Dynamic Rheological Properties of Flour, Gluten, and Gluten-Starch Doughs. I. Temperature-Dependent Changes During Heating[1]

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ABSTRACT

The dynamic rheological properties of flour-water doughs and doughs made from blends of commercial gluten and commercial starch were tested using a dynamic rheometer. Ambient temperature frequency scans of previously heated and cooled flour-water doughs showed that irreversible rheological changes were caused by heating doughs to ≥ 55°C. Dynamic rheological measurements of flour-water doughs during heating (temperature scans) indicated that G' (the storage modulus) increased and the tangent (ratio of G''/G' where G'' is the loss modulus) decreased rapidly between 55 and 75°C. The magnitude of the temperature-dependent rheological change (G') was proportional to the dough's starch content. Adding pregelatinized starch to a gluten-starch blend resulted in an increase in G' and a decrease in tangent, similar to the change caused by heating. Heating a gluten-water dough to 90°C and then cooling to 30°C caused only a small change in the dynamic rheological properties. This change appears to be caused by gelatinization of the gluten's residual starch. However, heating a gluten-water dough to 80°C increased the gluten's mixing time by more than 1,000%.

The rheological properties of a dough change greatly between the start of mixing and the end of baking. Because those changes can have significant effects on the machinability of the dough and the quality of the final product, accurate assessment of dough texture has spawned a great deal of research over the last 60 years (Bloksma 1971, Bushuk 1985). Much of that research employed traditional dough testing instruments such as the mixograph and extensigraph. This research has provided a great deal of information but is limited to empirical correlations.

Dynamic rheological techniques have been used to determine the fundamental mechanical properties of both synthetic and biological polymers (Whorlow 1980). Because most foods are polymer systems, dynamic tests have proven to be applicable to a variety of foods (Rao 1984). Over the past 20 years, one type of dynamic testing, sinusoidal stress-strain analysis, has proven to be a useful tool in examining the fundamental rheological properties of doughs (Faubion et al 1985). Beginning with studies by Hibberd and Wallace (1966) and Hibberd (1970a,b), the technique has been used to address a number of questions including flour protein content and absorption (Navickis et al 1982), mixing time (Bohlin and Carlson 1980), and breadmaking quality (Abdelrahman and Spies 1986).

Our objective in this study was to extend the use of sinusoidal stress-strain testing to examine the temperature-dependent rheological changes that occur as doughs are heated.

MATERIALS AND METHODS

Rheometer construction and operation at ambient temperature in shear have been described previously (Faubion et al 1985). A modification of the resistance oven technique (Junge and Hoseney 1981) to heat dough was used to measure the rheological changes occurring during heating. The top and bottom plates of the rheometer were insulated from the rest of the instrument with Delrin (DuPont, Wilmington, DE) blocks and attached to a variable transformer. The resulting current flow through the dough caused it to heat. Dough temperature was monitored by a thermocouple inserted in the middle of the sample. Heating rate was monitored at 2.5°C/min. The use of resistance heating eliminates the temperature gradient within the dough mass.

Commercial wheat gluten as well as native and commercially pregelatinized (drum-dried) wheat starches were obtained from Midwest Grain Processors (Atchison, KS). Commercial bread flour was obtained from Ross Industries (Cargill Inc., Wichita, KS) and contained 11.4% protein and 0.42% ash (14% mb). The

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commercial gluten contained 82.5% protein and 1.0% ash (db). Protein, moisture, and ash were measured by AACC methods 46-10, 44-15A, and 08-01, respectively (AACC 1983).

Absorption for the gluten-starch blends was calculated as 70% of starch weight and 100% of gluten weight. Doughs (80–100 g dry material) were mixed in a pin mixer (TMCO National Manufacturing, Lincoln, NE). Unless otherwise noted, all doughs were mixed to optimum development as judged subjectively by an experienced baker using dough appearance and feel.

After mixing, flour-water doughs and doughs made from gluten-starch blends with less than 40% (w/w, db) gluten were loaded immediately into the rheometer. Excess dough was trimmed away with a razor blade, and exposed dough surfaces at the edge of the plates were coated with lubricating grease (Mobil Oil Corp., NY) to prevent drying. Dough was allowed to rest in the rheometer for 5 min before testing began. The loading process took 10 min; therefore testing began 15 min after the completion of mixing.

Gluten-water doughs and doughs made from gluten-starch blends having 40% or more gluten were removed from the mixer and placed between Teflon-coated flat boards. Weights placed on the top board served to compress the dough to a thickness controlled by four adjustable set screws. After pressing for a total of 90 min, the dough was placed in the rheometer and the edges trimmed and greased as with the low-gluten samples.

The dimensions of the rheometer cell were 91 × 82 mm. Dough thickness was from 5 to 10 mm. Tests were conducted at 25°C and 2% strain.

The starch content of commercial gluten was determined by AACC method 76-11 (AACC 1976) modified to use 1*N* NaOH to gelatinize the starch.

Rheometer measurements were calculated from the voltage output of a Kistler model 9712A5 force transducer (Kistler Instrument Corp., Amherst, NY), and a Schaevitz model 050 DC-D linear variable differential transformer (LVDT; Schaevitz, Pennsauken, NJ). The force transducer was calibrated by the factory. The LVDT was calibrated with a Schaevitz model CAL-42M calibrator.

Values reported are the means of at least two (and generally more) replicate tests. Standard deviations between replicates were calculated as described by Snedecor and Cochran (1967) for samples by dividing the squared deviations by $n - 1$. Typical standard deviations for G' and tangent are shown in Table I.

RESULTS AND DISCUSSION

Frequency Scans Before and After Heating

Flour-water doughs were tested at 25°C before and after being heated to 45 or 55°C and then cooled to 25°C (Fig. 1). Heating the doughs to 45°C before testing caused no irreversible changes in either G' or the tangent. However, if heating was increased to 55°C, G' and tangent were irreversibly increased and reduced, respectively. Similar changes were reported previously by Bloksma and Nieman (1975), who speculated that the change might be caused by starch gelatinization. LeGrys et al (1980) reported an increase in the G' of gluten-water doughs as a result of heating but attributed the effect to increased gluten cross-linking rather than to starch gelatinization. To further investigate and characterize the irreversible change in dough rheology that occurs during heating from 45 to 55°C, doughs were tested while being heated (temperature scans).

TABLE I
Standard Deviation for Rheometer Readings

Frequency	Standard Deviation 1 Det.	2 Det.[a]	95% CI[b]
G' (log units)			
0.05 Hz	0.048	0.034	0.085
0.01 Hz	0.032	0.023	0.057
0.02–50 Hz	0.023	0.016	0.041
Tangent			
0.005 Hz	0.060	0.042	0.11
0.01 Hz	0.033	0.023	0.058
0.02–50 Hz	0.029	0.020	0.051

[a] The deviation for two determinations was calculated by dividing the one determination value by the square root of 2.

[b] The 95% confidence interval (CI) will be the mean value (which is plotted in the graphs) plus or minus the number listed in this table. The number in the table was calculated by multiplying the two determination standard deviation by 2.5.

Temperature Scans of Flour-Water Doughs

During the initial heating, the G' values decreased slowly as the dough temperature increased from 25 to 50°C (Fig. 2A). At approximately 55°C, G' began to increase rapidly, reaching a peak at approximately 75°C. The tangent (Fig. 2B) decreased at the same temperature that G' increased.

After the temperature reached 90°C, dough was cooled to 30°C and retested. The G' value did not return to its original level. Instead, G' was similar to those reached at 90°C. Reheating the dough to 85°C did not produce the rapid change in either G' or tangent seen with the first heating. The G' values decreased slowly and steadily over the full 30 to 85°C reheating range, while the tangent remained unchanged. Thus, the observed rheological change is complete after a single heating and is irreversible.

Temperature Scans of Gluten-Starch Blends

Starch gelatinization, gluten cross-linking, or both are possible explanations for the thermally induced rheological change occurring between 55 and 75°C (Fig. 2). If the effect were entirely caused by changes in the starch or by changes in the gluten fraction of the dough, the magnitude of the change should be proportional to the gluten-starch ratio in the dough. Therefore, blends of commercial gluten and commercial native wheat starch were mixed to doughs and tested while heating. An increase in G' and decrease in tangent during heating from 55 to 75°C were proportional to the starch content of the dough (Fig. 3).

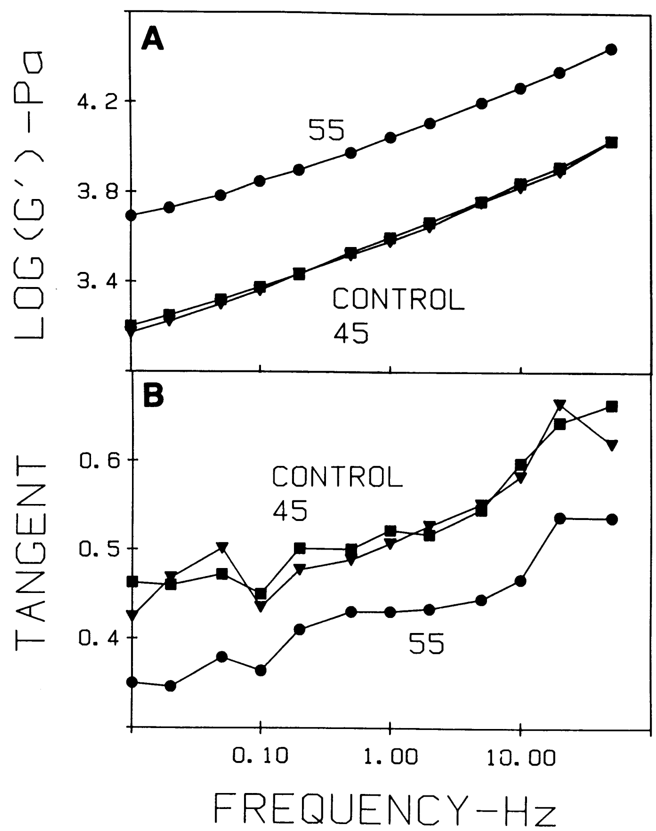

Fig. 1. Frequency scans of flour-water doughs. All doughs were tested at 25°C and had moisture contents of 44.9%. Control = no previous heating, 45 = heated to 45° C, 55 = heated to 55°C. Symbol identification is the same for A and B.

When those same doughs were reheated (Fig. 4), the tangent was inversely and G' directly proportional to the starch content. Therefore, rheological changes occurring as the dough was heated from 55 to 75°C were the result of changes in the starch fraction, presumably because of starch gelatinization.

Sucrose and NaCl

If the temperature-triggered change was, in fact, caused by starch gelatinization, then compounds known to affect starch gelatinization should affect the change as well. Sucrose and sodium chloride are known to increase starch gelatinization temperature (Spies and Hoseney 1982, Ghiasi et al 1983), so it seemed logical to expect that those solutes would increase the temperature at which the rheological change occurred.

This hypothesis was tested by adding 6% sucrose and 2% NaCl (based on flour weight) to flour-water doughs and testing those doughs during heating. Interpretation of results (Fig. 5) was complicated by the fact that the added sugar and salt made the dough slacker (lower G') and more viscous (higher tangent) before heating. As predicted, the rapid change in G' occurred at a higher temperature in the presence of sucrose and NaCl. However, the drop in tangent appears at a lower temperature. An explanation is not apparent.

Temperature Scans: Effect of Pregelatinized Starch on Gluten-Starch Blends

If the described increases in G' and decreases in tangent were due to starch gelatinization, the addition of pregelatinized starch to starch-gluten blends should cause the resulting doughs to rheologically mimic the effects of heating. This did occur, because unheated doughs containing pregelatinized starch had higher G' and lower tangent values than comparable doughs containing unmodified starch (Fig. 6). At higher levels (42%) it is clear that pregelatinized starch did not act in the same way as native starch does.

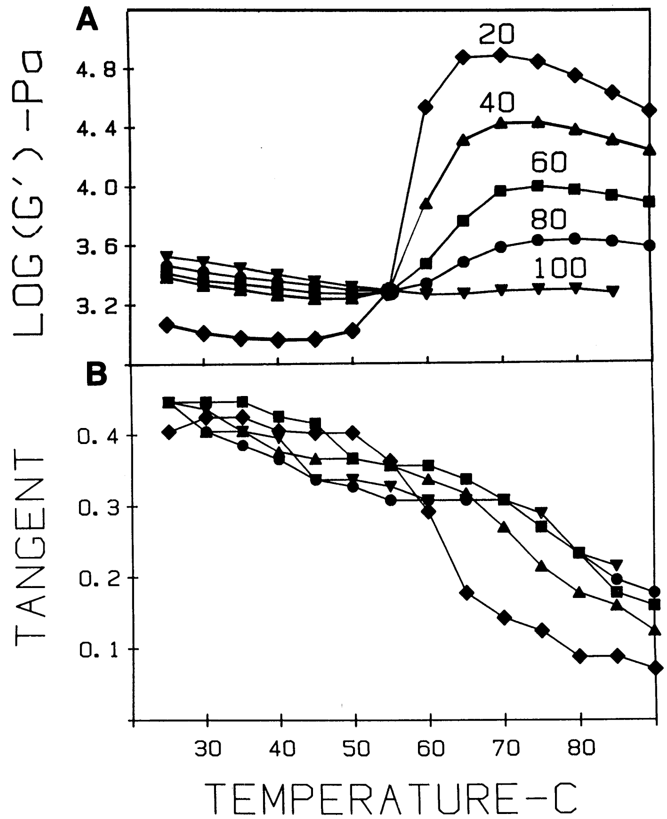

Fig. 3. Effect of first heating on the G' (2 Hz) and tangent of doughs made from blends of commercial gluten and commercial starch. Figures on the plots refer to the percent gluten in the blend. Symbol identification is the same for A and B.

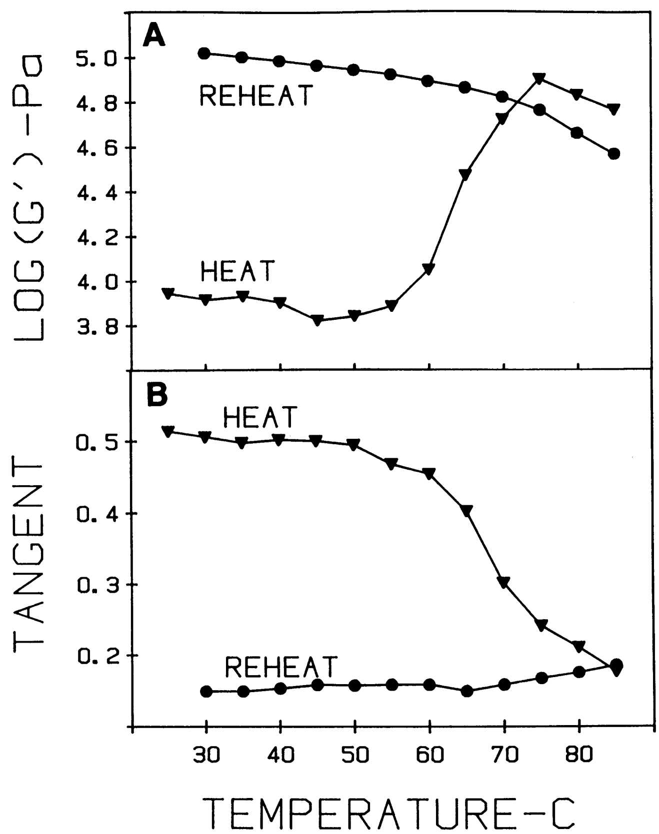

Fig. 2. Effect of heating on the G' (5 Hz) and tangent of flour-water doughs. HEAT = first heating, REHEAT = heating after dough was heated to 90°C and cooled. Dough moisture contents were 44.9%. Symbol identification is the same for A and B.

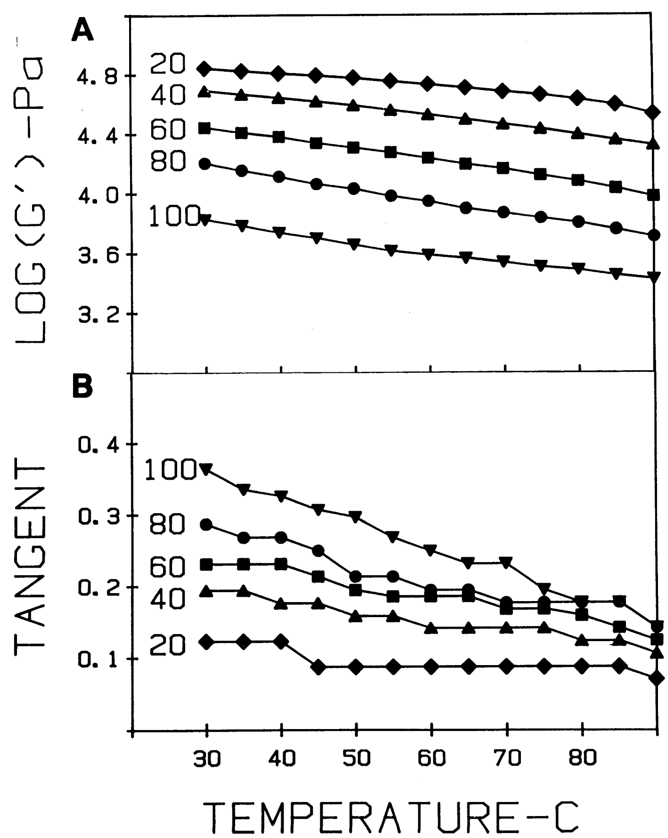

Fig. 4. Effect of reheating to 90°C on the G' (2 Hz) and tangent of doughs originally heated to 90°C and cooled (blends are as in Fig. 3). Symbol identification is the same for A and B.

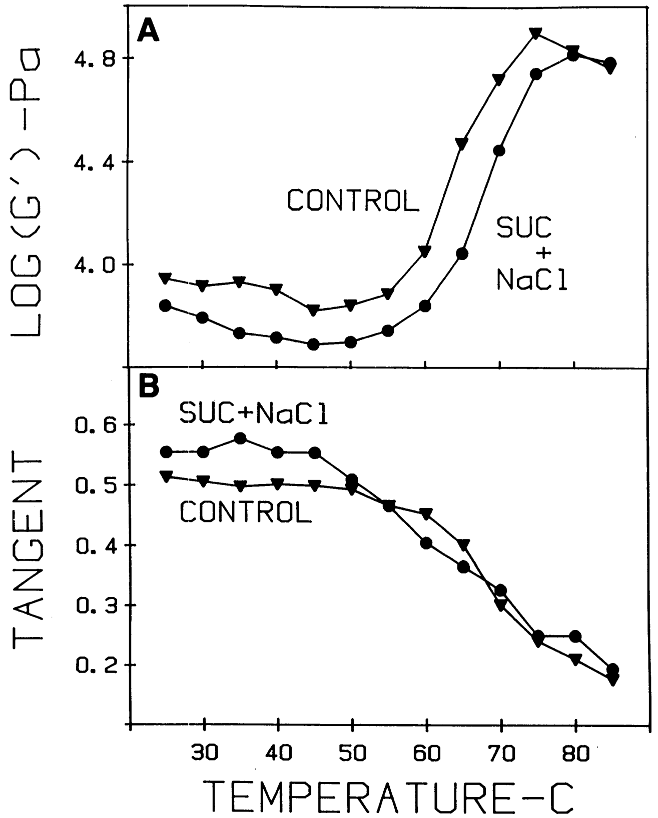

Fig. 5. Effect of the addition of 6% sucrose and 2% NaCl (flour weight basis) on G' (5 Hz) and tangent of doughs during initial heating. Dough moisture contents were 44.9 and 42.7% for the control and sucrose-NaCL doughs, respectively. Symbol identification is the same for A and B.

Temperature Scans: Effect of Dough Moisture Content

Doughs with different water contents were prepared and tested (Fig. 7). Although the drier doughs had higher G' values before being heated, the tangent was not affected by dough moisture content. Thus, decreasing dough moisture content did not cause the same rheological effect as did replacing a portion of the native starch in a blend by pregelatinized starch. Apparently, then, starch gelatinization affects dough rheology in ways other than simply absorbing water.

The increase in G' and decrease in tangent seen as doughs were heated indicate an increased number of rheologically effective cross-links in the system. This substantial effect of starch gelatinization (Fig. 3) suggests that starch is not just an inert filler in doughs undergoing heating. Below 55°C, the amount of native, unmodified starch present in the gluten-starch doughs had only a small effect on G'. However, above 55°C, the magnitude of change in G' was proportional to the amount of starch present in the dough. Gelatinization may provide the opportunity for increased hydrogen bonding between gluten polypeptides and starch molecules.

Temperature Scans of Gluten-Starch-Water Doughs

The rheological change occurring when doughs containing gluten, starch, and water were heated was a result of starch gelatinization (Figs. 3 and 6). In related work, Schofield et al (1983, 1984) showed that the volume-improving effect of added gluten in bread is irreversibly reduced or eliminated if the gluten is first heated above 55°C. We observed a small irreversible change in dynamic rheological properties when doughs made from 100% commercial gluten and water (i.e., no added starch) were heated. Is this change caused by the presence of residual starch, changes in the gluten protein itself, or both?

To shed light on this question, the data presented in Figures 3 and 4 were plotted with the heat and reheat cycles for each

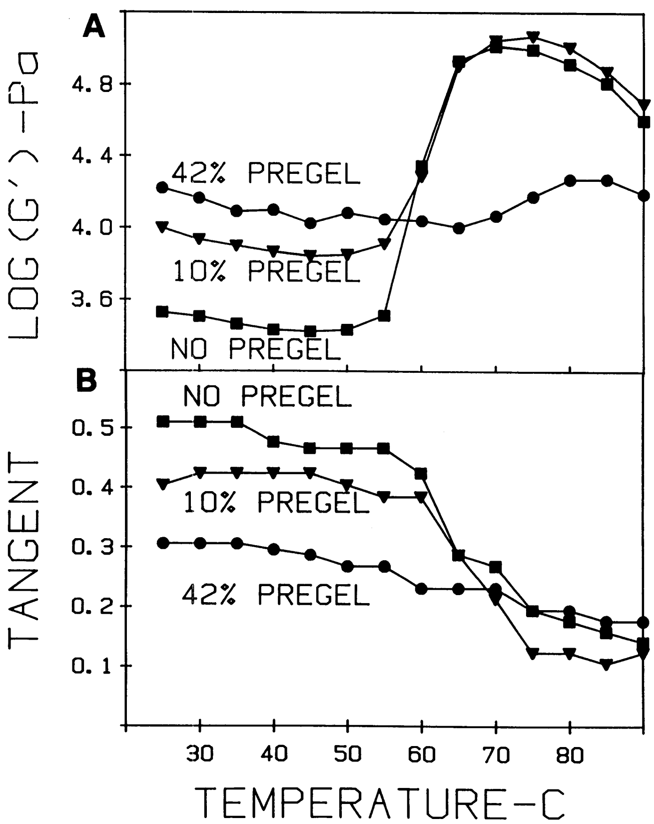

Fig. 6. G' (5 Hz) and tangent for the doughs made from blends of commercial gluten (15%), commercial pregelatinized wheat starch (shown on figure), and commercial unmodified wheat starch (remainder). Moisture content of all doughs was 44.9%. Symbol identification is the same for A and B.

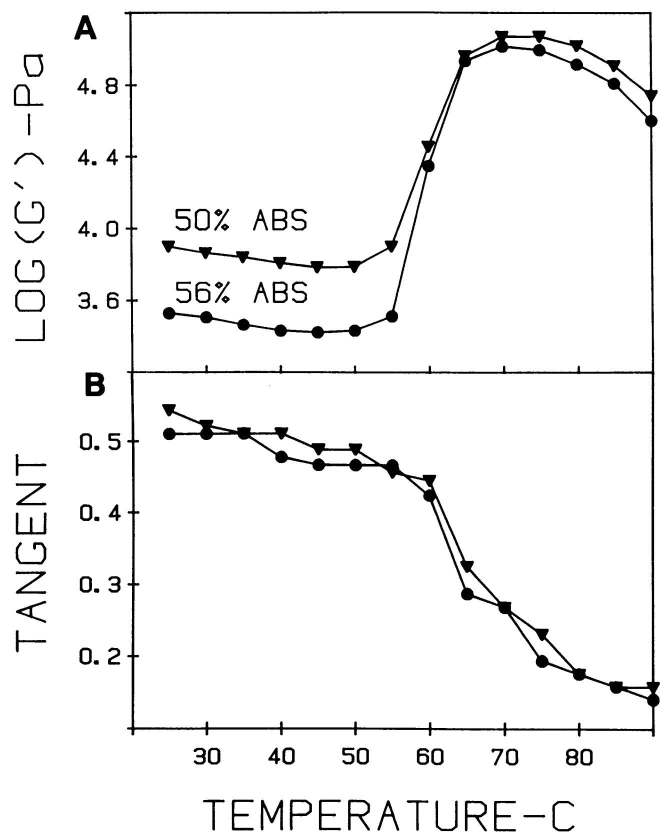

Fig. 7. G' (5 Hz) and tangent for dough made with 50% absorption (42.7% moisture) and for dough made with 56% absorption (44.9% moisture). All doughs were made from a blend of 15% commercial gluten and 85% commercial unmodified wheat starch. Symbol identification is the same for A and B.

formulation on the same graph. Plots for the 100% and 40% gluten doughs are shown in Figures 8 and 9, respectively. Examination of the data for the first heating showed that the rheological change was much greater for the dough containing added starch.

An objective measurement of the amount of irreversible change occurring can be derived from measurement of the difference in G' ($\Delta G'$) between the readings at 30°C before heating and at 30°C after heating to 90°C. The $\Delta G'$ was slightly over 0.3 log units for the 100% gluten dough and slightly over 1.3 log units for the 40% gluten dough. Similar measurements were made for doughs with other gluten-starch ratios.

The starch content of the commercial gluten was determined to be 8.4% db. The total starch (residual starch in the gluten plus commercial starch in blend) content of the doughs made from gluten-starch blends was then calculated. The $\Delta G'$ values described above were plotted versus the percent total starch (dry basis) in the doughs (Fig. 10).

If the nonstarch components of the doughs were not affected to heating to 90°C, then the plot would be expected to go through the origin, i.e., zero starch in dough should result in no change from heating to 90°C. The plot (Fig. 10) is very nearly linear and, if extrapolated to 0% starch, would give a $\Delta G'$ value very close to 0.0. The data, therefore, indicate that the nonstarch components of gluten are not affected by heating to 90°C.

Heated and Lyophilized Glutens

In all tests described thus far, doughs were mixed before being heated. Thus, the possible effects of heating on mixing properties were not yet tested. Heating of wheat samples (Finney et al 1962) or flour-water doughs (C. S. Lai et al, *personal communication*) was reported to increase mixing time. Therefore, a gluten-water dough was mixed to full development and then heated to 80°C in the resistance oven. After heating, the dough was removed, lyophilized, and ground in a Udy mill. An equivalent dough was mixed, lyophilized, and ground in the same manner but was not heated.

Both doughs were subsequently mixed to optimum and tested with the rheometer. The most prominent difference between heated-lyophilized and unheated glutens was in the amount of mixing each required to create a fully developed dough. Whereas lyophilization changed mixing time slightly (2.2 vs. 3.0 min.),

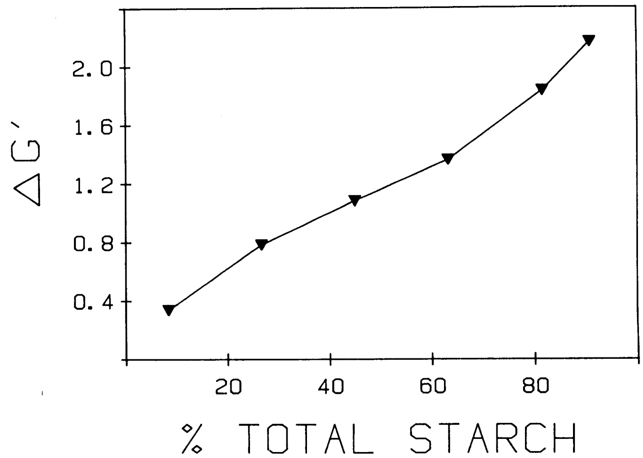

Fig. 10. $\Delta G'$ (differences in G' values at 30°C before and after heating to 90°C) for gluten-starch dough vs. percentage of total starch in dough. Measurements were at 5 Hz.

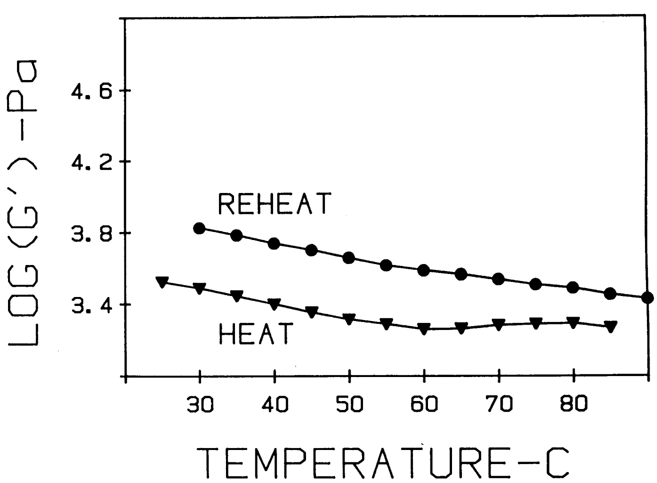

Fig. 8. Effect of first heating and reheating on G' (2 Hz) of gluten-water dough.

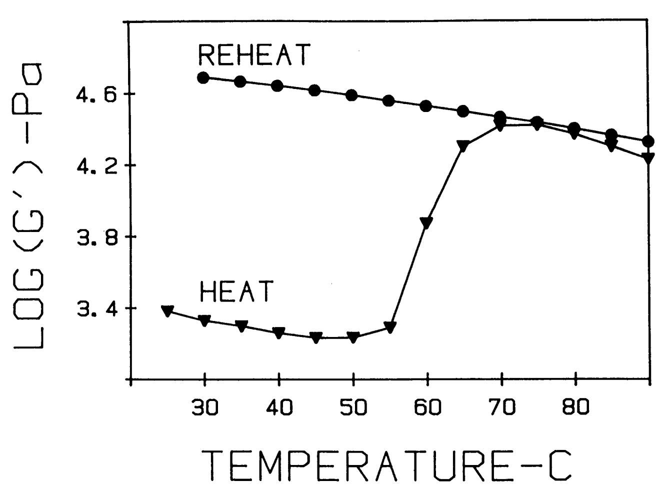

Fig. 9. Effect of first heating and reheating on G' (2 Hz) of gluten-starch-water dough.

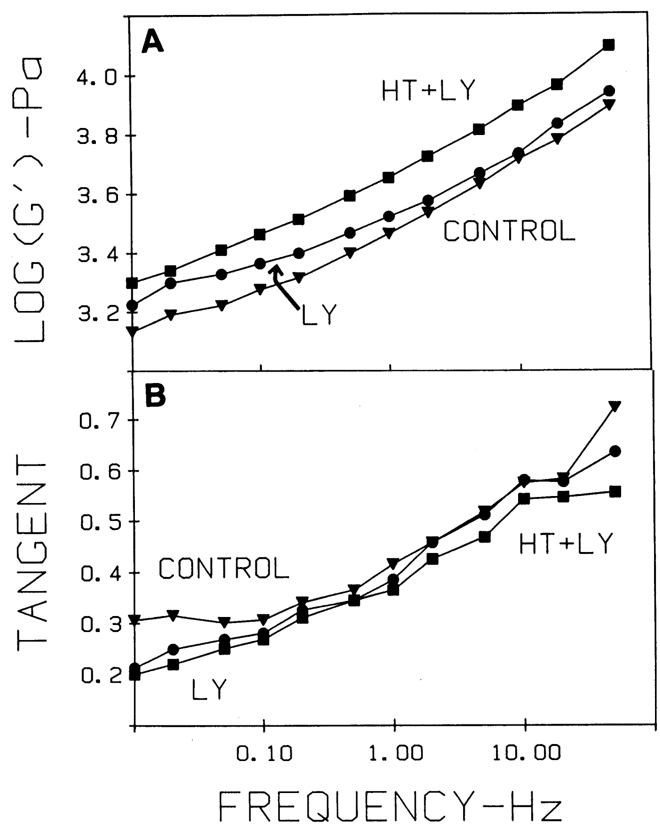

Fig. 11. Effect of prior heating and lyophilization of gluten on the G' and tangent of subsequently mixed doughs. CONTROL = no previous mixing. LY = previously mixed and lyophilized. HT + LY = previously mixed, heated to 80° C, and lyophilized. Test was conducted at 25°C.

heating the gluten-water dough, followed by lyophilizing gave a dried gluten with a mixing time of 25 min. However, at the end of this time, the dough produced was both cohesive and extensible. Frequency scans of these doughs (Fig. 11) demonstrated that the dough produced from the heated-lyophilized gluten had the higher G' and lower tangent expected because of the gelatinization of residual starch (Fig. 8). However, the addition of pregelatinized starch to gluten did not increase its mixing time significantly. Therefore, we must conclude that heating does, indeed, affect gluten protein but in ways not measurable by the dynamic rheometer.

One possibility is that the changes induced by heat affect the gluten proteins' ability to interact with itself in such a way that subsequent rehydration and mixing requires much more mechanical work. If mixed to complete development, differences due to heating are overcome.

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