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Stress Relaxation in Wheat Flour Doughs Following a Finite Period of Shearing. I. Qualitative Study

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ABSTRACT

The possibility of studying, with a classical cone and plate rheometer, the stress relaxation following shearing of finite and variable extent is shown. The half-relaxation time is the parameter chosen to evaluate the speed of stress relaxation; this time varies little with the extent of shearing, but is essentially dependent on the stress in the dough at the beginning of the relaxation. The effects on stress relaxation of the following factors are reviewed: flour type, dough-water content, mixing time, presence of sulfite or N-ethylmaleimide, and dough temperature; results are compared with those obtained by others. The curves representing the variation of the half-relaxation time in terms of the initial stress are similar to hyperbolas. It is suggested that the phenomenon of rapid stress relaxation can best be described as the rupture of a three-dimensional network which is at least partly constituted by secondary forces connecting the different chains.

Among all the rheological studies applied to food products, those concerned with the rheological properties of wheat flour doughs were probably the first to be published. Nevertheless, no simple idea about their behavior has been unanimously accepted, perhaps because for a long time equipment of cereal chemistry laboratories and methods of interpretation have been more or less empirical. Results obtained by various laboratories are often hard to compare and sometimes conflict. Bloksma (1-4) studied the creep of wheat flour doughs with a cone and plate rheometer built in his own laboratory and, as far as we know, his is the only recent work done with an apparatus of this kind.

However, there are several advantages in the use of the cone and plate viscometer: the positioning of the dough is easy; the shearing is nearly homogenous; and the apparatus is widely distributed. Consequently, we have adapted a viscometric method to the study of flour doughs (5). Moreover, this method appeared useful for following the storage and dissipation of elastic energy in dough by shear-stress relaxation. The study of stress relaxation has often been used for the characterization of the viscoelasticity of gluten or flour dough (6-26). Since Halton and Scott Blair's work (9,10) most authors acknowledge the significance of the relaxation rate for the assessment of the rheological properties and technological aptitudes of doughs.

Relaxation curves have usually been obtained following a stretching of gluten or dough, except for the compression tests by Webb et al. (25,26) with the Instron

TABLE I. RHEOLOGICAL CHARACTERISTICS OF THE DOUGHS

Flour Code	Protein ^a % dry matter	Chopin Alveograph				Rotovisko			
		Toughness mm.	Extensi- bility	P/L	Extension work at rupture (in 10 ³ erg/g.)	at σ max.		at γ_3	
						α_M	η'_M ^b	α_3	η'_3 ^b
f4	13.1	78.5	26.5	0.54	340.0	0.35	45	0.29	28
f5	13.2	100.0	23.5	0.88	365.0	0.33	43	0.32	26.5
f6	9.9	54.0	25.1	0.42	175.0	0.35	32	0.33	18.0
f8	10.0	52.5	24.9	0.41	175.0	0.34	30	0.33	20.5
f9	9.4	35.5	25.7	0.26	115.0	0.38	23

^aExpressed as N X 5.7.

^bIn 10³ poises.

machine. The first device used to this end, Schofield and Scott Blair's (6-10), stretched a dough cylinder floating on a mercury bath. Improved models were then used (17-19). Udy (14) modified a Jolly's balance, and Hlynka et al. (11-13,15) conceived a "relaxometer" working on a ball of dough. Some authors have recourse to devices specialized for study of flour doughs such as the Extensograph (16), the Neo-Laborograph (21-24), or a universal testing machine (20).

This work can be considered as the first attempt to study stress relaxation following simple shear.

MATERIALS AND METHODS

Flours

Five French baking flours of widely different strengths were used; their protein contents are given in Table I.

Dough Molding

All water contents are expressed as percent of dough weight. The doughs contained 0.83% NaCl (dough basis) and were obtained by mixing a constant weight of flour (250 g. dry matter) with the required amount of water in the farinograph bowl or the Chopin Alveograph kneader, according to manufacturers' instructions, except that the speed of the slow mixing blade of the farinograph was set at 75 r.p.m. After mixing, the dough was allowed to rest 20 min. in a closed plastic container at about 20°C.

Viscometry

The Rotovisko apparatus (Haake, Type BV) was used with a speed reducer (reducer 100), the MK 5000 measuring head, and the PK 1 cone (radius, 2.8 cm., angle, $5.471 \cdot 10^{-3}$ radian). The shear rate between cone and plate ($\dot{\gamma}$, in sec.⁻¹) is directly proportional to the speed of rotation of the cone (n , in turns per sec.):

$$\dot{\gamma} = 1148.27 \times n \quad (1)$$

The apparatus can work with 10 different shear rates (from 0.574 sec.⁻¹ to 93.01 sec.⁻¹). The torque applied to the cone, which is directly proportional to the shear

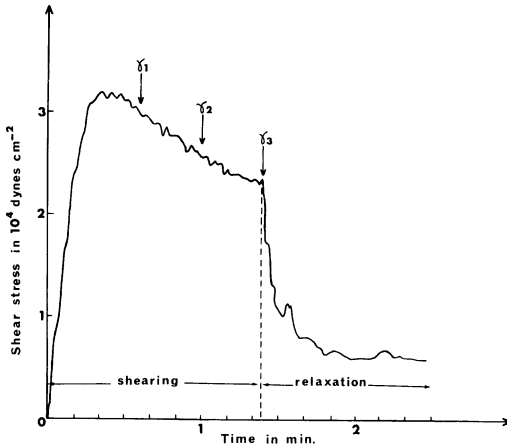


Fig. 1. Variation of shear stress with time. Arrows correspond to three values of the shear strain (γ_1 , γ_2 , and γ_3). Experimental conditions: f6-40%-6A-23.8° C. (flour, f6; water content, 40%; mixing, 6 min. in the Alveograph; temperature of dough in the viscometer, 23.8° C.).

stress (σ in dynes cm.⁻²), is measured with a galvanometer giving a lecture S:

$$\sigma = 9254.8 \times S \tag{2}$$

In fact the signal (S) operates on a potentiometer (MECI Speedomax) in view of having the variation of σ with the time.

The use of this cone-plate viscometer for studying wheat flour doughs has been described previously (5), and it must be said that, in such a case, equations 1 and 2 are approximate. Among other things they assume there is no interval between the apex of the cone and the plate, and no slippage on the surfaces.

The temperature of the plate is regulated by circulation of water. When the temperature is too different from the one in the laboratory, it is estimated by inserting a thermocouple in the dough, between cone and plate. Consequently the temperatures indicated are those of the dough during the measurement.

A piece of dough weighing 1 to 2 g. was compressed between cone and plate by means of an additional spring. The spring was withdrawn after 1 min., the surplus of dough cut off, and the shear stress at a specified shear rate recorded: a curve as shown in Fig. 1 was obtained. Such curves always have a maximum, whether sharp-pointed or plateaulike. Thus the values refer to the maximum shear stress and to three definite shear strains γ_1 (123.6), γ_2 (206.0), and γ_3 (288.3)(see Fig. 1) with η being the apparent viscosity ($\eta = \frac{\sigma}{\dot{\gamma}}$), the results can be expressed following a power-law:

$$\log \eta = (\alpha - 1) \log \dot{\gamma} + \log \eta' \tag{3}$$

The values of η' depend on whether the measurements refer to the maximum shear stress or to shear strains γ_1 , γ_2 , or γ_3 . On the other hand, the corresponding values of α are not significantly different from each other.

The rheological parameters of doughs prepared with various flours under identical conditions (water content = 43.2%, mixing 6 min. in the Alveograph

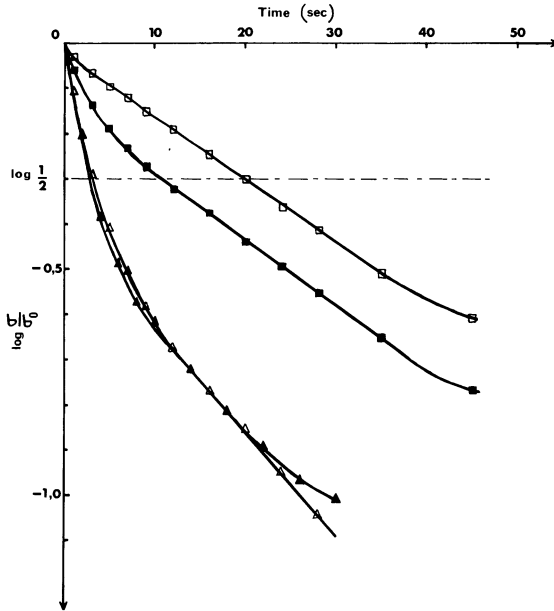


Fig. 2. Relaxation curves represented following the Maxwell model. Experimental conditions: f5-42%-6A-24.7° C. □, shear strain γ_3 , initial stress $\sigma_0 = 2.4 \times 10^4$ dynes cm.^{-2} ; ■, γ_3 , $\sigma_0 = 4.1 \times 10^4$ dynes cm.^{-2} ; ▲, γ_3 , $\sigma_0 = 8.3 \times 10^4$ dynes cm.^{-2} ; △, γ_2 , $\sigma_0 = 9.5 \times 10^4$ dynes cm.^{-2}

kneader) are given in Table I, as well as the extensimetric characteristics obtained with the Chopin Alveograph. Generally speaking, the stronger the flour, the higher the value of η' .

RESULTS

Study of the Shear Stress Relaxation

By rapidly stopping the cone with the help of the gearbox one can follow the relaxation of the shear stress, at a fixed shear strain γ_1 , γ_2 , or γ_3 (see Fig. 1). In fact, the dough is still slowly sheared as the torque-measuring spring slackens. However, Schremp et al. (27) have shown that, for time intervals much longer than a critical time $t_c = (b \cdot \eta)/c$, the relaxation is essentially at constant strain; b is an apparatus constant and c the torsional stiffness of the cone suspension, and here the final result is: $t_c = 3.4 \cdot 10^{-8} \times \eta$. It can be seen that with $\eta = 10^5$ poises and 10^6 poises it follows values of the critical time equal to 3.4×10^{-3} sec. and 3.4×10^{-2} sec., respectively; as a general rule the whole of the relaxation curve can be considered as obtained at a constant strain.

Note that these relaxation tests were done after a time of shearing variable with $\dot{\gamma}$ and γ (γ_1 , γ_2 , or γ_3) from 1.3 to 502.3 sec. This finite and, in most cases, rather long period of shearing differs with the generally applied procedure in which the deformation preceding relaxation is established quasi-instantaneously.

If the relaxation follows a Maxwell law with a relaxation time $\tau = \eta/G$, G being the shear modulus, we have:

$$\log \sigma/\sigma_0 = -t/\tau \quad (4)$$

Some relaxation curves are plotted in Fig. 2 following equation 4. As is already well known, the stress relaxation in wheat flour dough is not Maxwellian. Therefore, one can think that the shape of the relaxation curve depends upon the "mechanical history" of the material: shear rate $\dot{\gamma}$ and shear strain γ . The half-relaxation time ($t_{1/2}$), that is, the time required for a decrease of the stress from σ_0 to $\sigma_0/2$, is chosen as a parameter because it is quickly and easily evaluated. For each shear rate $\dot{\gamma}$ and each shear strain (γ_1, γ_2 , or γ_3) there is a σ_0 and a $t_{1/2}$. Some typical results are shown in Fig. 3. The following comments can be made:

1. All the points lie rather closely on the same curve and, at least in the present range of values, the half-relaxation time depends much more on the initial shear stress than on the shear strain, which seems to have only second-order effects on the relaxation.

2. The higher the initial stress, the faster the relaxation rate. This may explain why the half-relaxation times obtained were shorter than those corresponding to relaxation after stretching: in the latter case the initial stress was lower by a factor of about 10. Schofield and Scott Blair (6) were the first to claim that the relaxation rate increases when the initial stress increases or, which is equivalent, the deformation rate increases (see equation 3). This property is characteristic for stress relaxation following non-Newtonian flow (Ferry, 28, p. 57); in that case the use of a linear model is inappropriate and we cannot describe the relaxation curves by means of a generalized Maxwell body, which was previously used for dough (12,15,16,19) or gluten (20).

There is often no clear-cut distinction between the effects of stress and strain on the relaxation rate (6,19,21), although Cunningham et al. (12) have pointed out the influence of the initial stress on the relaxation curve. Generally, the relaxation tests on wheat flour doughs or gluten are performed after a constant stretching (12,15,16,20-24) and, hence, a variable initial stress. An exception is the work of

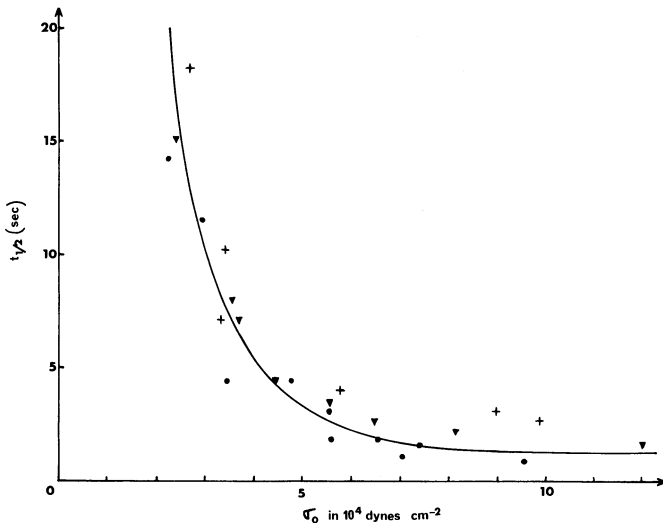


Fig. 3. Half-relaxation curve. Experimental conditions: f4-43.2%-6A-24.7°C.; +, ∇ , \bullet = $\gamma_1, \gamma_2, \gamma_3$, respectively.

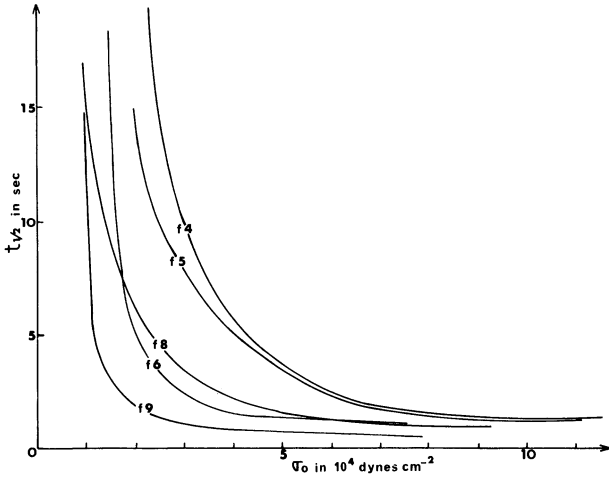


Fig. 4. Role of flour strength on relaxation. Experimental conditions: flour as indicated-43.2%-6A-24.7°C.

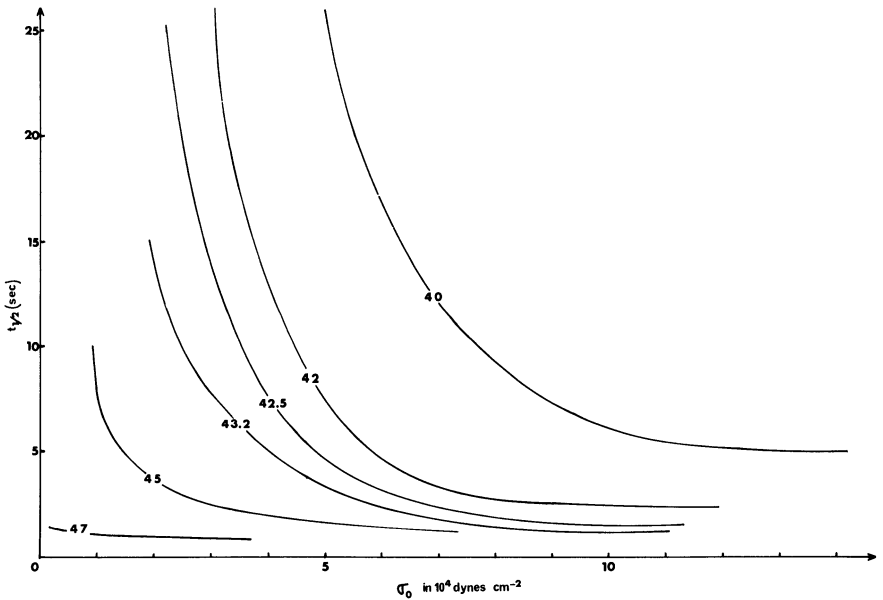


Fig. 5. Role of water content. Experimental conditions: f5-water content as indicated-6A-24.7°C.; shear strains γ_2 and γ_3 .

Heaps et al. (25,26) where the relaxation times are taken from a fixed compressive load.

Subsequently, half-relaxation times were used in this work for a qualitative evaluation of various factors on the relaxation rate. All the measures were done at

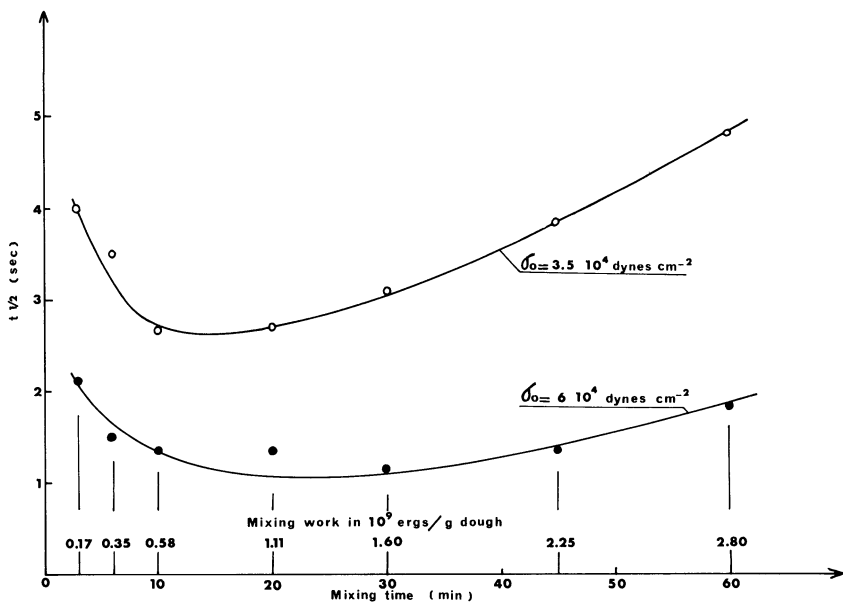


Fig. 6. Variation of the half-relaxation times corresponding to two values to σ_0 with the length of mixing in the farinograph. Experimental conditions: f6-42%-21.5° C.; shear strain γ_3 .

definite shear strains (γ_3 , γ_2 , and sometimes γ_1) in order to eliminate noteworthy effects due to the shear strain. The comparisons between half-relaxation times must be done at the same initial stress.

Effect of Various Parameters on the Relaxation

For the sake of clarity the experimental points in the σ_0 , $t_{1/2}$ curves are omitted.

Flour Type. The five flours in Table I can be ranked in three groups: high baking strength (f4 and f5), weak (f6 and f8) and very weak (f9). The corresponding doughs were prepared in an identical manner and particularly with the same water content. The half-relaxation times of these doughs are given in Fig. 4. Apparently the stronger the flour, the longer the half-relaxation times. Opinions on this subject differ widely. Halton and Scott Blair (9,10), as well as Webb et al. (26), with 14 flours, come to the conclusion that the relaxation times of doughs from strong flours are higher than those from weak flours. Shelef and Bousso (19) arrived at exactly the opposite conclusion with four flours, and Hlynka and Anderson (11) expressed the opinion that relaxation curves do not depend on flour type. However, these results are hard to compare with the present ones, because the doughs were never prepared at a fixed water content but, in most cases, at a constant consistency with the aim of having conditions close to industrial practice. This may explain, at least in part, the discrepancies.

Water Content. The half-relaxation times of doughs from the same flour, with water contents between 40 and 47%, are shown in Fig. 5. The effect of water content is rather sharp and, as said before, must be separated from that of flour

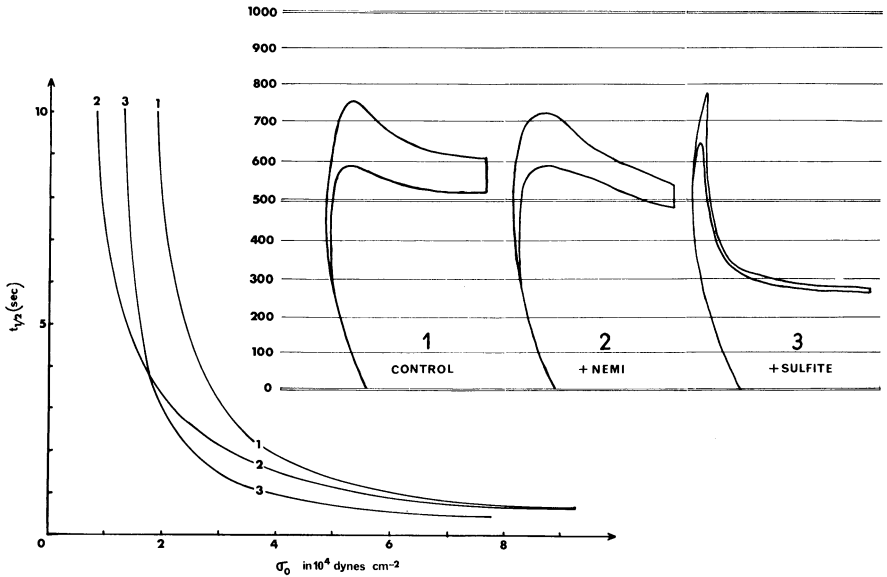


Fig. 7. Effect of two additives. Experimental conditions: f6-42%-8F (50-g. bowl)-24.7°C.; shear strains γ_2 and γ_3 . Additives (weight flour basis), NEMI, 140 p.p.m., sulfite, 0.22%.

strength. This result is in accord with previous ones: an increase in water content is said to bring about an increase in the stress-relaxation rates (10), or a decrease in relaxation times (11,21) and in relaxation moduli (12,13,15) of wheat flour doughs. In contrast, Barney et al. (20) assess the stress-relaxation rate in gluten to be independent of water content.

Mixing Time. Mixing times in the farinograph bowl varying from 3 to 60 min. were used. Expressed in term of work input, the corresponding scale is about the same as in Webb et al. (26). The various (σ_0 , $t_{1/2}$) curves are not far from each other and the half-relaxation times for two values of σ_0 are given in Fig. 6; the relaxation times are small and not greatly influenced by the mixing time, but the curves show a definite minimum. In Webb's work two types of response were noted when the mixing time increased: either a maximum or a slow decrease in relaxation time followed after compression of these doughs. The rate of work input, which is an important parameter for the rheological properties of doughs (29), is about three to four times smaller than in Webb's work. On the other hand, it is surprising that changes in mixing time, which were shown to produce great effects on the shearing characteristics of doughs (5), have so little influence on the relaxation curves.

Influence of Sodium Sulfite and N-Ethylmaleimide (NEMI). The 50-g. farinograph bowl was used and sodium sulfite or NEMI was introduced in water from the start of mixing. Mixing curves with the corresponding half-relaxation times are shown in Fig. 7. The effects of sulfite or NEMI are essentially the same: a decrease in the relaxation times. This result can be an indication that the sulfhydryl groups are not directly participating in the stress-relaxation phenomenon, at least in the high shear-stress range. There are some differences between the action of the two additives: sulfite increases the relaxation rate whatever the value of σ_0 , and

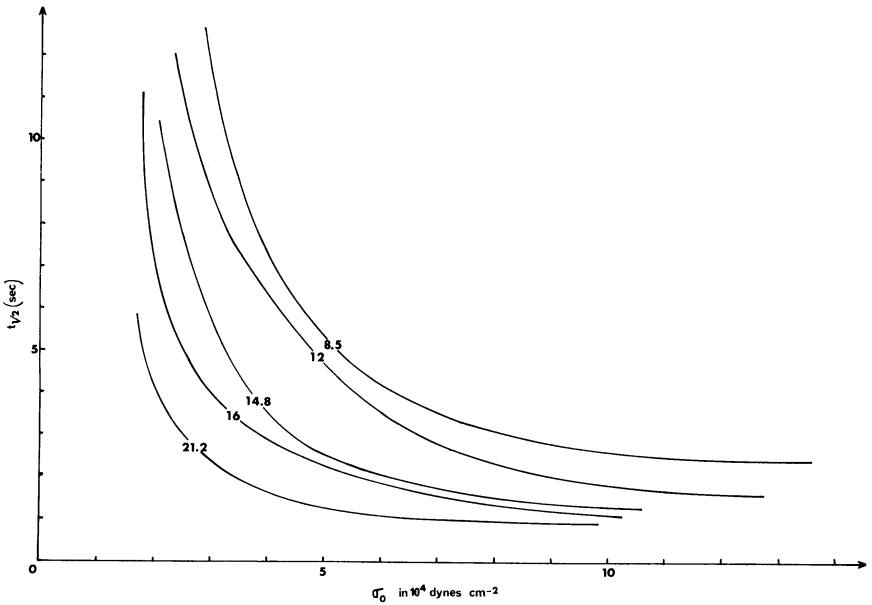


Fig. 8. Role of dough temperature. Experimental conditions: f6-43.2%-6F-temperature in °C, as indicated.

NEMI has a more pronounced effect when σ_0 is small.

Dough Temperature. As shown in Fig. 8 an increase in dough temperature provokes an increase in the relaxation rate, whatever the value of σ_0 , and a variation between 1° and 2°C. can be easily detected; this behavior has been previously established (9,10,15,21).

DISCUSSION

The use of a cone-plate viscometer enables one to study the shear-stress relaxation in wheat flour dough. In the range of strain and stress covered in the present work (γ from 123 to 288; σ_0 from 0.5 to 14×10^4 dynes cm^{-2} , approximately) the initial stress appears to be the major parameter; thus, it seems of prime importance to analyze the stress relaxation after deformation at a definite stress rather than a definite strain.

Some factors have a prominent influence on the half-relaxation times: flour type, water content, and temperature. So one can expect that the application of this method may prove useful for obtaining an index of dough quality, and also as a tool for a better insight into the stress-relaxation mechanism; such a development needs some quantitative interpretation of the course of stress relaxation. We want to draw attention to a means toward this end: generally the half-relaxation curves look like hyperbolas. Figure 9 shows that $t_{1/2}^{-1}$ is varying about in proportion with σ_0 . So the equation of the half-relaxation curves can be written as:

$$t_{1/2} = \frac{A}{\sigma_0 - \sigma_{lim}} \tag{5}$$

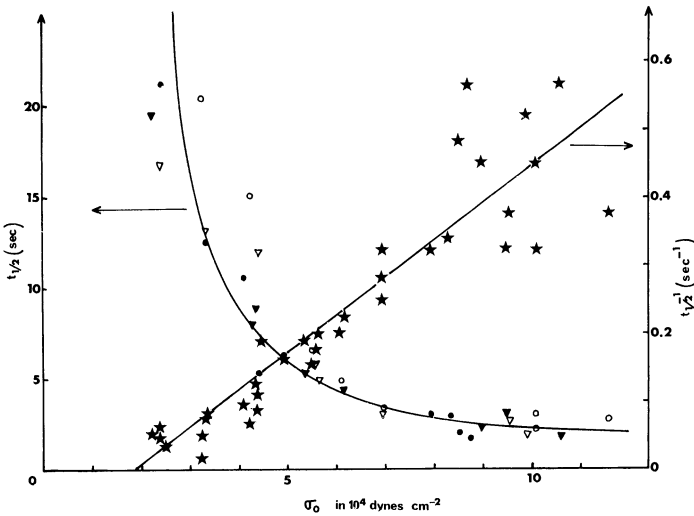


Fig. 9. Fitting of the half-relaxation curve to a hyperbola. Right hand scale: $\star = t_{1/2}^{-1}$ straight line is the least squares one. Left hand scale: $t_{1/2}$, the equation of the hyperbola is obtained from the straight line (see text). Experimental conditions, two separate runs (open and closed symbols): f5-42%-6A-24.7°C; shear strains γ_2 ($\nabla, \blacktriangledown$) and γ_3 (\circ, \bullet).

The curve corresponding to equation 5 with $A = 1.83 \cdot 10^5$ poises and $\sigma_{\text{lim}} = 1.9 \times 10^4$ dynes cm^{-2} is shown in Fig. 9 and, taking into account the experimental uncertainties, the agreement is rather good. The simplest Maxwellian model with the same half-relaxation time should have a viscosity η such that $\eta = A \times K \times L \eta_2$, and a shear modulus G such that $G = (\sigma_0 - \sigma_{\text{lim}}) \times K$, K being an indefinite constant. When $\sigma_0 = \sigma_{\text{lim}}$, equation 5 shows that $t_{1/2}$ might become infinite due to a zero value of the "equivalent modulus" G . However, close inspection of Fig. 9 seems to indicate some trend towards a deviation from equation 5 in the vicinity of σ_{lim} . In fact, this equation must be considered as purely empirical. We know stress relaxation in wheat flour doughs is not Maxwellian: the relaxation time increases during relaxation (see Fig. 2). The two simplest mechanisms for describing this effect with a Maxwell model are: (i) a decreasing modulus during relaxation, and (ii) an increasing viscosity during relaxation, and a linear elasticity. Mechanism (i) can be put in line with the variability of Young's modulus, observed in recoveries experiments by Lerchenthal (30) and attributed to a change in the number of elastic elements available per unit volume. Mechanism (ii) was suggested by Schofield and Scott Blair (6,8). Viscometric measurements with a cone-plate apparatus strongly suggest nonlinear viscous properties (5). Moreover, the existence of quasilinear elastic properties can be deduced from the creep experiments in shear of Bloksma (3) and has been shown by creep in simple tension by Smith and Tschogel (31).

Therefore, the next step for studying stress relaxation in doughs must be to test quantitatively a simple rheological model. It is expected that such a model can be useful for explaining at the molecular level the origin of the elastic properties of wheat flour doughs and, particularly, for deciding whether they are under the direct

dependence of disulfide bonds. The question was answered positively by Bloksma (4), who further refined his chemo-rheological interpretation of elasticity; the recent theories of Jones and Carnegie (32) and Ewart (33) offer another explanation. Jones and Carnegie assume that most of the disulfide bonds are intrachains and play only an indirect role in interchain bonding, which is predominantly hydrophobic or polar; Ewart claims that the disulfide bonds are exclusively engaged in linear chains and the three-dimensional network necessary to explain the elasticity of dough is obtained by entanglements via secondary forces. The most important point is that in the two hypotheses an external stress has to be supported, totally or at least in part, by an elastic network of chains connected by secondary forces. Such a picture implies that the stress relaxation is then no more under the direct dependence on disulfide-sulphydryl interchanges; this was already pointed out by Lastity (23,24) for stress relaxation in chemically modified gluten.

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