradation of the starch in those circumstances.

The retrogradation of pressurized starch can be quite different from that observed after heat gelatinization (1). In some instances, pressure induced the formation of new structures in the starch (33) that were not observed with heat gelatinization. In the case of heated rice starch, the crystal type of the starch converted from A type to a mixture of B and V types. Pressure, on the other hand, changed the crystal type of rice starch from A type to a mixture of A, B, and V types, due to the presence of residual A type crystals remaining after the pressurization treatment (14). Additionally, differences in the remaining crystallinity were observed in relation to different pressurization times and temperature (14).

In the case of pressurized starches created at relatively higher conditions of pressure and exhibiting complete gelatinization, the retrogradation kinetics tend to be lower than those observed with heated starches. Using relatively lower pressure conditions such that gelatinization of the starch after pressure treatment is incomplete, the pressurized starch tends to retain some initial crystallinity and shows a larger extent of retrogradation and different crystal structure than the corresponding heated starch.

NMR Analysis. Figure 4 plots the value of the water self-diffusion coefficient (D) of pressurized and heated wheat starches as a function of time during storage at 4 °C. The pressurized wheat starch showed a lower value of D than the heated starch at all of the storage time increments (similar to the ice-melting enthalpy obtained with DSC), indicating that the translational proton mobility in pressurized wheat starch is more restricted than in heat starch. A possible contributor to this observation might relate to the fact that pressurized wheat starch granules retained their intact structure, whereas some of the heated wheat starch granules lost their structure, which would result in higher water self-diffusion coefficients in heated

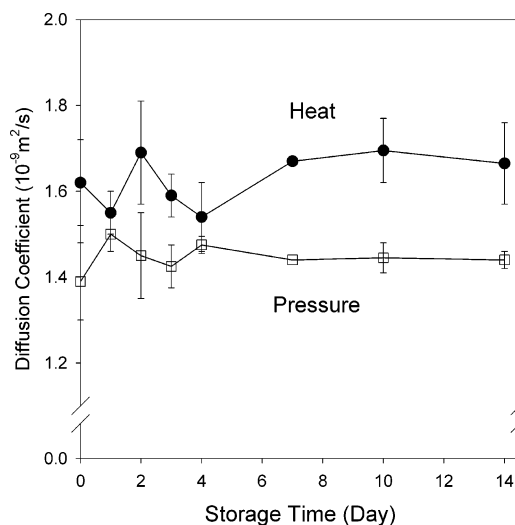


Figure 4. Change in water self-diffusion coefficient of heated and pressurized wheat starch gels during storage at 4 °C.

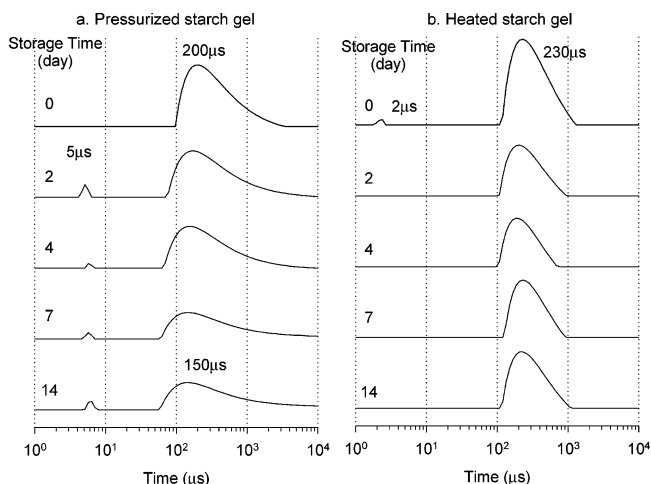


Figure 5. Continuous distribution of spin–spin relaxation time (T_{21} , obtained from one pulse sequence) of heated and pressurized wheat starch gels during storage at 4 °C.

wheat starches. In both the pressurized and the heated wheat starches, the value of D did not change significantly during storage at 4 °C, suggesting that only a very limited amount of water is entrapped in the starch crystal matrix. The water in pressurized and heated wheat starch may interact primarily with the amorphous regions of the starch gel matrix, which would not result in significant changes in the value of D during aging. Reports that the water in breadcrumb interacts with the amorphous structural components of starch to increase the rate of firming despite exhibiting less amylopectin recrystallization (29) may support this proposition.

Figure 5 shows the continuous distribution calculations of spin–spin relaxation time, T_{21} , obtained from one pulse experiments for pressurized and heated wheat starches at various time increments during storage at 4 °C. On day 0 (right after gelatinization and before storage commences), the pressurized starch shows one broad water population distribution peak at about 200 μs . As the storage time increases beyond 2 days, a small immobile water peak emerges at 5 μs , and the broad peak at 200 μs tends to shift toward shorter times (150 μs). On the other hand, the heated starch showed a small immobile peak at 2 s and a large peak at 230 μs on day 0. During progressive storage, the small peak at 2 μs disappears and the 230 μs peak remains relatively unchanged during storage.

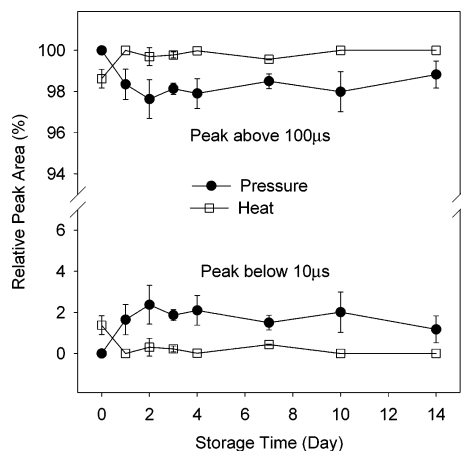


Figure 6. Changes in peak area of spin–spin relaxation time (T_{21} , obtained from one pulse sequence) of heated and pressurized wheat starch gels during storage at 4 °C.

As described above, the rigid or immobile proton signals occur in the microsecond (μ s) range and originate from protons on the solids or from water molecules tightly associated with the solids. The mobile proton signals occur in the millisecond (ms) range and originate from water molecules exhibiting relatively high mobility. Pressurized and heated wheat starches show differences in the relatively immobile water, which is in microsecond range. The changes in the peak areas for the T_{21} distributions during storage are plotted in **Figure 6**. In the case of the pressurized wheat starch, the more mobile fraction, which is the peak occurring above 100 μ s, decreases in day 1 and then maintains a constant value thereafter. The less mobile fraction, which corresponds to the peak below 10 μ s, increases in day 1 and then maintains a constant value for the remainder of the storage period. Results obtained for the heated wheat starch gels show the opposite tendencies, with the more mobile peak ($>100 \mu$ s) increasing to a constant value and the less mobile peak ($<10 \mu$ s) decreasing to a constant value after 1 day of storage. It is interesting to note that the more mobile fraction of the water in the pressurized wheat starch shifted to shorter relaxation times during storage but that the corresponding peak for the heated wheat starch did not move during aging. These results are indicative of the differences in the water dynamics for the pressurized wheat starch and the heated wheat starch.

Figure 7 shows the continuous distribution of spin–spin relaxation time, T_{22} , of pressurized and heated wheat starches obtained from CPMG pulse experiment during storage at 4 °C. This T_{22} distribution characterizes the mobile fraction of water, which occurs in the millisecond range. The T_{22} transverse relaxation time water population distribution confirms the differences in the water dynamics for the pressurized wheat starch as compared to the heated wheat starch. Pressurized wheat starch showed three distinctive populations occurring at 1, 6, and 30 ms in the fresh (0 day) and all of the aged samples. Heated wheat starch showed four populations for the fresh (0 day) sample occurring at 0.2, 1, 6, and 60 ms, and the 0.2 ms sample disappeared by day 2 of storage, and the three populations at 1, 6, and 60 ms were present at all of the storage times. Other noticeable differences were observed in the peak shape and in the mean value of the more mobile fraction: The 30 ms peak for pressurized starch showed a sharp and narrow distribution, and the 60 ms peak for the heated starch was a short and broad distribution. **Figure 8** shows the change of peak area of continuous distribution of T_{22} during storage. In both pressurized and heated wheat starches, the more mobile fractions increased,

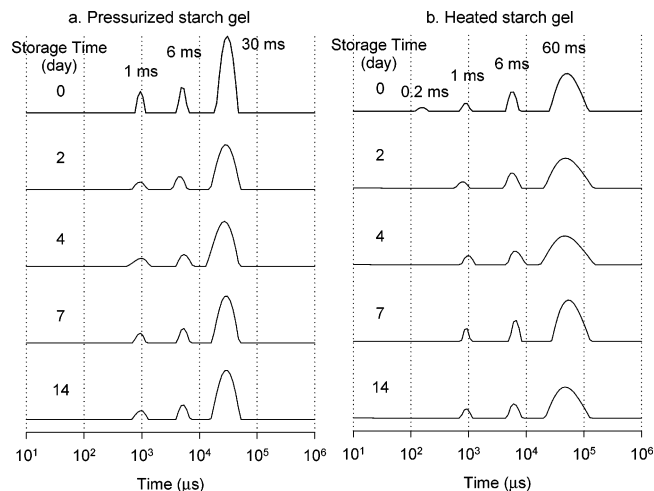


Figure 7. Continuous distribution of spin–spin relaxation time (T_{22} , obtained from CPMG pulse sequence) of heated and pressurized wheat starch gels during storage at 4 °C.

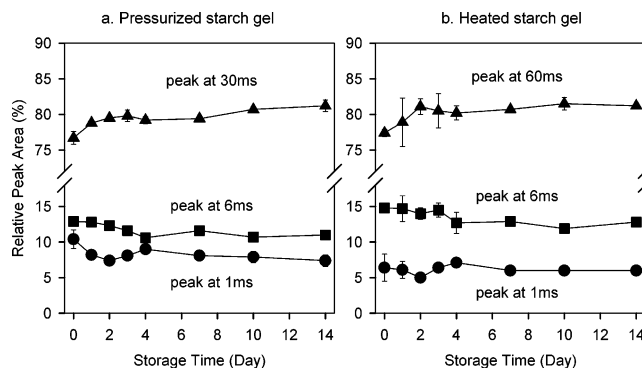


Figure 8. Changes in peak area of spin–spin relaxation time (T_{22} , obtained from CPMG pulse sequence) of heated and pressurized wheat starch gels during storage at 4 °C.

but the moderately mobile fraction and the less mobile fraction gradually decreased with aging. Although pressurized and heated wheat starches had different populations of transverse relaxation times, the change of the amount of each fraction showed a similar pattern during aging indicating that each fraction (e.g., less mobile, medium mobile, and more mobile fractions, respectively) in both wheat starches may play the same role in the retrogradation mechanism.

In conclusion, pressurized wheat starch features gelatinization characteristics and a retrogradation mechanism distinctive from those for heat-gelatinized wheat starch. Pressurized starch has different water dynamics as compared to traditionally heated starch that results in different retrogradation mechanisms and functionalities. The fundamental information and insight on the physicochemical properties of pressurized wheat starch that this study provides open the possibility for future investigations of the effects of high pressure on starches and other food biopolymers and the applications of HPP in the preservation of foods.

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