Quantitative Description of Fracturability Changes in Puffed Corn Extrudates Affected by Sorption of Low Levels of Moisture

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ABSTRACT

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Fracturability is a defining textural characteristic of extruded and crunchy products such as puffed snacks and cereals. Even low levels of moisture can significantly affect deformation properties and texture due to changes in the distribution of fracture intensities. The fracturability of puffed corn extrudates produced at two specific mechanical energy (SME) levels, which greatly influenced extrudate structure and deformation behavior, was measured by compression testing before and after equilibration of samples at 33% rh. Significant changes in fracturability due to moderate moisture sorption were manifest in a reduced total number of fractures occurring during compression, an indication of plasticization that was confirmed independently by differential scanning calorimetry (DSC) studies as reductions in glass transition temperature (T_g). However, in both instances, mean fracture intensity and average compressive resistance increased after equilibration, indicating a qualitative toughening or hardening of the products, despite increased moisture and decreased T_g. These textural developments were also reflected in changes in the parameters of fitted fracture intensity distributions. Thus, the influence of processing conditions (quantified in terms of SME) on the creation of new micro and macrostructures, and the effect of low levels of moisture on these structures, can be identified by using fracturability characteristics and T_g. Furthermore, fracturability parameters can demonstrate complexity in the deformation patterns of products that thermal measurements confirm to be plasticized.

Fracturability is an important and defining textural characteristic of brittle, crunchy products, particularly puffed, extruded foods such as cereals and snack items. This property is manifest in the progressive, incremental structural failure that occurs in response to strain due to the sequential breakage of small subunits of the material. Dried, porous products exhibit this type of deformation behavior because they comprise a network of cells and cell walls in which individual structural units can independently collapse even while the sample remains generally intact. Barrett et al (1994) defined sensory crunchiness arising from fracturability in terms of the perceived magnitude and frequency of repeated failures occurring in the sample during compression with the teeth.

However, fracture behavior and sensory characteristics arising from fracturability are highly subject to alteration by environmental conditions such as elevated humidity, which leads to plasticization by moisture sorption. Various methods have been proposed to quantify fracturability, such as fractal or Fourier analysis of the jagged-appearing stress-strain functions of specimens undergoing compression (Barrett et al 1992, 1994a; Peleg and Normand 1993; Rhode et al 1993), in which stress levels rapidly oscillate due to sequential fracturing of the material. A representative stress-strain relationship of a dry extruded product is shown in Fig. 1. Measured indices from these techniques, fractal dimension and mean magnitude of selected ranges of the power spectrum, respectively, have also been related to sensory texture (Barrett et al 1994a), physical structure (Barrett et al 1994a), and equilibration relative humidity (Barrett et al 1992, Rhode et al 1993).

More recently, Barrett et al (1994b) measured the intensity of fractures occurring during compression of (dried) puffed extrudates and fitted these sequential reductions in developed stress (determined by calculating the difference in stress level at subsequent strain data points) to an exponential function, the parameters of which were descriptors of product texture. Additionally, we found that samples equilibrated at 43 and 75% rh had lost between 71 and 98% of their initial fracturability as defined by cumulative fracture stress. Data from equilibrated samples were not fitted to distributions due to the extremely limited number of fractures occurring during compression. However, the relationship between texture and moisture at much lower moisture contents is potentially complex, as evidenced by observations by Harris and Peleg (1996), Barrett et al (1995), and Katz and Labuza (1981) of increased compressive resistance in extruded samples maintained at moderate humidity levels (before ultimate losses in strength and fracturability with absorption of greater quantities of water). Kaletunc and Breslauer (1993, 1996) showed that the influences of both the extrusion processing parameter specific mechanical energy (SME) and plasticization could be monitored by observing changes in the glass transition temperature (T_g) of extruded products. Furthermore, differential scanning calorimetry (DSC) studies of extrudates demonstrated that T_g is related to the textural attribute of crispness (Kaletunc and Breslauer 1993), which is a sensory indicator of the extent of brittleness of an extruded sample. Brittleness, in turn, can be quantified by the fracturability parameters of extruded products.

In this study, the effects of a low level of moisture that affects but does not eliminate fracturability were evaluated to quantitatively describe subtle changes in texture that can occur during storage of imperfectly sealed products (i.e., after opening of packages and exposure of product to moderate humidity levels). Two extrusion conditions, high moisture (low SME) and low moisture (high SME), were used to produce samples of widely differing physical properties. The thermal characteristics of the dried and humi-

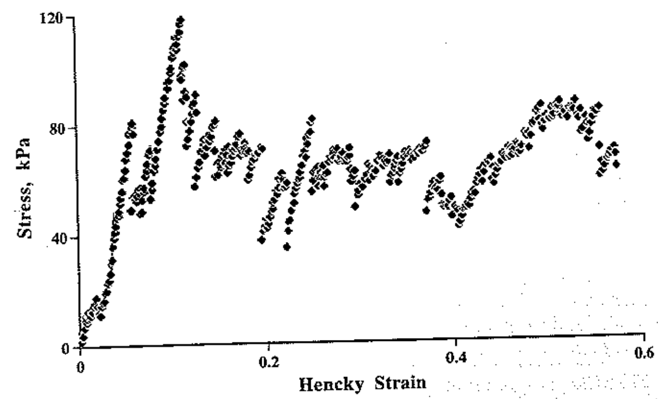

Fig. 1. Representative stress-strain curve for a brittle and fracturable corn meal extrudate.

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dified products were determined by DSC to assess moisture-induced differences in the state of the material.

MATERIALS AND METHODS

Sample Preparation

Corn meal extrudates (corn meal from Savage & Co., Burlington, MA) were extruded on a Werner and Pfliederer ZSK-30 twin-screw extruder using a solids feed rate of 27 kg/hr, a six-zone temperature profile of 38-38-116-116-138-138°C and a 4-mm diameter die. Two extrusion moisture contents, 18 and 25%, were tested yielding SME levels of 945 and 573 kJ/kg, respectively.

Equilibration

All products were initially freeze-dried. Humidified samples were equilibrated for 48 hr over a saturated solution of magnesium chloride, which yielded 33% rh (Greenspan 1976). Sample moisture contents were ≈7% (calculated by Computrac determination of initial, freeze-dried moisture content [≈1–2%] and measurement of weight gain after equilibration).

Compression

Specimens were sliced into disks 12 mm high and uniaxially compressed (circular testing fixture, 40 cm diameter) using a TX2 texture press (Texture Technologies, Scarsdale, NY) to 50% strain at a deformation rate of 0.2 mm/min and a data acquisition rate of 12.5 pts/sec. Before compression, three caliper measurements were taken of the specimen diameter and averaged. Six sample replicates were compressed.

TABLE I
Expansion Ratio and Bulk Density of Extrudates

SME[a]	Expansion Ratio[b]	Bulk Density (g/cm)
High	25.5	0.09
Low	14.1	0.18

[a] Specific mechanical energy.
[b] Extrudate cross-sectional area/die area.

TABLE II
Fracturability and Strength Parameters of Low Extrusion Moisture[a] Extrudates Under Compression

Attribute[b]	Freeze-Dried[c]	Equilibrated[d]
Number of fractures	72 (7.1)	54 (12)
Mean fracture intensity (kPa)	3.0 (22)	4.5 (11)
Cumulative fracture stress (kPa)	218 (27)	243 (13)
Exponent *b* of fitted distribution	0.32	0.25
Coefficient *C* of fitted distribution	9.3	5.2
r^2 of fit for distribution	0.79	0.67
Average compressive resistance (kPa)	21 (29)	31 (26)

[a] 945 kJ/kg of specific mechanical energy.
[b] Average of six compression replicates.
[c] Coefficient of variation ([standard deviation/mean] × 100).
[d] 7% moisture.

TABLE III
Fracturability and Strength Parameters of High-Extrusion Moisture[a] Extrudates Under Compression

Attribute[b]	Freeze-Dried[c]	Equilibrated[d]
Number of fractures	51 (9.3)	33 (14)
Mean fracture intensity (kPa)	24 (27)	31 (26)
Cumulative fracture stress (kPa)	1,230 (21)	992 (21)
Exponent *b* of fitted distribution	0.030	0.028
Coefficient *C* of fitted distribution	4.9	2.8
r^2 of fit for distribution	0.76	0.67
Average compressive resistance (kPa)	69 (32)	88 (28)

[a] 573 kJ/kg of specific mechanical energy.
[b] Average of six compression replicates.
[c] Coefficient of variation ([standard deviation/mean] × 100).
[d] 7% moisture.

Textural Data Analysis

Compression data were analyzed according to the procedure of Barrett et al (1994b), in which fractures in the samples were determined and measured by abrupt reductions in developed stress (i.e., negative differences in consecutive stress measurements) that occurred during compression. By this method, the distribution of these stress reductions, or fracture intensities, are evaluated within a preselected analysis range of 8–42% strain (middle two-thirds of compression curve).

Fracturability parameters, obtained from data averaged over all six replicates, were calculated using Minitab (State College, PA)

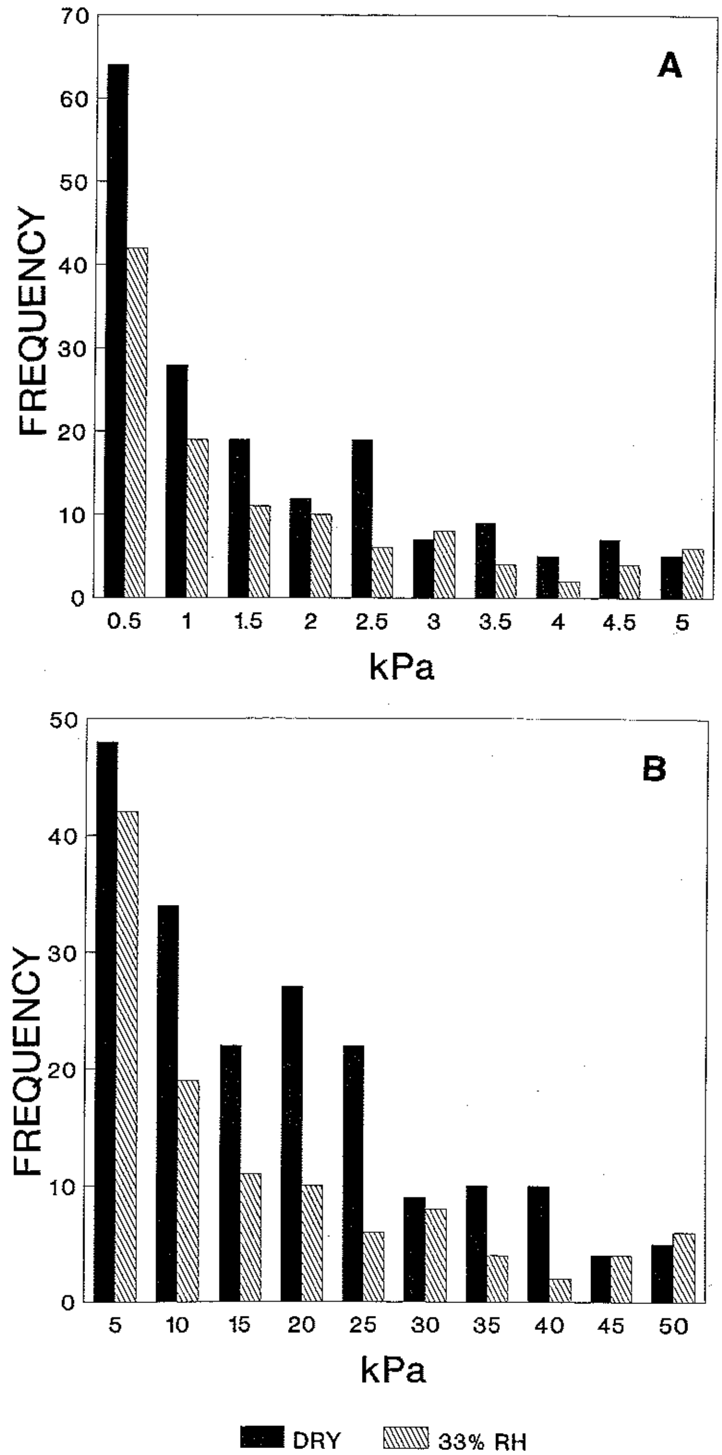

Fig. 2. Fracture intensity distributions for high specific mechanical energy (SME) extrudates (**A**) and low SME extrudates (**B**). Solid bars indicate dry sample, shaded bars indicate 33% rh equilibrated sample (7% moisture).

statistical software and included mean number of fractures occurring per compression, mean fracture intensity, cumulative fracture stress (sum of all fracture intensities per compression), mean level of stress developed during compression, and parameters from fitted distributions of fracture intensities (pooled data) using the function:

$$y = \text{frequency} = Ce^{(b\,\Delta x)}$$

where Δx refers to fracture intensity, and C and b are calculated coefficients. The exponent b indicates the relative preponderance of low-intensity versus high-intensity fractures. The coefficient C indicates the number of fractures below a minimal observable nonzero stress reduction (y-intercept). For example, a relatively larger value of b indicates a relatively larger proportion of low-intensity (and a relatively smaller proportion of high-intensity fractures) in the distribution. A relatively larger value of C indicates a relatively greater number of very low intensity fractures. Since fracture intensity distributions tend to be long-tailed, arbitrary cutoff limits of 100 kPa for the low SME sample and 10 kPa for the high SME sample were used when fitting data to the above function, analogous to the procedure employed by Barrett et al (1994b). Also, frequency data from all replicates were averaged (pooled then divided by six) before being fit to the above function.

Bulk Property Analysis

Extrudate expansion was calculated based on the average of three caliper measurements of the diameter of four different product specimens, and expressed as extrudate cross sectional area/die area. Bulk density was calculated (in quadruplicate) by measuring and weighing cylindrical sections.

DSC

DSC of the dry and moist samples was conducted using a Setaram DSC 111 (Caluira, France) computer-controlled, pressure-variable, temperature-scanning calorimeter. Sample preparation and analysis were conducted in accordance with the procedures of Barrett et al (1995).

RESULTS AND DISCUSSION

Structure and Fracture Behavior of Dry Samples

The samples varied considerably in structure (Table I), as indicated by ratios of almost 2:1 in bulk density (low to high SME sample) and radial expansion ratio (high to low SME sample). Accordingly, fracturability and strength parameters (Tables II and III) of the dry samples show a sixfold difference in mean fracture intensity and a threefold increase in average compressive resistance, with the higher values occurring in the less expanded, lower SME sample. However, the overall shape of the fracture intensity distributions for the samples are very similar (Fig. 2a,b) despite these differences in magnitude.

Parameters from the fitted fracture intensity distributions show a lower exponent b in the low SME product, indicating a relatively flatter distribution, more skewed toward higher intensity fractures as compared to that for the high SME extrudate. While the average number of fractures per compression was comparatively lower in the low SME sample (hence, a lower value for C), cumulative fracture stress was more than five times higher than that of the high SME product. The less expanded, denser extrudate was thus relatively much harder and relieved developed stress with much higher intensity fractures.

Effects of Low Levels of Moisture on Fracturability

Humidity-induced changes in fracturability followed similar patterns in both products, despite their structural differences. While cumulative fracture stress measurements indicated considerable retention of fracturability at 7% moisture content (>100 and 81% for the high and low SME extrudates, respectively) (Tables II and III), significant shifts occurred in the fracture intensity distribution parameters and in the general compression behavior of both samples.

In both instances, average compressive resistance increased with added moisture (48 and 28% for high and low SME extrudates, respectively), suggesting that the humidified samples did not fail exclusively by brittle fracture, as observed by Harris and Peleg (1996) and Barrett et al (1995). Additionally, while cumulative fracturability showed only modest changes, it is important to note that this parameter is a function of both the total number of fractures and the magnitude of individual fractures. For each sample, the total number of fractures occurring during compression declined markedly after moisture sorption, while the mean fracture intensity increased by approximately the same proportion. Correspondingly, parameters of the fitted exponential function show a slight flattening of the fracture intensity distributions after equilibration (i.e., a moderate lowering of b) which indicates a tendency toward higher intensity fractures. The decrease in the total number of fractures occurring during compression is reflected in a reduction of C. The manner in which these changes affect the form of the distributions is illustrated in Fig. 2.

These results show that alteration of texture by absorption of moderate levels of moisture is not necessarily manifested by a continuous softening of the samples, but rather by changes in the distribution of fracture intensities. While the total number of fractures occurring during compression by itself distinctly indicates textural loss, this effect must be considered in light of potential corresponding changes such as an increase in the magnitude of remaining fractures and an increase in overall compressive resistance. Such effects would most likely contribute to a perception of increased toughness or hardness during consumption of these products.

Thermal Stability of Dry Samples: Influence of SME

The T_g of the freeze-dried extrudates were 93 and 80°C for low (573 kJ/kg) and high (945 kJ/kg) SME extrusion processing conditions, respectively (Fig. 3). This is consistent with results of earlier studies on corn and wheat flour extrudates (Kaletunc and Breslauer 1993, 1996). As SME generated in the extruder increased, the extent of extrusion-induced fragmentation increased, which could be monitored by observed reductions in the T_g of the extrudates. The transition from the glassy to the rubbery state occurred over a broad temperature range, roughly 50°C for high SME samples and 70°C for low SME samples, from onset to end. The observation of a broad transition region was reported for wheat flour extrudates by Kaletunc and Breslauer (1996) and was attributed to the heterogeneous nature of preextruded material as well as to the molecular weight distribution resulting from fragmentation during extrusion. Because the raw material for both extrudates was identical, the difference in the T_g values and the broadness of the transitions can

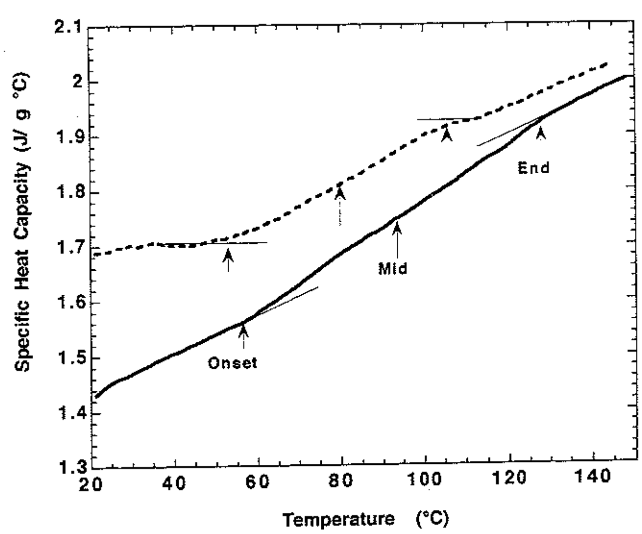

Fig. 3. Differential scanning calorimetry thermograms of dry corn extrudates for high SME (- - - -) and low SME (——) process conditions.

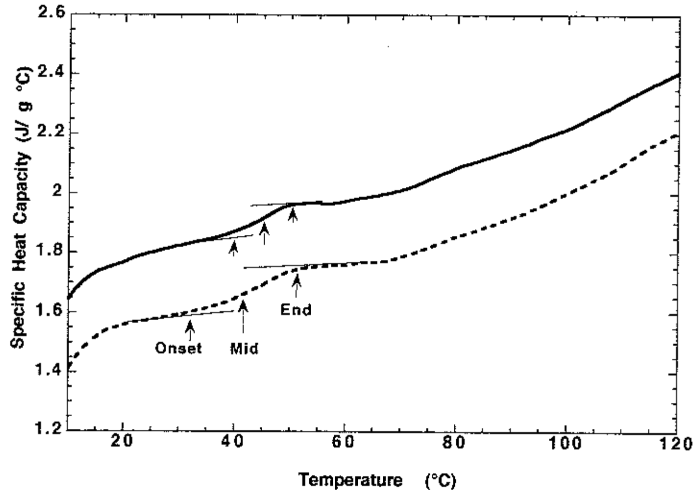

Fig. 4. Differential scanning calorimetry thermograms of corn extrudates with 7% moisture for high SME (- - - -) and low SME (——) process conditions. (Low SME scan is displaced by 0.2 J/g.)

be attributed to different extrusion processing conditions. A lower T_g for the dry, high SME extrudate indicates a lower M_w (Kaletunc and Breslauer 1996) in this sample. A relatively narrower glass transition range for the this product may indicate that increased shear further induced fragmentation of the higher molecular weight fraction, resulting in a narrower molecular weight distribution. Both the T_g and the fracturability characteristics of the dry extrudates are consistent in indicating that high and low SME conditions produced extrudates with different physical and structural properties.

Effects of Low Levels of Moisture on Thermal Stability

Plasticization of the extrudate samples by moisture was further confirmed by the DSC findings of reductions in T_g with increasing moisture content. Figure 4 shows thermograms of high and low SME moist (7% H_2O by weight) samples. The T_g of the equilibrated extrudates were 45C and 41°C for low and high SME processing conditions, respectively. The difference between the T_g values of high and low SME extrudates was less pronounced in the moist samples, demonstrating that while fragmentation defines the thermal stability of dry extrudates, plasticization by moisture becomes the dominant factor determining thermal stability of extrudates containing low levels of water. Increased mobility due to the presence of moisture far outweighs that due to any reduction in the size of starch polymers. Correspondingly, differences between most fracturability parameters of the two samples (mean fracture intensity, cumulative fracture stress, average compressive resistance), while still pronounced, were proportionally smaller after equilibration.

It should be noted that this dominance of moisture content was observable in environments as low as 33% rh. At this level, it is expected that, upon equilibration, the extrudates have a moisture content that is at the low end of that for intermediate moisture foods (Bourne 1987).

CONCLUSIONS

It is significant that changes in fracturability were pronounced, even though the measured T_g of both dry and 7% moisture samples were above the ambient temperature at which the mechanical tests were conducted. Nicholls et al (1995) demonstrated that starches can undergo brittle-ductile transitions that allow relatively greater deformation and stored energy (which is subsequently released in relatively higher intensity fractures) while still in the glassy state. It is also possible that toughening of the samples indicates some constituent association or structural development. In recently published work concerning storage of baked products, Ruan et al (1996) postulated that protein interactions (in addition to staling) contribute to firming. Our observations of toughening of other intermediate-moisture, flour-based extrudates maintained under high-temperature storage (Karydas and Wang 1995) also suggest the development of structures involving nonstarch components. Further investigation concerning physical phenomena underlying textural stability and potential complex formation in these products, which have water contents corresponding to the transition from low to intermediate moisture, is warranted.

LITERATURE CITED

Barrett, A. H., Normand, M., Peleg, M., and Ross, E. W. 1992. Characterization of the jagged stress-strain relationships of puffed extrudates using the fast Fourier transform and fractal analysis. J. Food Sci. 57:227-232, 235.

Barrett, A. H., Cardello, A. V., Lesher, L. L., and Taub, I. A. 1994a. Cellularity, mechanical failure, and textural perception of corn meal extrudates. J. Texture Stud. 25:77-95.

Barrett, A. H., Rosenberg, S., and Ross, E. W. 1994b. Fracture intensity distributions during compression of puffed corn meal extrudates: Method for quantifying fracturability. J. Food Sci. 59:617-620.

Barrett, A. H., Kaletunc, G., Rosenberg, S., and Breslauer, K. 1995. Effect of sucrose on the structure, mechanical strength and thermal properties of corn extrudates. Carbohydr. Polym. 26:261-269.

Bourne, M. 1987. Effects of water activity on textural properties of food. Pages 75-99 in: Water activity: Theory and Applications to Food. L. B. Rockland and L. R. Beuchat, eds. Marcel Dekker: New York.

Greenspan, L. 1976. Humidity fixed points of binary saturated aqueous solutions. J. Res. Nat'l Bur. Stand. 81A:89-94.

Harris, M., and Peleg, M. 1996. Patterns of textural changes in brittle cellular foods caused by moisture sorption. Cereal Chem. 73:225-231.

Kaletunc, G., and Breslauer, K. 1993. Glass transitions of extrudates: Relationship with processing-induced fragmentation and end-product attributes. Cereal Chem. 70:548-552.

Kaletunc, G., and Breslauer, K. 1996. Construction of a wheat-flour state diagram: Application to extrusion processing. J. Thermal Anal. 47:1267-1288.

Karydas, D., and Wang, C., 1995. Effect of screw configuration on quality of intermediate moisture extrudates. Final Report of the MIT David H. Koch School of Chemical Engineering Practice, Natick Station, Project MIT-NL-16. MIT: Cambridge, MA.

Katz, E. E., and Labuza, T. P. 1981. Effect of water activity on the sensory crispness and mechanical deformation of snack food products. J. Food Sci. 46:403-409.

Nicholls, R. J., Appelquist, I. A. M., Davies, A. P., Ingman, S. J., and Lillford, P. J. 1995. Glass transitions and the fracture behaviour of gluten and starches within the glassy state. J. Cereal Sci. 21:25-36.

Peleg, M., and Normand, M. D. 1993. Determination of the fractal dimension of the irregular, compressive stress-strain relationships of brittle, crumbly particulates. Part. Part. Syst. Charact. 10:301-307.

Rhode, F., Normand, M. D., and Peleg, M. 1993. Effect of equilibrium relative humidity on the mechanical signatures of brittle food materials. Biotechnol. Prog. 9:497.

Ruan, R., Almaer, S., Huang, V. T., Perkins, P., Chen, P., and Fulcher, R. G. 1996. Relationship between firming and water mobility in starch-based food systems during storage. Cereal Chem. 73:328-332.

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