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THE KIEFFER DOUGH AND GLUTEN EXTENSIBILITY RIG — AN EXPERIMENTAL EVALUATION

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ABSTRACT

Load-extension tests on flour dough are widely used by plant breeders, millers and bakers. The 'Kieffer dough and gluten extensibility rig' is a small-scale version of the Brabender extensograph, in which test pieces of about 0.4 g are extended. With the Kieffer rig, lower strain rates can be applied than in the Brabender extensograph and the experimental data can be expressed in terms of stress and strain. In this paper the performance of the Kieffer rig is illustrated by measurements on a weak and a strong dough. Formulas are given for the calculation of fundamental rheological parameters from the results of measurements with the Kieffer rig. Sagging and bending of the test pieces before measurements could be started, caused difficulties in the determination of the exact starting point of extension. The deformation was not purely uniaxial extension, because a shear component was also observed. The amount of dough that is extended did not increase throughout the test. This is probably due to the occurrence of a shear component fracture which occurred mainly near the hook. A relatively large variation in stress and strain at fracture was observed. The maximum in stress represents the strain at which the sample fractures macroscopically better than the maximum in force. Variation in deformation history and volume of the test pieces have a negative effect on the reproducibility.

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INTRODUCTION

Currently wheat is one of the most abundant crops in the world (Hoseney and Rogers 1990). From wheat flour a large variety of food products can be made, like breakfast cereals, breads, crackers, cakes, biscuits and pasta. By mixing, flour and water are transformed into a cohesive dough with viscoelastic properties (Schofield and Blair 1932). The rheological properties of wheat flour dough affect its behaviour during processing and consequently the quality of the finished loaf of bread (Bloksma 1990b). This conviction has led to the design of instruments with which rheological properties can be determined. Such measurements are widely used for the selection of new cultivars in breeding, for quality control in mills and bakeries and to study the effects of ingredients and adaptations of processes in the milling and baking industry. Fracture of dough membranes during fermentation and baking restricts the bread volume that can be obtained. Therefore tests determining large-deformation and fracture properties are relevant.

Information on the resistance to extension and the extensibility of dough can be obtained by load-extension instruments such as the Brabender extensograph (Bloksma 1990a). In this apparatus a cylindrical piece of dough is deformed in uniaxial extension by a hook which travels downwards at a constant rate. The required force to deform the dough is expressed in Brabender Units (BU) as a function of extension. From the extensograph load-extension curve several parameters can be derived, such as maximum resistance (R_{max}), extensibility to maximum resistance (E_{max}), total extensibility (E_{tot}) and the total area under the curve (A_{tot}) as a measure of applied energy (Muller *et al.* 1961). It has been used to predict baking performance of a flour based on these parameters (Bloksma and Bushuk 1988; Walker and Hazelton 1996). Although Bloksma (1962) has related the data obtained by this test to more fundamental parameters, the test still remains very empirical. The force and extension are not expressed in Newtons and strain, respectively. Major practical disadvantages of this test are that the position of the cradle (the clamp) depends on the force and that the amount of dough deformed increases with extension (Bloksma and Bushuk 1988). The conversion of extensograph load-extension curves into stress-strain curves is therefore seriously hampered. Under fermentation conditions, the rate of deformation of dough is three orders of magnitude smaller than the maximum rate of deformation of the test-piece in load-extension instruments like the Brabender extensograph (Bloksma 1990a). Therefore, the applied rates of deformation may be relevant for mixing and for the shock resistance of dough during processing, but the relevance of the data obtained at this high rate for baking performance can be questioned. Moreover, the dominant type of deformation during fermentation and oven rise is biaxial extension and not uniaxial (van Vliet *et al.* 1992). Finally, a large amount of flour (300 g) is needed to perform the test.

An apparatus similar to the Brabender extensograph was developed by Kieffer: the 'Kieffer dough and gluten extensibility rig', also called a micro-extensograph (Kieffer *et al.* 1981a, b). In this apparatus a test can be performed with only about 0.4 g of dough and the apparatus can be fitted on any materials testing machine, which gives us the possibility to measure the force in Newtons, to adjust the test speed and temperature and to store the data in computer files for further calculations. Kieffer *et al.* (1981b, 1998) compared the test with the Brabender extensograph and also related it to bread making performance. At a speed of about 410 mm/min the strain rate is about the same as that of the Brabender extensograph. Recently, Grausgruber *et al.* (2002) comparing both instruments, concluded that the micro-extensograph method is valuable in early-generation selection for wheat quality where the amount of available sample does not allow testing by the standard extensograph. The test method itself, however, has not been described extensively and formulas to calculate the stress and relative deformation rate are not given. Also only very little has been reported about the reproducibility of the test. Hence, to test this uniaxial extension instrument the rheological properties of a weak and a strong dough were evaluated with special attention to the correct determination of the type of deformation, the starting point of the actual extension and the reproducibility of the test.

THEORY

In Fig. 1A the extensibility rig is drawn schematically. Test pieces of dough are formed into 5 cm long pieces with a trapezium like cross-section (3/5 × 4 mm) in a teflon mould. After a resting period a dough piece is clamped between two plates by means of springs under the lower plate and extended in the upper direction with a hook (diameter 1.20 mm). As a small amount of the dough is squeezed out between the plates, the dough volume in between increases somewhat and due to gravity the sample will bend to a certain extent. This means that the point where the actual extension starts, lies above the surface of the lower plate. So the deformation is zero up to that specific point. In Fig. 1A, the distance which the hook has to travel from the surface of the lower plate to that point, is represented by y_0. The formulas for the calculation of fundamental rheological parameters can be derived from geometry. The initial length of the sample l_0 and the length l_t at time t are:

$$l_0 = 2 \cdot \sqrt{9^2 + y_0^2} \tag{1}$$

$$l_t = 2 \cdot \sqrt{9^2 + (y_t + y_0)^2} \tag{2}$$

in which y_t is the displacement of the hook from the point at which the actual extension starts, and 9 is half the width of the gap in the lower plate through which the hook passes (Fig. 1A). The relative deformation or Hencky strain and the strain rate can then be written as:

$$\varepsilon_H = \ln\left(\frac{l_t}{l_0}\right) = \ln\left(\frac{\sqrt{9^2 + (y_t + y_0)^2}}{\sqrt{9^2 + y_0^2}}\right) \tag{3}$$

$$\dot{\varepsilon} = \frac{d\varepsilon_H}{dt} = \frac{dl}{l_t\, dt} = \frac{1}{l_t} \cdot \frac{2(y_t + y_0)}{\sqrt{9^2 + (y_t + y_0)^2}} \cdot \frac{dy_t}{dt} = \frac{4 \cdot (y_t + y_0) \cdot v}{l_t^2} \tag{4}$$

in which v is the speed of the hook. The maximum in the extension rate curves for the micro-extensograph occurs when $y_0 + y_t$ is 9 (half the gap size) and the dough is at an angle of 45° to the plane of the clamps. In a Brabender extensograph l_0 is 37 mm and the speed is fixed at 840 mm/min (Bloksma 1962). As a result of the smaller initial length of the sample the strain at a certain hook displacement is higher in the micro-extensograph (Fig. 2A). The relative strain rate is very high in the Brabender extensograph (Fig. 2B). At a speed of about 410 mm/min the strain rate is about the same as that of the Brabender extensograph (Kieffer *et al.* 1981b).

The measured force F_m is not the force acting on the dough F_d. Assuming that the hook passes exactly through the centre of the gap, F_m is divided equally over both stretches of dough at each side of the hook. Sinα can therefore be expressed in forces as well as in lengths (Fig. 1B):

$$\sin\alpha = \frac{F_m / 2}{F_d} = \frac{y_t + y_0}{l_t / 2} \tag{5}$$

$$F_d = \frac{F_m l_t}{4 \cdot (y_t + y_0)} \tag{6}$$

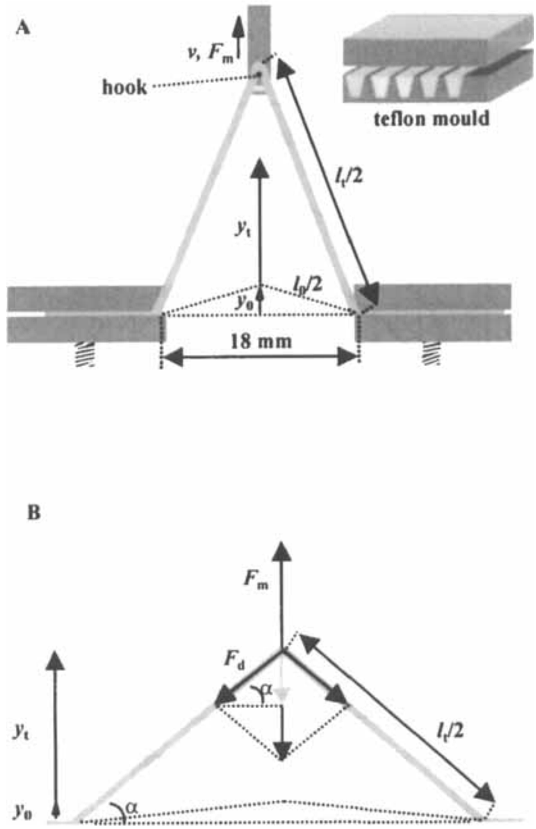


FIG 1A: SCHEMATIC DRAWING OF THE KIEFFER EXTENSIBILITY RIG WITH ITS TEFLON MOULD. 1B: SCHEMATIC DRAWING OF THE FORCES ACTING ON THE DOUGH PIECE (EQ. 5).

Assuming that the dough piece has the same cross-section over its whole length, the surface over which the force is acting is V/l_t, V being the volume of the dough piece that is extended. The stress σ can then be calculated according to:

$$\sigma = \frac{F_d}{V / l_t} \tag{7}$$

and the apparent extensional viscosity η_E as:

$$\eta_E = \frac{\sigma}{\dot{\varepsilon}} \tag{8}$$

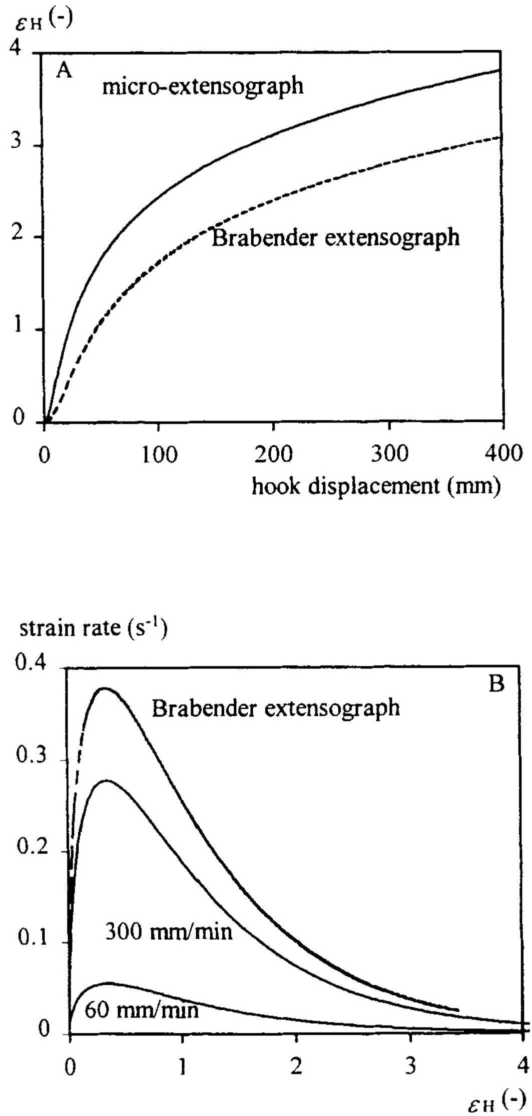


FIG. 2. COMPARISON OF MICRO-EXTENSOGRAPH (SOLID LINES) WITH BRABENDER EXTENSOGRAPH (BROKEN LINES)

2A: Hencky strain ε_h as a function of the displacement of the hook. 2B: Strain rate as a function of Hencky strain ε_h; hook displacement speed for the micro-extensograph indicated.

An uniaxial strain hardening parameter and strain rate thinning parameter can be calculated in a way similar to that in biaxial extension (Kokelaar *et al.* 1996).

EXPERIMENTAL

Materials

Two types of flour were used. One was the commercial, cookie-type mixture Kolibri with 15.0% moisture and 10.5% protein, obtained from Meneba. Inherently, a cookie-type flour has a poor bread making quality. The other was Vivant with 14.4% moisture and 10.3% protein, also obtained from Meneba and also having poor baking quality. Vivant gives a weak dough, while Kolibri, in combination with glucose oxidase, formed a strong dough. Other ingredients used were, NaCl (analytical grade) from Merck, Germany, Glucose oxidase (Oxygo L5) from Genencor, Finland (5364 glucose oxidase units/mL), and deionised water.

Methods

Dough preparation was as described for Kolibri (Dunnewind *et al.* 2003) and for Vivant (Sliwinski *et al.* 2003). Water addition was 60% (T = 20C) for Kolibri and 63% (T = 8C) for Vivant (on flour basis). In the case of Kolibri 6 mL water contained 0.2 g NaCl (2% on flour basis) and 20 μL glucose oxidase. The doughs were mixed in a mixograph (National Mfg. Co., Lincoln) for 7 (Vivant) and 4 (Kolibri) min.

For test piece preparation the dough was made into a roll, put on the lubricated (paraffin oil) lower plate of the teflon mould and compressed with the lubricated top plate (Fig. 1A). The doughs were left resting at 25 $\pm$ 1C for 60 min (Vivant) and at 30 $\pm$ 1C for 45 min (Kolibri + glucose oxidase).

Three methods for taking the test pieces out of the teflon mould were compared. In the first one the upper plate of the mould was slided away until a test piece was accessible. The test piece was then scooped out with a spatula. The second method made use of the fact that the ends of the test pieces are exposed at the side of the teflon mould. This makes it possible to press the test pieces out of the mould with a stick. To prevent the dough from flowing past the stick, first a small pellet of paper was put on one exposed end of the dough piece. When almost out of the mould the test piece was grabbed at the top with a pair of tweezers. For the third method the plastic strips belonging with the mould were used. They were laid in the grooves before the dough was put in it. After opening the mould, the dough pieces were taken out simply by lifting the strip.

Extension tests were performed with the Kieffer rig fitted on a Zwick materials testing machine equipped with a 50 N load cell and a plexiglas container and a heater to maintain the temperature. Tests were performed at 25 ± 1C and 30 ± 1C for Vivant and Kolibri dough, respectively. The plates and hook were lubricated with paraffin oil. Just before the start of the test both ends of the sample were clamped between the two plates of the Kieffer rig, which resulted in some sagging of the test piece. During the test the middle of the free hanging cylindrically shaped sample was pulled upwards by the hook until fracture occurred. The force required to do so and the displacement of the hook were recorded as a function of time and used for further calculations. Drying out of the dough during testing was prevented by lubrication of the test samples with paraffin oil and by preventing the relative humidity from becoming too low. The latter was done by placing a dish with demineralised water in the plexiglas container.

The sagging of the test piece after clamping in the extensibility rig, as a result of the squeezing of dough between the plates, was measured by a kathetometer. The distance between the bottom of the dough piece and the top of the lower plate was determined, which should be equal to y_0.

For determining the weight of the part of the dough piece that is extended, test pieces were clamped in the extensibility rig and the part of the test piece in the gap was cut off and weighed on an analytical balance.

By drawing lines on the surface of the dough pieces with an overhead marker, the types of deformation playing a role during clamping and extension of the test piece were made visible. The types of deformation were also determined for a thicker hook (diameter 4.55 mm).

RESULTS AND DISCUSSION

Taking Test Pieces from the Mould

After mixing the test pieces are put in the lubricated mould. Opening the mould and scooping the test piece out with a spatula, requires much experience. The test piece is easily deformed locally to a large extent and therefore this method was not further considered. Using the strips supplied with the mould also required much skill and the strips were hard to detach from flour doughs, leading again to unwanted deformations. Pressing the dough piece out of the mould using a stick required only a very small force as the surfaces of the mould were lubricated with paraffin oil. As a test piece is hanging down from the pair of tweezers gravity will deform it, but the force involved is small. This method is very easy and fast, requires no special training, and does not involve local damage of the dough piece. 'Using strip' and 'pressing out', test pieces were compared in extension tests at 3 different speeds (12, 60 and 300 mm/min). No differences in extensional properties were found (results not shown). The 'pressing out' method was used for all further tests.

Weight and Volume of Test Piece

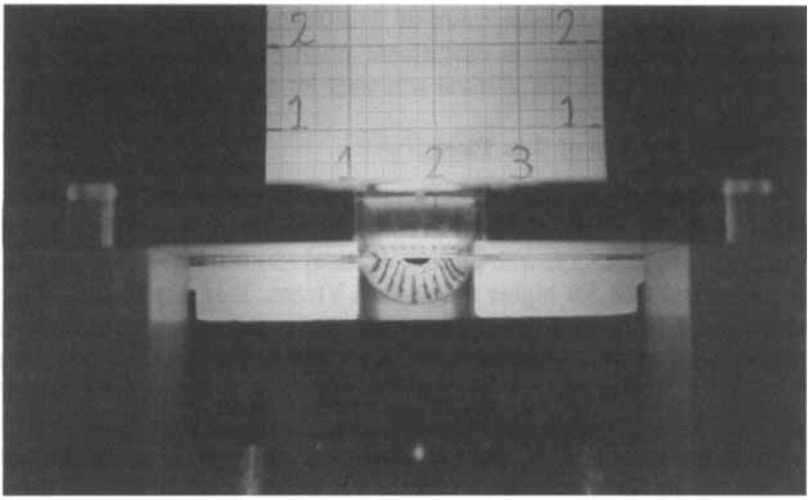
The weight of the dough piece in the gap of the apparatus was 0.345 g (± 0.01 st. dev.) for the weak dough (Vivant) and 0.395 g (± 0.018 st. dev.) for the strong dough (Kolibri with glucose oxidase). So Kolibri + glucose oxidase test pieces had a 14.5% higher weight than Vivant. This may be caused by the weaker dough being more compressed between the plates, thereby flowing in all directions and causing more friction, which retards the flow towards the gap. Assuming 1.24 g/cm^3 as the specific weight of both doughs (Baker and Mize 1941), the dough volume that is actually extended was about 0.278 and 0.319 cm^3 for Vivant and Kolibri, respectively. A varying volume has consequences for calculations that are based on volume. The influence of variation in the mass on the calculated stress was already discussed by Muller *et al.* (1961) in great detail for the extensograph.

Clamping of the Test Pieces

During clamping the dough is compressed between the plates whereby part of the test piece is squeezed. This results in an increase of dough volume and sagging of the test piece in the area of the gap between the clamps (Fig. 3A). After clamping the distance from the top of the lower plate to the bottom of the dough piece was on average 2.82 (± 0.42 st. dev.) and 1.32 (± 0.46 st. dev.) mm for Vivant and Kolibri, respectively. This corresponds to relative deformations of 0.05 and 0.01 for Vivant and Kolibri, respectively. It is likely that Vivant sagged more by its own weight due to its lower resistance to deformation. In several cases clamping led to changes in the test piece which complicated a correct analysis of the test. Clamping of test pieces of the stronger dough sometimes resulted in bending of the dough in a horizontal plane (Fig. 4A). In such a case the relative deformation at a certain hook displacement will be higher than assumed in the calculations. Sometimes after clamping the test piece was unevenly distributed between the plates at each side of the hook (Fig. 4B). This will result in an unequal deformation at each side of the hook.

These deformations shortly prior to testing will result in differences in deformation history between test pieces and therefore will have negative effects on the quality of the tests and the reproducibility of the results. Several other methods to determine the large deformation and fracture properties of wheat flour dough have been described in literature (Tschoegl *et al.* 1970; de Bruijne *et al.* 1990; Schweizer and Conde-Petit 1997; Uthayakumaran *et al.* 2002). In these cases sagging of the test-piece was prevented by submerging the sample

3A



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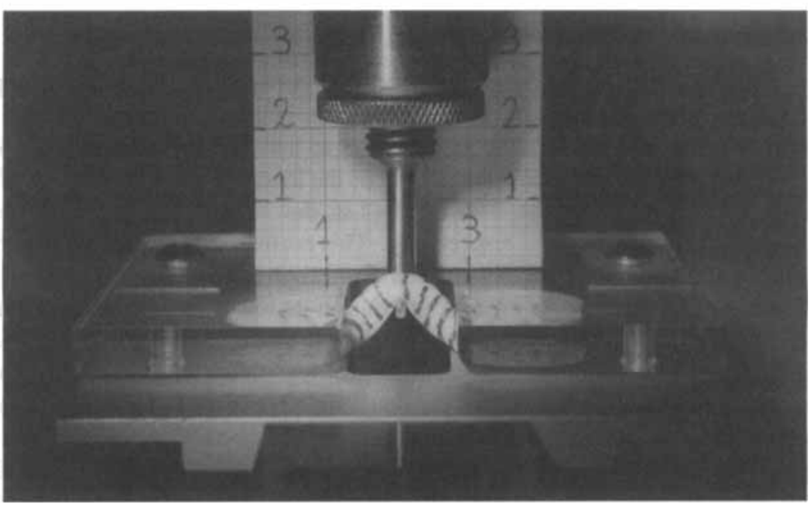

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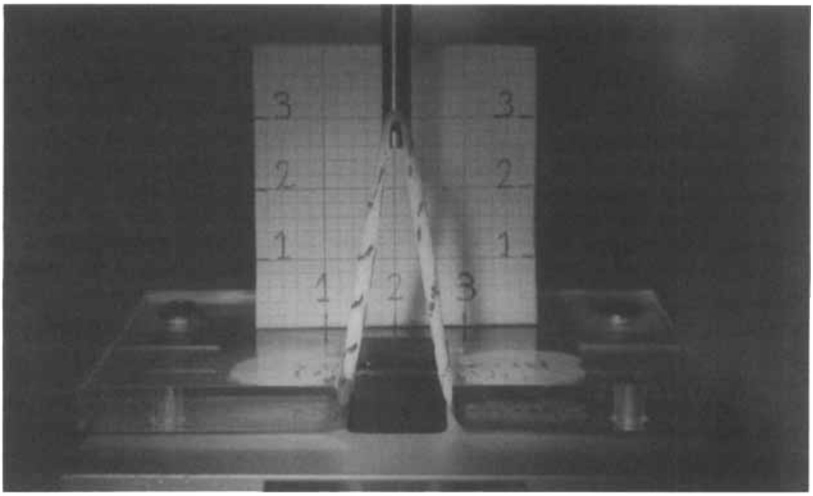
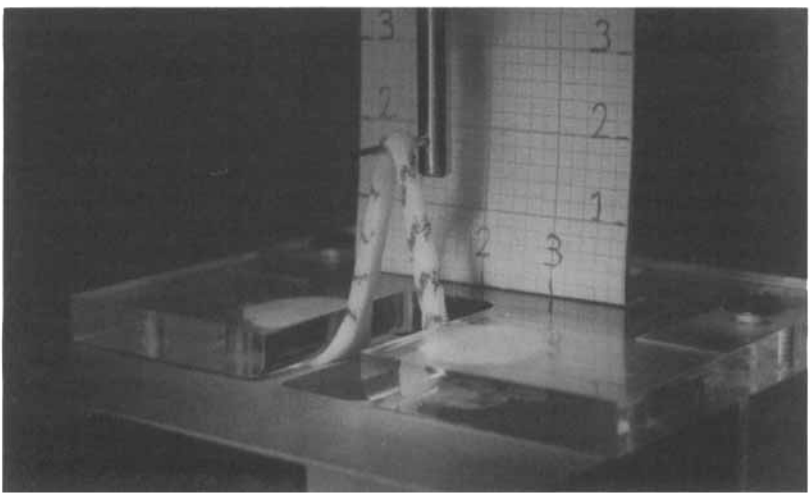
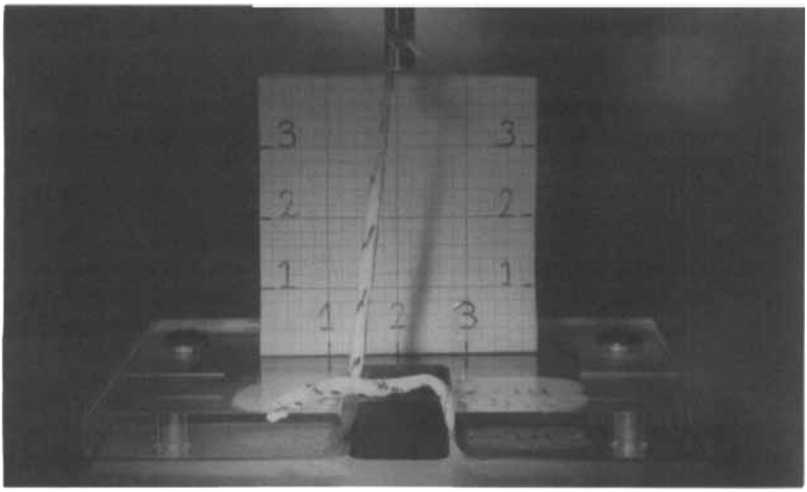


3D



3E



3F

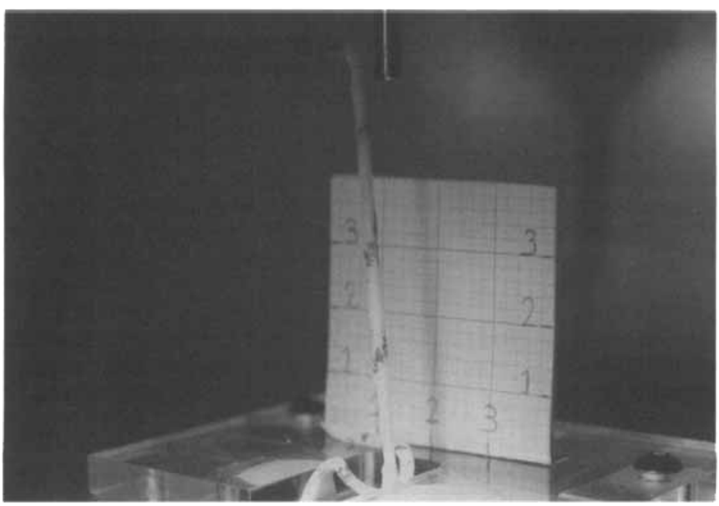


FIG. 3. SOME TYPICAL SITUATIONS AT DIFFERENT STAGES OF EXTENSION OF A FLOUR DOUGH SAMPLE IN THE KIEFFER EXTENSIBILITY RIG

Lines are drawn on the test sample for the determination of the type of deformation during extension. The numbers on the board refer to centimeters. 3A: Front view of the test piece after clamping before the start of the extension test. Note the sagging of the test piece. 3B: Side view of the test piece at an early stage of extension. 3C: Front view of the test piece at a further stage of extension. Note the tilted lines drawn on the test piece. 3D: Side view of the test piece at a further stage of extension. 3E: Front view of the extended test piece after fracture. Note the inhomogeneous deformation. 3F: Side view of the extended test piece after fracture.

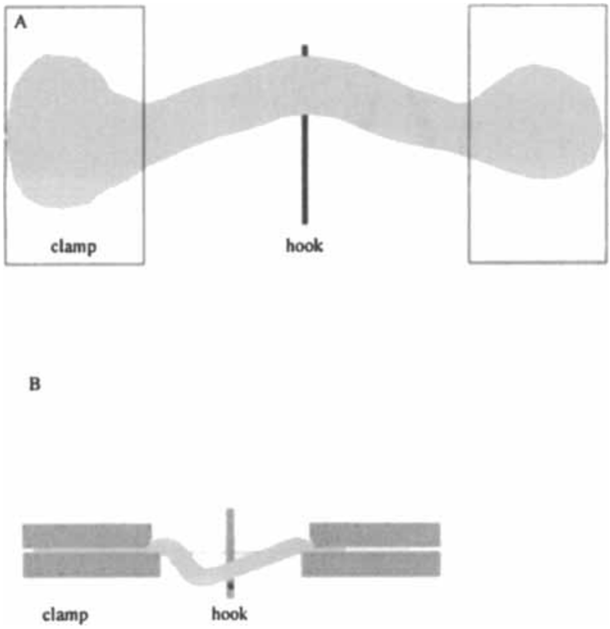


FIG. 4. DEFORMATION OF THE TEST PIECE IN THE GAP DURING CLAMPING NEXT TO SAGGING
4A: bending in a horizontal plane (top view). 4B: unequal distribution of dough over the gap as a result of unequal amounts of dough between the plates at each side of the gap (front view).

in a liquid of matching density (Tschoegl *et al.* 1970), by extending the dough on a mercury bath (de Bruijne *et al.* 1990), by flowing carrier gas between the sample and the sample table (Schweizer and Conde-Petit 1997) and by pulling apart a cylindrical dough piece that was adhered between a lower and upper grip using super-glue (Uthayakumaran *et al.* 2002).

Type of Deformation

As described above, the dough sample is deformed shortly before the start of the extension test due to the clamping of the test piece. During extension the dough just beside the clamps and on the hook became thinner in vertical direction and between 2 and 3 times as wide in horizontal direction as the rest of the sample (Fig. 5A and 5B at the points 1, 3, 4 and 6, and photograph in Fig. 3C). This unwanted deformation could be reduced by using a thicker hook (diameter 4.55 mm).

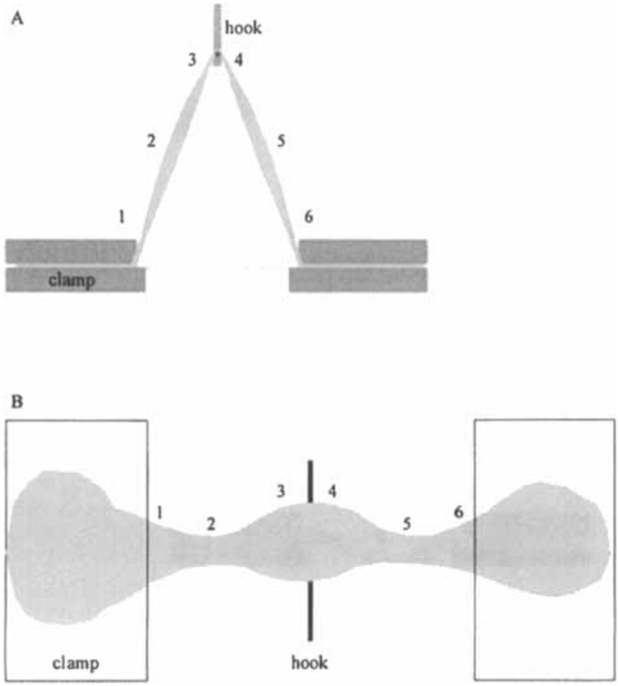


FIG. 5. THE DEFORMATION OF THE TEST PIECE IN THE MICRO-EXTENSOGRAPH
5A: front view. 5B: top view. The numbers are referred to in the text.

The type of deformation during clamping and extension was further investigated by marking test pieces with an overhead marker. Figure 3 shows typical pictures taken at different stages of a test. In Fig. 3A the sagging of the sample is very clear. Figure 3B shows the early stage of extension. The marks at the side and on the top of the sample are connected and perpendicular to the sides of the test piece. At further extension (Fig. 3C) the lines on the side of the test piece became tilted. The direction and extent of tilt of the lines varied along the sample. It implies a shear component in the x-y-direction. This complicates the strain calculation. In the calculation of the stress-strain curves we will neglect the contribution of the shear component to the total strain. During mixing, dough is deformed in combinations of shear and uniaxial elongation at high rates (MacRitchie 1986). According to Gras *et al.* (2000) dough mixing by a pin mixer can be viewed as a series of uniaxial extension tests. The Kieffer test therefore is related to mixing.

Figure 3D shows that the marks at the top were shifted compared to the lines on the side. Sometimes a shear component in the x-z-direction was noticed (not visible on the photographs). A tortuous sample or inhomogeneities in the dough may be the cause. Figures 3E and 3F show very inhomogeneous deformation; some parts were more extended than others and therefore fractured earlier. Contrary to what was observed for the extensograph (Bloksma and Bushuk 1988) and the mercury bath method (de Bruijne *et al.* 1990) the amount of dough that is extended remains more or less constant throughout the test with the Kieffer rig. Inhomogeneous deformation of dough samples during uniaxial extension was also described for the extensograph by Muller *et al.* (1961), for the stretching of dough rings by Tschoegl *et al.* (1970) and was very obvious in the extension of a cylindrical dough piece that was adhered using super-glue (Uthuyakumaran *et al.* 2000). In theory the best piece of equipment for this purpose is the Meissner caterpillar-type rheometer (Meissner and Hostettler 1994), however, more recently, Schweizer (2000) described a large collection of experimental difficulties associated with the measurement of uniaxial extension properties in this apparatus. It was also shown that to obtain a homogeneous deformation in the Meissner caterpillar-type rheometer, very careful sample preparation is of utmost importance (Schweizer and Conde-Petit 1997).

Force and Stress

Initially, the actual force exerted on a dough is higher than the measured force (Fig. 6). At the end of the measurement, however, it will be about half of the measured force. The latter is due to the change in angle under which the force is applied (Eq. 5 and 6). Because the deformation is never completely homogeneous and the force acts locally on an area smaller than the average one (Eq. 7), the calculated stress will always be somewhat lower than the maximum real stress in the test piece. In Fig. 6 it can be seen that the calculated stress, assuming a homogeneous deformation, reaches its maximum at a higher deformation than the force. The difference is the largest in case of the higher displacement speed, where fracture occurs at a high deformation. While the length of the dough cylinder increases steadily, the area on which the force acts decreases. If the force decrease is less than the decrease in cross section of the test piece the stress will still increase. The maximum in stress better represents the strain at which the sample fractures macroscopically than the maximum force. An increase of the force (and thus stress) was observed with increasing hook displacement speed (Fig. 6). Similar trends were reported for wheat flour dough in uniaxial extension tests (Tschoegl *et al.* 1970) and in biaxial extension tests (Janssen *et al.* 1996; Kokelaar *et al.* 1996).

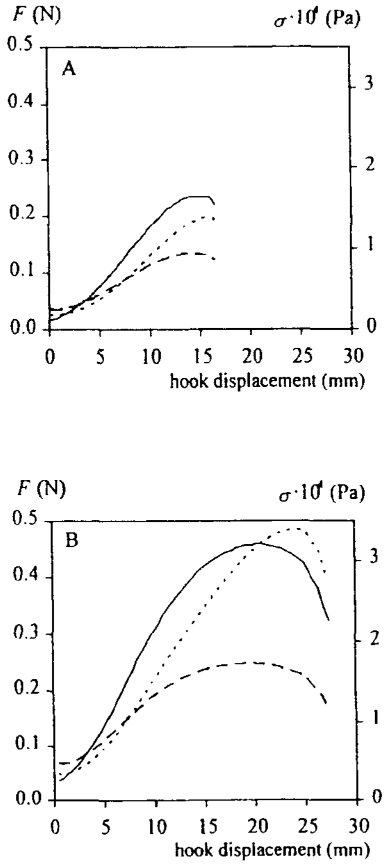


FIG. 6. AN AVERAGE (N = 10) CURVE OF MEASURED FORCE (F_m, SOLID LINE) AND CALCULATED FORCE (F_d, BROKEN LINE) AND STRESS (σ, DOTTED LINE) IN KOLIBRI + GLUCOSE OXIDASE DOUGH FOR A DISPLACEMENT SPEED OF 12 (A) AND 300 (B) mm/MIN

Position 2 mm above the surface of the lower plate was chosen as the start of extension.

Starting Point

Squeezing the dough out of the clamps results in a dough piece which is longer than the width of the gap (Fig. 3A). The starting point of the extension has to be defined as the hook position above the lower plate where the dough piece has the same length as before the hook touched it. This means that the hook has to travel to a point y_0 above the lower plate before it really starts to extend the test piece (Fig. 1A). For correct calculations of strain, strain rate and stress, this point has to be known exactly.

To determine this point for every separate test piece it can be reasoned that the extension actually starts when the force is greater than gravitational force. At that moment y_t is set to zero. Assuming a mass of 0.4 g for the sample the force exerted by gravity is $0.4.10^{-3} * 9.81 = 0.004$ N. However, it was observed that at any speed v, the force of 0.004 N was already exceeded when the hook was still below the surface of the upper plate (Fig. 7). This shows that a preload force cannot be used for determining the starting point of this extension test. The resistance against bending of the dough, while it is lifted by the hook, will be responsible for this phenomenon. Due to bending some parts of the test piece will be compressed and other parts somewhat extended. The higher the speed, the higher the bending rate and the higher the required force.

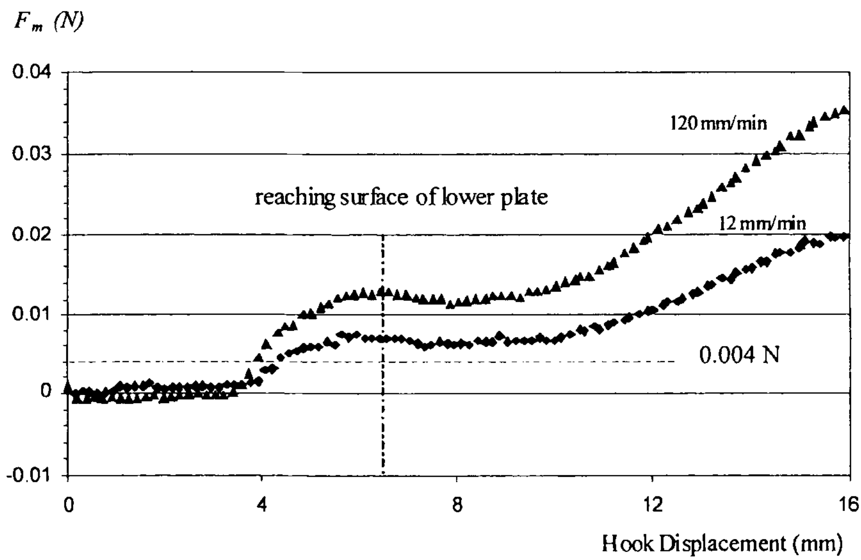


FIG. 7. MEASURED FORCE F_m AS A FUNCTION OF THE HOOK DISPLACEMENT FOR VIVANT DOUGH

The dotted vertical line indicates the displacement when the hook reaches the upper surface of the lower plate at the given speed (indicated in mm/min). The broken horizontal line indicates the displacement when a force of 0.004 N was reached.

Another possible way to determine the exact starting point of the test is by measuring the distance the hook moves between the point where it touches the dough piece, i.e., where the force starts to deviate from zero, and the surface of the lower plate. This difference should be equal to y_0. However, the point at which the force starts to deviate from zero was difficult to estimate accurately enough from the data, especially at lower speeds. So, when using a force to determine the starting point an error may be introduced.

Figure 7 shows that in the first part of the force-hook displacement curve three stages can be distinguished: an initial rise of the force, a part where the force increases less steeply with hook displacement or even decreases and a part where the force increases at an increasing rate. It can be argued that at y_0 the force will start to increase faster through actual extension of the dough, so one can also choose the inflection point in the force-displacement curve as starting point of the extension. As the inflection point in the force-displacement curve is hard to estimate, especially at high speeds, it would be better to use a method that is independent of the force-displacement curve.

Perhaps the best method to calculate the starting point is to combine the measured distance between the top of the lower plate and the bottom of the dough piece after clamping and the displacement of the hook where it has reached the surface of the lower plate. The starting point is then defined as the point where the hook has lifted the dough as much as it had sagged. This point should be determined for each type of dough again as sagging varies with cultivar, amount of water added, mixing time, etc.

Fracture

Photographs 3E and 3F show the fractured dough. The location at which the dough fractured, has been scored for 33 samples at different test speeds v (30 up to 300 mm/min). The score was 1, 0, 14, 16, 0 and 2 for the positions 1 to 6 indicated in Fig. 5. The test speed and type of dough had no influence. The bending over the hook, involving an important shear deformation above the tensile deformation, is probably the cause for the fracture occurring mainly at positions 3 and 4. When using a thicker hook (4.55 mm) fracture of Kolibri doughs occurred more often at positions 1 and 6, probably also caused by the relative stronger bending of the sample at these points.

It was noticed that just before fracture, extension was concentrated in a certain part of the test piece. At that position the dough piece became increasingly thinner and finally fractured. It depended on dough properties how fracture occurred. The weaker dough showed the strongest necking effect at the position where it was going to fracture. The stronger dough fractured more suddenly and faster. Sometimes holes were formed in the thinner regions close to the plates and the hook, in some cases leading to fracture.

As already mentioned, the maximum in stress is probably the best choice for the strain at macroscopic fracture. It is likely that fracture is initiated already before the strain where the force is highest. However, due to low crack speeds, as a consequence of extensive energy dissipation, it will take some time (depending on the dough) before the crack has extended over the whole cross-section of the test piece (slow fracture propagation) (van Vliet *et al.* 1993).

The deformation at fracture increased with increasing speed (Fig. 7 and Fig. 8). This is in accordance with measurements on flour doughs (Tschoegl *et al.* 1970) and gluten doughs (Rinde *et al.* 1970). However, it is opposite to the effect usually found for viscoelastic materials (van Vliet *et al.* 1993). For flour doughs this might be caused by the abundance of starch granules and gluten "particles" in the doughs. Much energy will be dissipated due to friction between them. The large energy dissipation causes crack growth in dough to proceed slowly. During the time the crack that ultimately will cause fracture grows, the dough is further deformed. The higher the speed the larger this deformation during crack growth. This results in a larger measured fracture strain at higher speeds (van Vliet *et al.* 1993).

Reproducibility

Figure 8 gives an example of a series of measurements at different speeds. No special precautions were taken to standardise the measuring procedure. At every speed the graph shows a large variation in force at certain values of extension and in force and deformation at fracture. Reproducibility of fracture stress and strain was determined for 44 samples of Kolibri dough at a displacement speed of the hook of 60 mm/min. Average fracture stress was 28.8 kPa (st. dev. 2.9) and average fracture strain was 1.0 (st. dev. 0.13). Presumably the large variation partly originates from the relative small sample size and the different preparations of small doughs in the mixograph. Furthermore variations in test piece volume and deformation history, as described above, will have their influence on the reproducibility of the test.

However, by using well standardised procedures much less variation between different determinations can be obtained (Kieffer *et al.* 1998; Verbruggen and Delcour 2003; Sliwinski *et al.* 2003). Figure 9 shows force-displacement (9A) and stress-strain (9B) data at fracture obtained from such measurements. Average results and coefficients of variation are shown in Table 1. Also from these results the effect of speed is obvious. Variation in the fracture force between the different measurements is relatively small, which can be explained by the fact that curves tend to flatten at larger displacements. Since the stress increases with increasing displacement (Fig. 6) the variation in fracture stress is larger. On the other hand the variation in displacement is larger than the variation in strain, which is due to the fact that the strain is a logarithmic value. It is nevertheless preferable to apply large range of speeds to obtain clear (significant) effects of deformation speed on the stress-strain curve.

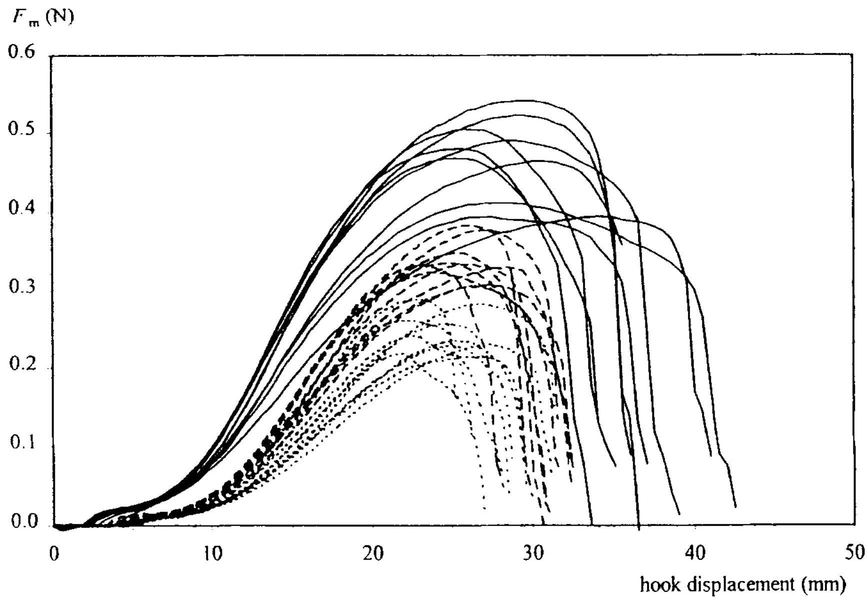
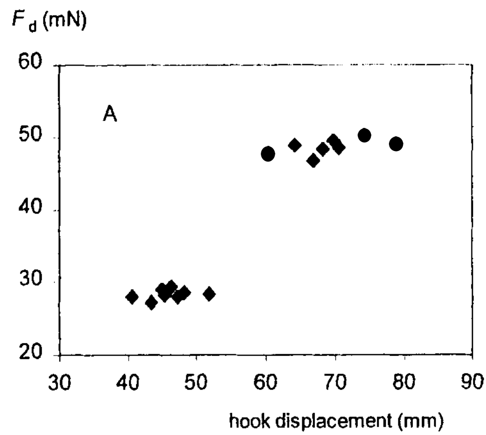


FIG. 8. MEASURED FORCE F_m AS A FUNCTION OF HOOK DISPLACEMENT AT THREE DIFFERENT SPEEDS MEASURED ON KOLIBRI + GLUCOSE OXIDASE
Solid lines 300, broken lines 60 and dotted lines 12 mm/min.

CONCLUSIONS

The 'Kieffer dough and gluten extensibility rig' is a load-extension instrument that can be considered as a micro-extensograph. Contrary to the extensograph, with the 'Kieffer dough and gluten extensibility rig' smaller dough samples can be tested lower, and therefore more relevant strain rates can be applied and experimental data can be expressed in terms of stress and strain. Test pieces are easiest taken from the mould by pressing them out of the mould using a stick. Clamping of the test pieces shortly before the start of experiments caused sagging of the dough cylinders resulting in a deformation of the test sample shortly before the start of the measurement and difficulties in the determination of the exact starting point of extension. To determine this point two methods are recommended. A hook position above the lower plate could be taken, which is based on the measured distance between the top of the lower plate and the bottom of the dough piece after clamping, but it is also possible to estimate the inflection point in the force-hook displacement curve. Deformation was not purely uniaxial extension. A shear component was also observed, which probably caused fracture to occur mainly near the hook. The maximum in stress represents better the strain at which the sample fractures macroscopically than the maximum in the force. A relatively large variation in stress and strain at fracture was observed. Variation in the deformation history and the volume of the test pieces have a negative effect on the reproducibility. Therefore a well-standardised test procedure has to be followed.



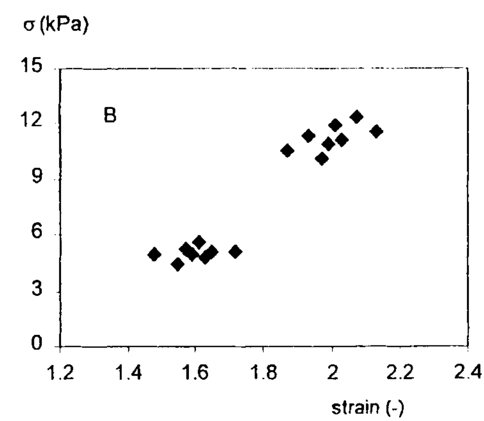


FIG. 9. FRACTURE PROPERTIES FOR VIVANT FLOUR DOUGH DETERMINED AT TWO DIFFERENT DISPLACEMENT SPEEDS OF THE HOOK; 12 mm/MIN AND 120 mm/MIN

9A: Calculated force F_d at fracture as a function of hook displacement. 9B: Fracture stress as a function of fracture strain.

TABLE 1.
FRACTURE STRESS, σ, AND FRACTURE STRAIN, ε_h, DETERMINED IN UNIAXIAL EXTENSION AT TWO DISPLACEMENT SPEEDS OF THE HOOK FOR FLOUR DOUGH OF VIVANT. DATA ARE AVERAGES OF 8 MEASUREMENTS. T = 25C

Hook speed (mm/min)	Force (mN)		Displacement (mm)		σ (kPa)		ε_h (-)	
	Average	CV	Average	CV	Average	CV	Average	CV
120	48.6	±2%	69.2	±8%	11.2	±6%	2.0	±4%
12	28.2	±2%	46.0	±7%	5.0	±6%	1.6	±4%

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REFERENCES

BAKER, J.C. and MIZE, M.D. 1941. The origin of the gas cell in bread dough. Cereal Chem. *18*, 19–34.

BLOKSMA, A.H. 1962. Slow creep of wheat flour doughs. Rheol. Acta *2*, 217-230.

BLOKSMA, A.H. 1990a. Rheology of the bread making process. Cereal Foods World *35*(2), 228-236, 959-960.

BLOKSMA, A.H. 1990b. Dough structure, dough rheology, and baking quality. Cereal Foods World *35*(2), 237-244.

BLOKSMA, A.H. and BUSHUK, W. 1988. Rheology and chemistry of dough. In *Wheat: Chemistry and Technology*, 3rd Ed., Volume II, (Y. Pomeranz, ed.) pp. 131-217, American Association of Cereal Chemists, St. Paul, Minnesota.

DE BRUIJNE, D.W., DE LOOF, J. and VAN EULEM, A. 1990. The rheological properties of bread dough and their relation to baking. In *Rheology of Food, Pharmaceutical and Biological Materials with General Rheology*, (R.E. Carter, ed.) pp. 269-283, Elsevier Applied Science, Barking, UK.

DUNNEWIND, B., VAN VLIET, T. and ORSEL, R. 2003. Effect of oxidative enzymes on bulk rheological properties of wheat flour doughs. J. Cereal Sci. *36*, 357–366.

GRAS, P.W., CARPENTER, H.C. and ANDERSSEN, R.S. 2000. Modelling the developmental rheology of wheat-flour dough using extension tests. J. Cereal Sci. *31*, 1–13.

GRAUSGRUBER, H., SCHÖGGL, G. and RUCKENBAUER, P. 2002. Investigations on the validity of the micro-extensigraph method to measure rheological properties of wheat doughs. Eur. Food Res. Technol. *214*, 79–82.

HOSENEY, R.C. and ROGERS, D.E. 1990. The formation and properties of wheat flour doughs. Crit. Rev. Food Sci. Nutr. *29*(2), 73–93.

JANSSEN, A.M., VAN VLIET, T. and VEREIJKEN, J.M. 1996. Fundamental and empirical rheological behaviour of wheat flour doughs and comparison with bread making performance. J. Cereal Sci. *23*, 43–54.

KIEFFER, R., KIM, J.-J. and BELITZ, H.-D. 1981a. Zugversuche mit Weizenkleber im Mikromaßstab. Z. Lebensm. Unters. Forsch. *172*, 190–192.

KIEFFER, R., GARNREITER, F. and BELITZ, H-D. 1981b. Beurteilung von Teigeigenschaften durch Zugversuche im Mikromaßstab. Z. Lebensm. Unters. Forsch. *172*, 193–194.

KIEFFER, R., WIESER, H., HENDERSON, M.H. and GRAVELAND, A. 1998. Correlations of the breadmaking performance of wheat flour with rheological measurements on a micro-scale. J. Cereal Sci. *27*, 53–60.

KOKELAAR, J.J., VAN VLIET, T. and PRINS, A. 1996. Strain hardening properties and extensibility of flour and gluten doughs in relation to breadmaking performance. J. Cereal Sci. *24*, 199–214.

MACRITCHIE, F. 1986. Physicochemical processes in mixing. In *Chemistry and Physics of Baking: Materials, Processes and Products*, (J.M.V. Blanshard, P.J. Frazier and T. Galliard, eds.) Roy. Soc. Chem., London.

MEISSNER, J. and HOSTETTLER, J. 1994. A new elongational rheometer for polymer melts and other highly viscoelastic liquids. Rheol. Acta *33*, 1–21.

MULLER, H.G., WILLIAMS, M.V., RUSSELL EGGITT, P.W. and COPPOCK, J.B.M. 1961. Fundamental studies on dough with the Brabender extensograph. I.-Determination of stress-strain curves. J. Sci. Food Agric. *12*, 513–523.

RINDE, J.A., TSCHOEGL, N.W. and SMITH, T.L. 1970. Large-deformation and rupture properties of wheat flour gluten. Cereal Chem. *47*, 225–235.

SCHOFIELD, R.K. and SCOTT BLAIR, G.W. 1932. The relationship between viscosity, elasticity and plastic strength of soft material as illustrated by some mechanical properties of flour doughs. Proc. Roy. Soc. (London) *A 138*, 707–719.

SCHWEIZER, T. 2000. The uniaxial elongational rheometer RME — six years of experience. Rheol. Acta *39*, 428–443.

SCHWEIZER, TH. and CONDE-PETIT, B. 1997. Bread dough elongation. In *Proceedings of 1st International Symposium on Food Rheology and Structure*, (J. Windhab and B. Wolf, eds.) pp. 391–394, ETH, Zurich.

SLIWINSKI, E.L., KOLSTER, P. and VAN VLIET, T. 2003. Large-deformation properties of wheat dough in uni- and biaxial extension. Part I: Flour dough. Rheol. Acta. (In press).

TSCHOEGL, N.W., RINDE, J.A. and SMITH, T.L. 1970. Rheological properties of wheat flour doughs. I. Method for determining the large deformation and rupture properties in simple tension. J. Sci. Food Agric. *21*, 65–70.

UTHAYAKUMARAN, S., NEWBERRY, M., KEENTOK, M., STODDARD, F.L. and BEKES, F. 2000. Basic rheology of bread dough with modified protein content and glutenin-to-gliadin ratios. Cereal Chem. *77*(6), 744–749.

UTHAYAKUMARAN, S., NEWBERRY, M., PHAN-TIEN, N. and TANNER, R. 2002. Small and large strain rheology of wheat gluten. Rheol. Acta *41*, 162–172.

VAN VLIET, T., JANSSEN, A.M., BLOKSMA, A.H. and WALSTRA, P. 1992. Strain hardening of dough as a requirement for gas retention. J. Texture Studies *23*, 439–460.

VAN VLIET, T., LUYTEN, H. and WALSTRA, P. 1993. Time dependent fracture behaviour of food. In *Food Colloids and Polymers: Stability and Mechanical Properties*, (E. Dickinson and P. Walstra, eds.) pp. 175–190, Roy. Soc. Chem., Cambridge, UK.

VERBRUGGEN, I.M. and DELCOUR, J.A. 2003. Extension properties of wheat flour dough fortified with characterised wheat gluten fractions. J. Cereal Sci. *37*(2), 151–156.

WALKER, C.E. and HAZELTON, J.L. 1996. Dough rheological tests. Cereal Foods World *41*, 23–28.