DOUGH RHEOLOGY AT LARGE DEFORMATIONS IN SIMPLE TENSILE MODE

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ABSTRACT

Modified extensigraph with variable hook speed and strain gage recording system and Universal testing machine (Instron) with special cross-head attachment were used for stress-strain studies on doughs at different extension rates in simple tensile mode. A higher accuracy of stress-strain curves obtained by Universal testing machine even at very low values of strain made it possible to interpret them in terms of isochronal constant strain rate modulus F(t*), and exponent n characterizing the time dependence of the time dependent constant strain rate modulus F(t). The sensitivity and practical significance of these two parameters were demonstrated on doughs prepared from chemically treated flours and mixtures of wheat flour and wheat starch as well as wheat flours supplemented with vital gluten. At strain values $\lambda-1>1.5$, extensigraph and Instron stress-strain curves were similar and could be easily transformed into deformation curves. Coefficients of absolute viscosity derived from the deformation curves over the shear stress range of 158-2,510 $kg{\cdot}m^{-1}{\cdot}s^{-2}$ were between 10^5 and 10^6 $g{\cdot}cm^{-1}{\cdot}s^{-1}$.

Because of the obvious importance of dough extensibility and resistance to extension during various stages of the bread-making process, load-extension tests have become an indispensable tool in both quality control and research in many cereal laboratories. The application of the Brabender Extensigraph and the Universal testing machine (Instron) for more fundamental rheological studies on dough in simple tensile mode was reported by several workers (1,2,3,4,5,6,7,8). This paper deals with the critical evaluation of the working techniques based on the application of these two instruments, as well as methods used for the interpretation of the results obtained. An attempt was made to determine the practical significance of the evaluated rheological parameters and to correlate them with the baking performance of the tested flours.

MATERIALS AND METHODS

Materials. Flours used in this study were commercial flours milled from Canadian hard red spring wheats (HRS), both untreated and chemically improved. Chemical analysis of these flours was done according to AACC Methods (9). Potassium bromate, used for chemical improvement, was reagent grade and was added to flour in the form of a water solution during dough mixing. Azodicarbonamide (ADA) was used in the form of the commercial improver MATUROX (kindly supplied by W&T Flour Service, Oakville, Ontario) in powdered form. The ADA content was determined analytically using AACC method 48-71 (9). Mixtures of wheat flour and wheat starch were prepared by replacing wheat flour with an equal amount of wheat starch (on a dry-solid basis). Mixtures of wheat flour and vital gluten were prepared in the same way.

Preparation of dough. All doughs were mixed in the 300-g farinograph mixing bowl to maximum development using constant flour weight method (9). Unless otherwise stated, the doughs were mixed at the water absorption required for a consistency of 500 B.U.

Extensigraph measurements. After 45-min reaction time, the dough test piece (150 g) were rounded in Brabender dough rounder and moulded by the three roller loaf moulder with stops allowing for maximum length of the dough cylinder of 13 cm. After additional 45 min of resting, the doughs were stretched at four different hook speeds: 100, 50, 20, and $\text{cm}\cdot\text{min}^{-1}$.

The extensigraph used was a modified type with strain gage electronic recording system and variable hook speed designed and constructed by the Engineering Research Service, Canada Department of Agriculture (10). Each extensigraph curve was replicated three times and the average curve was used for the final calculation. Shear stress values were calculated using the equation of Muller *et al.* (1).

Instron measurements. The Instron method, based on stretching dough rings of uniform and defined geometry, was described in earlier publications (8,11,12). The rings were tested in simple tension with an Instron Universal Testing Machine, Model TM using a 2,000 g tension-load cell and a special cross-head attachment (Fig. 1). The rings were stretched at four different cross-head speeds (100, 50, 20, 10 $\text{cm}\cdot\text{min}^{-1}$) while submerged in a buoying liquid of matching density. A mixture of Arochlor 1,221 and mineral oil of an average density of 1.19 $\text{g}\cdot\text{ml}^{-1}$ was used as recommended by Tschoegl *et al.* (11). Both reaction and resting times were 45 min, so that the condition of the dough at the time of measurement was exactly the same as in extensigraph tests. All reported data are based on the average of triplicate measurements.

The calculation of true stress and the evaluation of the stress-strain curves in terms of isochronal constant strain rate modulus for 0.1 min interval of extension F (0.1 min) and time-dependent modulus F(t) was done according to the procedure outlined by Tschoegl *et al.* (12). To obtain comparable data with extensigraph tests, tensile stress was transformed into shear stress by dividing by 3. Unless otherwise stated, all measurements were carried out at 25° C.

Baking tests. Baking tests were done according to the AACC straight-dough procedure (9) using the following formula: 100% flour, 9% sugar, 3.5% salt, 4.5% dried yeast, 4% shortening, 1% yeast food.

RESULTS AND DISCUSSION

Basic Characteristics of Flours Used

Some characteristics of flours used are summarized in Table I. All flours were of the strong type, but their load-extension characteristics covered a sufficiently wide range required by this study.

The Evaluation of Effective Mass

Both with Instron and extensigraph technique, the calculation of the effective mass, i.e., the mass actually involved in the development of the resistance to extension, appeared as one of the major problems in the conversion of the measured data into stress-strain data. With both methods, the volume, and hence, the effective mass of the dough subjected to stretching, change, as the stretching process progresses; the cross-sectional area of the dough piece does not change proportionally with its increasing length. This has an effect on the calculation of the true stress based on the actual cross-sectional area of the sample. Muller *et al.* (1) pointed out that errors in the measurement of effective mass were the most significant ones among those involved in extensigraph measurements. So far, direct weighing of the dough between the hook and the

TABLE I
Chemical, Rheological and Baking Characteristics of the Flours Used

			Extensigraph Characteristics					
Sample	Type of Flour	Protein %	Hook Speed $cm \cdot min^{-1}$	Maximum Tension g	Extensibility[a] s	Exponent n	Isochronal Modulus F (0.1 min) $kg \cdot m^{-1} \cdot s^{-2}$	Specific Loaf Volume $ml \cdot g^{-1}$
A	Patent	13.19	100	1090	13	−0.215	863	4.28
			50	870	22			
			20	620	53			
			10	480	110			
B	Patent	14.50	100	750	14	−0.287	778	4.55
			50	665	27			
			20	425	55			
			10	327	130			
C	Patent[b]	14.81	100	640	16	...	...	4.35
			50	535	35			
			20	400	65			
			10	345	120			
D	Patent[c]	13.63	100	690	16	−0.283	660	4.58
			50	572	35			
			20	380	80			
			10	315	130			
E	Patent	14.81	100	630	18	−0.368	490	4.95
			50	490	32			
			20	303	70			
			10	275	135			
F	Patent	13.63	100	595	16	−0.377	433	4.60
			50	462	30			
			20	350	70			
			10	247	140			

G	Straight run	12.19	100	442	16	−0.570	383	4.01
			50	320	30			
			20	220	70			
			10	...	...			

[a] Extensibility expressed as time required to reach maximum resistance.
[b] Flour "C" with 60 ppm $KBrO_3$ added.
[c] Flour "D" with 60 ppm $KBrO_3$ added.

TABLE II
Equations for Calculation of the Effective Mass Relative to Length Using Different Methods

Flour	**Straight Line** **Linear Regression**	 **Slope Method**	**Least Square Parabola**
A	$M=60.36+2.84\times(r=0.890)$	$M=60.70+3.57\times$	$M=47.74+7.47\times-0.29\times^2(R^2=0.13)$
B	$M=59.96+2.94\times(r=0.830)$	$M=57.93+3.33\times$	$M=51.32+6.09\times-0.20\times^2(R^2=0.07)$
C	$M=61.11+2.57\times(r=0.878)$	$M=57.14+3.12\times$	$M=46.96+7.72\times-0.32\times^2(R^2=0.19)$
D	$M=65.80+2.05\times(r=0.801)$	$M=59.63+3.03\times$	$M=51.78+7.29\times-0.33\times^2(R^2=0.27)$
E	$M=57.52+2.87\times(r=0.893)$	$M=57.62+2.86\times$	$M=43.41+8.02\times-0.32\times^2(R^2=0.16)$
F	$M=63.78+2.09\times(r=0.871)$	$M=62.17+2.35\times$	$M=52.20+5.95\times-0.24\times^2(R^2=0.16)$
Average	$M=61.40+2.56\times$	$M=59.20+3.06\times$	$M=49.07+7.09\times-0.289\times^2(R^2=0.15)$

$\times$ = half-length of the dough piece between the inner edges of the cradle.

Fig. 1. Cross-head attachment of the Universal testing machine (Instron) for stretching dough rings in simple tensile mode while submerged in buoying liquid of matching density.

inner edges of the cradle at different extensions was the only method used for the determination of the effective mass in extensigraph studies. A new technique, based on a principle similar to that used by Tschoegl *et al.* (11) for the determination of the effective circumference of dough rings stretched between two hooks was tried to replace the tedious direct weighing method. The principle is diagrammatically shown in Fig. 2. The displacement of the two marks on the

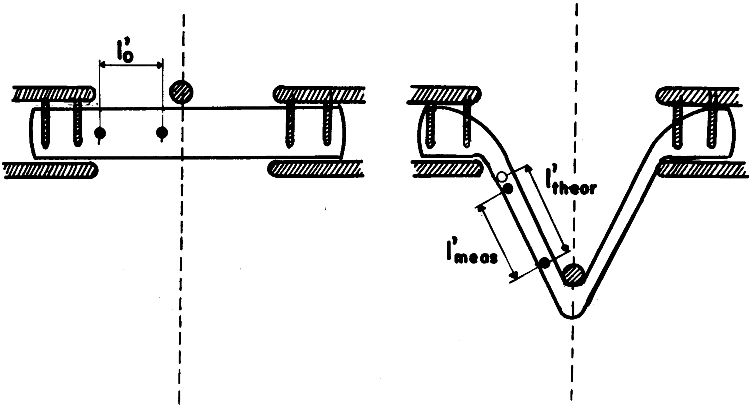

Fig. 2. Measurement of the reference marks displacement on the half-strand of the dough cylinder during its stretching with extensigraph.

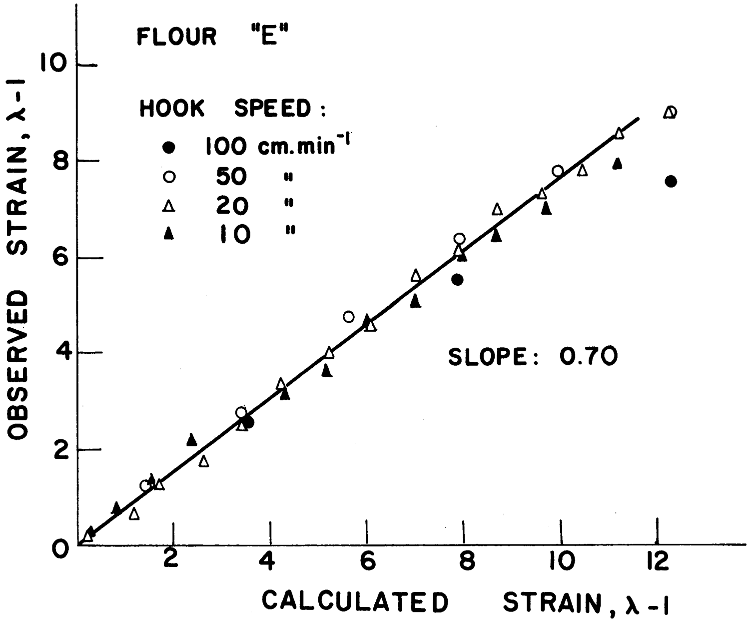

Fig. 3. Straight-line relation between the calculated and observed strain determined from the displacement of the reference marks on stretched dough piece.

half-strand of the dough was measured at different intervals of extension and values of strain, based on the theoretical and observed displacement, were plotted against each other. This plot (Fig. 3) resulted in a linear relationship over a sufficiently wide range of extensions without being affected by the rate of extension. The slope of the straight line was found dependent on the type of flour, decreasing with the increasing resistance of the dough to extension (Fig. 4). The following linear equation was then used for the calculation of the effective mass as function of the dough length.

$$M = A + \frac{2}{B} \cdot l \tag{1}$$

where B is the slope of the straight line resulting from the plot of observed to calculated strain, A is the intercept calculated for an experimentally determined effective mass of the dough between the inner edges of the cradle, l is the half-length of the dough piece in cm.

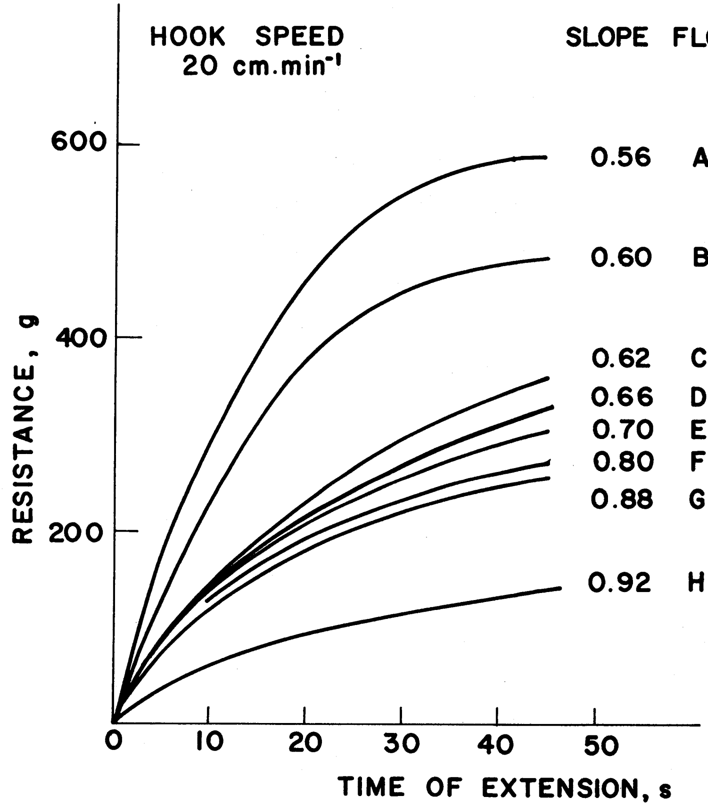

Fig. 4. Comparison of the extensigraph resistance (expressed in grams) with the slopes of the straight lines resulting from the plots of observed *vs.* calculated strains.

The calculation of the effective mass as linear function of dough length is certainly based on an approximation only. Prihoda and Bushuk (6) showed that parabola gave a closer fit, but they found a linear relationship a reasonably good approximation. In Table II, the linear equations for tested flours as obtained by this new method (referred to as "slope" method) and those obtained from the linear regression based on the directly determined masses, respectively, are summarized and compared with least square parabola equations. In Fig. 5, the correlation between data obtained by these different methods is shown for two flours which represented the extremes in the series of flours used. These two representative diagrams show clearly that in general, a better correlation was obtained for flours yielding doughs characterized by a lower resistance to extension. Data in Fig. 6 indicate to what extent the substitution of one method with another may affect the calculation of the stress values. A considerably wide spread of these values may be observed only with the "strongest" flours, especially at higher extensions.

For further calculations, the "slope" method equations were used for each individual type of flour and the mass values were substituted into equation of Muller *et al.* (1) for shear stress. The "slope" of the straight line resulting from the plot of observed against theoretical strain was also used in the calculation of the Cauchy strain ($\lambda-1$) based on the "effective" length of the dough. The modified equation was as follows:

$$\lambda-1 = \frac{[(H\cdot t)^2 + 3.52]^{.5} - l_o}{l_o} \cdot B \qquad (2)$$

where H is the hook speed in $cm\cdot s^{-1}$,

t is time in seconds,

l_o is the original length of the half

strand in cm.

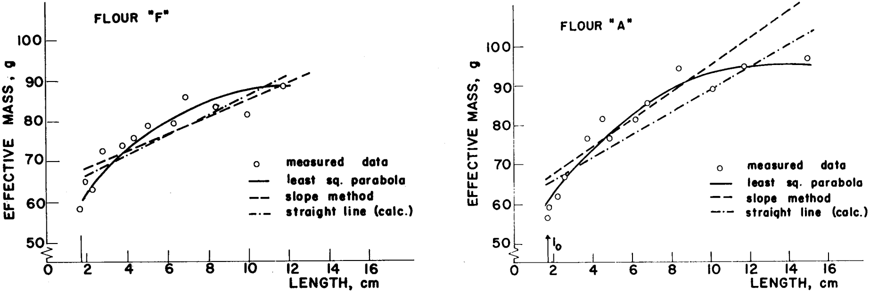

Fig. 5. Dependence of effective mass of dough strand on its length as determined by different methods. Least square parabola and straight line were calculated from the directly measured data.

The Comparison of Extensigraph and Instron Method

The stress-strain curves of doughs from some of the tested flours as obtained by both methods, are compared in Fig. 7. The Instron stress-strain curves could be interpreted easily in accordance with the present knowledge of fundamental dough rheology. The initial parts of the curves indicated the linear behavior of the dough up to stress values of approximately 150-200 kg·m^{-1}·s^{-2}. These critical stress values are in good agreement with those reported by Glucklich and Shelef (13). The nonlinear behavior at higher deformations and higher stresses was quite obvious; first the stress-softening effect was clearly predominant, being gradually replaced by strain-hardening. The curves also showed a more nonlinear relationship for less-resistant doughs, thus giving evidence of a greater effect of the dissipative (stress-relaxation) elements in the material. More linear character of the stress-strain curves of doughs with higher resistance to extension indicated their more pronounced elastic character. Lesser role of the dissipative elements was also demonstrated by a lesser effect of extension rate on the stress-strain relationship.

The stress-strain curves constructed from extensigraph data resembled the Instron stress-strain curves at higher values of strain only $(\lambda - 1 > 1.5)$. At lower deformations, the shape differed from that of the Instron curves. The separation of the curves for different hook speeds was rather difficult; in most cases the curves crossed each other. Unlike Instron curves, extensigraph curves do not start from the origin because the equation used for the calculation of stress gives a stress value due to the weight of the dough itself at zero strain. The downward

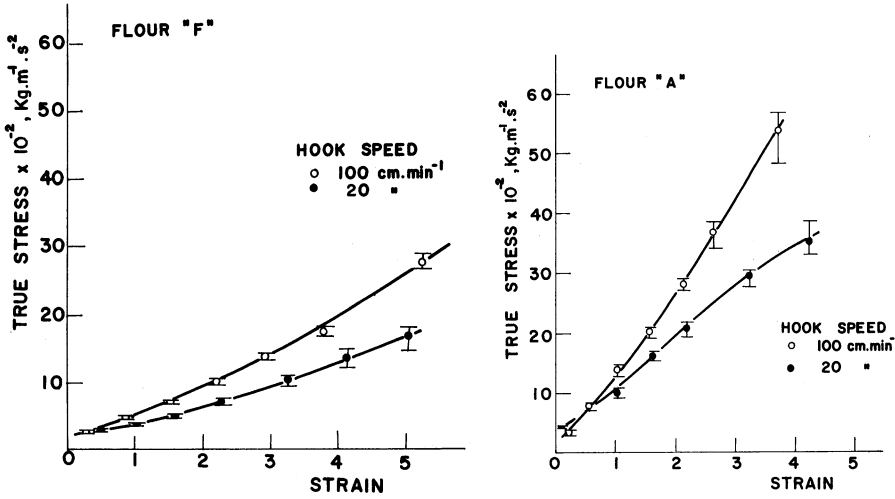

Fig. 6. Dependence of stress-strain curves on the effective mass determined by different methods. The vertical lines show the variations of values obtained by different methods. Effective masses calculated from the average equations for linear and parabolic regression are included. The points were calculated from the equations obtained by the "slope" method.

trend of the curves at low values of strain was explained by Muller *et al.* (1) as an artifact, due to the sudden large change in the angle of the dough strand with the vertical.

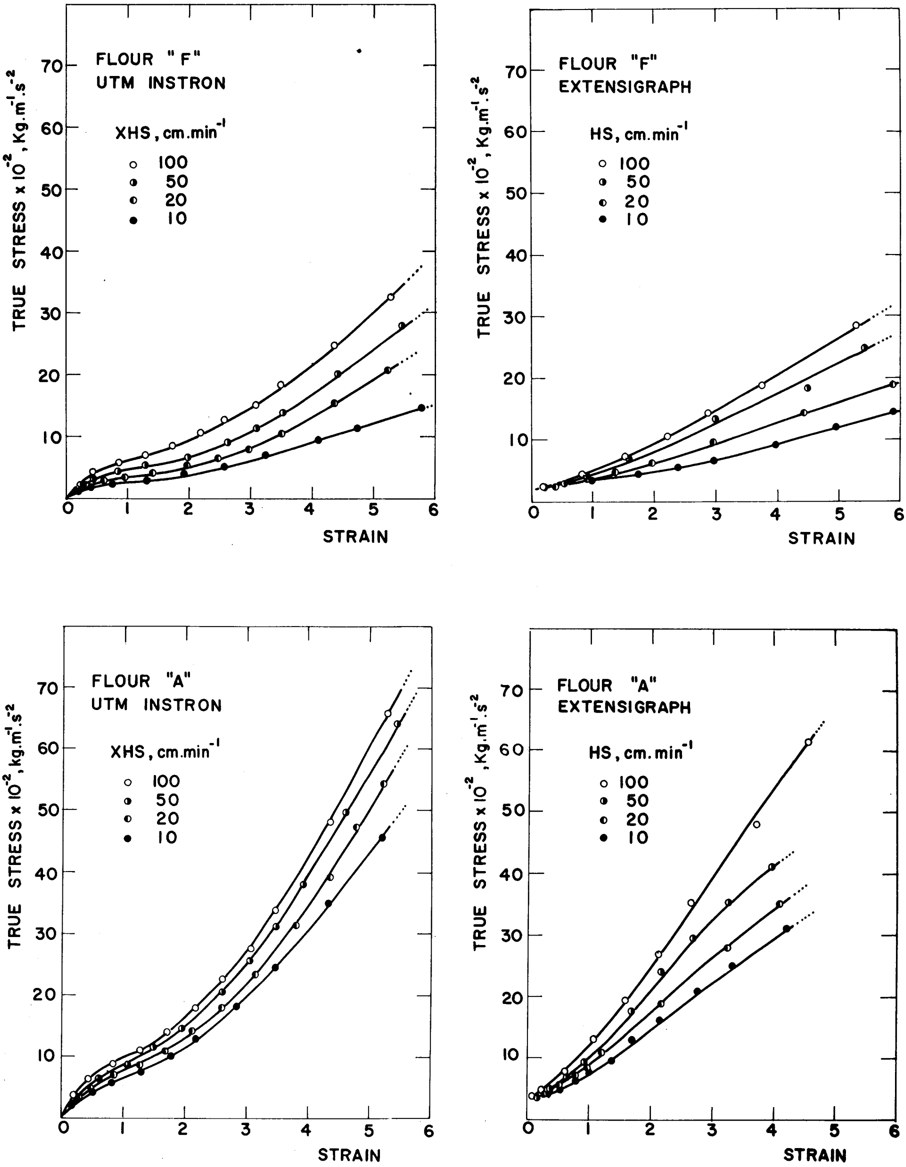

Fig. 7. Comparison of stress-strain curves obtained from extensigraph and Instron measurements for representative doughs.

Interpretation of Stress-Strain Curves

Because of a relatively high accuracy of the Instron stress-strain curves at very low extensions, the procedure based on the evaluation of the stress-strain relation in terms of isochronal modulus F(t*) and time-dependent modulus F(t) could be applied. The relation between these moduli can be expressed by the equation,

$$F(t) = F(t^*) \cdot (^t/t^*)^n \tag{3}$$

where the exponent n indicates the time dependence of the modulus. Since $n = 0$ indicates that the material is purely elastic, whereas $n = -1$ results from a purely viscous response of the material, any value within the mentioned limits characterizes a viscoelastic material. A stronger time dependence of F(t) results from a faster rate of the dissipation of the elastic strain energy which is characteristic of weaker flours. In Table I the values of n are compared with the extensigraph characteristics of the tested flours. A good correlation was found between the exponent and the "strength" of flour as determined from its maximum resistance and extensibility. The sensitivity of this method was further tested on flours treated with chemical improvers; the "strengthening" effect was quite evident from the values of the exponent n drifting closer to 0 (Tables III and IV). Similar results were obtained for flour supplemented with vital gluten, regardless of whether the dough had been prepared at constant farinograph water absorption or constant farinograph consistency (Table V). This confirmed earlier findings of Tschoegl *et al.* (12) that exponent n was independent of the water content in the dough. When the flour was mixed with wheat starch, a very small change in this exponent—more or less within the experimental error—was noticed at starch concentrations below 20% (Table VI); a more pronounced shift towards $n = -1$ was observed at higher concentrations. This leads to the conclusion that only changes in the component of the dough which is primarily responsible for its viscoelastic character, i.e., gluten complex, result in an immediate change of exponent n. A considerable decrease of this exponent at very high starch concentrations was obviously due to an excessive "diluting" effect on the continuous gluten phase.

The analysis of the 0.1-min moduli presented a more complex problem. An increase of this modulus was observed whenever a "strengthening" factor was introduced. Conversely, the addition of starch, while keeping the water content in the dough at the same level as in the control, resulted in lower values. The isochronal modulus, however, increased when starch was added under the conditions of constant farinograph consistency and decreasing water content in the dough. These results demonstrated clearly the combined effect of the intrinsic characteristics of the flour and the water content in the dough.

The discussed method was found sufficiently sensitive and fully applicable for a fundamental rheological work. However, it has to be kept in mind, that its applicability is limited to a rather narrow range of extensions and any extrapolation beyond these limits could lead to grossly erroneous results. The width of the applicable range depends very much on the type of the dough, generally becoming narrower with the increasing "strength" of flour. For the doughs used in this study, the range was not wider than $0.1 < \lambda - 1 < 1.8$.

The nature of the extensigraph stress-strain curves at these low extensions did not justify the application of the same procedure for their analysis.

TABLE III

Coefficients of Absolute Viscosity at Different Stresses Compared with the Values of Exponent n and 0.1 min Isochronal Constant Strain Rate Modulus of Chemically Treated Doughs Based on Measurements with the Instron

Sample	Isochronal Modulus F (0.1 min) $kg \cdot m^{-1} \cdot s^{-2}$	Exponent n	Coefficient of Absolute Viscosity × 10^{-6} in Poise True Stress in $kg \cdot m^{-1} \cdot s^{-2}$ 517	821	1,301	2,063	3,270	5,167
Control	499	−0.46	0.62	0.76	0.87	1.14	1.41	1.73
+ 30 ppm $KBrO_3$	758	−0.37	0.68	0.84	0.97	1.20	1.59	1.85
+ 60 ppm $KBrO_3$	803	−0.28	0.76	0.98	1.31	1.38	1.73	2.09
+ 90 ppm $KBrO_3$	840	−0.22	1.82	2.00	2.40	3.02	3.71	4.36
+120 ppm $KBrO_3$	938	−0.22	...	2.14	2.19	2.95	3.23	4.78

TABLE IV

Coefficients of Absolute Viscosity at Different Stresses Compared with the Values of Exponent n and 0.1 min Isochronal Constant Strain Rate Modulus of Chemically Treated Doughs Based on Measurements with the Instron

Sample	Isochronal Modulus F (0.1 min) $kg \cdot m^{-1} \cdot s^{-2}$	Exponent n	Coefficient of Absolute Viscosity × 10^{-6} in Poise True Stress in $kg \cdot m^{-1} \cdot s^{-2}$ 517	821	1,301	2,063	3,270	5,167
Control	499	−0.46	0.62	0.76	0.87	1.14	1.41	1.73
+ 10 ppm ADA	671	−0.29	0.83	1.00	1.35	1.82	2.13	2.45
+ 15 ppm ADA	792	−0.26	0.87	1.09	1.82	2.40	3.47	3.81
+ 20 ppm ADA	887	−0.21	1.90	2.51	3.09	3.81	4.36	4.90
+ 25 ppm ADA	1005	−0.19	2.24	2.76	3.63	5.75	6.02	6.76

TABLE V

Rheological Characteristics of Doughs Prepared from Flour Supplemented with Vital Gluten

Sample	Constant Farinograph Consistency (500 B.U.)			Constant Farinograph Water Absorption (65.9%)		
	Farinograph Water Absorption %	Isochronal Modulus F (0.1 min) $kg \cdot m^{-1} \cdot s^{-2}$	Exponent n	Farinograph Maximum Consistency B.U.	Isochronal Modulus F (0.1 min) $kg \cdot m^{-1} \cdot s^{-2}$	Exponent n
Control	65.9	499	−0.46	500	499	−0.46
+ 2% vital gluten	67.4	522	−0.44	565	594	−0.44
+ 4% vital gluten	69.4	609	−0.25	610	652	−0.28
+ 6% vital gluten	71.1	824	−0.22	650	824	−0.43

TABLE VI

Rheological Characteristics of Doughs Prepared from Mixtures of Flour and Wheat Starch

Sample	Constant Farinograph Consistency (500 B.U.)			Constant Farinograph Water Absorption (65.9%)		
	Farinograph Water Absorption %	Isochronal Modulus F (0.1 min) $kg \cdot m^{-1} \cdot s^{-2}$	Exponent n	Farinograph Maximum Consistency B.U.	Isochronal Modulus F (0.1 min) $kg \cdot m^{-1} \cdot s^{-2}$	Exponent n
Control	65.9	499	−0.46	500	499	−0.46
+ 10% starch	63.7	555	−0.46	460	475	−0.48
+ 20% starch	61.3	732	−0.48	390	396	−0.60
+ 30% starch	59.9	764	−0.51	360	300	−0.64
+ 40% starch	58.4	715	−0.50	290	...	...

Evaluation of Stress-Strain Curves at Larger Deformations

Prihoda and Bushuk (6,7) transformed extensigraph curves obtained at different rates of extension into deformation curves by plotting the length of the dough against the time of extension at arbitrarily chosen values of stress. The number of length/time data which can be read directly from the family of stress-strain curves for one value of stress is limited by the number of curves, i.e., by the number of extension rates applied. The number of these data (within the range of measured extensions and extension times) that can be obtained from diagrams like those shown in Fig. 8 is practically unlimited. These diagrams represent a double-logarithmic plot of stress values against corresponding times of extension for arbitrarily chosen values of strain at applied rates of extension. Parallel iso-strain lines show the time dependence of the stress at the indicated strains. [Identical diagrams are used as an intermediate step in the determination of the isochronal modulus by the procedure of Tschoegl *et al.* (12)]. A close correlation between the extensigraph and Instron measurements is obvious not only from the relative positions of the iso-strain lines on the coordinates, but also from their slopes, which were found in both cases, more or less identical. However, these diagrams could not be constructed from extensigraph data at low strains for reasons indicated above. Nevertheless, it was still possible to obtain a sufficient number of deformation curves like those shown in Fig. 9 from the limited extensigraph measurements. The slopes of the linear portions of these curves correspond to the coefficient of absolute viscosity (in centimeter-gram-second units) in the following way:

$$\text{Coefficient of abs viscosity} = 1/3 \cdot \frac{\text{stress}}{\text{relative velocity of extension}} \qquad (4)$$

where relative velocity of extension = slope/ l_o.

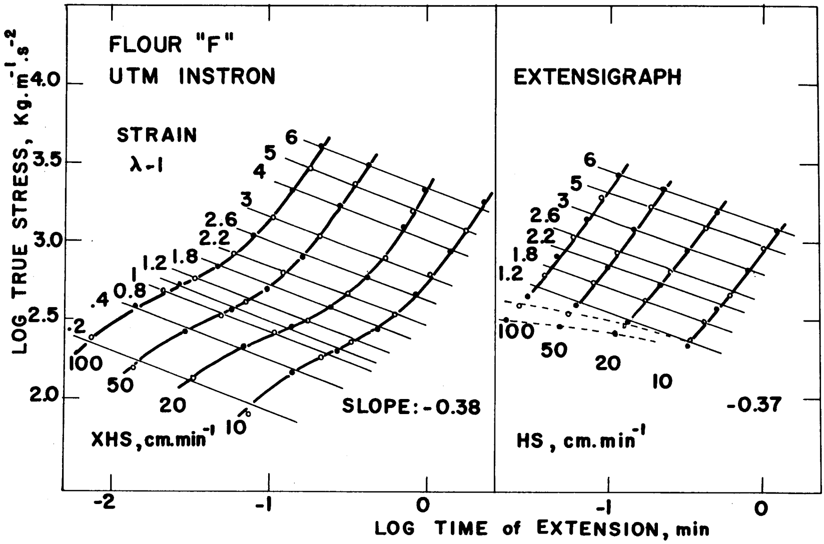

Fig. 8. Stress values obtained at four different extension rates and different values of strain plotted against time of extension on double logarithmic coordinates.

Table VII summarizes the coefficients of absolute viscosity over the stress range of 158 to 2,510 $kg{\cdot}m^{-1}{\cdot}s^{-2}$ for doughs prepared from flours, the characteristics of which are given in Table I and Fig. 4. Viscosity coefficients of dough reported in the literature lie between 10^4 to 10^6 $kg{\cdot}m^{-1}{\cdot}s^{-1}$ (14). Data in Table VII are within this range. Figure 10 shows the dependence of the coefficients of absolute viscosity on stress in double-logarithmic form for doughs prepared from flours treated with chemical improvers. These plots were essentially linear within the limits of experimental uncertainty. The intercept of the straight lines increased with increasing concentration of the improver, but the slope remained unchanged.

Rheological Parameters and Baking Performance

The comparison of the measured rheological parameters with the baking performance of the flours used gave some indications that a certain optimum range for these parameters could be established (Table I). This, however, requires more work with a larger number of flour samples with a wide spectrum of baking characteristics.

CONCLUSIONS

The results presented indicate that the effect of the flour quality on the change in the effective mass during extensigraph measurements is a very important factor in stress and strain calculations if large deformations and high stresses are involved. A method based on the measurement of the displacement of reference marks on the dough may replace the tedious technique of direct weighing of dough pieces cut out at different extensions. A reasonably good correlation was obtained between extensigraph and Instron stress-strain curves. The Instron method, however, was more sensitive and accurate at low values of strain which made it possible to evaluate the measurements in a greater number of fundamental rheological parameters. Another advantage of the Instron method is the small quantity of dough required.

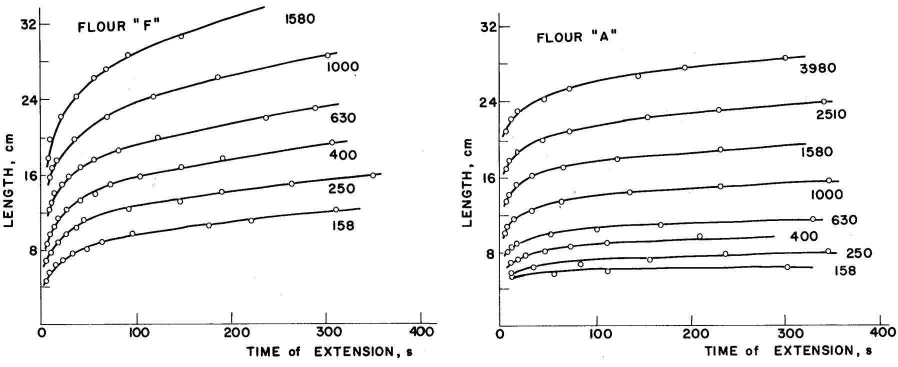

Fig. 9. Deformation curves for two representative samples of doughs based on the stress-strain data obtained by the Instron. Numbers indicate the stress in $kg{\cdot}m^{-1}{\cdot}s^{-2}$.

TABLE VII
Coefficients of Absolute Viscosity at Different Values of Stress for Doughs Prepared from Different Flours Based on Measurements with the Instron

Flour	**Coefficient of Absolute Viscosity × 10^{-5} in Poise**						
	True Stress in $kg \cdot m^{-1} \cdot s^{-2}$						
	158	**251**	**398**	**631**	**1000**	**1580**	**2510**
A	8.28	9.20	14.59	23.13	26.66	37.37	47.20
B	5.79	6.13	9.12	11.86	15.50	21.86	30.70
D	5.78	6.10	7.68	13.22	12.34	17.29	23.00
E	2.97	4.70	6.63	7.71	9.13	14.30	16.28
F	2.32	3.12	4.29	4.72	6.06	7.62	9.05
G	2.36	3.68	5.51	6.31	6.41	7.02	8.96

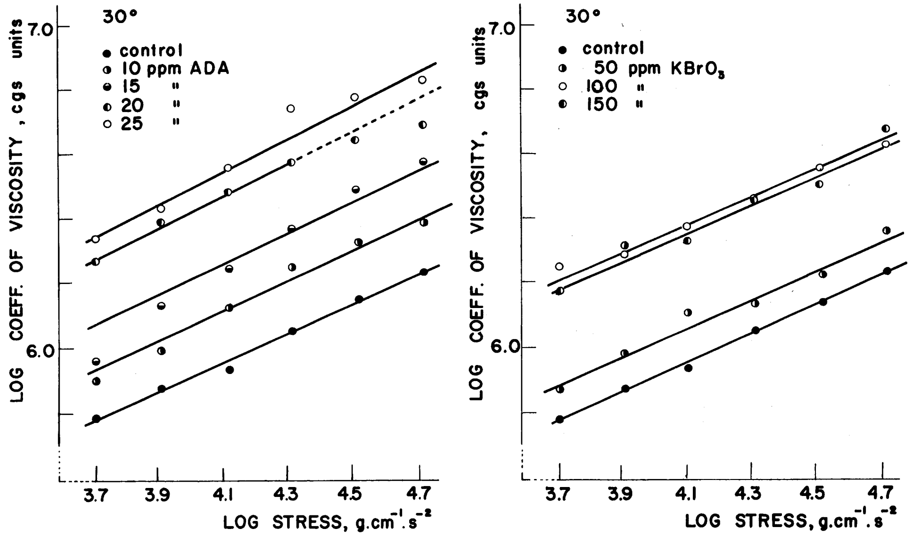

Fig. 10. Double-logarithmic plots of coefficients of absolute viscosity *vs.* stress for doughs from chemically treated flours.

Acknowledgments

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