Journal of Thermal Analysis, Vol. 47 (1996) 1361-1375

THERMOMECHANICAL PROPERTIES OF GLASSY CEREAL FOODS

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Abstract

The main objective of this paper is to discuss the relationship between physical state, fracture mechanism, and texture for low moisture cereal-based foods. Experiments were also carried out to get a better understanding of the role of water. At room temperature, extruded bread and white bread (previously) dehydrated, then rehydrated in atmospheres with controlled humidities) exhibited a brittle behavior up to around 9% moisture. At 13.7% moisture, they were ductile. A significant loss in the crispness of extruded bread was observed between 8.5 and 10% moisture. The glass transition temperature (T_g) was measured, using dynamic mechanical thermal analysis (DMTA), for samples with up to 40% moisture. The resulting T_g curve showed that the important changes in fracture mechanisms and crispness occurred while the samples were still in the glassy state. The viscoelastic behavior of both extruded and white breads suggested that a secondary relaxation occurred around 10°C. Another event was observed around 70°C for low moisture sample, using DMTA. This event was attributed to disruption of low energy interactions.

Keywords: aging, bread, fracture mechanism, glass transition, viscoelasticity, sub-T_g relaxation

Introduction

It is a likely assumption that to be perceived as crispy, a food material should exhibit a brittle behavior. Moreover, its fracture should be accompanied by a sound [1], reflecting the way energy is released within the material during fracture. Two main properties can be considered to control the quality of crispy foods: their sensory crispness and their resistance to fracture. Both properties reflect the mechanical behavior of the food material.

Foods such as crispy, low moisture, cereal foods are generally stored and consumed in the glassy state. Indeed, since cooking is accompanied by the disappearance of most crystalline structures, and since storage below the glass transition strongly inhibits recrystallization, such foods remain mostly amorphous.

Most crispy cereal foods are known [2, 3, 4] to be highly sensitive to changes in moisture content. Upon hydration above a critical moisture content, crispness is lost and mechanical behavior changes from brittle to ductile; moreover, due to collapse phenomena, the shape of expanded foods may change. A

0368–4466/96/ $ 5.00

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close relationship was observed between loss in sensory crispness and changes in mechanical properties, when the moisture content of breakfast cereals was increased above 9% [4].

Extensive studies performed on synthetic polymers have demonstrated that mechanical behavior is highly dependant on the physical state and organization of the polymer network [5]. Studies have recently been performed, on the one hand, to correlate changes in mechanical properties (e.g. the brittle–ductile transition) with relaxation processes exhibited by polymers [6], and on the other hand, to explain the consequences of aging on these mechanical properties [7]. Discussion on the relationship between texture and physical state of food products is still rare, particularly when amorphous materials are concerned [8]. Moreover, the molecular mechanisms involved in the modifications induced by rehydration are still poorly understood.

The objective of this paper is to discuss the relationships between texture, mechanical behavior, and the molecular relaxation processes occurring within low-moisture cereal foods. Our interest was also focussed on the role played by water. Results obtained with extruded bread have been compared to those obtained with white bread, in order to evaluate the contribution of the organization of the polymer network. White bread can be considered as a solid foam [9–11] (or sponge, depending on whether the cells are closed or open), in which the cell walls are composed of a very concentrated dispersion of particles (partially swollen and gelatinized starch granules occupy most of the sample volume) in a continuous phase of mixed polymers [12] (i.e. amylose, being the main starch component in this phase, and gluten). In extruded bread, the granular structure of starch is expected to have disappeared [13], and thus the continuous phase surrounding the air cells is composed predominantly of the starch components, mainly amylopectin. Moreover, some reduction in the mean molecular weight, and some orientation of the biopolymers, may result from the extrusion process [13].

Materials and methods

Commercial extruded and white breads were obtained from a local supermarket. For the extruded product, the composition, as provided by the manufacturer, was: wheat flour (72%), rye flour, wheat germ (5%), sugar, vegetable fat, lactose, skimmed milk powder, salt. The initial moisture content was 5.3 % (wet basis). For baked white bread, the composition, as provided by the manufacturer was: wheat flour, sugar, honey, milk powder, milk fat, salt, yeast, lecithin, calcium propionate, water.

The samples were dried over P_2O_5 at room temperature for three weeks (unless when specified). Then, they were equilibrated in different atmospheres with relative humidities ranging from 11 to 84% (saturated salt solutions, 25°C). Moisture content was measured by weighing samples before and after drying for 5 h at 103 °C. Moisture content values are expressed as percent water on a wet basis (w/w).

Mechanical tests

The deformation rates used (crosshead speeds of 20 and 10 mm min^{-1} for compression and tensile tests, respectively) were chosen in order to provide good sensitivity and good precision, and to limit sample dehydration during the analysis.

Strips (30×15×7.5 mm) were subjected to uniaxial compression between two parallel plates, on an Instron 1122 machine, at room temperature.
For the extruded samples, two orientations were tested:

- stress parallel to the extrusion direction;
- stress perpendicular to the extrusion direction.

The extrusion direction was assumed to correspond to a preferential orientation of the molecular chains and air cells imposed by the extrusion process. Samples were compressed up to 50% strain. The fracture behavior was characterized by the fracture stress and apparent Young's modulus (E; $E_{\parallel}$: stress parallel to the extrusion direction; $E_{\perp}$: stress perpendicular to the extrusion direction). For each moisture content, five samples were tested and a mean value calculated.

Tensile tests were performed on extruded samples only. The samples had the same shape and size as in compression tests.The extremities of the samples were glued onto aluminium plates fixed to the machine with double-faced cellotape. Samples were subjected to uniaxial tension until rupture. The parallel and perpendicular orientations were tested. The fracture behaviour was characterized by the fracture stress and Young's modulus, and by the percent of deformation at rupture. For each moisture content, three samples were tested and a mean value calculated.The variation coefficients were in the range 10 to 25% for all the calculated parameters and for the two types of tests.

Dynamic viscoelastic measurements

During dynamic mechanical measurements, samples were subjected to a sinusoidal tension-compression test. With the Viscoanalyzer (Metravib R.D.S; Ecully, France) used in this study, the stiffness K and the loss angle δ were obtained and allowed the calculation of the dynamic parameters: the dynamic Young modulus E^*, its elastic (or storage) component E', and its viscous (dissipative) component E''.

Sample dimensions were approximately: 30×15×10 mm. They were glued (Super dur, Duratrode) onto sample holders and protected from dehydration with silicon grease (Rhone-Poulenc). A 5 μm ($1.7\times10^{-2}\%$) deformation was applied to the samples; the range of frequencies used was between 5 and 40 Hz. The experiments were performed within the linear region of viscoelasticity.

Differential Scanning Calorimetry (DSC)

Samples with different moisture contents and commercial samples "as is" were analyzed by differential scanning calorimetry (Perkin-Elmer DSC 2), in order to detect either any eventual melting peak of crystalline material or any enthalpic relaxation event. Bread samples (approximately 10 mg) were weighed into pre-weighed aluminium pans. To detect crystallinity, distilled water was added, in order to analyse the samples in the presence of excess water (75% wet basis), whereas enthalpic relaxation was studied without excess water. The pans were sealed and scanned from 27 to 147°C at a rate of 10°C min^{-1}. Then, they were cooled to 27°C and rescanned at the same rate and within the same temperature range. The DSC was calibrated with azobenzol and indium.

Results and discussion

Effect of water on the mechanical behavior of cereal-based foods

The mechanical behavior of cereal foams is controlled by the density of the material, the geometric characteristics of the foam, and the viscoelastic behavior of the cell walls [9]. In the following, we will concentrate on the fracture mechanism of foams and on their viscoelastic properties. It can be considered that a product's response to large deformations is related to its handling and eating quality, whereas experiments at small deformations yield information on the structure and physical state of the material.

Fracture mechanism (large deformations)

Figure 1 illustrates the effect of water on the stress/strain curves for extruded bread, obtained by both compression and tension tests. Up to 9% moisture (w/w on a wet basis), extruded bread exhibited brittle behavior, which then changed to ductile behavior. Similar results were obtained for white bread by compression tests.

Figure 2 shows the apparent Young's modulus as a function of moisture content, determined from the results in Fig. 1a for extruded bread. Increases in modulus and fracture stress [14] (e.g. fracture stress from 0.25 to 0.5 MPa, for compression test with the stress parallel to the extrusion direction) were observed between 4 and 9% moisture by both compression and tension tests; then, both quantities decreased with increasing moisture content. The amplitude of the increase in modulus was strongly dependant upon the orientation of the

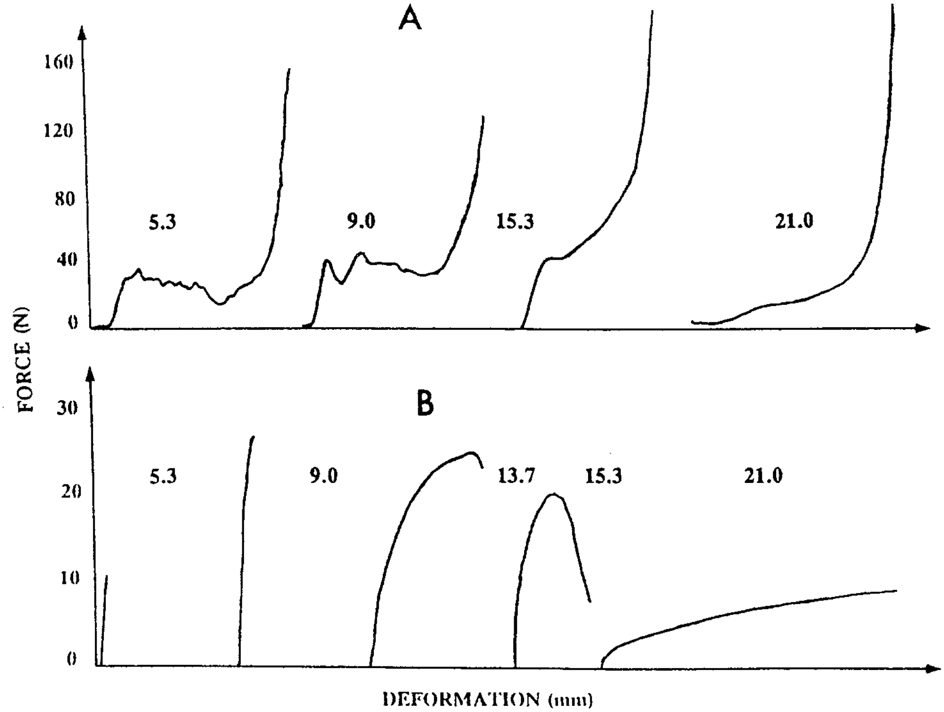

Fig. 1 Influence of moisture content on force-deformation curves for extruded bread: A) compression; B) tension experiments. Moisture contents in % (w/w wet basis) are given in brackets

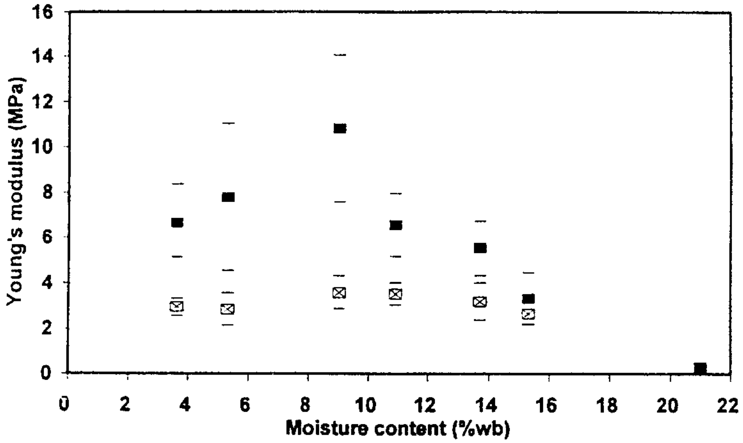

Fig. 2 Effect of water on the apparent Young's modulus of extruded bread, calculated from the force deformation curves (compression tests) in Fig. 1. (■): stress parallel to the extrusion direction; (⊠): stress perpendicular to the extrusion direction

polymer chains and air cells, resulting from the extrusion process. The increase was particularly significant when the deformation was parallel to the direction of the polymer chains. The ratio, $E_{\parallel}/E_{\perp}$, reflecting the anisotropy of the sample, was 2.2 at 3.6% moisture, increased to 3.0 at 9% moisture, then decreased with increasing moisture. A similar change was observed with the anisotropy ratio of fracture stresses [14]. Above 15% moisture, no effect assignable to anisotropy was observed with the extruded sample. Collapse of the foam structure occurred above this level of hydration, as was revealed by an increase in the material's apparent density (from 0.13 g cm^{-3} below 14% moisture to 0.4 g cm^{-3} at 20% moisture) (no change in density was observed with white bread). Thus, two steps in the process could be distinguished: up to 9% , the increase in modulus could result from an apparent stiffening effect of water on the polymeric network; then at higher moisture contents, the softening effect of water would become predominant. The stiffening process was reversible upon dehydration, i.e. rigidity decreased as moisture content decreased from 9 to 5%.

The change in the apparent Young's modulus observed for white bread was different from that for extruded bread, since no apparent stiffening effect of water was observed for white bread (data not shown). The apparent Young's modulus decreased slightly and continuously from 2.5 to 1.1 MPa, as moisture content increased from 3 to 9%. Within the same hydration range, the fracture stress was almost constant, on the order of 0.5 MPa.

An apparent hardening effect of water (maximum in the fracture stress at 10% moisture) on the mechanical behavior of starch has previously been reported [15]. Nicholls *et al.* [16] compared the fracture mechanism for extruded gluten, wheat starch, and waxy maize starch, within the hydration range from 4 to 18% moisture. They observed that gluten exhibited a fracture behavior very different from that of starch. Gluten failed by unstable brittle fracture up to a moisture content of 12.5%; then at 15.5% moisture, it became more ductile. Those authors concluded that the brittle/ductile transition for gluten was closely associated with the glass transition of the protein. In contrast, starch exhibited an increase in ductility while still in the glassy state.

X-ray diffraction measurements, as well as DSC in the presence of excess water, confirmed that both our extruded and white bread samples, at ≤9% moisture, were mostly amorphous (no crystallinity detected). Thus, we focussed our attention on the relaxation processes affecting the amorphous materials, i.e. the glass transition and relaxations occurring below the glass transition temperature (T_g).

Viscoelasticity determined from mechanical spectroscopy (small deformations)

Evidence of a glass transition occurring in white bread was previouly demonstrated [17] using thermomechanical analyses (TMA), then confirmed using dynamic mechanical thermal analyses (DMTA) [18]. The α relaxation associated with the glass transition of amorphous materials is characterized by a drop in the elastic component (E') of the complex Young's modulus (E^*), a peak in the loss component (E'') and a peak in tanδ (E''/E') as a function of temperature; these features are shifted to higher temperatures at higher frequencies [19]. A peak in tanδ associated with sub-T_g relaxations would exhibit a lower sensitivity to frequency [20] than whould the T_g peak, and a peak due to the melting of an organized structure would not be sensitive to frequency. The value of the modulus reflects the extent of intermolecular coupling that controls the resistance of the molecules to deformation [21]. The value of tanδ represents the dissipation factor.

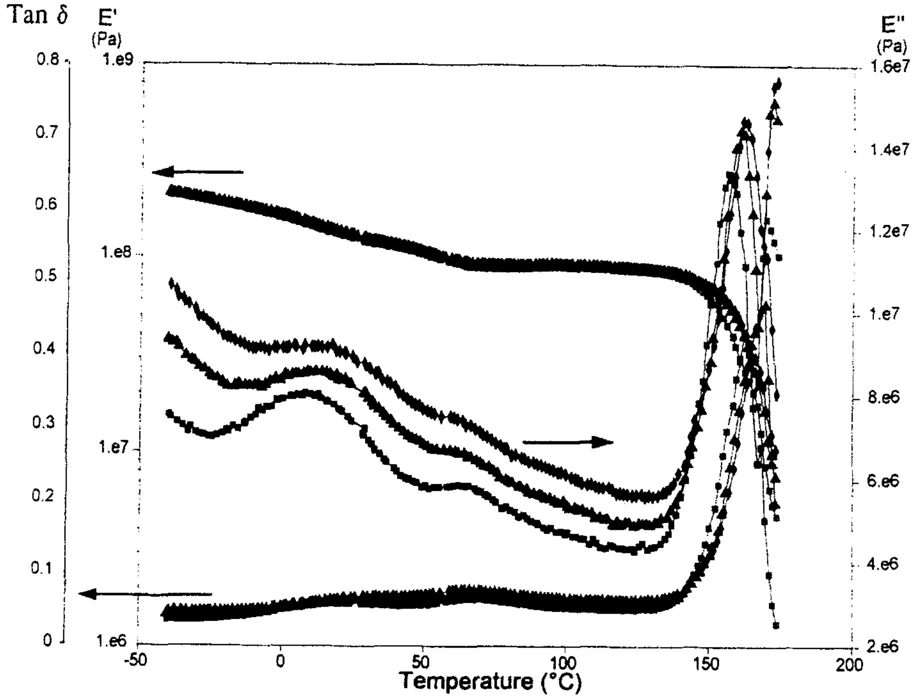

Fig. 3 Viscoelastic behavior of extruded bread with 5 % moisture. Measurement frequencies: ■ 5 Hz, ▲ 20 Hz, ♦ 40 Hz; deformation: 5 μm (1.7 $\times 10^{-2}$%)

The viscoelastic behavior of extruded bread with 5% moisture is illustrated in Fig. 3. A glass transition was observed to start around 140°C (drop in E', peaks in E'' and tanδ, all shifted to higher temperatures at higher frequencies). Peaks in tanδ and E'' with much lower amplitudes were also observed at lower temperatures. A peak in tanδ (and E''), centered at 10°C, exhibited a lower sensitivity to frequency than did the main relaxation peak. A broad tanδ peak was also observed around 70°C; its position was not sensitive to frequency. The same features were observed for a sample of white bread with 5% moisture.

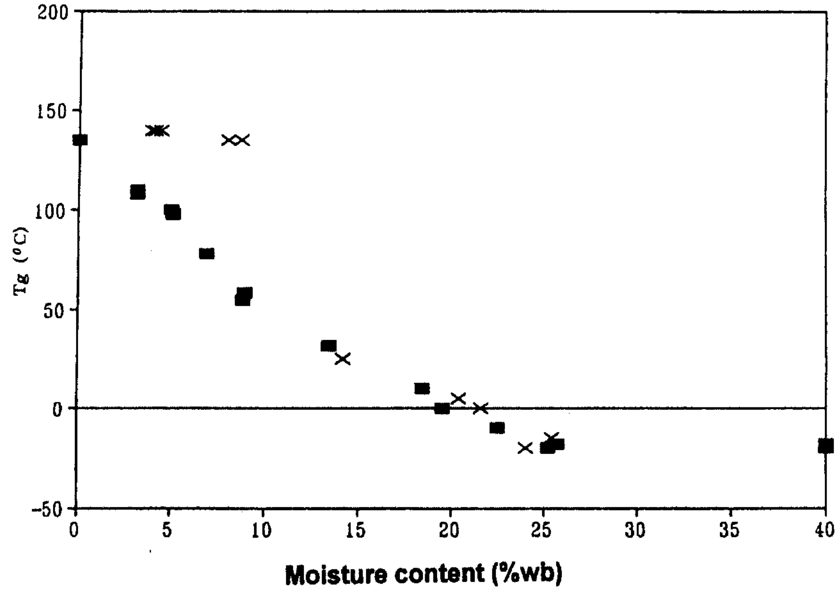

Fig. 4 Influence of water on the glass transition temperature (T_g) of extruded bread (×) and white bread (■). T_g was determined from the onset of the drop in E' measured at 5 Hz

As the moisture content of white bread increased, the peak in tanδ , characteristic of the glass transition, shifted to lower temperatures. This same plasticizing effect of water on the T_g of extruded bread could only be observed at moisture contents higher than 14%. Figure 4 shows T_g *vs*. moisture content for white bread and extruded bread. At room temperature, both products went through their glass transition when their moisture content exceeded about 15%.

The glass transition can be made to occur either by increasing the temperature of a sample at constant hydration or by increasing its moisture content at constant temperature. (It could also be made to occur by changing the measurement frequency, or, in industrial practice, by changing both hydration and temperature.) Thus, when the values of tanδ, measured at 20°C (for both the extruded and baked breads are plotted *vs*. moisture content (Fig. 5), a strong increase is observed above 15% moisture; this increase could be attributed to the glass transition. This would confirm that, at room temperature, both white bread and extruded bread passed through their glass transition when their moisture content exceeded 15%. The peak in tanδ, associated with the glass transition, was much higher for the extruded sample than for white bread. This could be related to a higher number of relaxing segments [21] within the glass transition region in the extruded bread than in the white bread sample. This difference between the extruded and white breads should be attributable to differences in the structure of the materials (e.g. a more homogeneous [19] structure for the extruded samples) or organization of the biopolymer network (e.g. differences in intermolecular interactions).

The apparent activation energy for the glass transition of white bread was calculated from the influence of frequency on the temperature of the maximum in the tanδ peak (calculated from the slope of the ln(frequency) *vs.* $1/T_{max}$ plot). Values of 424 and 370 kJ mol^{-1} were obtained for white bread samples with almost 0 and 5% moisture, respectively. Approximately the same value (415 kJ mol^{-1}) was obtained for extruded samples with 4 to 10% moisture. From these values, a "fragility" parameter (*m*) could be determined [22]. According to Angell's classification, low-moisture breads could thus be categorized as relatively "strong", i.e. structurally quite resistant to heating above T_g. These values of activation energy for the α relaxation of white bread were similar to that reported for synthetic polymers in the absence of plasticizer (351 kJ mol^{-1} [19]. Values of 242 kJ mol^{-1} for gluten with 13% moisture and 300 kJ mol^{-1} for amylopectin with 10% moisture have also been reported [23, 24].

The positions of the minor peaks at 10 and 70°C were not significantly affected by hydration. Their sensitivity to frequency suggests that a secondary relaxation (sub-T_g) occurred around 10°C for both the extruded and baked low-moisture breads. β (or secondary) relaxations are generally described as resonances associated with reorientational or vibrational motions of small molecules, side groups, or small segments of polymers [25]. They involve localized motions occurring in high entropy regions dispersed in the otherwise rigid matrix of a glass. These motions are associated with low energy barriers. Secondary relaxations, which are almost undetectable by calorimetry, are easily detected by dielectric or mechanical relaxation spectroscopy [26, 27]. In proteins, they have been attributed to water-sensitive motions of side chains [26]. In glucose-water glasses, the motions of water molecules were credited with being only partly responsible for the β relaxation, since anhydrous glucose also showed a well-separated β process, observed in dielectric relaxation studies [27]. The latter authors mentioned that in polymers, water did not seem to affect the location of the β process.

The origin of the peak at 70°C (Fig. 3) is still unclear; however, the fact that its position was not sensitive to measurement frequency suggests that the underlying molecular process could be associated with a melting process, i.e. with a disruption of low energy interactions. Moreover, in contrast to the glass transition and the minor relaxation observed at 10°C, where the peaks in tanδ were associated with a steeper decrease in the *E′* component, the broad tanδ peak observed around 70°C was accompanied by a stabilization of sample rigidity *(E′)*. Thus, one interpretation for this behavior could be that the disruption of low energy interactions suggested above could involve the disruption of water-biopo-

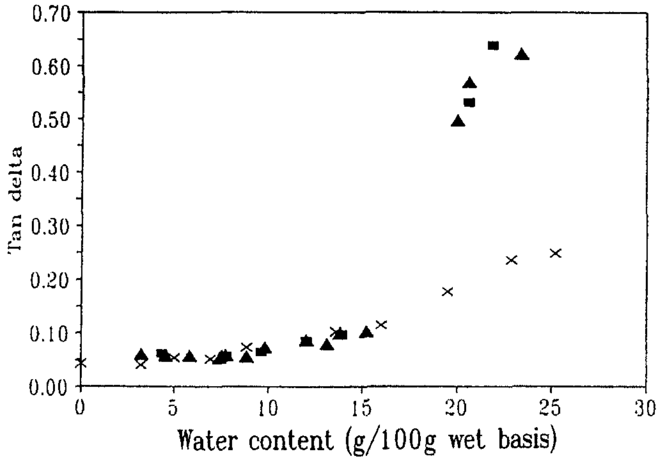

Fig. 5 Influence of water content on tanδ values measured at 20°C and 5 Hz for extruded bread (▲): stress perpendicular to the extrusion direction; (■): stress parallel to the extrusion direction; and white bread (x)

lymer hydrogen bonds. Experiments are currently underway to study the nature and reversibility of the events at 10 and 70°C.

Dynamic measurements of Young's modulus (measured at 20°C) were also sensitive to the anisotropy of an extruded sample, and we observed that the ratio, $E_{\|}/E_{\perp}$, which was close to 1 at moisture contents >15% , was >1 below T_g (e.g. around 2 at 5% moisture). Tanδ values were not sensitive to sample anisotropy (Fig. 5). The disappearance of an anisotropic effect, on the apparent Young's modulus measured at large deformations, and on the complex Young's modulus measured at small deformations, may be related to the increase in density, as the extruded sample started to collapse when it approached the glass transition. This collapse of the macroscopic structure of the extruded sample might be a reflection of structural relaxation of the polymer network. This behavior might be due to the fact that, at the end of the extrusion process, the biomolecules could be captured in a conformational state of relatively high free-energy. When the mobility of the polymer network increases due to increased hydration, the network would relax to a more stable configuration. No significant effect of time was observed on the complex Young's modulus of extruded bread (with 5 to 9% moisture) measured at 25°C.

DSC studies

From the DSC results, no thermal event assignable to a glass transition was detected for the bread samples described above. The change in specific heat at T_g is expected to decrease with increasing chain stiffness, cross-linking density, or distribution of structural relaxation times [22, 25, 26], Two main explanations can be proposed for the absence of ΔC_p shift in DSC curves for the cereal-based foods studied:

– because of the intrinsic heterogeneity of these kinds of food products, the transition is spread over a large temperature range, and thus, the progressive increase in heat capacity becomes undetectable;

– a smaller increase in heat capacity would be associated with the glass transition, as expected for materials classified as "strong" by Angell [22]. "Strong" materials are those that exhibit a strong resistance against structural degradation when heated through their supercooled liquid regime. Indeed, as reported above, breads behaved as relatively "strong" materials.

Although no thermal event (step in the change in specific heat) assignable to a glass transition was observed with these cereal foods, an endothermic peak did appear upon aging both the white and extruded bread samples below or above T_g (Fig. 6). This peak was shifted to higher temperatures as the aging temperature was increased (below T_g), and its enthalpy increased slightly. When a bread sample with 9% moisture was aged at 25°C, the maximum of the endothermic peak was at 56°C; after aging at 40°C, the peak maximum was at 70°C. Moisture content and aging time had only limited effects on the temperature and enthalpy (on the order of 2.5 J g^{-1}, for both the extruded and baked breads stored at 25°C) of the endotherm. This endotherm disappeared on an immediate second scan and gradually returned on storage. As previously [28] observed, the endotherm also disappeared upon dehydrating white bread samples at room temperature.

An endotherm around 50–70°C, i.e. at or below T_g, with the same characteristics (sensitivity to changes in hydration, and to thermal treatments) was also reported for other cereal foods and for a wide range of aged biopolymers (polysaccharides or proteins) with 5 to 25% moisture [23, 29–32]. Different interpretations have been proposed to explain such an event:

– A similar sub-T_g endotherm, frequently described for synthetic polymers, is generally attributed to enthalpic relaxation associated with the glass transition [33]. The same interpretation has been proposed for cereal products [29].

– Disruption of a long-range hydrogen-bonding network [32].

– Interactions between water and polysaccharides could lead to endothermic melting [30].

– After one year of storage, the enthalpy of the endothermic peak was on the order of 1 J g^{-1} for wafers (6% moisture). Based on wide-angle X-ray scattering and Fourier transform infra-red results, it was concluded that the thermal event corresponded to the melting of some short-range ordered structure, such as a single-helical structure [31].

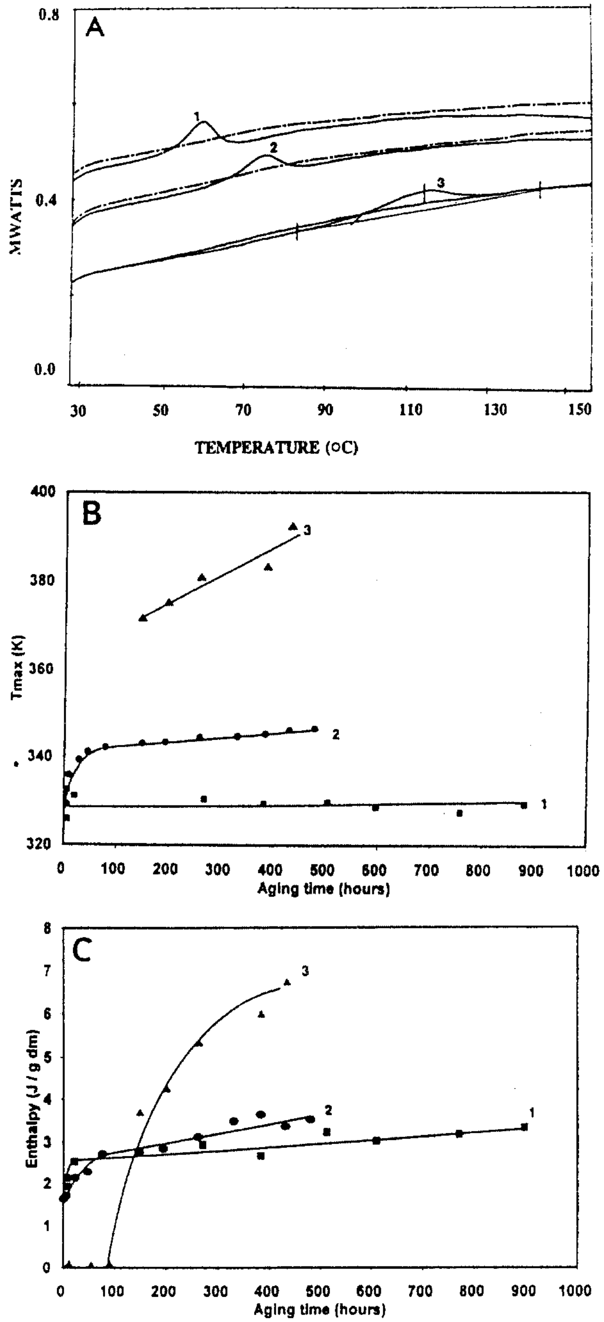

Fig. 6 A) DSC curves for white bread with 9% moisture, after 11 days of aging at (1) 25°C, (2) 40°C, and (3) 70°C; heating rate 10°C min^{-1}; dotted line = second scan. Influence of aging time on B) the T_{max} endotherm and C) enthalpy; aging temperature: (1) 25°C, (2) 40°C, (3) 70°C

Upon aging the 9% moisture extruded bread at 70°C, an endothermic peak was observed at 108°C, but with a different rate of appearance (i.e. corresponding, probably, to a different event): until 4 days of storage, no endotherm was observed; after that, the endotherm appeared and increased strongly with increasing time (enthalpy: 6.5 J g^{-1} after 20 days) (Fig. 6). This endotherm was assumed to reflect a phenomenon such as the melting of starch retrograded during aging.

Relationship between mechanical properties and physical state

White bread and extruded bread both exhibited a brittle-to-ductile transition in the same range of moisture contents: between 9 and 13.7% . More precise measurements of the hydration conditions that induce the brittle-to-ductile transition would be useful to demonstrate any eventual differences in behavior between the extruded and baked breads. A pronounced increase in density, probably due to collapse of the foam structure, was observed to occur above 12% moisture for the extruded bread. For the same food product, a loss of crispness was observed between 8.5 and 10% moisture [3].

Figures 4 and 5 suggest that, for both types of cereal foods, these modifications occurred below T_g. The importance of sub-T_g relaxations to the mechanical behavior of synthetic polymers was recently demonstrated [6]. When these relaxations involve localized skeletal motions, they may correlate with the ductility or toughness of a polymer. Wu [6] suggested that the brittle-ductile transition arises from the onset of skeletal-bond rotations under an applied stress (i.e. the onset of conformational rotations of skeletal bonds). Depending on the polymer's flexibility, this onset could occur more or less closely to T_g.

The peaks in tanδ, observed with mechanical spectroscopy, at 10 and 70°C can not be directly related to a change in fracture behavior. However, Fig. 5 suggests that, for the extruded sample at 20°C, tanδ values started to increase slightly around 10% moisture, i.e. before the onset of the main peak attributed to the glass transition. Mechanical behavior at large deformation indicated that this corresponded to conditions under which water induced a softening of the extruded bread (a decrease in the apparent Young's modulus). A significant loss of sensory crispness for the extruded sample also occurred within the same hydration range. These results would thus agree with the hypothesis that the onset of localized skeletal motions of the biopolymers (probably starch) would require approximately 10% moisture at room temperature, and that these motions would cause important changes in the mechanical properties of extruded bread. Moreover, the extruded sample and starch [15, 16] appear to exhibit similar mechanical behavior.

Other similarities between the baked and extruded samples can be noted. They exhibited the same stages in the relaxation processes observed with

DMTA, in particular, the occurrence of a relaxation, just below room temperature (in the frequency range studied), showing the same characteristics as that of a secondary relaxation. As discussed above, a secondary relaxation corresponds to the onset of localized, relatively fast and low-amplitude motions of water-sensitive small molecules or polymers. This increased degree of mobility would be expected to allow some local reorganization of the glassy matrix to occur during storage. Physical aging, which is the change in properties observed when a thermodynamically non-equilibrium glassy state tends to recover toward an equilibrium state, at constant temperature and zero stress [7], has been shown to affect the mechanical behavior of synthetic polymers within the glassy state. The enthalpy relaxation (endothermic event) associated with the glass transition is generally observed after aging below T_g, but above secondary relaxation processes [22]. Even though, with DSC, an endotherm was observed below T_g for both low-moisture white bread and extruded bread, no significant effect of aging time or of hydration (i.e. of proximity to T_g) was observed, as would have been expected for physical aging. Moreover, no significant effect of time on the complex Young's modulus was observed in this study. It was thus difficult to conclude anything about the possible contribution of physical aging to the observed results.

Upon hydration, the extruded sample became harder. This hardening was reversible upon dehydration. A possible interpretation for the mechanical behavior of the extruded bread would be that water would enhance the intermolecular, low-energy interactions between biopolymers, and thus improve the sample's resistance to fracture. This strengthening effect of water on the extruded sample would be favored by the orientation of the polymer chains and by the instability of the polymeric glass, since the extruded material is assumed to be quenched, at the end of the extrusion process, in a state far from the equilibrium conformation of its constituting polymers.

Conclusion

Despite a better knowledge of the physical state of cereal-based foods, the understanding of the molecular mechanisms controlling their mechanical properties is still incomplete. Thorough studies on the role of water are highly desirable. Magnetic resonance studies are currently underway to measure the molecular mobilities of both the water and the solid matrix, under different conditions of hydration and temperature.

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