AN INVESTIGATION INTO THE RHEOLOGICAL PROPERTIES OF FLOUR DOUGH. STUDIES IN SHEAR AND COMPRESSION[1]

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ABSTRACT

Doughs made from durum wheat and common wheat flours exhibited similar rheological behavior, namely instantaneous elastic strain, delayed elastic strain, and viscous flow.

At ordinary stresses, durum dough was more rigid and more viscous than vulgare dough. At higher stresses it was still more viscous, but its rigidity became equal to that of vulgare. Both kinds exhibited nonlinearity, more pronounced for viscous flow in durum and for elastic strain in vulgare. The nonlinearity was a result of both strain-hardening and stress-softening.

In most rheological studies, to date, only the behavior of doughs suitable for breadmaking has been investigated. Schofield and Scott Blair (10–13), using the extensimeter, were the first to establish the Maxwellian character of the material. Later, Halton and Scott Blair (5,6,7) and Halton (3,4) found physical interpretations for the domestic terms "spring" and "shortness," referring to properties related to a high relaxation time for the former and to brittleness for the latter. Reiner (9) interpreted Schofield's and Scott Blair's findings in terms of a rheological model.

Hlynka, Anderson, and Cunningham (1,2,8) dealt mainly with the relaxation process of stretched doughs at constant extension, emphasizing the importance of this process in determining bread quality. Their investigation showed the existence of a generalized Maxwell body with a spectrum of relaxation times.

The present work was undertaken in order to extend these studies to include doughs not commonly used for breadmaking, with a view to bringing out the significant properties required for the baking

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process, as well as to subsequent improvement of the properties of doughs of poor baking quality.

One variety of the vulgare class was studied as representative of doughs suitable for baking, and one variety of the durum class as representative of unsuitable doughs. Conclusions were confined to these two particular varieties.

The work was conducted on lines similar to those of the above-mentioned investigations, and similar instruments were used.

Materials and Methods

Materials. Flour-water doughs were prepared from two flours, one a commercial flour obtained from a mixture of 80% hard red winter wheat and 20% soft wheat; the other, a durum flour obtained experimentally from locally grown durum wheat. The protein and ash contents of the vulgare wheat flour were 13 and 0.69%; those of the durum wheat flour were 12.5 and 1.2%, respectively. (All values calculated to 13% moisture basis.)

The absorption employed in preparing the vulgare and durum doughs was 54 and 64%, respectively; these values yielded doughs with the same rate of extrusion by the Halton device (4).

Preparation of Samples. The doughs were prepared in the small mixing unit of a Brabender Farinograph of 50-g. flour capacity. The slower arm of the farinograph rotated at 65 r.p.m. The water was added to the flour drop by drop at a constant rate after 2.5 minutes of mixing. The dough was placed in an extruder, where it was allowed 10 minutes for relaxation and then loaded by means of a piston and a 40-kg. weight. The dough cylinder extruded in the first minute was discarded and that extruded in the second minute was weighed; if the weight had reached about 5 g. the batch was considered to be of the desired consistency and the cylinder extruded in the third minute was tested.

The cylindrical specimens showed swelling upon emergence from the extruder, their diameter increasing from the original 0.60 cm. of the extruder to 0.73 cm. in durum and 0.80 cm. in vulgare.

Two paper cuffs to which the dough readily adhered were attached to the respective ends of the specimen immediately after extrusion. The part of the specimen to be in contact with mercury in the apparatus (see below) was coated with a thin film of glycerin to minimize friction. Apparatus, flour, and water were maintained at a constant temperature of 23.5°C. throughout the experiment.

Apparatus and Experiments. Figure 1 gives a schematic description

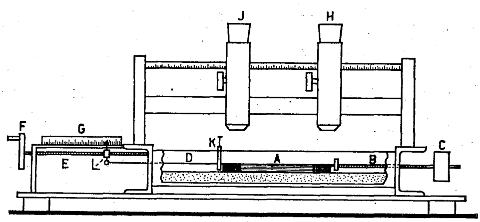

Fig. 1. Extensimeter (not to scale). A, dough cylinder; B, E, threaded rods; C, wheel; D, rubber band; F, wheel; G, scale; H, J, traveling microscopes; K, stop; L, nut.

of the extensimeter. A dough cylinder, *A,* about 4 cm. long, is floated on mercury in a metal bath, with one cuff rigidly fixed to a threaded rod, *B*. By rotating a wheel, *C,* the dough cylinder is shifted axially. The other cuff is attached to one end of a thin rubber strand, *D,* the other end of which is attached, in turn, to a nut, *L,* with an indicator, moving along another threaded rod, *E*. The rubber strand can be stretched to a desired length by rotating a graduated wheel, *F* (readings to 0.025 cm.), the position of the indicator being read on a scale, *G,* graduated in half-millimeters. Two traveling microscopes *H* and *J* are trained on the cuffs, permitting readings to 0.001 cm.; an additional scale on the objective of microscope *J,* graduated in 0.004 cm., permits rapid displacement readings without shifting the microscope. The stop *K* enables an instantaneous transference of load from the rubber strand to the specimen. The extension of the rubber strand (previously calibrated by means of weights, its elastic properties having been found satisfactory) is a direct measure of the stress.

Four measurements were made on the extensimeter, namely, instantaneous elasticity, deformation-time relations at constant load, stress-relaxation, and stress-deformation relations at constant rate of stress.

Instantaneous Elasticity. An instantaneous load was applied by stretching the rubber strand with the stop *K* held in position and subsequently released. The instantaneous elongation was noted through the microscope, *J*. The load was then removed and the instantaneous recovered elongation noted in turn. The latter is a measure of the instantaneous elasticity.

Deformation-Time Relations at Constant Load. A given load was applied as before, except that the load was kept constant by adjusting wheel *C* after the stop was released. Elongation readings were noted through microscope *H* at given time intervals.

Stress Relaxation. In this case, the instantaneous elongation was kept constant by adjusting wheel *F* so as to relax the load. Load readings were noted on scale *G* at given time intervals.

Stress-Deformation Relations at Constant Rate of Stress. Nut *L* was adjusted so that rubber strand *D* was just free of stress. Wheel *C* was then rotated so that both ends of the specimen were moved to the right, the left-hand one at a constant rate (which loaded the specimen at a constant rate). Readings were noted through both miscroscopes, that of microscope *J* indicating load, and their difference indicating the elongation of the specimen.

A volumetric test consisted in measuring the *bulk elasticity*. The sample was extruded as before, but with a diameter of 5 cm. It was enclosed in a rubber bag and placed inside a transparent cell filled with glycerin (see Fig. 2). By applying varying pressures to the glycerin,

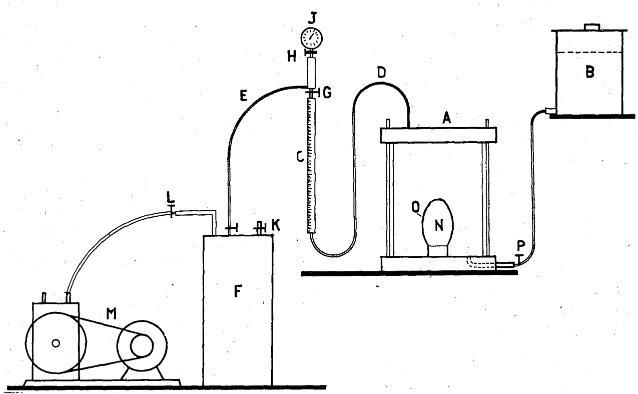

Fig. 2. Volumetric compression apparatus (not to scale). A, cylindrical cell; B, glycerin container; C, buret; D, E, rigid tubes; F, air reservoir; G, H, K, L, P, stopcocks; J, pressure gage; M, pump; N, dough cylinder; O, rubber bag.

varying volumetric compressions of the sample ensued, which were measured by the drop in glycerin level in the buret, allowing for volume changes of the system.

Definitions of Rheological Terms and Quantities

Symbols

A = cross-sectional area
E = modulus of elasticity
e = extension
e = base of natural logarithms
l = length
P = load
p = isotropic pressure
T = time constant
t = time
V = volume
Δ = increment
ε = strain (elastic extension)
ϵ_v = cubical dilation

η = coefficient of viscosity
ϑ = yield point
κ — bulk modulus
λ = coefficient of viscous traction
ν = Poisson's ratio
σ = tensile stress

Subscripts

d, delayed
o, original
rel, relaxation
ret, retardation
s, solid

Cylindrical specimens of original length l_0 and cross section A_0 are deformed by stretching to various values of l and A. Elongation Δl is given by

$$\Delta l = l - l_0 \tag{1}$$

and *extension* is defined by the relative deformation

$$e = \Delta l / l_0 \tag{2}$$

With the specimen subjected to pull P, *tensile stress* is defined by

$$\sigma = P/A \tag{3}$$

On unloading, part of the elongation, termed "elastic," is recovered. *Elastic extension* or *strain* is denoted by ϵ. In the relationship

$$\epsilon = \sigma/E \tag{4}$$

the parameter E is called *modulus of elasticity*. With E constant, equation 4 represents a straight line passing through the origin. As the cylinder is stretched through the action of the tensile stress σ, it undergoes lateral contraction; the ratio of this lateral contraction and the extension ϵ, known as Poisson's ratio ν, is given by

$$\nu = \tfrac{1}{2}(1 - E/3\kappa) \tag{5}$$

where κ is the bulk modulus, defined further down. If the contraction on unloading is equal to the extension on loading, the process is purely elastic. If the modulus of elasticity drops with increasing stress, one speaks of *stress-softening;* its increase with strain is termed *strain-hardening*. When a cylinder is extended, there is a tendency for the weaker section to show greater deformation accompanied by additional contraction; this produces stress concentrations, and unless the process is arrested by work hardening it will lead to still greater deformation, and eventually to rupture. Uneven contraction of this kind is called *necking*.

Through elongation from l_0 to l, the cross-sectional area is reduced from A_0 to A. If the volume is assumed constant, we have

$$A = A_0 \frac{l_0}{l} \tag{6}$$

and from equations 3 and 2

$$\sigma = \frac{P}{A_0}\frac{l}{l_0} = \frac{P}{A_0}\frac{l}{\Delta l}e \tag{7}$$

Therefore, if we measure extension by $\Delta l/l$ instead of $\Delta l/l_0$, we may express σ as P/A_0. This procedure permits study of the case of constant stress σ, by keeping pull P constant.

Elastic recovery can be *instantaneous* (at acoustical speed) or *delayed*. It takes theoretically infinite, but practically some finite, time for the delayed elastic strain to be completed, in which case the time required for the strain to be reduced to its e^{th} part is the *time of re-*

tardation (T_{ret}). In general, part of the elongation is irrecoverable, irrespective of the time lapse. When this permanent part appears under the smallest load, increases continuously with time, and its rate of deformation increases with the applied stress, it is referred to as *viscous flow*. If the permanent deformation appears only after a certain stress has been exceeded, it is referred to as plastic deformation, with the limiting stress defining the *yield point,* usually designated by ϑ. In linear viscous flow the extension or relative deformation per unit time is proportional to the stress. We have, in analogy to equation 4,

$$\frac{e}{t}=\frac{\sigma}{\lambda} \tag{8}$$

where the parameter λ is called the coefficient of viscous traction measured in poises, and equal to three times the coefficient of viscosity as commonly defined.

When the elastic strain is not instantaneous but delayed, this delay is due to another kind of viscosity, known as solid viscosity (λ_s) to distinguish it from the first kind, which may then be termed liquid viscosity. The corresponding time of retardation is

$$T_{ret} = \lambda_s/E_d \tag{9}$$

where E_d is the modulus of the delayed elastic response.

If an elastic deformation is produced in a viscoelastic material by the application of a given stress with provision for maintaining the deformation constant, the stress will gradually decrease. The phenomenon is known as *stress-relaxation,* and the time T for the stress to be reduced to its e^{th} part is the *time of relaxation* (T_{rel}). In analogy to equation 9 we have

$$T_{rel} = \lambda/E \tag{10}$$

A pressure acting upon a body in a direction normal to its surface and with equal magnitude in all directions is called *isotropic pressure,* denoted by p. Hydrostatic pressure is a typical example. Such pressure produces a relative reduction in volume, or negative cubical dilatation ϵ_v, where

$$\epsilon_v = \Delta V/V_o \tag{11}$$

For defining compressibility, an *elastic bulk modulus* κ is used, where

$$\kappa = p/\epsilon_v \tag{12}$$

Results

Instantaneous Elasticity. The instantaneous relative deformation of both doughs as a function of the stress is shown in Fig. 3. The loading and unloading curves coincided, the slope of the curve at the origin being a measure of the modulus of elasticity at zero stress. The curves

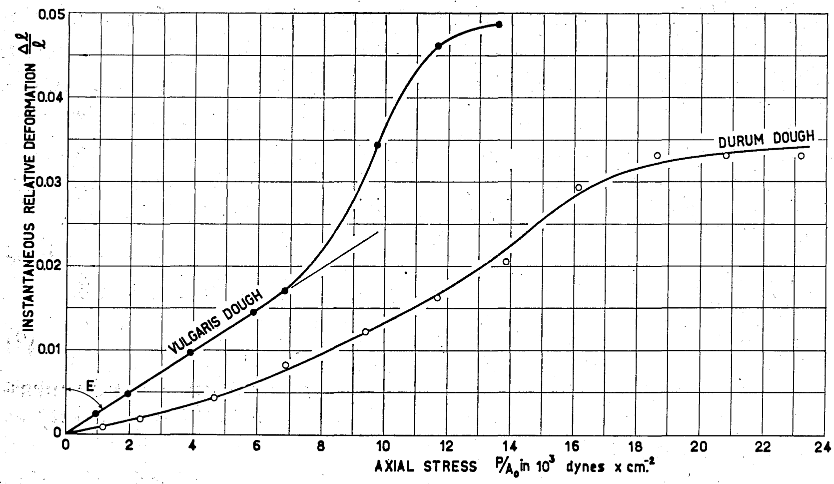

Fig. 3. Instantaneous elasticity. Each point represents deformation both on loading and unloading, the two branches being coincident.

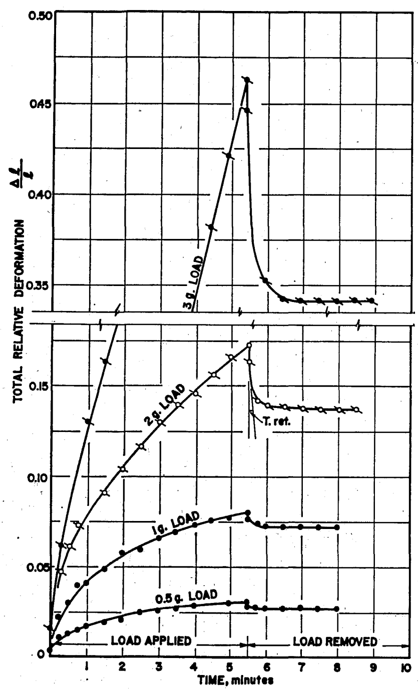

Fig. 4. Deformation-time relations at constant load, for vulgare dough. Each curve is a mean of 4 to 5 tests.

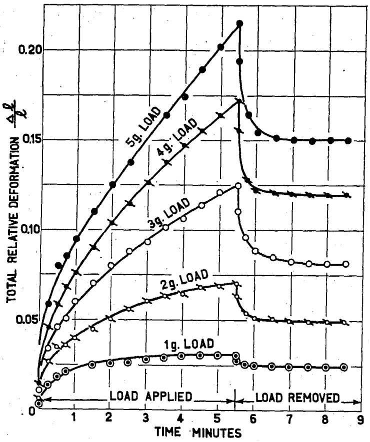

Fig. 5. Deformation-time relations at constant load, for durum dough. Each curve is a mean of 4 to 5 tests.

of both doughs were linear at low stresses, and nonlinear as stress and deformation increased. This nonlinearity, more pronounced in the vulgare dough, was apparently due in both cases to a combination of effects: stress-softening, predominant in the early stages, resulted in a

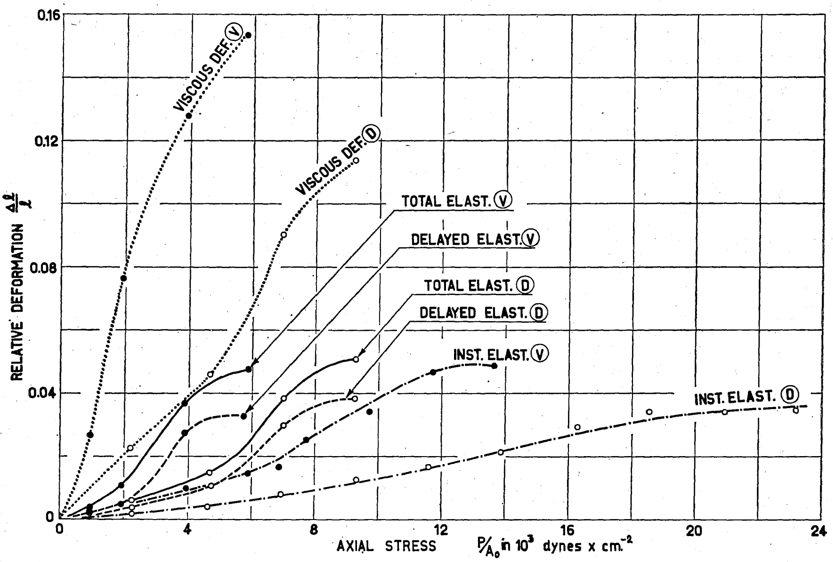

Fig. 6. Three types of deformation vs. stress in durum and vulgare doughs. The viscous deformation is that produced in 5½ minutes. The curves of instantaneous elasticity above 6 × 10³ dynes/cm.² for vulgare and 10⁴ dynes/cm.² for durum are those of Fig. 3. D = durum; V = vulgare.

convex-downwards curve. This was gradually offset by strain-hardening, the curve becoming convex upwards.

Deformation-time relations at constant load are shown in Fig. 4 for vulgare dough and in Fig. 5 for durum dough. Loads were limited to a maximum of 3 g. for vulgare and 5 g. for durum in view of the set-

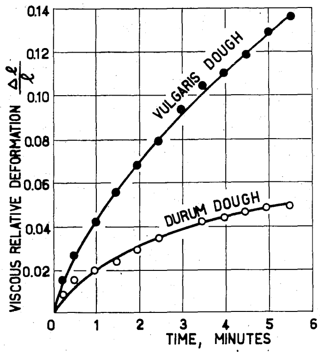

Fig. 7. Viscous deformation under 2 g. load. The curves were derived from the total deformation on loading after subtracting the mirror images of the recoverable deformation on unloading.

ting-in of necking. The curves show three kinds of deformation in flour dough, namely: instantaneous elastic, delayed elastic, and nonrecoverable viscous. This series of tests permitted isolating the various components of deformation in the two doughs, and plotting each of them against the stress, as shown in Fig. 6. All three deformations showed

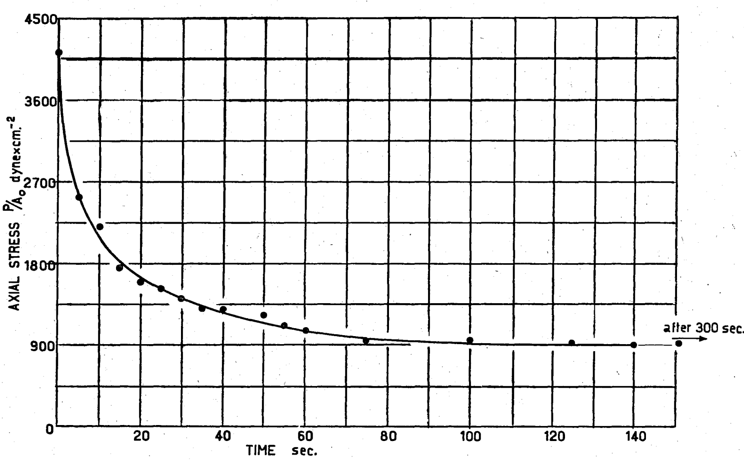

Fig. 8. Stress relaxation, vulgare dough. The curve is a mean of three tests.

nonlinearity due to stress-softening and strain-hardening.

The viscous deformations shown as functions of stress in Fig. 6 were determined by subtracting the recoverable component from the total deformation obtained in 5.5 minutes. Nonlinearity was more pronounced in durum dough and, again, was the result of stress-softening and strain-hardening. As functions of time, they are shown in Fig. 7 for a load of 2 g. These curves were derived from the total deformation curves of Figs. 4 and 5, by subtracting the mirror images of recovery curves from loading curves. This was justified, for, as can be seen from these figures, a loading time of 5.5 minutes may be regarded as "infinite" in respect to delayed elastic deformations. The viscous flow (Fig. 7) at constant load was nonlinear with respect to time; there was a strain-hardening effect, more pronounced in durum dough.

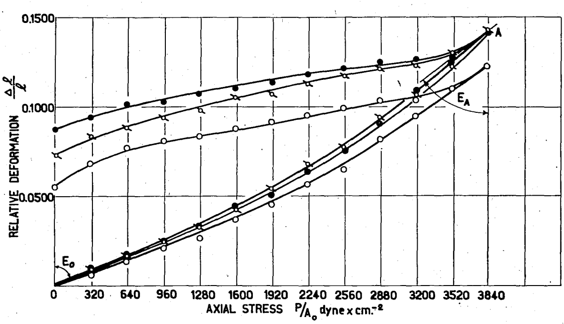

Fig. 9. Deformation-stress relations, vulgare dough. Rate of loading and unloading: 640 dynes/cm.² per minute.

Stress-relaxation measurements were carried out with vulgare dough only, with an initial instantaneous elastic deformation of 0.01, brought about by a load of 2 g. (Fig. 8). The relaxation of this load proved that the unrecoverable deformations (Figs. 4 and 7) were indeed viscous. Relaxation, however, was incomplete, indicating a yield point at 900 dynes per cm.², the asymptotic value of the curve.

Deformation-Stress Relations at Constant Rate of Loading. All three kinds of deformation were involved in this test (Fig. 9). The recovered part consisted of the instantaneous elastic and part of the delayed elastic deformation. The nonrecovered part consisted of the viscous deformation and part of the delayed elastic deformation still unrecovered.

The shape of the curve was characteristic for doughs, the concavity of the loading branch with respect to the deformation axis being due to delayed elasticity. When the sense of the stress was changed, the

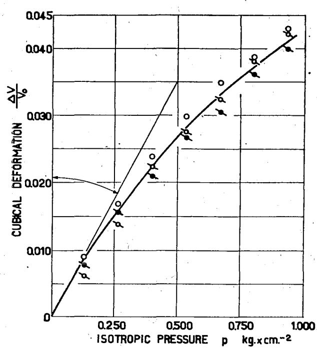

Fig. 10. Volumetric compression test, vulgare dough.

slope of the unloading branch at that point represented the modulus of instantaneous elasticity at that stress. This was smaller than the slope at the origin, showing the elasticity to be nonlinear ($E_A < E_o$). This effect, combined with the delayed elasticity effect, was responsible for the sigmoidal shape of the recovery curve.

It should also be noted here that the elasticity was linear up to about 900 dynes per cm., and nonlinear above it.

Bulk Elasticity. Figure 10 shows results of three tests. The isotropic stress applied was much higher than that produced during the baking

TABLE I

NUMERICAL VALUES OF RHEOLOGICAL PARAMETERS AT ZERO STRESS

	DOUGH MADE OF *Triticum vulgare*	DOUGH MADE OF *Triticum durum*
Elastic bulk modulus: κ in dynes/cm.^{2}	1.4×10^7	[a]
Modulus of elasticity: E in dynes/cm.^{2}	4.2×10^5	9×10^5
Poisson's ratio, ν	0.5	[a]
Liquid coefficient of viscosity: λ in poises	18×10^6	36×10^6
Time of relaxation: T_{rel} in seconds	43	40
Modulus of delayed elasticity: E_d in dynes/cm.^{2}	2.7×10^5	9×10^5
Time of retardation: T_{ret} in seconds	9	20
Solid coefficient of viscosity: λ_s in poises	2.4×10^6	18×10^6

[a] Not determined.

process, hence only a very limited stretch of the curve is of practical importance. The bulk modulus is given by the slope at the origin. Its calculated value, compared with that of the modulus of shear elasticity, shows dough to be an almost incompressible material. The nonlinearity of the curve at higher stresses is common to most materials.

The rheological parameters of the two doughs, calculated from these results and based on the rheological considerations, are shown in Table I.

Discussion

The experiments described above substantially confirmed the results obtained by the investigators reviewed in the introductory section, with regard to the Maxwellian character of dough. More specifically, it was found here that:

1. There exists an instantaneous elastic deformation, linear at stresses below a certain yield point and nonlinear at higher stresses owing to stress-softening and strain-hardening.

2. There exists a viscous deformation constituting a major part of the deformation in all time-dependent tests. This deformation, which could be isolated only in recovery tests (Fig. 7), is nonlinear (indicating strain-hardening) and is combined with plastic deformation with a finite yield point, as evident from the incomplete relaxation.

3. Delayed elastic deformation was also observed in the deformation-time curves in series with the above-mentioned deformations.

4. The calculated bulk modulus (see Table I), being very large compared with that of shear, shows that dough may be regarded as almost incompressible.

5. Qualitatively, no difference was found between the durum and vulgare doughs tested.

6. Quantitatively, with the criterion of water absorption as basis of comparison, as proposed by Halton (6), durum dough at ordinary stresses was found more rigid and more viscous than vulgare dough.

7. Neither the rigidity nor the viscosity was found constant. The elastic deformation curve was less linear for vulgare than for the durum dough, and the viscous deformation curve less linear for durum than for vulgare. Linearity and nonlinearity may be due to stress-softening and strain-hardening.

8. It can be seen (Fig. 6) that for the elastic deformations at high stresses, the moduli (as measured by the slope of the secants) of both durum and vulgare tended to become equal. For viscous deformation, however, the vulgare curve was always above that of durum; hence the coefficient of vulgare would always be less than that of durum at the same stress. As a result, the relaxation time (coefficient/modulus ratio) of vulgare dough would be shorter than that of durum dough with increasing stress.

In addition to all this, the results gave the desired information with regard to properties required for the baking process:

Cylinders of vulgare flour showed necking at the early stress of 6×10^3 dynes per cm.^{2}, whereas cylinders of durum flour withstood much higher stresses. This apparent paradox can be explained by the viscous strain-hardening, which affected durum more than vulgare, as explained above (paragraph 7). Had it been possible to continue with the stretching, the durum cylinder, having higher relaxation time, would most probably have undergone brittle rupture (a phenomenon related to "shortness"), whereas the vulgare cylinder, having lower relaxation time, would have continued its excessive deformation much longer and ended in plastic failure.

The capacity to extend under stresses without rupture prevents escape of gas and is therefore the desired property for baking. This is possible when the time of relaxation is sufficiently short to deal with impending fracture, and is the case in vulgare dough, but not in durum, apparently owing to viscous strain-hardening.

On the following points, the findings of this work are in apparent disagreement with those of others, although in most cases it was due to the different water contents used:

(a) Schofield and Scott Blair (10–13) obtained a yield point of 5,000 dynes/cm.^{2}, whereas in this work it was found below 1,000 dynes/cm.^{2}.

(b) The high yield point in the case of Schofield and Scott Blair enabled them to work below it. This explains the equal initial moduli of the loading and unloading phases in the "deformation-stress" curve (12, Fig. 5). In the present work, the initial modulus of the unloading phase was smaller than that of the loading one (Fig. 9).

(c) According to Halton (4), doughs made with different flours adjusted to have equal rates of extrusion should have equal moduli, whereas in the present work the moduli varied by as much as 100%. It must be realized, however, that the values in the table refer to zero stress, whereas the stresses in Halton's experiments were quite high. In fact, it can be seen from Fig. 6 that at high stresses all elastic curves approach a more or less constant value with $E_{vulg} = E_{dur} = 2.25 \times 10^5$ dynes/cm.^{2} (see paragraph 8 above). Thus, Halton's criterion seems to be substantiated.

(d) Cunningham and Hlynka (1) related the relaxation to a generalized Maxwell body, with a corresponding spectrum of relaxation times. The relaxation curve reported here also suggests such a configuration, and consequently, the single value presented is a simplification.

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