Dynamic Rheological Properties of Flour, Gluten, and Gluten-Starch Doughs.

II. Effect of Various Processing and Ingredient Changes

P. C. DREESE, J. M. FAUBION, and R. C. HOSENEY

ABSTRACT

The dynamic rheological properties of gluten-starch, gluten, and flour doughs were tested at 25°C and 2% strain, over a frequency range of 0.1 to 50 Hz. Cysteine and mercaptoethanol reduced the storage modulus (G') and increased the loss tangent (G'')/G' where G' is the loss modulus. When flour was fractionated into gluten, starch, and water-soluble fractions, the presence of the water-soluble fraction affected G' and loss tangent in a manner similar to cysteine or mercaptoethanol. The fast-acting oxidant, potassium iodate, increased G' but had no effect on the tangent. Increased dough moisture resulted in lower values of G' at all frequencies tested. Over the range used, the moisture of gluten-water doughs did not affect the tangent at any frequency tested. The moisture content of flour-water doughs did not affect the tangent at frequencies of 10 Hz and below, but at 20 and 50Hz the results were erratic. Overmixing flour-water doughs had essentially the same rheological effect as increasing dough moisture.

The fundamental mechanical properties of wheat flour doughs are important in determining both the handling properties of the dough during processing and the quality of the finished product. In spite of its importance, knowledge of the fundamental rheological behavior of dough has been difficult to obtain because of its complicated, nonlinear, viscoelastic behavior and the empirical nature of many popular testing instruments (Parker and Hibberd 1974, Hibberd and Parker 1975).

Dynamic force-deformation testing methods, developed for use in polymer rheology (Ferry 1980), are potentially useful in evaluating a number of rheological properties of foods (Rao 1984) including doughs. These methods have been applied to the analysis of dough systems (Hibberd and Wallace 1966; Hibberd and Parker 1970 a,b; Navickis et al 1982) with various degrees of success. The goal of this study was to use dynamic force-deformation testing to better understand what factors affect the rheological properties of flour and gluten-starch doughs.

MATERIALS AND METHODS

Rheometer construction and operation has been described by Faubion et al (1985). Commercial wheat gluten was provided by Midwest Grain Products, Atchison, KS, and had protein and ash contents (db) of 82.5% and 1.0%, respectively. Native wheat starch was also from Midwest Grain Products. Unless otherwise noted, the flour used was a commercial, untreated bread flour (11.4% protein, 0.42% ash, 14% mb) from Ross Industries (Cargill, Wichita, KS). A soft red winter wheat flour (9.63% protein, 0.47% ash, 14% mb) from Nabisco Brands Inc., Toledo, OH, was tested to show the effect of wheat classes. Protein, moisture, and ash were measured by AACC methods 46-10, 44-15A, and 08-01, respectively (AACC 1983).

Flour was fractionated into gluten, starch, and water-soluble fractions according to the procedure shown in Figure 1. The resulting lyophilized gluten had protein and ash contents (db) of 80.6% and 0.40%, respectively. Flour was reconstituted by making a dry blend of the gluten, starch, and water solubles in the same proportion (w/w, db) as they were produced from the parent flour. Dough preparation and rheometer testing procedures were given by Dreese et al (1988).

Doughs were tested in the rheometer for 100 min while seven readings were taken at times evenly spaced on a logarithmic time scale. In addition to control (flour-water) doughs, doughs containing 30 ppm (flour weight basis) cysteine (added as cysteine HCl), and dough containing 30 ppm potassium iodate were tested. A second flour-water dough was tested for only 1 min at each of the seven reading times. Readings were taken at 0.5, 5, and 20 Hz.

Flour-water doughs made from bread flour (hard red winter wheat) and soft red winter wheat flour were tested at frequencies from 0.01 to 50 Hz. Bread doughs were made from the following formula (flour weight basis): flour, 100; water, 60 (optimum); sugar (sucrose), 6; salt, 1.5; shortening, 3; instant dry yeast, 0.76; potassium bromate, 0.001 (optimum). Doughs were tested at 8 min after the mixing, first punch, second punch, pan, and oven stages of the pup-loaf baking procedure (Finney and Barmore 1943). Doughs made from blends of commercial gluten and commercial unmodified wheat starch were tested at 100, 40, 20, and 10% gluten (Table I). Gluten-water doughs containing 0, 25, 50, 100, 200, and 400 ppm (gluten basis) cysteine (added as cysteine HCl) were tested at 0.5, 5, and 20 Hz. Mercaptoethanol was added to doughs made from blends of 15% gluten (commercial and lyophilized) and 85% commercial wheat starch. Cysteine hydrochloride and mercaptoethanol were from Fisher Scientific (Fairlawn, NJ) and were reagent grade.

All data reported are the means of at least two (and generally more) replicate tests. Standard deviations between replicates were as given by Dreese et al (1988).

**Fig. 1.** Flour fractionation procedure.
RESULTS AND DISCUSSION

Effects of Testing Time, Cysteine, and Iodate

Results from tests at higher frequencies were similar to those at 0.5 Hz, so only those data are shown. Relative to the controls, cysteine decreased $G'$ and increased the tangent (Fig. 2). Iodate (Fig. 2) increased $G'$ relative to controls but had little effect on the tangent. Cysteine or iodate are known to affect dough properties (Bloksma 1975), yet the changes we found in $G'$ and tangent were small. Thus, this dynamic analysis technique appears to be no better at demonstrating the effects of these additives than conventional tests such as the extensigraph (Dempster et al 1954).

The $G'$ values for all doughs decreased slowly with time when held under vibration (Fig. 2). Cysteine and iodate did not affect the rate at which $G'$ decreased, but the rate of decrease was less for doughs not continuously vibrated. The tangent did not change significantly with time.

Doughs are known to relax after mixing. The fact that resting time in the rheometer had only a small effect on $G'$ and no effect on the tangent indicates that the rheometer will not be useful for measuring dough relaxation.

Effect of Flour Type

Results (Fig. 3) showed that $G'$ and tangent increased with frequency, and that the frequency dependence (slope), was essentially equal for the two flours.

Fig. 2. The effect of time in the rheometer with vibration at 0.5 Hz and 1% strain on flour-water doughs with 44.9% moisture. Cysteine and iodate doughs contained 30 ppm (flour basis) of L-cysteine and potassium iodate, respectively. The NO-VIB doughs were not vibrated in the rheometer except for 1-min intervals at each reading. Symbol identification is the same for A and B.

<table>
<thead>
<tr>
<th>TABLE 1</th>
<th>Dough Moisture Contents*</th>
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<tr>
<td>Gluten (%)</td>
<td>Starch (%)</td>
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<tr>
<td>10</td>
<td>90</td>
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<tr>
<td>20</td>
<td>80</td>
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<tr>
<td>40</td>
<td>60</td>
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<tr>
<td>100</td>
<td>0</td>
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*Data presented in Fig. 5.

Fig. 3. Rheological properties of doughs (44.9% moisture) from hard red winter and soft red winter wheat flours.

Fig. 4. The effect of fermentation time on tangent and $G'$ for bread doughs. Fermentation times for the different treatments are shown as 1P = first punch, 113 min; 2P = second punch, 163 min; PAN = 188 min; and OVEN = 243 min. Symbol identification is the same for A and B.
Fermenting Doughs
Immediately after mixing (Fig. 4), dough had a much higher
dynamic storage modulus and lower loss tangent than did dough
with longer fermentation. Because the densities of these doughs
were not measured, it is possible that some of these differences
resulted from variations in density. The loss tangents of fermented
doughs varied inversely with frequency at frequencies below 1 Hz.
This was the opposite of results for flour-water doughs (Fig. 3).
Low modulus values (and hence, low force transducer readings)
precluded taking measurements below 0.1 Hz, so it is not known at
what, if any, frequency the tangent values would reach a
maximum. A peak in a tangent versus frequency plot is generally
interpreted as a molecular vibration at that frequency (Armeniades
and Baer 1977, Allen 1978). With the exception of the doughs
measured at the oven stage, the increase in tangent at lower
frequencies was positively correlated with fermentation time.

Gluten-Starch Blends
The slope of log $G'$ versus log frequency plots (Fig. 5A) increased
as the gluten content of the blend increased. The water absorption
used in preparing all doughs was 103% of the gluten weight and
59% of the starch weight. With these water absorptions, the
absolute value of $G'$ was nearly constant for blends containing 40%
gluten or greater. At gluten contents $\leq$ 40%, $G'$ varied inversely
with gluten. Hibberd and Parker (1970b) reported that $G'$
increased as the gluten content of gluten-starch blend increased.
However, their work was done at a constant dough moisture, and
the higher water absorption of gluten relative to starch caused
doughs with higher gluten contents to be drier. Gluten content of
the dough had little or no effect on the value of the tangent (Fig.
5B).

Flour Versus Gluten-Starch Blend
When doughs made from flour were tested and compared to
doughs produced from a 15% gluten-starch blend, plots of $G'$
versus frequency were significantly different (Fig. 6). Although the
protein contents (db) of the gluten-starch blend and flour were
similar (12.4 and 13.3%, respectively), the slopes of the two $G'$ plots
were much different. The slope of the flour-water dough plot was
similar to that produced by testing a 100% gluten dough (Fig. 5). At
frequencies below 10 Hz, the loss tangent of the flour dough was
lower than for the 15% gluten blend. The data show that other flour
components, in addition to gluten and starch, influence dough
rheology.

Lyophilized Gluten, Starch, and Water Solubles
When flour was fractionated into gluten, starch, and water
solubles, the reconstituted flour dough (lyophilized gluten +
lyophilized starch + water solubles) had tangent values that were
essentially equal to those of the parent flour, except at the lowest
frequencies tested (Fig. 6B). A plot of $G'$ versus frequency for the
reconstituted flour was roughly parallel to that for the parent flour
but was lower by roughly 0.1 log $G'$ units (Fig. 6A).

Omitting the water solubles from the reconstituted dough
causcd the tangent to be significantly lower and $G'$ to be
significantly higher. Thus, the observed effect of water solubles,
making the dough relatively slack and more viscous, was
reflected by the dynamic test.

Doughs produced from lyophilized gluten plus lyophilized
starch had higher $G'$ and lower tangent values than did equivalent
doughs produced from commercial gluten plus commercial starch.
An increase in the cross-linking of a polymer system will cause the
$G'$ to increase and the loss tangent to decrease (Ferry 1980). These
results suggest that the lyophilized gluten is a more cross-linked
polymer system than the commercial gluten. This is contrary to
what might be expected. Commercial gluten is heated during
drying but lyophilized gluten is not. Heating is thought to cause
increased cross-linking in gluten (Bale and Muller 1970, Schofield
et al 1983). It is possible that other differences (chemical or
physical) between the commercial and hand-washed-lyophilized
gluten more than compensate for the effect of heating during
drying. Because the starch in commercial gluten is not gelatinized

Fig. 5. $G'$ and loss tangent for doughs from blends of commercial gluten
and commercial unmodified wheat starch. Dough moisture contents for
each blend are given in Table I. Symbol identification is the same for A and
B.

Fig. 6. Comparison of doughs made from flour, commercial gluten +
commercial starch, lyophilized gluten + lyophilized starch, and lyophilized
gluten + lyophilized starch + water solubles. Moisture content for all
doughs was 44.9%.
(Dreese et al. 1988), the heat used in the commercial drying process cannot be great.

Effect of Moisture in Gluten-Water Doughs

Gluten-water doughs with higher moisture (Fig. 7) had a lower storage modulus and a tangent equal to dough with lower moisture. The decrease in $G'$ with increasing dough water content is in agreement with the observation that dough is more easily deformed when its water content is greater. These observations are in agreement with Hibberd and Parker (1970b).

Effect of Moisture in Flour-Water Doughs

The results of mixing and testing flour-water doughs at different absorptions (Fig. 8) were similar to those for gluten-water doughs. $G'$ values decreased with increasing dough moisture. Dough moisture content did not affect tangent values at frequencies of 10 Hz and below. For the driest doughs (43.4% moisture) the tangent at 20 Hz was higher than the tangent at 10 Hz and, thus, followed the general trend of higher frequencies producing higher tangents. For dough with 44.9% or higher moisture content, the tangent at 20 Hz was lower than at 10 Hz.

Effect of Mixing Time in Flour-Water Doughs

Flour water doughs mixed for different times before being tested gave results (Fig. 9) similar to those for the dough moisture study (Fig. 8). Doughs with longer mixing times behaved like doughs with higher moisture. $G'$ values decreased with increasing mixing times. Tangent values were essentially unaffected by mixing time.

The appearance and feel of an overmixed dough are similar to those of a dough with excess water. The fact that the dynamic rheological properties of an overmixed dough also are similar to those of a dough with excess water indicate that overmixing may decrease the water-binding capacity of gluten and that the water that is thereby released may be responsible for many of the properties of an overmixed dough.

Effect of Cysteine on Gluten-Water Doughs

Results (Fig. 10) were similar to those produced by flour-water doughs containing cysteine (Fig. 2) in that cysteine decreased $G'$ and increased the loss tangent. A reduction in polymer cross-linking will cause lower $G'$ and higher tangent values (Ferry 1980). The rheometer measurements confirm the commonly held view that cysteine reduces the water-binding capacity of gluten and that the water that is thereby released may be responsible for many of the properties of an overmixed dough.

Fig. 7. Effect of moisture content on doughs made from commercial gluten and water. Symbol identification is the same for A and B.

Fig. 8. Effect of moisture content in flour-water doughs. Numbers indicate percent moisture. Symbol identification is the same for A and B.

Fig. 9. Effect of mixing time on flour-water doughs with 44.9% moisture. Numbers indicate mixing time in min. The optimum mix time was 3 min. Symbol identification is the same for A and B.
that cysteine breaks disulfide cross-links or limits their formation in gluten (Bloksma 1964).

**Effect of Mercaptoethanol**

Mercaptoethanol also breaks disulfide cross-links in gluten and is generally more effective at causing gluten breakdown than cysteine. When added to dough, mercaptoethanol increased the tangent and decreased $G'$ (Fig. 11) in a manner similar to cysteine. Because cysteine was tested in gluten-water doughs, whereas mercaptoethanol was tested in doughs made from gluten-starch blends, it is impossible to draw conclusions about the relative effectiveness of the two reagents.

When the molecular weight of a polymer increases beyond a critical molecular weight ($M_c$), further increases in molecular weight have minimal effects on the dynamic rheological parameters of the system (Runt and Harrison 1980, Graessley 1984). The molecular weight of most gliadin proteins is in the 25,000–50,000 range. The molecular weight of glutenin proteins ranges from 40,000 to several million (Kasarda et al 1971). Thus, it is probable that the average molecular weight of the proteins in the dough system is above the $M_c$, and changes in molecular weight would have only a small effect on the dynamically measured rheological parameters. The addition of mercaptoethanol probably decreased the average molecular weight of the gluten by cleaving intermolecular disulfide bonds. When the molecular weight decreased below $M_c$, the effect was observed as a decrease in $G'$. The fact that the commercial and the lyophilized gluten showed reduced $G'$ at the same levels of mercaptoethanol is an indication that the molecular weights of the commercial and of the handwashed lyophilized gluten were nearly equal.

**LITERATURE CITED**


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