

# Rothamsted Repository Download

## A - Papers appearing in refereed journals

Halton, P. and Blair, G. W. S. 1936. The relationship between conditions governing rupture and flow. *Journal of Physical Chemistry (1952)*. 40 (6), pp. 811-819.

The output can be accessed at: <https://repository.rothamsted.ac.uk/item/95x71/the-relationship-between-conditions-governing-rupture-and-flow>.

© Please contact [library@rothamsted.ac.uk](mailto:library@rothamsted.ac.uk) for copyright queries.

# THE RELATIONSHIP BETWEEN CONDITIONS GOVERNING RUPTURE AND FLOW IN FLOUR DOUGHS

P. HALTON<sup>1</sup>

*Research Association of British Flour Millers, St. Albans, England*

AND

G. W. SCOTT BLAIR

*Physics Department, Rothamsted Experimental Station, Harpenden, England*

*Received April 8, 1936*

## INTRODUCTION

In an earlier paper (6) it was shown that there is a general correlation between the bread-making qualities of flour doughs and certain of their physical properties, chiefly relaxation time (viscosity/shear modulus) and the rate of fall of viscosity during fermentation. The importance of a further dough property, the tendency to tear when stretched, was also realized, but the discussion of the significance of this in terms of viscosity, modulus, etc., was left until further experiments had been completed. The tendency to tear varies from dough to dough, but only becomes apparent to the baker when it reaches the stage when the dough "tears" easily under bake-house manipulation. Such doughs are called "short" and the baker speaks of varying degrees of "shortness," such as "slight shortness" or "extreme shortness." Before this stage is reached, however, the baker does not recognize this tendency to tear, although it is there and probably influences the bread-making quality of the dough. It may, for example, be an important factor in determining whether a dough bakes into a loaf having the fine vesiculated crumb generally associated with an all-Manitoba flour or the coarse and open crum from an all-English flour.

A direct measure of the tensile strength of a flour dough is at the moment impossible, partly because the value obtained depends so greatly on the conditions of stress application, and partly because the excessive flow and consequent thinning of the test piece before rupture makes a determination of the cross section impossible.

The conception of "shortness" as determined by the baker is a complex one, and probably depends partly on ductility as well as on tensile strength. Ductility represents the critical deformation of the material at the point

<sup>1</sup> By mutual agreement the authors' names are given in alphabetical order and no seniority is implied.

of rupture, the tensile strength being the critical stress. The relationship between the two is complex, and it is sufficient for the present purpose to point out that ductility is a function of viscosity, shear modulus, and elastic after-effect as well as of tensile strength. When a piece of dough is extended,<sup>2</sup> since the stress built up in the dough is proportional to the viscosity and to the rate of extension, the tensile strength will be exceeded after quite a small elongation if the extension is rapid, whereas a slow pull will allow a big deformation before rupture occurs. Thus for a direct test carefully controlled conditions are essential, whereas the baker judges shortness partly by extending the dough at quite uncontrollable rates and stresses, and partly by observing the stretching and tearing of a mass of dough under its own weight. Shortness is therefore not a very sharply defined property, and only comparatively wide differences can be reliably observed. Such wide differences do, however, occur even within the range of commercial flours, and some quantitative measure of them is essential for a further understanding of the nature of shortness. Measurements of ductility at unknown stress but controlled rates of deformation, although fairly reproducible, do not correlate with "shortness" as determined by handling. It has been shown (15) that the size of a bubble which can be blown in a dough is only a measure of "shortness" for doughs in which this property is predominant. In more extensible doughs, the shear modulus plays an important part in determining extensibility (*vide infra*).

Although the relationships between rupture and flow conditions are not yet fully understood, certain phenomena have been observed which indicate a method by which shortness may be measured. This method depends on the relationship between the rate at which viscosity falls with increasing stress (structural viscosity)<sup>3</sup> and the brittleness of materials (13). If a dough were a true highly viscous fluid, it would extend until the test piece narrowed to a thread before rupture; if it were a solid, up to a limiting stress (tensile strength) it would hardly deform at all (viscosity infinite) and at this stress it would break right across. This suggests that the more a dough approximates to a true fluid, the less "short" it is likely to be.

A certain correlation between high work-hardening and shortness had been observed in the rheogram experiments,<sup>4</sup> and it has been known for some time that high work-hardening is generally associated with big structural viscosity, but owing to the very limited range of stress available in the rheogram method, the full implications of this were not realized, although the significance of the distribution of relaxation times in determining plastic properties had already been appreciated (14).

<sup>2</sup> It is here assumed that the elastic extension is small in comparison with the non-recoverable extension.

<sup>3</sup> The use of this term is convenient, but does not imply agreement with the theoretical treatment of the Ostwald school.

<sup>4</sup> For a description of the rheogram method see references 6 and 12.

If a dough is extended until it breaks, a fibrous structure<sup>5</sup> can be observed with the naked eye, especially clearly in the case of a short dough. Rupture occurs as a result of the tearing apart of the fibers; the more coarse the fiber structure, the sooner the mass disintegrates, because the tearing of a single fiber makes a big rent and greatly decreases the area over which the load is distributed. Local rupture results, as Griffith (5) has emphasized, in a big conversion of potential into kinetic energy, producing a local fall in viscosity. Griffith claims that in the case of metals this effect is responsible for the fact that measured tensile strengths are always so much smaller than the theoretical ones.

The formation of gross fibers in the dough as it is deformed not only causes an increase in viscosity and shear modulus (work-hardening) but also, by producing a heterogeneity of structure, effects a progressive slipping of fibers as stress increases, which shows itself in high structural viscosity. The shear angle gets more and more distorted the grosser the fibers become. Tammann and Rejtö (*vide* Goubkin (4) consider that work-hardening in metals may be ascribed entirely to this effect, and Nádaï (7) states that it is responsible for brittleness, although it is now claimed (3) that this is an exaggeration.

When the junction between two fibers slips and breaks, a rent is formed and these previously extended fibers contract, making the rent worse. It is clear that the extent of widening of the rent will depend on how much the elastic elements were extended prior to rupture. The modulus of the fibers thus affects the conditions of rupture, and in comparing the structural viscosities of doughs it is best to adjust their moisture contents such that comparisons can be made at a fixed modulus (6). It must be remembered that whereas the measurable viscosity of a heterogeneous material is probably a function of the viscosities of all its parts at the stress in question, the tensile strength depends on the resistance of the weakest part. If there is a wide distribution of viscosities amongst the elements making up the dough, this will result both in a high structural viscosity and in a tensile strength which is low for the mean viscosity. Heterogeneity, reflecting a high structural viscosity, thus always tends to favor rupture and homogeneity favors flow.

The nature of the process of fiber formation in dough is very little understood, nor is it known why some flours give doughs which are abnormally prone to it, though it would be surprising if there were no natural variation in this, such as is found in all other physical properties.

Potel (8) and Potel and Chaminade (9) have shown that mild oxidation increases shortness in dough, while reduction diminishes it. The processes may be followed by observing changes in oxidation-reduction potential.

<sup>5</sup> The term "fibrous structure" really implies no more than the existence of mechanical anisotropy, but is conveniently used in this sense even in describing properties of crystals (11).

In view of the recent work of Astbury, Dickinson, and Bailey (2) one might suggest that oxidation effects some type of mild denaturation involving "the liberation or generation of peptide chains which aggregate on coagulation into parallel bundles like those found in the structure of  $\beta$ -keratin and similar fibres." In the case of dough, these fibers only manifest themselves when the dough is extended. Whether the process is actually reversible on subsequent reduction, or whether the reducing agent acts at different spots in the dough structure is not known.

The fiber structure of dough is also enhanced by the addition of many other materials such as the fats, which, by modifying the viscosity at certain points in the dough, tend to increase heterogeneity, and hence structural viscosity.

Astbury (1) has pointed out that the amino acid<sup>6</sup> cystine ( $\text{HOOC}\cdot\text{CH}(\text{NH}_2)\text{CH}_2\text{S}\cdot\text{SCH}_2\text{CH}(\text{NH}_2)\cdot\text{COOH}$ ) can form strong cross linkages between protein chains, like the rungs in a ladder. It has been found that cystine does make unyeasted dough feel less short. Cysteine ( $\text{HOOC}\cdot\text{CH}(\text{NH}_2)\text{CH}_2\text{SH}$ ) would be expected to be less effective in this respect, because only one end of the molecule can attach itself to the protein chain, but on the other hand, it is a reducing agent, which may account for the fact that in practice it is found to be not much less effective than cystine. Other amino acids, such as aspartic acid and *m*-aminobenzoic acid, become less effective as their polar properties diminish.<sup>7</sup>

The connection between structural viscosity and shortness has a profound influence on the interpretation of viscosity and modulus data in terms of baking values. The extent of elastic recovery (spring) of a short dough, as estimated at the high stresses used in handling, will not bear a normal relation to that determined at low stresses intended to correspond to those obtaining in the fermenting dough. Good spring has been shown to depend on high viscosity and low modulus, and since the latter does not alter very drastically with stress, a dough whose viscosity falls to any abnormal degree as stress is increased will also appear progressively to deteriorate in spring. This has been amply verified in the baker's experience. This means that in order to compare relaxation times (viscosity/modulus) with bakehouse data, the stresses at which they are determined should correspond with the internal stress built up in the dough during fermentation. The latter cannot yet be measured directly. This diffi-

<sup>6</sup> Amino acids are found as products of proteolysis in the fermentation of flour dough, and in view of the above experiments it is tempting to suggest that changes in physical properties during fermentation may be ascribed to them. This is unlikely, however, since it has been shown (6) that these physical changes are only slightly affected by the presence of normal quantities of yeast, and Samuel (10) has demonstrated that in yeasted doughs the amino acids produced by fermentation are used up by the yeast in its metabolism (*vide infra*).

<sup>7</sup> Some of these experiments were suggested to us by M. Potel.

culty has not been fully overcome, but it really means that both spring as measured by relaxation time and shortness as measured by structural viscosity in the test described below, have to be taken into account in assessing the quality of a flour. The extent to which these two properties are present in a dough determines its quality, but their relationship to each other, and how this varies in different flours, is not yet fully understood.

The experiments described below are designed to test whether the effect of various materials on the shortness of doughs as judged qualitatively by handling, can be shown to be paralleled by quantitative measurements of structural viscosity. If this can be done, although shortness is realized to be an extremely complex property, we shall be in a position to say that at least big differences in shortness can be measured by means of structural viscosity. In the later part of this paper, the question of smaller differences is discussed.

#### EXPERIMENTAL

Dough cylinders for viscosity measurements<sup>8</sup> are prepared by extruding the dough through a metal "gun" at high pressure. It was noticed that when doughs showed about the same viscosity at the standard shearing stress of the order of 500 dynes per square centimeter the "shorter" doughs always came out of the gun faster and thus evidently had lower viscosities than the non-short doughs under the very high stress applied in the gun. To test this quantitatively, a flour was made short by the addition of different quantities of lard, and the viscosity ( $\eta$ ) at 500 dynes per square centimeter under set conditions, together with the time ( $T$ ) taken for a certain quantity of dough to be extruded from the "gun" under a load of 7 lb., were determined. The results, which are included in table 1, clearly indicate that over a big range of stress increasing shortness is accompanied by a rapidly increasing structural viscosity.

The time of extrusion from a gun is not a very satisfactory way of measuring viscosity, and it was of interest to determine whether the ratios of viscosities at two different stresses which could be applied in the standard technique would indicate big differences in shortness. The viscosities of a dough made without shortening agents were measured at 250 and 600 dynes per square centimeter, respectively, the ratio of these viscosities being 1.29. A similar test on a dough in which 2 per cent of lard had been incorporated showed a ratio of 1.54, 5 per cent of lard gave a ratio of 2.23, and 10 per cent of lard 2.25, although in the last case the stresses employed were no longer suitable. The structural viscosity had evidently been progressively increased by the shortening.

A test was then arranged in which other materials which were known to

<sup>8</sup> The technique for measuring viscosity and modulus is described in the earlier paper (6).

affect shortness were added to flour when making doughs, varying quantities being used. The viscosity ( $\eta$ ) and the modulus ( $n$ ) at 500 dynes per square centimeter and the time of flow ( $T$ ) out of the gun were measured under standard arbitrary conditions and compared in each case with a separate control. (This was done because temperature varied somewhat between the different experiments.) The results are given in table 1.  $\eta/T$  is taken as a measure of structural viscosity. It is clear that the effects, although perhaps not very accurately determined, are in entire

TABLE 1

*Effect of certain substances on the viscosity, modulus, and structural viscosity of flour doughs*

SUBSTANCE		DOSE IN PARTS PER MILLION OF FLOUR	$\eta/\eta_{\text{control}}$	$n/n_{\text{control}}$	$T/T_{\text{control}}$	$\eta/T/(\eta/T)_{\text{control}}$
decrease shortness	Cystine	4	0.86	1.07	0.89	0.96
		20	0.79	0.88	0.98?	0.80
		1,000	0.31	0.63	0.88	0.35
		2,000	Very low	Very low	0.82	Very low
	Cysteine	4	0.86	0.99	0.82	1.05
		20	0.71	0.77	0.73	0.98
		1,000	Very low	Very low	0.13	Very low
	Aspartic acid	500	0.59	0.85	0.91	0.65
		2,000	0.36	0.52	0.65	0.57
	<i>m</i> -Aminoben- zoic acid	500	0.61?	0.82?	0.85	0.72?
		2,000	0.64	0.76	0.77	0.84
	increase shortness	Lard	40,000	0.97	1.25	0.63
100,000			1.61	1.85	0.38	4.22
200,000			1.48	3.10	0.10	14.8
FeCl <sub>3</sub>		High?	5.75	2.98	0.49	11.8
HPO <sub>3</sub>		8,000	1.71	0.93	0.40	4.3

agreement with the earlier findings, thus justifying the conclusion that for the range of variation considered the structural viscosity test gives a valid measure of shortness.

The treated and untreated doughs were compared at the same moisture contents. The addition of amino acids decreased the viscosity, modulus, and structural viscosity and in this way rendered the dough both softer and less short. The addition of extra water to a dough also lowers  $\eta$ ,  $n$ , and  $\eta/T$ , and to compare the relative effects of cystine and water on struc-

tural viscosity it is necessary to make measurements at an arbitrarily chosen condition of consistency, such as a constant modulus.

A further experiment was therefore made in which doughs, with and without cystine, were compared at a modulus of  $1.0 \times 10^4$  dynes per square centimeter. For the control dough  $\eta = 0.85 \times 10^6$  dynes per second per square centimeter,  $T = 40$  sec., and hence  $\eta/T = 2.1 \times 10^4$ . For the dough containing 5 parts of cystine per 100 parts of flour  $\eta = 0.79 \times 10^6$ ,  $T = 46$ , and hence  $\eta/T = 1.7 \times 10^4$ .

Similar measurements were made at other moduli, and in each case the dough containing cystine had a slightly lower structural viscosity than the control dough. Thus a series of doughs of increasing water content and having the same moduli as the cystine doughs in table 1 would, dough for dough, have higher structural viscosities than the latter. Cystine thus makes dough less short partly by rendering it softer, as does water, and also by reducing the structural viscosity for a given consistency. To pro-

TABLE 2  
*Structural viscosity of doughs from some English flours*

FLOUR NO.	$T$	$\eta$	$\eta/T$
893	54	$0.6 \times 10^6$	$1.1 \times 10^4$
898	45	$0.7 \times 10^6$	$1.5 \times 10^4$
903	38	$0.6 \times 10^6$	$1.6 \times 10^4$
902	40	$0.65 \times 10^6$	$1.6 \times 10^4$
911	35	$0.6 \times 10^6$	$1.7 \times 10^4$
892	43	$0.8 \times 10^6$	$1.9 \times 10^4$
899	32	$0.7 \times 10^6$	$2.2 \times 10^4$
904	21	$0.55 \times 10^6$	$2.6 \times 10^4$

duce similar effects on consistency much larger quantities of water than of cystine are necessary. Although water and amino acids diminish shortness in a somewhat analogous way, namely through their effect on viscosity, the mechanism must be entirely different. Some tentative suggestions about the amino acid mechanism have already been discussed.

In addition to the above tests on flours whose degree of shortness had been artificially altered, a number of flours, all milled from English wheat and which when tested in the bakehouse had shown varying degrees of natural shortness, have also been examined.

These flours were tested under carefully controlled conditions in a constant-temperature room<sup>9</sup> at 80°F., the temperature at which the flours had been examined in the bakehouse.

Doughs were made from each, and at intervals samples were taken on

<sup>9</sup> This constant-temperature room was not available for the earlier experiments quoted in table 1.

which the values of  $T$ ,  $\eta$ , and  $n$  were obtained. Curves were then drawn relating  $T$ ,  $\eta$ , and  $n$  to the age of the dough, and from these, values of  $T$  and  $\eta$  corresponding to a modulus of  $1.0 \times 10^4$  were obtained for each flour. These values, together with the ratio of  $\eta$  to  $T$ , are given in table 2.

Of these flours, which are arranged in table 2 in order of increasing  $\eta/T$ , the first two, Nos. 893 and 898, did not, according to the baker, show any signs of shortness. On the other hand, the last three, Nos. 892, 899, and 904, were all stated to be decidedly short. Of the three intermediate flours, Nos. 903 and 902 were rather short, while No. 911 was not short.

The correlation between  $\eta/T$  and shortness is therefore not perfect, and this may be due to one or more of the following reasons:

- (1) The impossibility of differentiating by feel any but comparatively large differences in shortness.
- (2) Shortness, while being mainly determined by structural viscosity and to a less extent by elastic modulus, may also be influenced by other factors which are not apparent at the moment.
- (3) The shortness of a test piece of dough from which the gas has been expelled may not be the same as that of the inflated parent dough which is examined by the baker. Shortness is more easily detected by feel in an inflated dough and is possibly influenced by the vesiculated structure of the inflated dough.
- (4) While  $T$  gives a rough measure of viscosity, the value obtained may be so influenced by other factors that  $\eta/T$  may give only an approximate value of structural viscosity.
- (5) Both  $\eta$  and  $T$  are not constants but are dependent on the stresses used in their determination. These stresses should approximate to those operating in the dough if exact correlation with bake-house experience is to be expected. The correct values for these stresses will only be found after considerable experience. In this connection it may be mentioned that the viscosities of the flours in table 2 were measured both at 600 and 200 dynes per square centimeter, but that the values of structural viscosities, as given by the ratio  $\eta_{200}/\eta_{600}$ , were so similar as to fail to differentiate one flour from another.

While therefore it is realized that the present methods of measuring structural viscosity and assessing shortness by feel are too imperfect to expect a close correlation between them in the case of the comparatively small differences between natural flours, there is little doubt that the two are intimately connected.

#### SUMMARY

1. It has been shown that the shortness (i.e., ease of tearing) of flour doughs is closely paralleled by the rate at which viscosity falls with increas-

ing stress (structural viscosity). A perfect correlation is not obtained, partly because neither property can be determined with great accuracy.

2. The effect on structural viscosity of certain substances (fats, amino acids, etc.) known to alter the shortness of dough has been measured, and the nature of the processes involved discussed.

3. The structural viscosities of doughs made from a batch of English flours have been determined, and it is found that the data are paralleled by fairly large differences in shortness as observed in the bakehouse.

4. The significance of shortness in terms of heterogeneity of dough, and its relation to tensile strength and ductility are tentatively discussed.

The authors wish to acknowledge their indebtedness to Dr. E. A. Fisher, Director of the Research Association of British Flour Millers, for his help and criticism in the writing of this paper.

#### REFERENCES

- (1) ASTBURY, W. T.: *Fundamentals of Fibre Structure*, p. 154. Oxford University Press (1933).
- (2) ASTBURY, W. T., DICKINSON, S., AND BAILEY, K.: *Biochem. J.* **29**, 2351 (1935).
- (3) BURGERS, W. G., AND BURGERS, J. M.: *First Report on Viscosity and Plasticity*, p. 188. Amsterdam (1935), Dutch Royal Acad. Sci.
- (4) GOUBKIN, S. I.: *J. Rheology* **3**, 501 (1932).
- (5) GRIFFITH, A. A.: *Proc. 1st Intern. Cong. App. Mech.*, p. 55 (1924).
- (6) HALTON, P., AND SCOTT BLAIR, G. W.: *J. Phys. Chem.* **40**, 561 (1936).
- (7) NÁDAI, A.: *Plasticity*. McGraw-Hill Book Co., New York (1931).
- (8) POTEL, P.: *Compt. rend. acad. agr. France* **21**, 115 (1935); *Ann. agron.* **5**, 691 (1935).
- (9) POTEL, P., AND CHAMINADE, R.: *Compt. rend.* **200**, 2215 (1935).
- (10) SAMUEL, L. W.: *Biochem. J.* **28**, 273 (1934); **29**, 2331 (1935).
- (11) SCHMID, E., AND BOAS, W.: *Krystallplastizität*. J. Springer, Berlin (1935).
- (12) SCHOFIELD, R. K., AND SCOTT BLAIR, G. W.: *Proc. Roy. Soc. London* **138A**, 707 (1932); **139A**, 557 (1933); **141A**, 72 (1933).
- (13) SCHOFIELD, R. K., AND SCOTT BLAIR, G. W.: *Nature* **136**, 147 (1935).
- (14) SCOTT BLAIR, G. W.: *Physics* **4**, 113 (1933).
- (15) SCOTT BLAIR, G. W., AND POTEL, P.: 1936. In process of publication, probably in *Bulletin des anciens élèves de l'école de meunerie*.