Frequency Dependence of Viscoelastic Properties of Bread Crumb and Relation to Bread Staling[1]

F. C. Wang[2] and X. S. Sun[2,3]

ABSTRACT

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The viscoelastic behavior of bread crumb was studied using dynamic mechanical analysis (DMA) in the compression mode with the frequency sweep. The dynamic storage modulus (E′), loss modulus (E″), and tanδ (E″/E′) were measured for bread crumb aged up to three days at ambient temperature. The viscoelastic properties of bread crumb showed a characteristic frequency dependence similar to that of a soft rubberlike solid. Typical behavior of bread crumb involved a transition from rubberlike to glasslike consistency with increasing frequency. At a low frequency region, the E′ and E″ values were relatively small and nearly constant, showing characteristics of the rubbery plateau. Then, they increased rapidly with increasing frequencies and approached a glasslike state. Tanδ was low and almost constant at low frequencies before the transition, then went through a prominent peak with increasing frequency. The frequency at which the tanδ of bread crumb started to rapidly increase was defined as the onset frequency (f_o) of the transition. The f_o values increased with the aging of bread crumb samples, which correlated highly to bread staling (r = 0.942). Both dynamic moduli E′ and E″at f_o also increased with the aging of bread, which correlated highly to firmness obtained using a texture analyzer in a static compression mode (r = 0.941 and 0.943, respectively). DMA measurements could be helpful in characterizing bread staling.

Bread texture is an important quality indicator to both consumers and producers. From a rheological point of view, the change in hardening or firming of a bread crumb often is accompanied by loss of resilience during staling (MacRitchie 1980). Therefore, firmness often is used as a measure of bread staling, which has been determined successfully by using instruments such as a Baker Compressimeter, an Instron universal testing machine, or a texture analyzer (Ponte and Ovadia 1997) in a static compression mode. A standard method (AACC 2000) for bread staling based on force-deformation for firmness in the static compression mode has been available. However, these traditional instruments do not provide fundamental rheological data and the results are limited to empirical correlations. Bread crumb is a complicated viscoelastic foam material, the applied force is generally not related linearly to the corresponding deformation, and the results also depend on the rates of deformation (Hibberd and Parker 1985). Thus, assessing and comparing the results from many of the published reports is often difficult when the specific details of the experimental instruments and procedures differ.

Dynamic mechanical analysis (DMA) has been used as a tool to determine dynamic rheological properties of polymeric materials (Murayama 1978) including cellular polymeric foams (Meinecks and Clark 1973; Hilyard 1985). Unlike static force-deformation, dynamic measurements simultaneously record viscous and elastic responses of a specimen and provide fundamental rheological data. DMA recently has been used for textural characterization of foods (Matthiesen and Ibar 1991; Hallberg and Chinachoti 1992; Vodovotz et al 1996; Campanella and Peleg 1997; Weipert 1997). Bread crumb is a porous solid matrix with cellular structure (Taranto 1983) composed mainly of gluten, starch, and water, representing a typical viscoelastic biopolymer foam system. Persaud et al (1990a,b) initiated the use of a dynamic rheometer to determine bread staling in a simple shear mode at a fixed frequency of 5 Hz. They found that the shear elastic modulus increased with bread staling, and the changes in the storage modulus with aging were temperature-dependent. Unfortunately, the shear dynamic rheometer was not sensitive enough to detect significant effects of the single surfactant at a relatively low level (Persaud et al 1990b) on the modulus of bread crumb, which could be observed by using a traditional static instrument (Ponte and Ovadia 1997). This might be because the dynamic measurements were performed in a shear oscillatory deformation mode. As pointed out by Weipert (1997), the shear principle rheometer was less suitable for measuring bread crumb, a relatively solid specimen, because of a fixed gap between the parallel plates, whereas compressive oscillatory DMA should be advantageous for those measurements. More recently, Ovadia and Walker (1996) used a similar dynamic rheometer in compression mode at the same frequency with a nondestructive option to reexamine the firming curve of aged bread and confirmed that the storage modulus could be used to predict bread staling. Selection of the oscillatory frequency also is particularly important in dynamic tests because the viscoelastic properties of different foods or different states of a food may have different frequency dependencies (Campanella and Peleg 1997). Therefore, the objectives of the present study were to characterize the dynamic viscoelastic properties of bread crumb with DMA in the compression mode and investigate the viscoelastic behavior during bread staling as related to dynamic frequency.

MATERIALS AND METHODS

Bread Preparation

Commercial bread flour obtained from Ross Industries (Cargill Inc., Wichita, KS) was used for making breads. It contained 10.45% protein and 0.52% ash on a 14% moisture basis.

A 100-g optimized straight-dough breadmaking procedure (Approved Method 10-10B, AACC 2000) was followed. A 100-g mixer (Swanson-Working pin-type; National Manufacturing Co., Lincoln, NE) was used to mix the bread dough to optimum. Fermentation time was 90 min, proof time was 33 min at 30°C and 85% rh, and baking time was 24 min at 215°C.

In this experiment, variations in bread staling were created using seven bread formulas (Table I). Formula 1, used as control, was a standard dough formula with optimum water absorption (64%) based on the AACC method 10-10B (AACC 2000). Formulas 2 and 3 reduced the level of water absorption to 56 and 60%, respectively; formulas 4 and 5 reduced the level of shortening to 0 and 1.5%, respectively; and formulas 6 and 7 had additions of 0.5 and 1.0%, respectively, of sodium stearoyl-2-lactylate (SSL) (Emplex, American Ingredients Co., Kansas City, MO).

Sample Preparation for DMA Measurements

All freshly baked loaves were cooled for 2 hr at room temperature (≈25°C). Then they were sliced cross-sectionally into slices 12.5 mm thick (Oliver Machinery Co., Grand Rapids, MI)

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[1] Contribution 00-461-J from the Kansas Agric. Exp. Stn., Manhattan, KS 66506.

[2] Dept. Grain Science and Industry, Kansas State University, Manhattan KS 66506.

[3] Corresponding author. E-mail: xss@wheat.ksu.edu Phone: 785-532-4077. Fax: 785-532-7010.

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and placed in double-sealed plastic bags. Cylindrical crumb samples (15 mm diameter) were cut from the centers of the three centermost slices of each bread loaf using a circular cutter and were stored at original positions in double-sealed plastic bags at ambient temperature (≈25°C) for DMA measurements.

Density Determination

The bread loaves were allowed to cool for 2 hr, weighed, and then measured for volume by rapeseed displacement. The apparent density of bread loaf (after 2 hr of cooling, as day 0) was calculated by dividing the weight by the volume of a bread loaf.

The crumb density of the cylindrical crumb specimen was determined by measuring its mass and volume. The mass of the crumb sample was measured before initial DMA measurement (on day 0). The volume of the cylindrical crumb sample was the product of the area of the cross section and the thickness of the sample.

Moisture Content Determination

The apparent moisture content (MC) of bread loaf was determined by the equation: $MC = ([W_b - W_d]/W_b) \times 100\%$, where W_d is the total dry matter weight of flour and other ingredients, and W_b is the weight of bread loaf corresponding to days 0, 1, and 3 of storage. For example, the moisture content of the fresh bread loaf (on day 0) made with control formula could be calculated as: W_d = 86 g of flour + 1.5 g of salt + 6 g of sugar + 4 g of nonfat dry milk + 3 g of shortening = 100.5 g, W_b = 147.65 g (after 2 hr of cooling on day 0), and, thus, MC = 31.93%.

The moisture content of the crumb sample from day 3 was determined by Approved Method for two-stage bread procedure (AACC 2000) immediately after DMA measurement on day 3. The moisture content of the crumb sample from day 1 was calculated from the sample mass weighed on day 1 and the dry matter weight on day 3. A similar method was used for calculating the moisture content of the fresh crumb sample (day 0).

DMA Measurement

Dynamic mechanical analysis using a DMA 7e (Perkin-Elmer, Norwalk, CT) was used to measure dynamic viscoelastic properties of the bread crumb samples. A 10-mm parallel plate and disk measurement system was installed. The cylindrical sample was placed between the parallel plate and disk, and then a static force of 20 mN and a dynamic force (sinusoidal) of 16 mN were applied in the compression mode. Both static and dynamic forces used in this experiment were sufficiently small and, thus, in the relatively linear viscoelastic region, which was preliminarily determined (Fig. 1). The configuration of the DMA 7e required that the static force be ≈10 to 20% greater than the dynamic force to keep the probe in continuous contact with the sample during the entire analysis. A frequency scan was performed and the dynamic storage modulus (E′), loss modulus (E″), and tanδ (E″/E′) were calculated and plotted as functions of frequency (Pyris Software for Windows, Perkin-Elmer).

Firmness Measurement

The firmness of the bread samples was determined using a texture analyzer (TA-XT2, Texture Technologies Corp., Scarsdale, NY) with a cylindrical probe (1 in. diameter) following Approved Method 74-09 (AACC 2000). On the day of baking (day 0) and day 1 and day 3 after baking, three center slices of each loaf were measured at room temperature (≈25°C). The rate of compression was set at 1.7 mm/sec. Firmness was the force (N) required to compress the slices (12.5 mm thick) by 25% strain.

Statistical Analyses

A completely randomized experimental design was used to compare the measurement responses of seven formulation treatments (changes in level of water, shortening, and sodium stearoyl-2-lactylate [SSL]) in duplicate at three aging times (days 0, 1, and 3). The three centermost slices taken from each bread loaf were subsamples. The Statistical Analysis System (SAS Institute Inc., Cary, NC) was used for analysis of variance and correlation of data at $P < 0.05$.

RESULTS AND DISCUSSION

Moisture Content

Moisture contents of bread loaves and crumbs with various formulas during storage are presented in Table II. Moisture content increased as water level increased. The bread loaf and crumb with-

TABLE I
Bread Dough Formulas (Baker's %)

Ingredient	1 (Control)	2 (56% water)	3 (60% water)	4 (0% shortening)	5 (1.5% shortening)	6 (0.5% SSL)[a]	7 (1% SSL)
Flour[b]	100	100	100	100	100	100	100
Water	64	56	60	64	64	64	64
Yeast (Instant dry)	2	2	2	2	2	2	2
Salt (sodium chloride)	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Sugar (sucrose)	6	6	6	6	6	6	6
Nonfat dry milk	4	4	4	4	4	4	4
Shortening	3	3	3	0	1.5	3	3
SSL	0	0	0	0	0	0.5	1.0

[a] SSL = sodium stearoyl-2-lactylate.

[b] 14% moisture basis.

TABLE II
Moisture Content (%) of Bread Loaves and Crumb Samples Made with Various Formulas at Different Aging Times[a]

	Loaf			Crumb		
Formula[b]	Day 0	Day 1	Day 3	Day 0	Day 1	Day 3
0% Shortening	34.7a	34.66a	34.58a	47.71a	47.49a	46.67a
1.5% Shortening	32.51b	32.48b	32.41b	45.03b	44.84b	44.13b
56% Water	29.84e	29.81e	29.73e	41.33d	41.13d	40.31d
60% Water	30.69d	30.65d	30.58d	42.50c	42.31c	41.49c
Control	31.93bc	31.89bc	31.83bc	44.55b	44.35b	43.55b
0.5% SSL	31.8bc	31.76bc	31.68bc	43.19c	42.98c	42.17c
1.0% SSL	31.6c	31.55c	31.47c	42.82c	42.61c	41.79c
Mean[c]	31.87y	31.83y	31.76y	43.88z	43.68z	42.87z

[a] Values represent mean of two replicates; mean comparisons within a column followed by the same letter are not significantly different ($P > 0.05$) among formulas.

[b] SSL = sodium stearoyl-2-lactylate.

[c] Values represent the average of the seven means from different formulas; value comparisons within the row followed by the same letter are not significantly different ($P > 0.05$).

out shortening had the highest moisture contents. The bread loaves with SSL had moisture content similar to that of the control, but the crumbs with SSL had a slightly lower moisture content than that of the control. The crumb samples had a higher moisture content than the loaves because they were from the center of the loaf.

Moisture contents of both bread loaves and crumb samples decreased as storage time increased, but such a decrease was not statistically significant in this experiment (Table II). In addition, ≈1% moisture loss (after day 3 of storage) had no significant effects on the bread staling measurement (Baik and Chinachoti 2000). Therefore, the effect of moisture loss on crumb staling was negligible in this study.

Frequency Dependence of Viscoelastic Properties

Typical results of the frequency dependence of dynamic viscoelastic properties of fresh (day 0) and aged (days 1 and 3) crumb samples of bread made with a control formula are shown in Fig. 2. At low frequencies, the E′ and E″ values were relatively small and nearly independent of the oscillatory frequency, presenting a rubbery state or plateau. Then, they rapidly increased by $\approx 10^{2.5}$ to a glasslike state at the frequency beyond ≈35 Hz. A maximum in E″ appeared at the rather high frequency. In the low-frequency region, tanδ was small and almost constant at ≈0.5; then it rapidly rose to a peak and sharply dropped.

Bread can be considered as a continuous biopolymer network of amorphous gluten filled with partially crystalline starch granules (LeMeste et al 1992). The viscoelastic behavior of the bread crumb was grossly similar to that of a lightly cross-linked rubbery material (Ferry 1980; Nielsen and Landel 1994; Davidou et al 1996).

The rapid increase in moduli and the peak in tanδ could be considered a transition from rubberlike to glasslike consistency (Ferry 1980; Nielsen and Landel 1994). The term transition was used to describe the frequency dependence of viscoelastic properties distinct from the glass transition as a function of temperature. As frequency increased, the measurements of moduli and tanδ changed from rubberlike to glasslike, but there was no change in the thermodynamic state of the material.

The transmission of stress within the bread crumb at higher frequencies may have an effect similar to that of an increase in cross-linkage of bread crumb material because of an increase in vibration on filled particles (Murayama 1978). Modulus increases with increasing dynamic frequency (strain rate) because there is less time for stress relaxation during the compression.

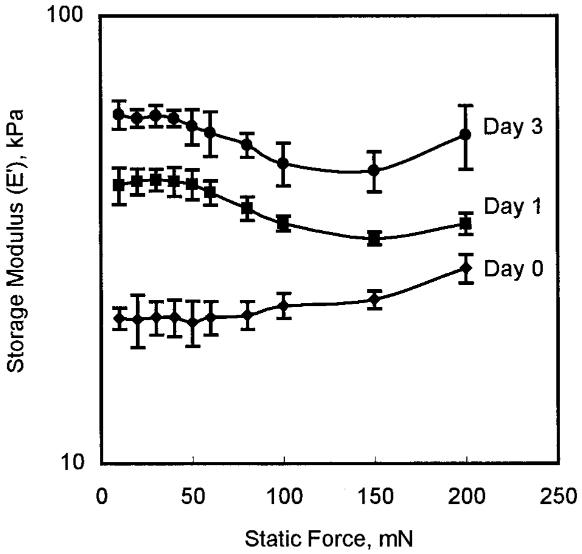

Fig. 1. Force (dynamic force = 80% static force) dependence of dynamic storage modulus (E′) at a frequency of 4 Hz and room temperature with parallel plate disk in compression mode for fresh (day 0) and aged (days 1, 3) crumb samples of bread made with a control formula.

Tanδ is a measure of mechanical damping because it is proportional to the frictional energy dissipated per deformation cycle (Murayama 1978). The angle δ reflects the time lag between the applied stress and the strain. Therefore, the transition could also be explained by the motion theory of polymer molecules because

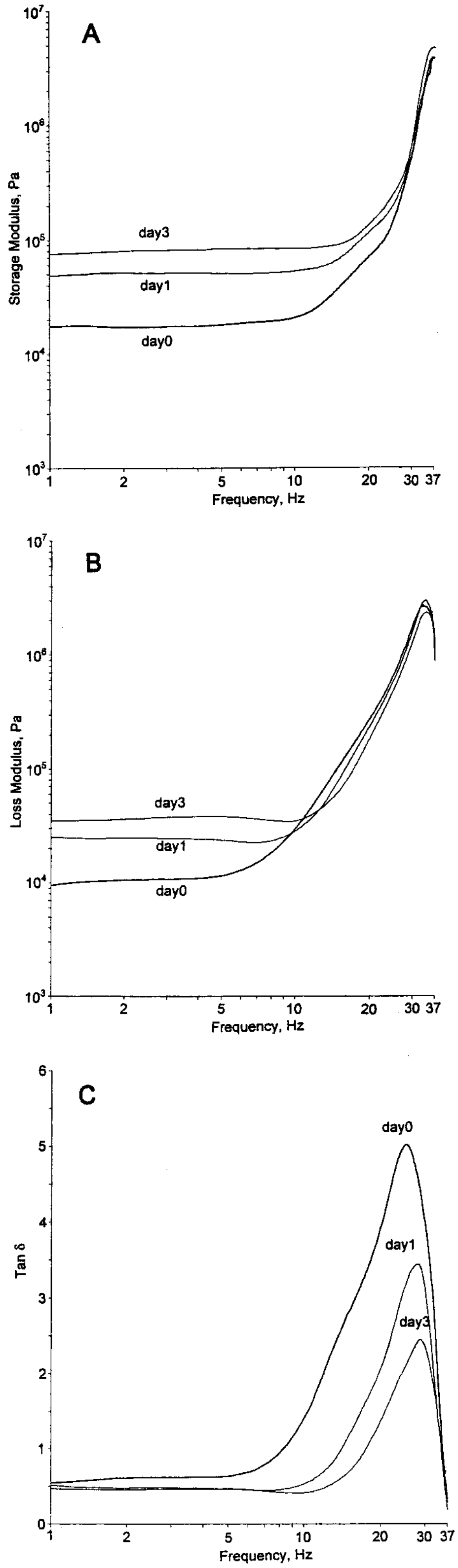

Fig. 2. Typical dynamic mechanical analyzer curves in the wide frequency sweep for fresh (day 0) and aged (days 1 and 3) crumb samples of bread made with a control formula measured at room temperature with 20 mN static force, 16 mN dynamic force, and parallel plate disk. **A,** storage modulus (E′); **B,** loss modulus (E″); and **C,** tanδ (E″/E′).

the phase lag phenomenon results from the internal friction caused by the motion of molecular chain segments. At low oscillatory frequencies, the time was sufficient for molecular rearrangements and relaxation and, hence, the motions of the chain segments could follow the dynamic force applied, resulting in a small phase lag. However, the phase lag increased greatly at a higher frequency because the sinusoidally oscillating force varied so fast that the motion of the molecular chain segments was no longer able to follow. In this case, the damping was high owing to the stronger friction from the motion in some molecular chain segments, which dissipated more heat energy.

Effects of Aging and Formulas on Viscoelastic Properties of Bread Crumb

The E′ and E″, in the low frequency region, increased as bread storage time increased (Fig. 2). The tanδ peak became smaller and its location shifted to a higher frequency with aging of bread. The DMA curves show the transition from rubberlike to glasslike consistency with a frequency scanning range from 1 to 37 Hz (Fig. 2). The wide frequency range, however, was not used in the main experiments because of a long scanning time that caused great moisture loss in crumb samples. To reduce scanning time and minimize moisture loss, preliminary experiments were conducted to determine the effective frequency range. The effective frequency range in this study was determined to observe the onset of the transition. The effective frequency ranges were 3–7, 4–8, and 5–10 Hz for bread crumb samples from days 0, 1, and 3, respectively. The E′, E″, and tanδ curves obtained in the effective frequency ranges for fresh (day 0) and aged crumb samples (days 1 and 3) of bread made with the control formula are shown in Fig. 3. For practical purposes, the onset frequency of the transition (f_o), at which tanδ started to rapidly increase, would be an important and useful parameter to locate the characteristic transition. The f_o was designated as the onset frequency at the intersection point of two tangential lines of the logarithmic tanδ frequency curve (Pyris Software for Windows, Perkin-Elmer).

The mean values of f_o and E′ and E″ at f_o of seven bread formulas are summarized in Table III. As the bread aged, the mean E′, E″, and f_o values of crumb significantly increased, but the increases in E′, E″, and f_o were much greater from day 0 to day 1 than those from day 1 to day 3 (Table IV). The bread with 0 or 1.5% shortening had the largest increase from day 0 to day 1, and the bread with 1% SSL had the smallest increase from day 0 to day 1. The increases from day 1 to day 3 were almost parallel for all formulas.

For shortening effect, all E′, E″, and f_o values significantly increased as the shortening level was reduced from 3 to 0% (Table III). The increase from day 0 to day 1 was not significantly different between 0 and 1.5% of shortening (Table IV), but was greater than that of the bread with 3% shortening (control). Similar phenomena were observed for water levels of 56–64%.

For SSL effects, the E′, E″, and f_o values on day 0 were higher for the breads with SSL than that of the control bread without SSL (Table III). After day 3 of storage however, the E′, E″, and f_o values of the bread with 1% SSL became significantly lower than those of the control bread without SSL. The increasing rates of E′, E″, and f_o during the first day (Table IV) of the bread with 1% SSL were significantly slower than those of the control bread.

TABLE III
Mean and Statistical Significance by Analysis of Variance for Dynamic Mechanical Analysis (DMA) Measurements on Fresh (Day 0) and Aged (Days 1 and 3) Crumb Samples of Breads Made with Various Formulas[a]

	E′ (kPa)			E″ (kPa)			f_o (Hz)		
Formula[b]	Day 0	Day 1	Day 3	Day 0	Day 1	Day 3	Day 0	Day 1	Day 3
0% Shortening	57.74a	90.14a	116.29a	27.66a	35.64a	41.15a	7.89a	10.46a	11.91a
1.5% Shortening	20.35b	46.68b	69.65b	11.79b	22.58b	27.77b	5.11b	7.68b	9.23b
56% Water	19.15b	39.71b	56.62c	11.70b	21.80b	25.99bc	5.17b	6.92bc	8.66bc
60% Water	12.56c	29.13c	51.48c	7.85c	16.82c	24.62c	4.37cd	6.21cd	8.15cd
Control	8.62d	18.06d	39.90d	5.93d	11.71d	19.84d	3.93d	5.31d	7.49bde
0.5% SSL	12.36c	20.02d	36.12d	7.52c	11.83d	17.97d	4.83bc	5.56d	7.15ef
1.0% SSL	12.41c	15.93d	26.25e	8.05c	10.66d	14.67e	5.00bc	5.37d	6.57f
Mean[c]	20.46x	37.09y	56.61z	11.50x	18.72y	24.57z	5.19x	6.79y	8.45z

[a] Mean values represent mean of two measurement replicates; mean comparisons within a column followed by the same letter are not significantly different ($P > 0.05$) among bread crumbs of varying formulas. E′ = storage modulus at onset frequency of tanδ peak (f_o) and E″ = loss modulus at f_o.

[b] SSL = sodium stearoyl-2-lactylate.

[c] Values represent the average of the seven means from different formulas; value comparisons within the row followed by the same letter within a row are not significantly different ($P > 0.05$) for each specific measurement parameter.

TABLE IV
Mean and Statistical Significance by Analysis of Variance for Increasing Rates of Dynamic Mechanical Analysis (DMA) Measurements on Crumb Samples of Breads Made with Various Formulas to Day 3 of Aging[a]

	Increase in E′ (kPa/day)			Increase in E″ (kPa/day)			Increase in f_o (Hz/day)		
Formula[b]	Day (0–1)	Day (1–3)	Mean[b]	Day (0–1)	Day (1–3)	Mean[b]	Day (0–1)	Day (1–3)	Mean[c]
0% Shortening	32.41a	13.07a	22.74a	7.97ab	2.76bc	5.36ab	2.57a	0.72a	1.64a
1.5% Shortening	26.32ab	11.49a	18.91ab	10.79a	2.60bc	6.69a	2.57a	0.77a	1.67a
56% Water	20.56bc	8.46a	14.51bc	10.11a	2.10bc	6.1a	1.74ab	0.87a	1.31a
60% Water	16.58cd	11.18a	13.88bc	8.98ab	3.91a	6.44a	1.84ab	0.97a	1.40a
Control	9.44d	10.93a	10.18cd	5.79bc	4.07a	4.93ab	1.38bc	1.09a	1.23a
0.5% SSL	7.66de	8.05a	7.85cd	4.31cd	3.08ab	3.69ab	0.74d	0.79a	0.76b
1.0% SSL	3.52e	5.16a	4.34d	2.62d	2.01c	2.31b	0.37d	0.60a	0.48b
Mean[d]	16.64x	9.76y	…	7.22x	2.93y	…	1.60x	0.83y	…

[a] DMA = dynamic mechanical analysis; mean values represent mean of two replicates; mean comparisons within a column followed by the same letter are not significantly different ($P > 0.05$) among bread crumbs of varying formulas; E′ = storage modulus at onset frequency of tanδ peak (f_o) and E″ = loss modulus at f_o.

[b] SSL = sodium stearoyl-2-lactylate.

[c] Values represent average of two increasing rates (day 0–1 and day 1–3), average increasing rate during three days of aging; value comparisons within a column followed by the same letter is not significantly different ($P > 0.05$) among bread crumbs of varying formulas.

[d] Values represent the average of the seven means from different formulas; value comparison within the row followed by the same letter is not significantly different ($P > 0.05$) for each specific parameter.

TABLE V
Density of Fresh Bread Loaves and Crumb Samples (Day 0) Made with Various Formulas[a]

Formula[b]	Loaf			Crumb
	Volume (cm^3)	Specific Volume (cm^3/g)	Density (g/cm^3)	Density (g/cm^3)
0% Shortening	565a	3.78a	0.2643a	0.2854a
1.5% Shortening	770b	5.25b	0.1906b	0.2001b
56% Water	773b	5.39bc	0.1854bc	0.1928b
60% Water	800c	5.52c	0.1813cd	0.1831c
Control	825d	5.59cd	0.1790cd	0.1753c
0.5% SSL	850e	5.74de	0.1742de	0.1655d
1.0% SSL	878f	5.91e	0.1691e	0.1573e

[a] Values represent mean of two replicates; mean comparisons within a column followed by the same letter are not significantly different ($P > 0.05$).

[b] SSL = sodium stearoyl-2-lactylate.

The results reflected the effects of changes in formulation ingredients such as shortening, water, and SSL on the dynamic properties of bread crumb during staling. As expected, SSL was a good antistaling reagent (Ponte and Ovadia 1997) that could delay starch retrogradation or recrystallization and improve moisture retention of the heat-coagulated gluten (Davidou et al 1996). Shortening might be a kind of lubricant, reducing the friction between molecules; however, no direct reason has been suggested. Water as a plasticizer in the crumb would result in less resistance to deformation.

In polymer systems in the rubbery region, increasing E′ can be used as an indicator of the development of, or an increase in, crystallization and cross-linking (Murayama 1978, Ferry 1980, Nielsen and Landel 1994). In partially crystalline polymers, crystals may act as cross-links by tying segments of many molecules together (Nielsen and Landel 1994). Bread, as a food polymer system, is composed mainly of an amorphous gluten network filled with partially crystalline starch granules (LeMeste et al 1992). During bread aging, retrogradation or recrystallization of starch (Schoch and French 1947, Schoch 1965, Krog et al 1989, Zobel and Kulp 1996) and cross-linking between gluten and starch (Martin and Hoseney 1991, Martin et al 1991, Davidou et al 1996, Ovadia and Walker 1996) would occur, restricting molecular motion, and water might migrate from the gluten to the starch fraction of the crumb, causing gluten network firming (Breaden and Willhoft 1971; Cross et al 1971; Kay and Willhoft 1972; Willhoft 1971a,b, 1973). All these could contribute to the increase in E′ with negligible moisture loss. The increase in E″ might be explained mainly by the behavior of the noncrystalline part of the crumb such as the entanglement and plasticization of the amorphous polymeric network. The tanδ also is sensitive to these changes in bread staling (Murayama 1978, Ferry 1980, Nielsen and Landel 1994). Therefore, to avoid moisture loss caused by temperature scan, tanδ frequency transition can be used for characterizing bread staling, which can be measured at an ambient temperature (Ferry 1980, Nielsen and Landel 1994).

Crumb Density and Its Relationship to DMA Measurements

The volume, specific volume, density of bread loaves, and crumb density for fresh breads made with various formulas are shown in Table V. Shortening, water, and SSL had a significant effect on crumb density through increasing loaf volume or specific volume.

Bread crumb has a porous structure with mainly open cells. Bread loaves with equivalent weight but different volume generally imply that they have differences in crumb cell sizes and cell wall thickness. As expected, crumb density had significant influence on crumb rheology and firming.

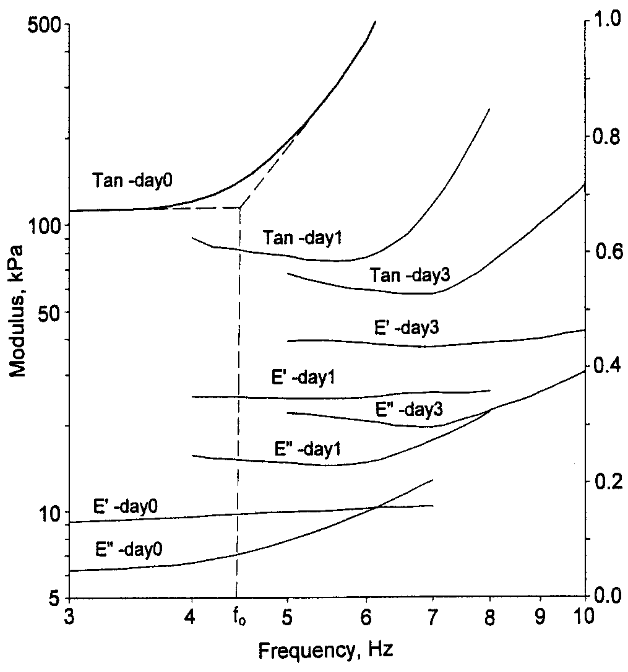

Fig. 3. Dynamic mechanical analyzer measurements of crumb samples of bread made with the control formula as functions of frequency at room temperature with 20 mN static force, 16 mN dynamic force, and parallel plate disk to day 3 of storage. Storage modulus (E′), loss modulus (E″), and tanδ (E″/E′).

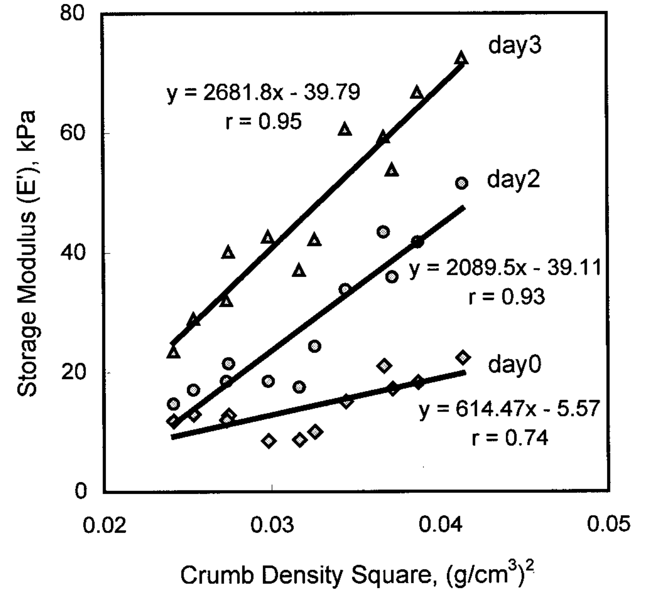

Fig. 4. Relationship between bread crumb density and dynamic mechanical analyzer storage modulus (E′) at onset frequency for fresh (day 0) and aged (days 1 and 3) crumb samples of breads made with six different formulas to day 3 of aging.

With an assumption of constant density for the same crumb sample to day 3 of storage, the storage modulus E′ of fresh and aged (days 1 and 3) crumb samples are plotted against crumb density squared (ρ^2) (Fig. 4). The crumb density for the bread without shortening was much larger than crumb densities for the rest of breads (Table V), and it was not included in the correlation to avoid masking the correlation between E′ and ρ^2 of the rest of breads. The results showed positively linear relationships between E′ and ρ^2 for both fresh and aged breads. The relationship between DMA storage modulus and crumb density squared roughly fits the model developed by Gibson and Ashby (1997), expressing the behavior of cellular polymer solids with open cells in linear elastic compression: $E' \propto (E'_s/\rho_s^2)\rho^2$ where E′ is the Young's modulus of polymer foam and E'_s is that of the material from which the cellular solid is made, and ρ and ρ_s are the density of foam and cell-

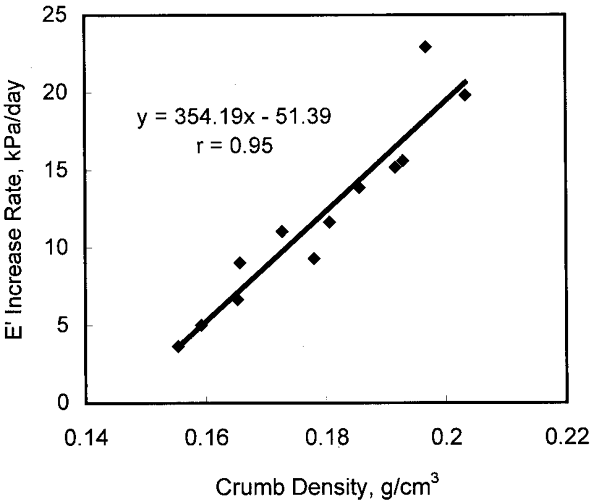

Fig. 5. Relationship between bread crumb density and average increase per day of dynamic mechanical analyzer storage modulus (E') at onset frequency for the crumb samples of breads made with six different formulas to day 3 of aging.

wall material, respectively. In this study, the E' is the DMA storage modulus of bread crumb, E'_s is the storage modulus of the cell wall material, and ρ and ρ_s are the density of bread crumb and cell wall material, respectively.

As mentioned above, an increase in water, shortening, or SSL led to a decrease in E', which was caused indirectly through the effect on crumb density. However, the slopes (E'_s/ρ_s^2) of the linear regression lines in Fig. 4 increased as bread aged. If the density (ρ_s) of solid material of the bread cell wall was assumed to be the same for all formulas and to be constant during aging, the storage modulus of the crumb cell wall material (E'_s) increased with aging. These results indicate that both density and material staling of crumb are major contributors to DMA measurements. The average increase per day of E' to day 3 of measurements is plotted against fresh crumb density for all breads except for the bread without shortening (Fig. 5). A positive linear correlation with an r value of 0.95 showed that the bread with high density tended to stale faster, and vice versa. Therefore, an increase of water, shortening, or SSL in the formula not only softened the bread through decreasing crumb density or increasing loaf volume, but also slowed the rate of firming by other physical and chemical mechanisms in antistaling, as previously discussed.

Correlation Between Dynamic and Static Measurements

Changes in viscoelastic properties (E', E'', and f_o) for the seven formula breads with aging were similar to the firming curves obtained by static compression testing using the texture analyzer. The linear regressions between static (in large deformation) firmness and dynamic (in small dynamic deformation) viscoelastic parameters (in fundamental units) of breads during staling are shown in Fig. 6. The E', E'', and f_o values all were correlated highly to the static firmness measurements (r = 0.941, 0.943, and 0.942, respectively). This indicates that the dynamic viscoelastic parameters (E', E'', and f_o) would have significant importance in studying bread staling.

CONCLUSIONS

DMA in the compression mode was effective in detecting rheological changes in bread related to staling and formula. The bread crumb behaved similarly to a soft rubberlike solid in the frequency sweep. Typical viscoelastic behaviors of bread crumb involved a transition from rubberlike to glasslike consistency with increasing frequency. The rubbery or plateau moduli (E' and E'') and onset frequency (f_o) of tanδ peak increased as bread aged in a manner similar to the firming curves. Correlations between the dynamic (DMA) and static (texture analysis) methods were r = 0.941, 0.943, and 0.942 for firmness force versus E', E'', and f_o, respectively.

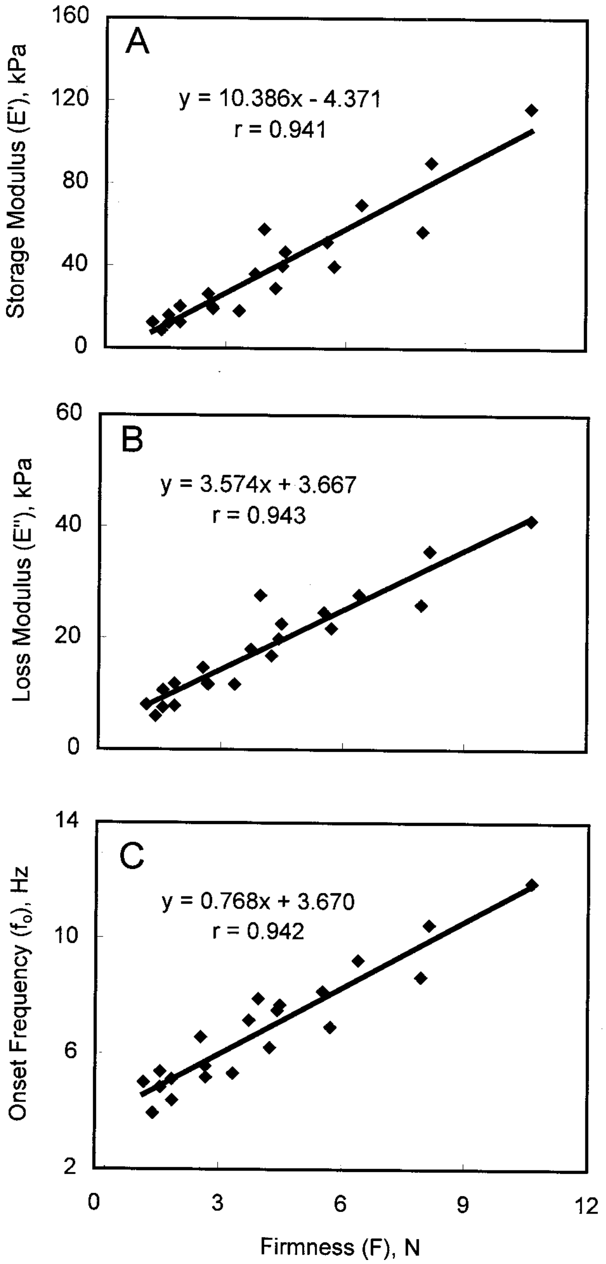

Fig. 6. Correlation between static firmness (F) obtained with a texture analyzer and dynamic mechanical analyzer measurements. **A,** storage modulus (E') at onset frequency (f_o); **B,** loss modulus (E'') at f_o; and **C,** f_o of tanδ peak.

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