

**Mechanical Properties of Short Doughs and their
Corresponding Biscuits**

Promotor: dr. ir. P. Walstra
hoogleraar in de zuivelkunde

Co-promotor: dr. ir. T. van Vliet
universitair hoofddocent in de levensmideleennatuurkunde

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Alexios Baltasvias

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Theorems

1. The continuity of fat in a short dough may be manipulated by changing the fat content, the fat type, or by adjusting mixing time.

This thesis

2. In short doughs sucrose has a dual role; it facilitates formation of a non-fat continuous phase via its effect on solvent quantity and retards, or even inhibits, gluten development by affecting solvent quality.

This thesis

3. It has been known for several years that the addition of solid fat makes biscuits more tender to eat. This is at least partly due to the fat forming a continuous phase and to the higher air volume fraction that it induces.

This thesis

4. Whether biscuits are in a glassy or rubbery state cannot be predicted from their water contents in relation to the state diagrams of individual components.

This thesis

5. The observation of Miller that doughs with low water content are more sensitive to temperature than those with high water content is likely to be explained by the former doughs being "more bicontinuous" than the latter.

Miller, A.R. In "Rheology of Wheat Products" (H. Faridi, ed.) Am. Assoc. Cereal Chem., St. Paul, Minnesota (1985) pp 117-132.

6. Doescher *et al.* suggest that the spreading of short dough in the oven stops shortly after reaching the glass transition temperature of gluten. In their experiments, however, flour was replaced by gluten and, therefore, the affinity of starch and pentosans for water was neglected.

Doescher, L.C., Hosney, R.C. and Milliken, G.A. *Cereal Chemistry* 64 (1987) 158-163.

7. Slade and Levine suggest that a sucrose solution has a smaller plasticising action on gluten as compared to a fructose solution. To examine the validity of this idea, large deformation experiments would be a valuable tool, provided that differences in quantity and viscosity of sugar solutions are taken into account.

Slade, L. and Levine, H. In "The Science of Cookie and Cracker Production" (H. Faridi, ed.) Chapman & Hall, New York (1994) pp 23-141.

8. The most widely used method for monitoring the hydrogenation of edible oils is the solid fat index. However, the relationship between the solid fat index and the consistency of the finished product depends on the oil and the way it is subsequently handled.

9. Warnings of health hazards are printed on all cigarette packets and advertising of cigarettes on commercial television is banned. Similar measures are not taken for alcoholic beverages, possibly because the social climate is more favourable to drinking.

10. Frequently, when people are judged as being "different", they are also judged as being "wrong".

Preface

I am particularly indebted to my promotor, Pieter Walstra, and my co-promotor, Ton van Vliet, for their constant and friendly supervision and guidance during the course of this work.

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Abstract

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Keywords: Short doughs, short-dough biscuits, rheology, fracture, glassy state, fat, gluten, sucrose.

The mechanical properties of short doughs of various composition were determined in small amplitude oscillatory experiments and in uniaxial compression. Regardless of composition, the linear region was very limited; beyond that, pronounced yielding and flow occurred. Conductimetry was also used to evaluate the effect of fat type and fat content on dough structure. Short doughs showed large differences in mechanical spectra, conductivity and apparent biaxial extensional viscosity, according to fat and sucrose contents, fat type and mixing time. It is concluded that short doughs are bicontinuous systems; reducing the fat content or changing the rheological properties of the fat relative to those of the non-fat phase results in fat-dispersed systems. The rheological properties of the non-fat phase are largely determined by intact flour particles present in a concentrated sucrose syrup. Sucrose delays, if not inhibits, gluten development through its effect on solvent quality and facilitates formation of a non-fat continuous phase via its effect on solvent quantity. Mixing promotes formation of a continuous fat phase.

Mechanical properties of short-dough biscuits of various composition were determined in three-point bending tests. Increasing fat content or omitting sucrose from the recipe decreased the modulus and the fracture stress of the biscuits. The effect of fat content, however, was dependent on fat type. Temperature during dough preparation, dough water content and temperature during bending tests affected the mechanical properties of biscuits to an extent which depended on fat content. Diffusion of Sudan III into the biscuits indicated that low-fat biscuits are fat-dispersed systems and high-fat biscuits are bicontinuous. Differential scanning calorimetry showed that, irrespective of composition, starch gelatinisation was slight, if not absent, presumably due to the limited water content coupled with the low baking temperature. Under certain storage conditions, biscuits are in a glassy state. Upon water uptake, the matrix undergoes a glass-rubber transition. It is concluded that the mechanical properties of biscuits are mainly determined by air volume fraction, fat continuity, size of inhomogeneities, and physical state of the non-fat phase.

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Chapter 1

Introduction

1.1 Definitions

The terminology used for both doughs and biscuits varies among countries. In the UK the name short dough is related to recipes relatively rich in fat and sucrose with small amounts of water¹. A basic difference between short doughs and others is that the former lack extensibility, are not coherent under tension and break easily^{2,3}. In the USA short doughs are classified according to the method of dough piece formation, *e.g.* rotary-moulded, wire-cut and rout-/bar-press (sometimes called deposited). Differences in these methods are related to variation in dough consistency, *e.g.* stiff doughs are formed on a rotary moulder. This variation in dough consistency reflects variation in dough composition². For example, fat content ranges between 20 and 60% and sucrose content between 25 and 55% (expressed as percentage of weight of flour)³. Table 1.1 shows some typical short-dough recipes³⁻⁷.

In the UK short-dough biscuits are known as sweet biscuits¹. In the USA they are again subdivided according to the method of dough piece formation, *i.e.* rotary-moulded, wire-cut (*e.g.* sugar-snaps, ginger-snaps), etc².

Table 1.1 Typical recipes for various short doughs.

Ingredient (g)	Rotary moulded	Wire cut	Rout/bar press	Sugar snap
Flour	100	100	100	100
Sucrose	29-33	43-45	40-45	60
Fat	27-32	40-45	63-70	30
Water	10-14	17-23	6-12	17-22
Salt	1-2	1	1	1
Non-fat dry milk	1-2	1-2	3	0-3
Leavening agents	1	1	0-2	1-2

Short-dough biscuits, though they comprise widely varying products, are distinguished from other types in that the former tend to become larger in width and length during the early stages of the baking process (so-called "spread"). It is believed that the dough piece tends to spread during baking due to the absence of a continuous

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protein network³. The spread of short-dough biscuits during baking is taken as a criterion of flour quality and is traditionally evaluated by either biscuit width or diameter alone, or by the so-called "spread factor", which is the ratio of biscuit width or diameter to biscuit thickness; the larger the values the better the flour quality would be⁸.

1.2 Principal ingredients

1.2.1 Wheat flour

Wheat flour is the principal component of nearly all biscuits. The properties of the flour obtained on milling vary with the variety and with the agronomic and climatic conditions under which the wheat is grown and harvested. The terms "hard" and "soft", as used to describe wheat, relate to the milling characteristics of the wheat grains. When a grain of soft wheat is milled the resulting flour contains, not only fragments of endosperm, but also free starch granules and fragments of free protein. The endosperm of hard wheat on the other hand breaks into larger fragments, resulting in the production of a coarser flour containing many damaged starch granules¹. Hard wheats tend to have higher protein contents than soft wheats. However, the hardness of a given cultivar of wheat is genetically controlled and is not directly correlated with the protein content of the kernel². Flour from hard wheats is primarily used for bread making and flour milled from soft cultivars for making biscuits and cakes. In general, biscuits made from soft wheat flour have a better appearance, softer bite and greater tenderness than those made from hard wheat flour². It was observed that flours from soft wheat cultivars produced biscuits with larger width or diameter⁹⁻¹³, smaller height⁸ and lower bulk density¹⁴ than hard flours did. Biscuits baked from soft wheat flours continued to spread for a longer period during baking than did biscuits baked from hard wheat flours; doughs from the latter flours showed a rapid viscosity increase at an earlier stage and lower temperatures of the baking process, although rates of dough temperature increase and water loss were comparable¹⁵⁻¹⁷.

The principal reasons for evaluating flour quality in short doughs are, firstly, to produce reproducible dough consistencies, appropriate to the forming method and machinery and, secondly, to give satisfactory biscuits. The problems of specifying flour quality for biscuit making arise very much from the recipe of the product and the effect on the baked biscuit³. It has been stated^{5,7,18} that the suitability of instrumental measurements, and of measurements of biscuit width or thickness, to ascertain soft wheat quality depended on the biscuit recipe and on mixing time; the "spread factor" should not be used as a flour quality index^{19,20}.

The water absorption characteristics of the flour appear to be important; the amount of added water required to obtain a dough of satisfactory consistency was appreciably lower with flours from soft wheats than with those from hard wheats¹⁴. A negative correlation between the amount of water absorbed by the flour and biscuit diameter was observed^{10,12,21-25}. However, Gaines and Tsen⁵ found that such correlations depended on biscuit recipe. Fractionation and reconstitution studies showed that the tailings and the water-soluble fractions decrease biscuit diameter^{26,27} and increase biscuit hardness²⁸. The tailings fraction consists of damaged starch, small starch granules, proteins, lipids, cellulosic material and water-insoluble pentosans²⁹. The water-soluble fraction comprises albumins, globulins, glycoproteins, and water-soluble pentosans³⁰. The detrimental effect of water-solubles and tailings is associated with their high water absorption characteristics^{23,27}. Krishnarau and Hosene³¹ replaced sucrose in biscuits by hydrolysed starch tailings; biscuits containing a sample hydrolysed for 0.5 hour had the least spread, whereas those containing starch tailings hydrolysed for 2 hours spread the most. The effect was attributed to the molecular weight of the hydrolysis products.

Another possible quality attribute of soft wheat flours is particle size. It was observed that soft wheat flours generally had smaller particle size and produced larger biscuits than hard wheat flours^{10,32}. Wheat flours passed over a 325-mesh sieve yielded two fractions; for soft flour, the fine fraction resulted in larger biscuits than the coarse counterpart, whereas no differences were observed between the two fractions of hard flour⁹. On the other hand, changes in granularity of flour, resulting from the milling of wheat tempered to various water levels, were not associated with significant biscuit diameter changes³³. However, when soft flour from higher-tempered wheat was sieved, the fine fraction produced larger biscuits than did the coarse fraction³³. Reducing the mean particle size of flours from both hard and soft wheats substantially increased the levels of dough water required in short dough formulations¹⁴. Bulk density¹⁴ and diameter^{10,34,35} of short-dough biscuits decreased with increasing extent of flour grinding, *i.e.* with decreasing flour particle size. However, extensive grinding also results in a high level of damaged starch^{33,34,36}.

1.2.1.1 Protein

Much importance is attached to the flour protein level, because the protein absorbs twice its weight of water and, therefore, affects the flour water absorption³. Because larger biscuit spread is associated with soft wheats of low protein content^{25,32}, it is generally believed that the protein content should be relatively low.

One approach to study the effect of protein content is through different flour types. Bettge *et al.*³⁷ reported an inverse relationship between protein content and biscuit diameter, that could account for flour quality. However, other researchers^{10,12,22,24}

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found that the protein content could not predict the baking performance of flours from different wheat cultivars. It was observed that flours of higher protein content resulted in harder biscuits; because the lowest protein flours did not produce the softest biscuits, factors such as protein quality may have also contributed to biscuit hardness^{28,38}. Protein content was also negatively correlated with biscuit density⁵.

Protein content within a cultivar can be manipulated by growth conditions. Gaines *et al.*²⁸ found no relation between protein content and biscuit diameter; conversely, Abboud *et al.*²⁴ found a positive correlation.

Another approach involves fractionation and reconstitution methods. Larger biscuits were obtained from reconstituted flours in which gluten had been omitted²⁷.

Finally, the effect of protein content can be studied in flours supplemented with protein. The diameter of biscuits baked from soft flour fortified with increasing levels of commercial vital glutens was not affected; increasing concentration of laboratory-prepared gluten markedly reduced biscuit spread. It was suggested that processing treatments may account for the observed differences between commercial and freeze-dried glutens³⁹.

Information on flour protein quality is incomplete. Gaines and Finney⁴⁰ added commercial proteases to short doughs and found that some proteases (papain and a *Bacillus subtilis* protease) were more effective in increasing biscuit spread than others (an *Aspergillus oryzae* and a bovine pancreatic proteases); the effect of enzymes on dough consistency did not follow any pattern. Gaines²⁵ treated soft wheat flours with two reducing agents that cleave disulphide bonds and obtained contradictory results: L-cysteine gave larger biscuit diameters relative to the control, but dithioerythritol (DTE) decreased the diameter. He also treated the flours with potassium iodate, which oxidises thiol groups, or *N*-ethylmaleimide (NEMI), which blocks thiol-disulphide interchange; the effect on biscuit diameter depended on the cultivar from which flour was obtained. DTE reduced dough consistency, whereas the other agents had relatively little effect.

Although the water level in short doughs is low, it is possible to develop gluten if mixing is excessive; therefore, mixing should be minimal. On the other hand, mixing conditions should be such that adequate dispersion of ingredients is achieved. This is usually done by a two-stage mixing procedure. In the first stage, known as the "cream-up", all the ingredients except the flour are mixed for several minutes at gentle speed. This is done to dissolve as much of the sucrose as possible in the available water, and to disperse the syrup and the undissolved sucrose in the fat. The flour is then added to this "cream" and mixing proceeds again at a gentle speed for less than one minute during which a uniform mixture is formed. This stage is known as the "dough-up" and will yield at one extreme a crumbly dough, which can be moulded with a rotary moulder, or at the other extreme a soft plastic mass suitable for extrusion in various types of machinery. The length of mixing for "cream-up" is not

critical but that for the second stage is. Some manufacturers adopt an "all-in" or single-stage mixing method, which requires prolonged mixing to achieve adequate dispersion of all the ingredients. As a result, the potential for gluten development is greatly increased and the biscuits are regarded as inferior to the products of the two-stage process^{1,3}. However, biscuits made with the "all-in" mixing method tend to spread more than biscuits made with the two-stage mixing method^{24,41}. It was found that biscuit diameter^{18,25} and dough consistency²⁵ changed negligibly with mixing time; however, longer mixing times increased sensory ranking of biscuit hardness²⁵. On the other hand, Olewnik and Kulp⁶ found that, as mixing time increased, the consistency of wire-cut and deposited doughs decreased.

1.2.1.2 Damaged starch

Damaged starch granules are known to absorb much higher levels of water than do undamaged starch granules at low temperatures; the latter absorb about 30% of their weight, whereas the former absorb three times that amount³. A high level of damaged starch decreased biscuit diameter^{24,36,42,43} and increased dough consistency³⁶.

1.2.1.3 Pentosans

Pentosans, although making up only 2-3% of the flour, are important due to their ability to absorb up to 10 times their weight of water². A purified starch tailings fraction isolated from soft wheat flour, which was rich in pentosans, had a great water absorption capacity; this fraction greatly decreased biscuit spread when added in small quantities to a control flour, the effect being proportional to the quantity added^{42,44,45}. Sollars⁴⁶ studied the water-solubles of wheat flour, which were further subfractionated into two fractions: a low molecular weight (68-78% of the original water-solubles) and a high molecular weight. The polysaccharides from the latter fraction greatly reduced biscuit diameter. Contrarily, Abboud *et al.*²⁴ found a poor correlation between the total pentosan content of 56 samples of flour and the spread of biscuits baked from these flours.

1.2.1.4 Water

Variation in dough consistency is commonly blamed on the flour, and adjustment is tried by increasing or reducing dough water level³. However, water present in the flour and added water are not equivalent in terms of their effect on dough properties. If a sample of flour is divided into two parts and the water content of one part is reduced, by even 1-2%, by air drying or tempering, then doughs made from the two parts by adding sufficient water to give the same total water content did not have the

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same consistency¹; biscuit spread was also decreased¹⁰. Gaines *et al.*³⁶ observed that, for a constant added water content, increasing flour water content resulted in a larger biscuit diameter and a lower dough consistency.

1.2.1.5 Flour lipids

It has been shown⁴⁷⁻⁵¹ that biscuits, baked from flours from which the natural lipid had been extracted, had a smaller diameter than those baked from untreated flours; addition of the extracted lipid to the defatted flours restored the original spread. Polar and non-polar lipid fractions alone were only partially effective in improving defatted flour; both were required for full restoration of quality⁴⁸. Biscuit spread increased progressively when both whole and defatted flours were treated with free lipids at one and a half, two, and three times the normal levels^{39,48}. When the lipids of two flours, which differed in biscuit-spread properties, were interchanged, the diameter of the biscuits was the same as that of biscuits from untreated flours⁴⁷. Fractionation of lipids showed that a phosphorus-containing fraction was the functional component⁴⁷. Later, it was suggested⁵¹ that digalactosyldiglyceride and/or phosphatidylcholine are the primary contributors to functionality.

1.2.2 Fats

The addition of fat to the dough has the effect of reducing the amount of water required to achieve a dough of desired consistency and of making the product more tender-eating¹.

Commercial fats consist of a mixture of solid and liquid triglycerides. The presence of some solid fat in the dough during mixing and shaping is essential; the use of liquid oil, or too high a dough temperature resulting in melting of the fat, produces adverse changes in the handling characteristics of the dough². The solid content of the fat at mixing also affects dough density. If rotary moulded, doughs with lower solid fat have higher densities³. On the other hand, the fat must be sufficiently deformable to become well mixed with the other components². It is recommended that there is at least 15-20% solid fat at the dough temperature³. The fraction solid in the fat, and thereby the rheological properties of the commercial fat used in biscuits, are very sensitive to temperature⁵². However, the proportion of solid fat in a given sample is not only dependent on the sample temperature but also depends on the recent temperature history. Thus, two samples of the same fat examined at the same temperature may have different properties, according to their temperature histories¹. Therefore, a constant temperature history during the various processing steps is essential⁵². The structure of fats is also time-dependent even at constant temperature. Most triglycerides below their melting points exhibit more than one crystal modification. In

a mixture of solid and liquid triglycerides recrystallisation from one modification to another takes place at constant temperature.

The ingredient that is principally responsible for binding the other ingredients together in the dough is the fat. This explains why the temperature of the dough plays such an important part in controlling dough consistency¹. Dough temperature should preferably be between 21 °C and 27 °C, or even lower (18-22 °C) if the fat content is high^{3,52}. However, while dough temperature does undoubtedly affect dough consistency (via its effect on the solids content of the fat), mixing to constant final temperature does not appear to offer a suitable method of defining the end point of mixing¹. It should be mentioned that the effect of dough temperature on dough consistency depends on water content; doughs with low water content were more sensitive to temperature than those with high water content⁴.

Miller⁴ and Olewnik and Kulp⁶ found that dough consistency was greatly reduced with increasing fat content. A dough containing 8% soybean oil was found to be softer than one containing 15% fat⁵³ (percentages on flour weight). It was found that neither fat type nor fat content affected biscuit spread^{24,54}. However, it was also found that, in recipes with certain flours and fine sugar granulation, both fat type and fat content influenced biscuit spread^{55,56}. When fat was partly replaced by soybean oil, sensory crispness scores of the biscuits were adversely affected⁵⁷. Burt and Thacker⁵⁸ found that the hardness of biscuits increased with decreasing fat content. Wade⁵⁹ reported that biscuits made with butter fat were harder than those made with either "commercial fat" or lard. Observations of Hornstein *et al.*⁶⁰ suggest that, for a given fat type, the physical properties of the fat is an important variable.

1.2.3 Sugars

The quantity and type of sugar has significant effects on short dough rheology, as well as on the texture, appearance and flavour of the finished product⁵². The principal sugar in short doughs is sucrose. The addition of sucrose to short doughs also has the effect of reducing the added water requirements of short doughs¹. The water levels in most short doughs preclude complete dissolution of the sucrose³. Crystal size affects the rate of sucrose dissolution in water, during dough mixing; the smaller crystals will dissolve more rapidly. When the mean size of sucrose crystals was reduced an increase in biscuit spread was observed^{24,41,55,61}. Greater spread was also obtained by increasing the sucrose level^{8,12,24,41,54,56}. Miller⁴ found that the consistency of rotary-moulded doughs was not sensitive to the recipe level of sucrose. Conversely, Olewnik and Kulp⁶ reported that rotary-moulded and deposited doughs were much softer and stiffer, respectively, with increasing levels of sucrose. Slade and Levine⁸ reported that biscuit diameter increased, whereas biscuit height and dough consistency decreased with increasing sucrose level. Biscuits made with fructose and glucose were thicker and had

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smaller diameter than those made with sucrose; these effects were attributed to differences in melting temperature and rate and extent of dissolution of the various sugars in water^{8,62}. However, Doescher *et al.*¹² observed no differences in diameter of biscuits made with crystalline or predissolved sucrose. When high-fructose corn syrup was substituted for sucrose at a constant dough water content, the biscuit spread factor decreased; in three-point bending tests, deformation to fracture increased, the more so for a higher level of substitution^{63,64}. Similar results were obtained with glucose syrups at a constant volume of solution (water and dissolved sucrose)^{11,12}. The force required to compress doughs to 3 mm was higher for doughs containing sucrose syrup than for those containing glucose syrup or fructose syrup; when doughs were heated to increasing temperatures between 30 and 50 °C and cooled to the measuring temperature (27 °C), the force increased regardless of sugar type¹¹. Bullock *et al.*⁶⁵ found that the resulting biscuits required a smaller force to break, when sucrose was replaced by polydextrose; sensory hardness of biscuits with polydextrose decreased⁶⁶. Contrarily, Campbell *et al.*⁶⁷ reported that polydextrose appears to be a suitable substitute for sucrose, since it had no significant effect on the mechanical properties of biscuits.

1.2.4 Water

Water may be considered a catalyst in short doughs as it allows changes to occur in the other ingredients. From a commercial point of view, since nearly all water must be removed during baking, it is important that the amount of water added be kept to a minimum; it is adjusted so as to produce a dough of acceptable consistency for processing^{2,3}. Miller⁴ observed that consistency of rotary-moulded doughs decreased with increasing water content. Olewnik and Kulp⁶ found that consistency of rotary-moulded and wire-cut doughs decreased and increased with increasing water content, respectively. Slightly increasing dough water content first increased and then decreased biscuit diameter^{10,18,25}; sensory ranking of biscuit hardness increased²⁵ and dough consistency immediately after mixing decreased^{25,68}. The effect of water content on dough consistency was less pronounced in doughs tested 1 hour after mixing⁶⁸. The changes in dough consistency occurring during 1 hour after mixing differ among flours and were found to be a function of water content and initial dough consistency after completion of mixing^{4,68}.

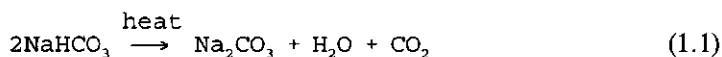
1.2.5 Emulsifiers

The addition of emulsifiers to short doughs appears to provide a means of reducing the amount of fat in the recipe. Incorporation of MDG (mono-diglycerides) in a dough with soybean oil as the fat phase, made it softer⁵³. Gaines⁶⁸ observed that the addition

of lecithin (0.63% on flour weight) caused a decrease in dough consistency, as determined 1 hour after mixing. Commercial soy lecithins (1-2% on flour weight) gave a significant increase in biscuit spread and also restored the spread of biscuits enriched with gluten^{39,69-72}. Burt and Thacker⁵⁸ found that DATEM (diacetyl tartaric acid esters of monoglycerides), SSL (sodium stearoyl lactylate) and CAEM (citric acid esters of monoglycerides) were the most effective in producing softer biscuits. A 20-30% reduction in fat can be successfully achieved on short doughs by using these emulsifiers at 0.5% on a dry ingredients basis. However, the reduction in fat resulted in a much stiffer dough, which necessitated some alteration in the recipe water level to allow proper dough handling properties. It was found that SSL, SSF (sodium stearoyl fumarate) and sucrose esters such as ST (sucrose tallowate), SMP (sucrose monopalmitate), SMDS (sucrose mono- and distearate) and SDS (sucrose distearate) at 0.5% on flour weight effectively increased the spread factor and reduced the fat required in biscuits⁷³. These emulsifiers also improved the spread of biscuits made from hard wheat flour as an alternative to soft flour, and this may be important to counteract variations in flour which occur from year to year⁷⁴. Increasing concentrations of sucrose esters up to 1% on flour weight increased spread factor in biscuits⁷⁵, but little further improvement was seen beyond 1%. In addition to SSL and DATEM, Hutchinson *et al.*⁷⁶ also tested MDG, LAEM (lactic acid esters of monoglycerides), SMG (succinylated monoglycerides), EMG (ethoxylated monoglycerides) and PGE (polyglycerol esters). They found that the hardness of biscuits was dependent on the type of emulsifier, the amount used, the fat content and the storage time. Similar results were reported by Del Vecchio⁷⁷ and Campbell *et al.*⁶⁷. SSL in biscuits fortified with cottonseed flour improved eating quality after one or two months of storage⁷⁸.

1.2.6 Leavening agents

Leavening involves the introduction and expansion of some type of gas in the dough. Mixing the dough incorporates air; air is not very soluble in water and forms cells, which serve as nucleation sites for the expansion of gases produced later in baking. Air expands during baking and therefore provides a leavening action. Water also contributes to leavening when converted to vapour. Carbon dioxide is widely used as a leavening gas. The most common source of carbon dioxide is sodium bicarbonate (baking soda)². Sodium bicarbonate alone will partially decompose late in the baking stage¹:



However, this is too late for optimum biscuit volume and the unreacted sodium bicarbonate gives the product an unpleasant taste⁷⁹. Acidic salts, such as certain ortho- and polyphosphates, provide hydrogen ions in the presence of which sodium

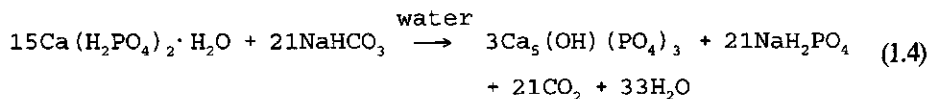
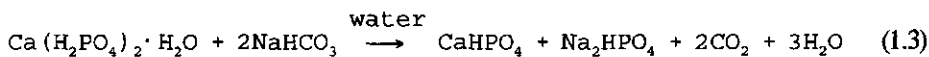
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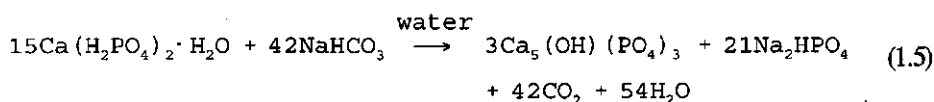
bicarbonate reacts to release more carbon dioxide⁸⁰. Table 1.2 shows some of the leavening acids in commercial use. The trade names frequently applied to specific compounds within this group are somewhat misleading. This is more readily apparent by comparing the trade and scientific names⁸¹ as shown in Table 1.2.

Table 1.2 Nomenclatures of some leavening acids in commercial use.

Leavening acid	Trade name	Scientific name	Chemical formula
MCP·H ₂ O	monocalcium phosphate monohydrate	monocalcium dihydrogen orthophosphate monohydrate	Ca(H ₂ PO ₄) ₂ ·H ₂ O
AMCP	anhydrous monocalcium phosphate	anhydrous monocalcium dihydrogen orthophosphate	Ca(H ₂ PO ₄) ₂
DCP·2H ₂ O	dicalcium phosphate dihydrate	monocalcium hydrogen orthophosphate dihydrate	CaHPO ₄ ·2H ₂ O
SAPP	sodium acid pyrophosphate	disodium dihydrogen phosphate	Na ₂ H ₂ P ₂ O ₇

The reaction of any of the phosphates with sodium bicarbonate to yield carbon dioxide is quite complicated. For example, various combinations of the following four chemical reactions are needed to describe the relatively simple leavening action of MCP·H₂O:





In reactions (1.2) through (1.5), an approximately neutral pH of the baked product must correspond to nearly equimolar amounts of NaH_2PO_4 and Na_2HPO_4 . Thus reactions (1.2) and (1.4) must be balanced by reactions (1.3) and (1.5) to give the proper ratio of NaH_2PO_4 to Na_2HPO_4 , so that the baked product will be buffered to a pH of about 7. It should be noted that the chemical reactions for the other phosphate leavening agents are considerably more complex⁷⁹. Therefore, the concept of NV (neutralising value) of a leavening acid was developed. It represents the grams of sodium bicarbonate which will be neutralised by 100 grams of the leavening acid and is determined by titration of the acid. Another term customarily encountered with leavening acids is DRR (dough rate of reaction). It is a measure of the reactivity of the leavening acid with baking soda during the mixing stage and during bench action (the holding period after mixing). % CO_2 evolution at a given temperature is plotted against time. Table 1.3 shows the reactivities of various leavening acids⁸².

Table 1.3 Properties of some commercially available leavening acids.

Leavening acid	Neutralising value	% CO_2 released		
		During mixing	Bench action	During baking
MCP·H ₂ O	80	60	0	40
AMCP	83.5	20	30	50
DCP·2H ₂ O	33	0	0	100
SAPP	72	22-40	8-11	52-67

The rate of acid dissolution governs the rate of carbon dioxide release and is the basis for classifying leavening acids according to speed. For example, if the compound is highly soluble, carbon dioxide is rapidly evolved and the acid is referred to as fast-acting. Conversely, if the acid dissolves slowly, it is a slow-acting leavening acid. In most leavening systems, the rate at which carbon dioxide is released is greatly accelerated when the temperature is elevated even slightly. However, some leavening acids are less sensitive to high temperatures than others⁸⁰.

MCP·H₂O was one of the first acidic phosphates used in baking. This phosphate is classified as a fast-acting leavening acid. Approximately 60% of the theoretical CO_2 is released during the mixing stage. Little more gas is then released until the product is placed in the oven and the temperature reaches approximately 60 °C. This is due

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to MCP·H₂O reacting upon contact with water to form some slow reacting DCP·2H₂O:

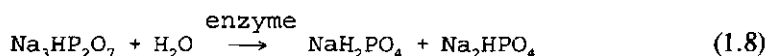
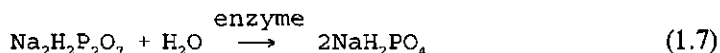


Because of the rapid rate of reaction, MCP·H₂O has limited application as used alone⁸².

AMCP is used more often than the monohydrate. The crystals of the commercial product are coated with alkali-metal phosphate compounds⁸². Coated and uncoated AMCP dissolve at nearly the same rate in water. However, in alkaline solutions of both types, there is a definite time delay in the rate of dissolution of the coated materials, hence a delayed release of acid⁸³. This behaviour is very desirable in some products that are allowed to rest for some time prior to baking⁸².

DCP·2H₂O is only slightly soluble and it forms a slightly alkaline solution at normal temperatures of mixing and holding of doughs. As a result, it does not release acidity for reaction with baking soda until the temperature is raised to about 65 °C or higher⁸².

At least five grades of SAPP, each having a different rate of reaction, are currently marketed. Differences are achieved by variation in the manufacturing procedure. All react very quickly with sodium bicarbonate in an aqueous solution. In dough, however, they interact with other ingredients, particularly the calcium of milk, to form an insoluble coating on the surface of each particle⁸². This protective coating in the dough may give delay times of nearly 3 hours at room temperature, but the coating may still be disrupted by heating⁷⁹. Another ingredient having a large effect on leavening rate is sucrose. Sucrose depresses the rate by 5 to 10% of the evolved gas, depending on sucrose content⁸⁴. The fastest reacting SAPP is seldom used alone in baking applications. A slower grade can be used if the dough temperature is kept at about 16 °C. The slowest SAPP can be utilised if dough temperatures of around 21 °C are maintained⁸². The reaction between SAPP and baking soda results in the formation of some trisodium monohydrogen pyrophosphate. This, as well as any unreacted SAPP, can be hydrolysed by the action of the pyrophosphatase naturally present in the dough, the process being accelerated with increasing temperature during baking until the enzyme inactivation temperature is reached^{82,83}:



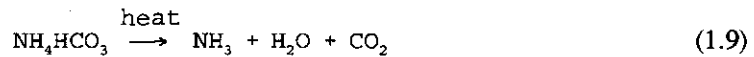
About 60% of the pyrophosphate in the dough is hydrolysed after 1 hour at room temperature and after 24 hours only 2% pyrophosphate remains⁸².

A further important effect, which may be controlled by choice of the proper leavening acid, is the rheological properties of a dough during handling and baking.

Part of this influence may be interpreted in terms of a charge effect on the protein; in addition, there are specific interactions between the gluten and the ions of the leavening acids. In general, sulphate anions weaken and phosphates strengthen the gluten structure, pyrophosphates being more effective than orthophosphates; calcium cations give thicker doughs than do sodium cations^{83,84}.

Baking powder is a leavening system consisting of a mixture of baking soda, leavening acid and a diluent, such as dried starch, which separates the ingredients so that they will not interact with each other during storage. There are two types of baking powder. The "single-acting" ones usually contain MCP·H₂O or AMCP. The "double-acting" ones usually contain MCP·H₂O and SAPP. With this mixture, there is an initial rapid reaction during the mixing stage with very slow release of gas during the bench period and retention of most of the acid for release during the baking stage⁸².

Carbon dioxide can also be produced by ammonium bicarbonate, which does not require the presence of a leavening acid. On heating, ammonium bicarbonate breaks down into three volatile components¹:



Ammonia gas provides an additional leavening action. The major disadvantage of ammonium bicarbonate is that the products must be baked to near dryness. Even relatively small amounts of water left in the product retain ammonia, and even small amounts of ammonia make the product inedible². Increases in ammonium bicarbonate were found to produce proportional increases in biscuit diameter^{18,54}.

1.2.7 Minor ingredients

This group comprises ingredients based on dairy products such as evaporated or condensed milk, whole milk powder, skimmed milk powder and demineralised whey powder. Tsen *et al.*⁷³ found that biscuit spread was reduced slightly by adding non-fat dry milk.

1.3 Mechanical properties

1.3.1 Importance of mechanical properties

Nowadays, automation and mechanisation is an important goal for the baking industry. As a corollary, the need for control of the relevant food quality attributes of the intermediate (dough) and finished products (biscuits) has increased. Mechanical properties are paramount, as they are directly associated with handling and eating

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characteristics. Understanding the mechanical properties of short doughs and their corresponding biscuits may provide guidelines for:

- Quality control of raw materials. Variations in dough consistency are considered to be a major source of problems in controlling rate of extrusion and dough piece weight³. In fact, it is dough consistency which determines the method of dough piece formation. Control of biscuit thickness and density are prevalent problems germane to commercial packaging of short-dough biscuits³. This variability in spread is obviously related to dough consistency.
- Product innovation. Currently, consumer attention is directed towards nutritional aspects. As a result, the demand for low-fat, low-sugar foods retaining the characteristics of the original products has increased. Fracture behaviour affects the perception of sensory properties during mastication and thereby has important implications for consumer acceptance of such products. Fracture properties may also be an important issue, for instance, when choosing proper storage conditions and/or packaging materials.
- Use of alternative raw materials. The use of emulsifiers and/or liquid oils as a substitute for fats would be desirable for biscuit manufacturers in order to reduce production costs⁵³.

1.3.2 Assessment of mechanical properties

The routine method of assessing dough consistency is usually subjective, based on the operator's experience; the sample is often squeezed between the fingers. To date, no method is generally accepted for objective measurement of the mechanical properties of short doughs⁸⁵. Several instruments have been used to study the mechanical properties of short doughs. These include the Farinograph⁶, the Extensograph⁵³, a modification of the Mixograph^{15,16}, a modification of "The Simon Research water absorption meter"^{5,68}, penetrometers¹⁴, the Stevens-LFRA Texture Analyser^{4,8} and the Instron^{11,17,25,36,40,64}.

For short-dough biscuits, other instruments such as the FMBRA Biscuit Texture Meter^{5,58,59}, the General Foods Texturometer⁸⁶ and the Kramer Shear Press⁸⁷⁻⁹¹ have been employed, in addition to the Instron^{7,28,38,76,92-94} and the Stevens-LFRA Texture Analyser⁶⁷. In the above mentioned studies the tests performed were empirical in nature; the parameters measured are not well defined and the results are uniquely related to the particular equipment design and geometry besides on specimen properties⁹⁵.

Another approach adopted involves more "fundamental" methods. Examples are:

- compression tests^{11,96}
- impact tests⁹⁷
- three-point bending tests^{60,63-65,78,98-102}

In the foregoing studies the results are expressed in force and deformation units, *e.g.* slope of force-deformation curve (wrongly referred to as Young's modulus) etc. This does not allow comparison of results because the force-deformation relationship is affected by the size of the test-piece. Mechanical properties, such as Young's modulus, ought to be independent of specimen dimensions⁹⁵.

1.4 Aim and outline of thesis

An essential aspect for biscuit manufacturers is to objectively assess the mechanical properties and to know how to modify them by adjusting composition and/or processing variables. In general, studies on short doughs and their biscuits, albeit empirical in nature, are fragmentary and in certain cases the results are contradictory; the role of basic ingredients is still not clear. Determination of true material properties may yield much better understanding of the relation between added ingredients, process parameters and the characteristics of the intermediate and the final product. At present, publications on short doughs and short-dough biscuits, in which fundamental parameters are determined, are rare^{52,103-107}. Likewise, little is known about the relationship between structure and mechanical properties.

This thesis sets out to:

- study the effect of certain composition and processing variables on the mechanical properties of short doughs and the corresponding biscuits
- relate mechanical properties to the internal structure.

Chapter 2 describes the rheological properties of short doughs of various composition at small deformation. In addition to small-amplitude oscillatory experiments, conductimetry was applied to obtain information on the structure of short doughs. The rheological properties of various short doughs at large deformation and at various deformation rates are discussed in chapter 3; such experiments are essential in order to characterise the dough response during various stages of the manufacturing process. The effect of sucrose on the large deformation properties of short doughs was investigated in more detail, with particular reference to mixing time. The results are presented in Chapter 4. The invariably inhomogeneous structure of short-dough biscuits in combination with instrumentation limitations, necessitated the use of a particular experimental set-up; in Chapter 5 the development of a procedure for the determination of fracture properties of short-dough biscuits is described. Chapter 6 reports the effect of composition on fracture properties of short-dough biscuits. The relationship between the mechanical properties of short-dough biscuits and their underlying structure is discussed in Chapter 7. The main conclusions of this thesis are summarised in the last Chapter.

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Chapter 2

Rheological Properties of Short Doughs at Small Deformation

Abstract

The rheological properties of short doughs of various composition were determined in dynamic shear experiments at small deformation. Regardless of composition, the linear region was very limited; beyond, significant structure breakdown occurred. Furthermore, an increase in storage modulus was invariably observed during ageing. In addition to oscillatory experiments, conductimetry was used to evaluate short doughs with different fat types and fat content. Short doughs showed differences in mechanical spectra and in conductivity, depending on fat content and fat type. These results are considered in terms of the structure of short doughs. It is concluded that short doughs are bicontinuous systems; reducing the fat content or changing its rheological properties relative to those of the non-fat phase results in fat-dispersed systems. The rheological properties of the non-fat phase are largely determined by flour/starch particles. It appears that gluten contributes only to a limited extent to these properties.

2.1 Introduction

Each food has a certain physical structure (macroscopic, microscopic and molecular) which governs its appearance, handling properties and eating characteristics. To consistently manufacture a food of acceptable quality (*e.g.* a short-dough biscuit), one has to evaluate and control the relevant food quality attributes. In several products, the mechanical properties are paramount, as they are directly associated with handling and eating characteristics.

Currently, there is a growing public interest focusing on nutritional aspects of food. The result is an increasing demand for foods with lower fat and/or sugar levels, while retaining the characteristics of the original products. Information on the functional role of the component under consideration is of primary importance for new product development, control of manufacturing processes and quality assurance.

Short doughs are distinguished from others in that they lack extensibility. They are not coherent under tension but have a small fracture strain and break easily¹. Short doughs usually contain an appreciable amount of fat. When the fat level is high, less water has to be added to make a dough of desired consistency². The presence of some

solid fat in the dough during mixing is essential. The use of liquid oil or too high a dough temperature, resulting in the complete melting of the fat, produces adverse changes in the handling characteristics of the dough; it becomes very soft. On the other hand, the fat must be sufficiently deformable to become well mixed with the other components¹. The temperature of the dough strongly affects its physical properties mainly because it determines the proportion of solid fat present³. Therefore, constant temperature histories during the various processing steps are essential.

Sucrose is also added in relatively high amounts in short doughs³. The proportion of sucrose that is in the crystalline state and the size of the sucrose crystals, would affect its rate of dissolution in water and therefore would affect dough consistency. Generally, the greater the proportion of crystalline sucrose and the larger the sucrose crystals, the slower would be the rate of dissolution in water². The water levels in short doughs preclude complete dissolution of the sucrose.

From the processing point of view, the quality control of raw materials for short doughs is associated predominantly with dough consistency. Variations in dough consistency are commonly blamed on the flour and adjustment is tried by increase or reduction of the dough water level. This variability is considered to be a major source of problems in controlling dough piece weight³. Moreover, the depositing machine used to form dough pieces cannot handle doughs that are either very soft or very stiff. The routine method of assessing dough consistency is usually subjective, based on the mixer operator's experience. To date, no method is generally accepted for objective measurement of the mechanical properties of short doughs². Moreover, there is insufficient understanding about what would be the desirable mechanical properties.

Several instruments have been used to study the rheological properties of short doughs. These include the Brabender Farinograph⁴, a modification of the Mixograph^{5,6} the Stevens-LFRA Texture Analyser^{7,8}, a penetrometer⁹, a modification of "The Simon Research water absorption meter"^{10,11}, the Instron for texture profile analysis^{12,13} and compression-penetration tests¹⁴⁻¹⁷ and the Brabender Extensograph¹⁸. All these test methods are empirical in nature. The parameters measured are poorly defined and the results cannot be described in terms of fundamental rheological properties; they are uniquely related to the particular equipment design and geometry and affected by size and shape of the test-piece^{19,20}.

By using small amplitude oscillatory measurements the deformation can be kept so small that the structure of the material is not irreversibly altered during measurement. The rheological parameters so obtained may provide information on the structure of the material²⁰. This knowledge can be further supplemented by other techniques, such as microscopy and conductimetry. The latter technique may be useful for systems composed of a conductive and a non-conductive phase. Since the conductivity of an aqueous phase is much higher than that of an oil phase, a crude but simple conductiv-

ity measurement readily discriminates between o/w and w/o emulsions^{21,22}. It has also been employed to monitor gas production in bread dough during fermentation²³ and evaluate foaming properties of proteins²⁴.

At present, published fundamental rheological studies on short doughs are rare. Likewise, little is known concerning the role of certain ingredients in dough consistency and there is some controversy about the effects. For example, Olewnik and Kulp⁴ found that rotary-moulded doughs with increasing levels of sucrose were much softer, whereas Miller⁷ reported that consistency of rotary-moulded doughs is not sensitive to the recipe level of sucrose.

The aims of the present investigation are:

1. To develop methods for the characterisation of the structure of short doughs.
2. To determine the small deformation rheological properties of short doughs of various composition in relation to their structure.

This Chapter deals with dynamic rheological measurements at small deformation, and conductivity experiments, with various short doughs. Large deformation properties of short doughs will be the subject of Chapters 3 and 4.

2.2 Experimental

2.2.1 Materials

Commercial wheat flour was obtained from Meneba Meel BV (Rotterdam, the Netherlands) and native wheat starch (water content 12.32%) from Latenstein Zetmeel BV (Nijmegen, the Netherlands). The chemical composition of the flour is presented in Table 2.1.

Table 2.1 Chemical composition of flour

	weight %
Starch	72.3
Protein (N × 5.7)	12.2
Lipids	1.35
Water	11.94
Ash	0.5

Starch, protein, water and ash were determined by Netherlands Standardisation Institute (NNI) procedures²⁵⁻²⁸. Determination of lipids was carried out according to an International Organization for Standardisation (ISO) method²⁹. Granulated sucrose

was provided by Suiker Unie (Breda, the Netherlands). Three fat types were used, namely, a standard fat, a firm fat and sunflower oil. The doughs are designated as standard dough, firm-fat dough and liquid-oil dough, respectively. The other doughs examined contained the standard fat which is commonly used for short doughs. The firm fat is more appropriate for puff pastry doughs. Fats were supplied by Bakkerol BV (Rotterdam, the Netherlands) and commercial sunflower oil was purchased at a local retailer. In both fats the fraction solid was determined as a function of temperature by pulse proton NMR. Prior to the determination, the fats were tempered as follows; melted at 80 °C, held for 30 min at 60 °C, then held for 2 hours at 0 °C followed by 2 hours at 20 °C and finally overnight at 0 °C. The tempered fats were held for at least 30 min at every measuring temperature. The results are given in Table 2.2.

Table 2.2 Solid fat content as a function of temperature for the fats investigated

Solid fat content		
°C	Standard fat	Firm fat
10	45.5	51.8
15	38.2	44.1
20	34.1	36.4
25	25.1	33.3
30	13.1	22.5
35	3.6	13.5
40	0	4.3
45	0	0

2.2.2 Preparation of doughs

Dough compositions are given in Table 2.3. A typical short dough composition was used as the standard (standard dough). The others were chosen in such a way that, whenever omitting an ingredient or reducing its amount, the weight ratios of the other components were constant. For conductivity experiments the compositions were somewhat different, allowing comparison of doughs with various fats at the same fat volume fraction. Deionised water was used in all experiments.

Doughs were prepared in a Farinograph employing a 50 g mixing bowl. Mixing was carried out for 8 min. Initially fat, sucrose, water and salt were premixed for half a minute at low speed (29.7 rpm), flour was then added followed by mixing for half a

minute at the same speed. A high speed (59.3 rpm) was then employed for 7 min, during which mixing was stopped twice to scrape the dough from the bowl sides to ensure uniform mixing. For comparison, worked fats were also studied using the same mixing procedure. The operation temperature and the temperature at which the fats were stored were adjusted to 20 °C by means of water baths. The other ingredients were stored at room temperature.

Table 2.3 Composition of the doughs (% w/w) investigated

Ingredient	low sugar			
	standard dough ^a	starch dough	fat dough	free dough
Flour ^b	39.8	-	47.7	50
Starch ^b	-	35.2	-	-
Fat/oil	24.7	26.6	9.8	31
Sucrose	20.4	21.9	24.4	-
Water	14.4	15.5	17.2	18
Salt	0.7	0.8	0.9	1

^a Apart from fat type, the firm-fat dough and the liquid-oil dough had the same composition

^b Dry matter

2.2.3 Density measurements

Bulk densities were determined with a disk of known volume (90 mm in diameter and 15 mm in height). A piece of dough was placed into the disk, the excess was scraped off and the weight was measured. Care was taken to fill the disk completely and prevent void formation. The average coefficient of variation for the densities was 1.5%.

2.2.4 Conductimetry

Two types of cell were used in combination with a Jenway 4020 conductivity meter. The frequency is automatically selected by the instrument; for conductivity values $< 2 \times 10^{-3} \Omega^{-1} \text{m}^{-1}$ the frequency is 100 Hz, for higher values the frequency is 400 Hz. The first type of cell is a standard one with platinum electrodes and a known constant (96 m^{-1}). It is more suitable for liquid samples, because stiff materials may damage

the electrodes. The other cell consists of two stainless steel electrodes of which one is fixed. A spindle of a micrometer is attached to the other, thereby permitting adjusting the separation between the two electrodes.

It was found that the deviation of the cell constant from the theoretical one increased with increasing gap size. In addition, for a given gap size, the cell constant was seen to be lower when the conductivity reached low values ($< 5 \times 10^{-6} \Omega^{-1} \text{m}^{-1}$). Therefore, for such conductivity values, the cell constant of the variable gap cell was determined by comparison with the standard cell. A dough with a fat volume fraction of 0.4 was used as a reference material. For higher conductivity values, the variable gap cell was calibrated with potassium chloride solutions.

The doughs were tested at room temperature. Dough temperature (monitored with a probe) and conductivity were recorded simultaneously, so that all values could be automatically recalculated to 20 °C using a compensation factor of 2% per °C. This factor is recommended in the operating manual of the instrument when performing measurements on substances with unknown temperature coefficients. The experiments were performed at least in duplicate and, where possible, measurements were done at two gap sizes. Values reported are means of these determinations. The coefficient of variation increased with increasing fat content, and ranged from 5% at low fat contents to 27% at the standard fat content.

2.2.5 Rheological experiments

In these tests a sinusoidally varying stress is applied to a test-piece and the resulting sinusoidally varying strain is measured. Viscoelastic materials are characterised by the phase difference δ (known as loss angle) between stress and strain, originating from the viscous properties of the material. G' is defined as the elastic or storage modulus and represents the energy stored in the material and recovered per cycle of imposed stress or strain. G'' is referred to as the viscous or loss modulus and represents the energy dissipated in the material. The ratio of the loss modulus to the storage modulus is called the loss tangent or $\tan \delta$. The results are meaningful only in the region where the material being tested behaves linearly, *i.e.* the stress is linearly proportional to the strain³⁰.

A Carri-Med CSL 500 rheometer was used for oscillation experiments. Its software was used to calculate G' , G'' and $\tan \delta$. Cone and plate geometry could not be used due to the presence of large suspended particles which may clog the gap in the centre. Therefore, roughened parallel plates were employed with a diameter of 2 cm. To determine whether slippage occurred, preliminary experiments were carried out with various gap sizes (1, 2, 3 and 5 mm). It was found that the moduli curves overlapped, apart from those for the 5 mm gap where the results were somewhat lower. Con-

sidering that the gap size should be relatively large with respect to sample inhomogeneities (*e.g.* sucrose crystals of 0.5 mm), the gap size for subsequent measurements was set to 3 mm. A tube of about 2 cm internal diameter was punched into the dough to form a cylinder. After removal of the tube a disc-like test-piece (thickness of 3.5 mm) was cut by means of a ring and a wire.

The rheometer was operated in various modes:

(i) stress sweep at a fixed angular frequency ω of 6.28 rad/s; (ii) time sweep at a fixed strain amplitude γ_{\max} of 2×10^{-4} and ω of 6.28 rad/s; and (iii) frequency sweep at a fixed γ_{\max} of 2×10^{-4} . All measurements were carried out at 20 °C. For the time-sweep experiments, the dough was tested immediately after mixing. For the frequency and stress sweeps, the dough was allowed to rest for 1 hour after its insertion between the plates. For every experiment a freshly prepared dough was used and the test was repeated at least once. For every system studied the averages of duplicates are plotted. The time to complete a stress sweep was 15 min whereas a frequency sweep took 20 min.

To minimise drying out of the doughs a humidity chamber was used. The relative humidity in it was controlled by placing a saturated solution of potassium bromide in a cavity in the bottom of the chamber. This salt solution has about the same water activity (0.83 at 25 °C)³¹ as those of the samples, although the latter differed from sample to sample (Table 2.4).

Table 2.4 Densities and water activities (a_w) at various temperatures (T) of materials studied

	density kg/m ³	a_w -	T °C
Unworked standard fat	870	-	
Unworked firm fat	920	-	
Liquid oil	900	-	
Worked standard fat	840	-	
Worked firm fat	880	-	
Standard dough	940	0.781	24.9
Firm-fat dough	1040	0.783	24.7
Liquid-oil dough	1180	0.761	25.2
Low-fat dough	1250	0.792	24.1
Sugar-free dough	1010	0.919	24.6
Starch dough	1020	0.811	23.7

The water activities were measured about 5 min after dough preparation using an Aqualab CX-2 water activity system. Given that the weight loss of a spherical standard-dough piece (about 3 cm in diameter) exposed to ambient temperature and relative humidity for 8 hours was found to be 1.4%, changes in water content during measurements were expected to be insignificant.

2.2.6 Standardisation of rheological measurements

The instrument is a controlled-stress rheometer, allowing the user to set either the stress or both stress and strain. In the latter option the rheometer applies a starting stress value, specified by the user, which will be adjusted, if necessary, by the rheometer until the required strain is achieved; this option was selected. Avoiding permanent change in structure, that is, working within the linear region (see below), is essential. Setting only the stress such that the strain does not exceed the linear region at low frequencies was not effective, because with increasing angular frequency or ageing time the resulting strain decreased. At a strain of 10^{-5} the measurements were no longer reliable, as was shown by the waveforms ceasing to be sinusoidal. On the other hand it took more time to perform a "stress and strain set" rather than a "stress set" experiment.

The viscoelastic properties of the doughs changed less with time after 1 hour had elapsed (see below). This, coupled with the importance of separating ageing and frequency effects, necessitated the use of a 1 hour equilibrium time before starting the measurements.

Frequency sweeps were carried out in order of increasing ω . To check whether the results coincided, regardless of whether the tests were run in ascending or descending frequencies, a firm-fat dough was examined in both modes. At angular frequencies greater than 0.5 rad/s the moduli were almost identical, while at lower they were somewhat scattered (results not shown). However, the pattern of the curves was by no means altered.

It should be emphasised that the fraction solid in the standard fat is very sensitive to temperature (see Table 2.2). Within the usual working range (20-25 °C), the amount of fat solid drops by 26%. Temperature fluctuations may give rise to fat recrystallisation, which alters its physical properties³². Moreover, the mechanical properties of the fat will determine the extent of its dispersion during mixing which, in turn, will affect the consistency of the dough. Thus, attention was paid to keep the storage temperature of the fat as constant as possible at 20 °C.

Initial experiments yielded results with significant scatter. The source of variation was traced to the uncontrolled temperature of the room in which the dynamic moduli were determined. It was found critical to ensure that the temperature of the

environment does not deviate more than ± 1 °C from the rheometer temperature. In a temperature sweep experiment, it was observed that with increasing temperature of the test-piece (20-23 °C) the storage moduli of the unworked standard fat and of the standard dough dropped by 25% and 20%, respectively.

In Table 2.5 the means and the coefficients of variation of 6 experiments (for every system studied) are summarised. Considering the extreme sensitivity of these systems to temperature as well as that the treatment of individual test-pieces could not be strictly uniform, the reproducibility of the results presented here was satisfactory.

Table 2.5 Means and coefficients of variation (%) of G' and $\tan \delta$ for all materials tested. Results taken 1 hour after the end of mixing. $T = 20$ °C, $\omega = 6.28$ rad/s and $\gamma_{\max} = 2 \times 10^{-4}$

	Mean		Coefficient of variation %	
	$G' \times 10^{-5}$ N/m ²	$\tan \delta$	G'	$\tan \delta$
Worked standard fat	6.53	0.079	2.4	3.5
Worked firm fat	19.36	0.082	7.5	4.7
Standard dough	6.69	0.255	4.7	1.6
Firm-fat dough	8.73	0.277	6.7	1.4
Low-fat dough	4.34	0.39	4.9	1.1
Liquid-oil dough	0.22	0.525	7.4	3.2
Sugar-free dough	7.50	0.238	5.1	2.5
Starch dough	5.36	0.202	5.1	3.3

2.3 Results

2.3.1 Density

The densities of the fats and the doughs studied are given in Table 2.4. It is seen that the dough density depended on the type of fat used. The standard fat and the firm fat in the worked state contained 3.5% and 4.4% more air than the corresponding fats in the unworked state. The calculated air volume fractions for the liquid-oil dough, the firm-fat dough and the standard dough are 3.3%, 14.8% and 23%, respectively. It is believed that the solid content of the fat at mixing affects dough density; doughs with lower solid fat have higher densities³. As is evident from Table 2.4, a function of the

fat is to entrap tiny air bubbles, but the air content is not directly related to the solid fat content. The air volume fraction does not seem to be critical at this stage, but probably it may be of primary importance for the resulting biscuit properties in which case the ratio between solid and liquid fat may play a part.

2.3.2 Conductivity

Results are shown in Fig. 2.1 and clearly indicate a different phase behaviour ranging from a fat-continuous to a fat-dispersed system, as the fat level is reduced. The solid lines represent theoretical values calculated according to Bruggeman's equation²²:

$$\frac{k - k_p}{k_m - k_p} \left(\frac{k_m}{k} \right)^{\frac{1}{3}} = 1 - \phi \quad (2.1)$$

where k , k_p , k_m are the electrical conductivities of the dispersion, disperse phase and continuous medium, respectively ($\Omega^{-1} \text{m}^{-1}$) and ϕ is the volume fraction of the disperse phase.

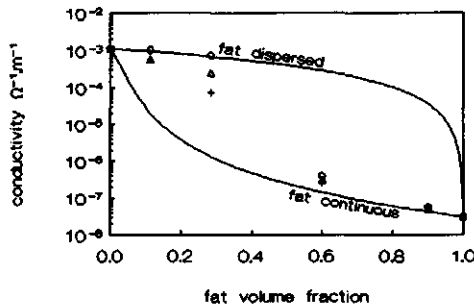


Figure 2.1 Conductivity values of various doughs as a function of fat volume fraction: (○) liquid-oil dough; (Δ) firm-fat dough; (+) standard dough. Solid lines represent theoretical values calculated according to Bruggeman's equation (see text).

Strictly speaking, Bruggeman's theory was derived for concentrated dispersed systems of spherical particles. The mathematical theories of dispersions of non-spherical particles are very complicated and not always applicable. Nevertheless, Bruggeman's equation is still useful for heterogeneous mixtures consisting of dispersed particles with various shapes. According to Hanai²², the dependence of the conductivity of disperse systems on volume fraction is generally well represented by Bruggeman's equation as seen from a variety of experimental results.

In applying Bruggeman's equation it is assumed that the conductivity of the non-fat phase is not appreciably affected by the degree of mixing, which may be different in doughs of various fat content. MacRitchie³³ quoted small changes in conductivity of bread doughs with degree of mixing. However, fat content and fat type have an impact on the air volume fraction, as can be readily seen in Table 2.4. To compare the experimental values with those predicted by theory, the former were recalculated to zero air volume fraction as follows: Since the air bubbles, which are assumed to have a far lower conductivity than the rest of the dough, are expected to be dispersed in a continuous phase, equation (2.1) can be reduced to²¹

$$\frac{k}{k_m} = (1 - \phi)^{\frac{3}{2}} \quad (2.2)$$

The air volume fraction is given by³⁴

$$\phi = \frac{\rho_s - \rho}{\rho_s} \quad (2.3)$$

where ρ_s and ρ are the solid and bulk densities of doughs, respectively (kg/m^3). The solid density can be estimated from³⁴

$$\rho_s = \frac{1}{\sum_{i=1}^n \frac{m_i}{\rho_i}} \quad (2.4)$$

where n is the number of constituents, m_i is the mass fraction of constituent i and ρ_i is the solid density of constituent i (kg/m^3). The solid densities of several constituents have been summarised by Peleg³⁵.

The difference in the conductivity values of the fats tested was negligible and therefore only two "Bruggeman's" lines are plotted, representing average values. At intermediate fat levels, *i.e.* $\phi \approx 0.3$, the continuity of the fat was influenced by the physical state of the fat incorporated (Fig. 2.1). The liquid-oil dough was found to be fat-dispersed as opposed to the standard dough which appears to be more bicontinuous. The effect of fat type seems to be smaller at high and low fat contents.

2.3.3 Dynamic moduli

2.3.3.1 Effect of stress amplitude

Figures 2.2 and 2.3 show representative examples of the behaviour of the materials investigated when imposing increasing stresses. In a preliminary experiment with a standard dough it had been found that the G' , G'' curves cross over at a strain of the order of 10^{-1} showing that the material changed from solid-like to more liquid-like. Eventually, the upper plate slipped over the sample. Considering the likelihood of instrumental artefacts at very small strain it is very difficult to pinpoint precisely the onset of non-linearity. In certain cases it is even doubtful whether an experimentally

accessible linear region exists. Such a typical example is the worked firm fat (results not shown).

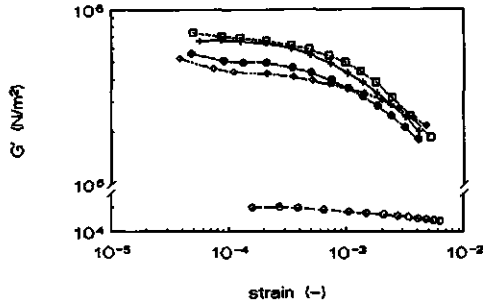


Figure 2.2 Strain dependence of storage modulus for the worked standard fat and various doughs: (□) worked standard fat; (+) standard dough; (●) starch dough; (◇) low-fat dough; (○) liquid-oil dough. Measurements performed 1 hour after mixing. $T = 20\text{ }^{\circ}\text{C}$ and $\omega = 6.28\text{ rad/s}$.

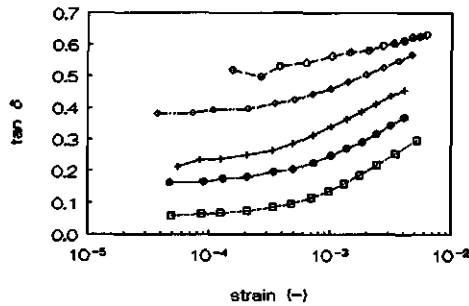


Figure 2.3 Strain dependence of $\tan \delta$ for the worked standard fat and various doughs. The symbols and the operation conditions are the same as in Fig. 2.2.

Accepting this limitation, it seems that most of the samples studied had some common characteristics. They became clearly non-linear at a strain of about 2×10^{-4} apart from the liquid-oil dough which was linear up to a strain of approximately 4×10^{-4} . The shape of the curves of the storage modulus versus strain did not vary; G'

values fell dramatically with increasing strain. This behaviour is exemplified by the standard dough (Fig. 2.2). Obviously, the doughs and the fats are extremely sensitive to large deformation. Their structure is readily broken down by very small mechanical action. The changes induced by shear appeared to be less severe in the case of the low-fat and the liquid-oil dough, since the shape of the curves was much flatter. Another interesting feature is that both had a significantly lower G' and higher $\tan \delta$ as compared to the standard dough. Consequently, they are relatively more liquid-like and more easily deformed. Apparently, fat reinforces the rigidity of the dough. At large deformation, it acts in the opposite direction since the G' curves for the standard dough and the low-fat dough crossed over at a strain of $\approx 3 \times 10^3$.

In general, the loss moduli did not decrease as sharply as G' , tending to be linear over a wider range of strain (results not shown). In the case of the worked fats they increased, passing through a maximum at a strain of 2×10^3 beyond which the reverse was observed. The mechanism causing this peak may be associated with some yield phenomenon. Regardless of composition, $\tan \delta$ increased with increasing strain, indicating a more liquid-like behaviour at large deformation (Fig. 2.3).

2.3.3.2 Ageing effects

The changes in G' and $\tan \delta$ between 15 and 150 min after mixing are tabulated in Table 2.6.

Table 2.6 Relative changes in G' and $\tan \delta$ taking place between 15 and 150 min after mixing. $T = 20^\circ\text{C}$, $\omega = 6.28 \text{ rad/s}$ and $\gamma_{\text{max}} = 2 \times 10^4$

	$\Delta G' \times 10^{-5}$ N/m ²	$\Delta G'$ %	$\Delta(\tan \delta)$ %
Worked standard fat	0.92	16	-15
Worked firm fat	5.60	33	-22
Standard dough	2.45	42	-10
Firm-fat dough	4.50	66	-13
Low-fat dough	1.80	56	-7
Liquid-oil dough	0.15	92	-3
Sugar-free dough	1.25	19	-2
Starch dough	1.54	41	-8

It took a few minutes for the test-piece to reach the "apparent" measuring temperature. When the temperature of a standard-dough piece was monitored by means of a thermocouple, a drop of 1.5 K took place during the first 15 min and the temperature

attained a constant value thereafter (22.1 °C) without reaching the set temperature. In addition, effects due to relaxation of stresses induced during the loading procedure will occur during that time. Therefore, results obtained during the first 15 min were disregarded.

Throughout the time scale studied, G' was invariably higher than G'' irrespective of the sample tested, implying that the materials behave mainly elastically. G' as a function of ageing time for several materials is given in Figure 2.4.

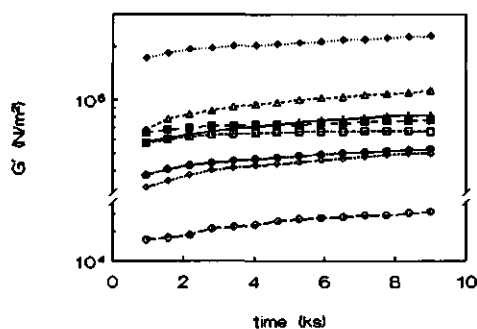


Figure 2.4 Storage modulus as a function of ageing time for all materials tested: (□) worked standard fat; (◆) worked firm fat; (+) standard dough; (Δ) firm-fat dough; (●) starch dough; (◇) low-fat dough; (○) liquid-oil dough; (■) sugar-free dough. $T = 20\text{ °C}$, $\omega = 6.28\text{ rad/s}$ and $\gamma_{\max} = 2 \times 10^{-4}$.

As can be seen, at first, the storage modulus increased rapidly and then more slowly but steadily. G' of the worked standard fat appears to become constant after 1 hour. Its value together with that of G'' were lower by a factor of 3 with respect to those for the worked firm fat. This reflects its lower resistance to deformation, which may partly stem from its lower solid fat content. Both exhibit a predominantly solid like behaviour (Table 2.5). No changes in the loss modulus with time were observed for both worked fats. In contrast, for all doughs G'' increased at a rather slow rate (results not shown). In all cases a relatively more elastic structure is formed on ageing, as indicated by $\tan \delta$ which tended to decrease slightly with time (Fig. 2.5). As expected, changing the type of the fat resulted in doughs differing in dynamic moduli, but not to the same degree as observed for the corresponding fats. The firm-fat dough was relatively more liquid-like than the standard dough: $\tan \delta$ was slightly, yet consistently, higher (Table 2.5).

The curves of G' and $\tan \delta$ versus ageing time for the liquid-oil dough are qualitatively similar to those of the standard dough (Figs. 2.4 and 2.5). The high $\tan \delta$ of the former shows that replacing fat by sunflower oil renders a dough more liquid-like in character as well as markedly less firm.

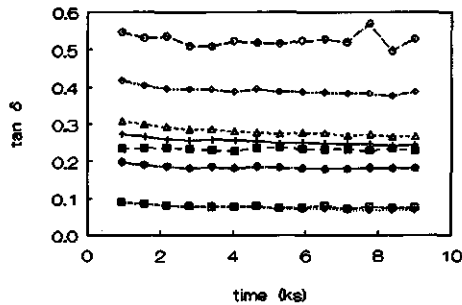


Figure 2.5 $\tan \delta$ as a function of ageing time for all materials tested. The symbols and the operation conditions are the same as in Fig. 2.4.

The curves for the low-fat dough and the starch dough followed the same pattern. In other words, the evolution of the dynamic viscoelastic parameters with time was practically similar to that observed for the standard dough. Again, no limiting values were reached within the experimental time. Relative to the standard dough the starch dough was more easily deformed (lower G') and more solid-like (lower $\tan \delta$).

As is evident from Figure 2.4, the shape of the curve of G' versus ageing time was somewhat different for the sugar-free dough. Initially G' was slightly higher than the corresponding value for the standard dough and it increased slowly with time. Unlike the standard dough, after a period of about 1 hour it reached a roughly constant value. G'' remained unaffected during ageing and was virtually equal to that of the standard dough. As a result, $\tan \delta$ values tended to converge during ageing. It seems that addition of sucrose led to a dough with a slightly more liquid-like behaviour as compared to the sugar-free dough (Fig. 2.5).

2.3.3.3 Effect of angular frequency

Plots of the mechanical spectra of several samples versus ω are depicted in Figures 2.6 and 2.7. The rheological response of the worked standard fat was found to be largely independent of ω . There was a slight increase in G' and a moderate decline of $\tan \delta$. For the worked firm fat the changes with ω were in the same direction, G' only

differing in absolute values (results not shown for sake of clarity). The shape of the curves suggests a pronounced solid-like behaviour for the worked fats.

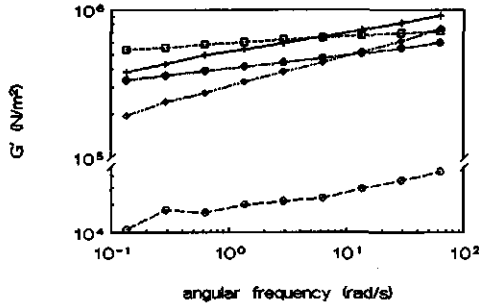


Figure 2.6 Frequency dependence of the storage modulus of the worked standard fat and various doughs: (□) worked standard fat; (+) standard dough; (●) starch dough; (◇) low-fat dough; (○) liquid-oil dough. Measurements performed 1 hour after mixing. $T = 20\text{ }^{\circ}\text{C}$ and $\gamma_{\max} = 2 \times 10^{-4}$.

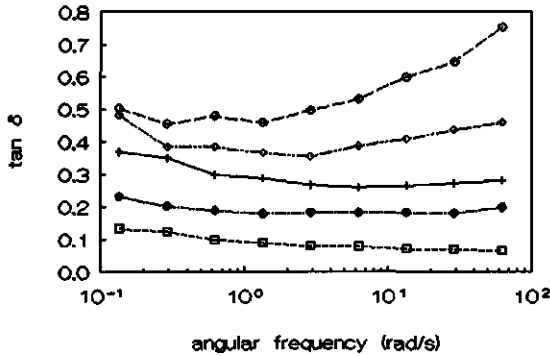


Figure 2.7 Frequency dependence of $\tan \delta$ of the worked standard fat and various doughs. The symbols and the operation conditions are the same as in Fig. 2.6.

The doughs appeared to differ from the corresponding worked fats. The frequency dependence of the storage modulus was enhanced (Fig. 2.6). The loss modulus was almost constant up to an angular frequency of about 6 rad/s, beyond which it increased with ω (results not shown). This resulted in a small decrease of $\tan \delta$ with increasing

ω at low ω , and about constant values or a slight increase at higher ones (Fig. 2.7). Compared to the firm-fat dough, the lower modulus of the standard dough (Table 2.5) may have resulted, to some extent, from the higher air volume fraction. The standard dough contains about 8% more air than the firm-fat dough. However, this difference is too small to fully explain the lower G' for the former.

For the liquid-oil dough, both dynamic moduli increased with increasing ω , but G' did not keep pace with G'' at angular frequencies over 1 rad/s, as follows from the noticeable increase in $\tan \delta$.

Reducing the fat content brings about substantial changes in the resulting dough. The elastic component became more frequency dependent (Fig. 2.6); $\tan \delta$ increased with increasing angular frequency at $\omega > 3$ rad/s (Fig. 2.7).

The frequency dependencies of G' for the starch dough (Fig. 2.6) and the sugar-free dough (results not shown) are rather similar to that of the standard dough. As ω decreased, so did the storage modulus. Initially, $\tan \delta$ decreased with increasing ω , followed by a region where it was roughly independent of ω (Fig. 2.7).

2.4 Discussion

The conductivity studies described above (Fig. 2.1) show a transition from a fat continuous to a fat dispersed system with decreasing fat content. At intermediate fat levels, typical of short dough recipes, the physical state of the added fat had an influence on its distribution in the dough.

The most important factor determining the distribution of the fat throughout the non-fat phase after mixing, is the rheological properties of the latter relative to those of the former. Although changes occur in these properties due to shear, it seems reasonable to assume that the firm fat was much stiffer than the non-fat phase in contrast to the standard fat. So, on mixing, the firm fat will be broken down to large lumps, whereas the standard fat will be smeared out over the flour particles. In addition, the standard fat may, partly, be present in oil droplets formed during mixing. Fat crystals protruding from these globules facilitate globule contact. After the crystals pierce the film between two globules, liquid oil forms a neck resulting in partial coalescence³⁶. Large irregular clusters may form, which implies that the effective fat volume fraction is greater than the calculated one. However, it is unlikely that the effective fat volume fraction is 0.84, which would be needed for the standard dough to be classified as fat dispersed (see Fig. 2.1). Consequently, this dough is, at least, partly bicontinuous. The results shown in Fig. 2.1 are consistent with observations from Shepherd and Yoell³⁷, who used conductivity experiments to investigate cake batters. Although their results were not corrected for air volume fraction, they reported increasing resistance, hence decreasing conductivity, with increasing fat content. The values deviated from

those predicted by Bruggeman's equation for an o/w emulsion. They attributed this effect mainly to irregularly shaped fat particles, enclosing areas of the aqueous phase and thus increasing the effective volume fraction.

Qualitatively, the results of our dynamic measurements are in agreement with those presented by Menjivar and Faridi³⁸. The limited extent of the linear region for all the doughs tested strongly suggests that the elastic properties stem from a network of particles rather than from a macromolecular one. Fig. 2.2 shows a clear difference between the moduli of the starch dough and the standard dough. The lower modulus of the former may be due to the lower ratio of non-fat solids to water. However, in that case, $\tan \delta$ would be similar to that of a standard dough, if not higher. Yet, $\tan \delta$ of the starch dough was lower than that of the standard dough. The absence of gluten may affect the distribution of water and thereby the rheological properties of the non-fat phase and so the mixing properties. Then, the extent of dispersion of the various components may be affected, giving rise to a changed contribution of the starch and/or the fat to the dough properties. It should be realised that, in this study, gluten was not separated from the flour. Instead, flour was replaced by commercial native starch. Presumably, components other than gluten were also lost. For instance, pentosans, which form a small fraction of the flour (2-3%), have a pronounced effect on its water absorption²⁰. Several researchers have noticed that pentosans decrease G' and increase $\tan \delta$ of either gluten^{39,40} or bread doughs⁴¹⁻⁴³. The level of damaged starch also affects the added water requirements of short doughs³; high levels of damaged starch increase dough consistency¹². Interpretation of our results in this light is complicated, since the effects due to gluten, pentosans or to damaged starch, cannot be separated.

As far as sucrose is concerned, it slightly increased the liquid-like character of the dough (Table 2.5). Sucrose possibly modifies the properties of the non-fat phase via its influence on the amount of solvent. When sucrose dissolves in water, the volume of the aqueous phase in the system is increased. One gram of sucrose, when dissolved in 1 g of water, produces 1.63 ml of total solution⁴⁴. Sucrose may also affect solvent quality. When a constant volume of a sucrose solution was added to a constant weight of flour, increased mixograph development times were observed with increasing concentration of sucrose. The effect was ascribed to decreased mobility of solutes in the system, indicative of the retarded rate of water uptake by gluten, caused by the smaller plasticising action of sucrose solution (compared to water alone) on glassy polymers such as gluten⁸.

The next question is what forms the non-fat phase (gluten or starch particles). It has been reported that oscillatory measurements at small deformation were unable to detect qualitative differences in gluten development between cracker doughs and short doughs; the reason is said to be the long-range interactions associated with a gluten

network³⁸. On the other hand, according to Flint *et al.*⁴⁵, microscopic examination of short doughs showed no evidence of gluten development; the protein is associated with starch granules as intact endosperm particles. As can be seen in Fig. 2.2, the moduli of most of the doughs investigated have a similar strain dependence. In addition, the shape of the curves exhibits no qualitative differences from that observed for the worked fats. Reduction of fat content caused a considerable decrease in the stiffness of the dough. This implies that fat is a crucial structural component of the system. If present in high quantities, it is the fat which imparts cohesiveness to the dough¹. Indeed, preparation of a dough without added water was still feasible. Also shown in Fig. 2.2 is the cross over of the moduli curves for the standard dough and the low-fat dough. Combining this observation with those by Flint *et al.*⁴⁵ and by Menjivar and Faridi³⁸ (discussed above), it may be concluded that, at small deformation, the response of the standard dough is primarily governed by fat particles. In contrast, the strain dependence of the low-fat dough may reflect more the properties of a network formed by flour particles. Figure 2.6 shows that, for both worked fats, the storage modulus was hardly dependent on ω . Conversely, a positive frequency dependence was observed for all doughs containing fat. These data signify that short doughs are not merely fat continuous, with the other components dispersed in it, but bicontinuous. Lowering the fat content yields a more fat dispersed dough, which may account for the enhanced frequency dependence of the moduli of the low-fat dough.

Substituting liquid oil for solid fat drastically decreased the modulus of the dough, which indicates that the non-fat phase must have a low modulus. In view of the absence of a gluten network the non-fat phase may be visualised as being made up of a saturated sucrose solution surrounding and bridging flour/starch particles. Moreover, a moulded liquid-oil dough readily appeared to flow upon standing and oil was seen to leak out. This underlines that the liquid-oil dough was not entirely fat dispersed. This finding does not readily agree with the conductivity results. It is conceivable that, upon mixing, the oil will be divided to rather spherical, fine droplets which may partly coalesce to form some sort of channels in certain regions.

Ageing effects have important implications for the processing characteristics of short doughs. Wade⁴⁶ has suggested that short doughs undergo changes that are similar to those caused by drying out, but are likely to be caused by the slow uptake of added water in the doughs by the hydrophilic components. These changes slow down after about 30 min. In commercial practice, dough is allowed to rest for 30 min despite the stiffening continuing for longer periods. It has been considered that during that period the changes in consistency and the loss of stickiness are such as to enable proper dough handling by the dough forming machinery³. It has been demonstrated that the changes in consistency occurring between mixing and processing were affected by the initial dough consistency after the completion of mixing⁷, by the amount of water added and the type of flour¹¹, and by the addition of enzymes¹³.

Regarding the worked fats, comparable results have been obtained, among others, by Nederveen⁴⁷, who also observed a stiffening process following kneading of fat dispersions. It has been stated that such structure recovery in fats originates from flocculation and sintering of fat crystals³². If this reasoning would hold for short doughs, one would expect:

- the ageing effects to vanish in the case of the liquid-oil dough which does not contain any solid fat.
- the extent of the stiffening process to be reduced in the case of the low-fat dough which is assumed to be fat dispersed.

It is evident from Fig. 2.4 that another mechanism than that directly due to the fat must, at least partly, be responsible for the observed changes during ageing.

It has been suggested that the principal flour polymers prone to swelling and network formation include gluten, damaged starch and pentosans and that sucrose delays the rate of swelling⁸. The latter argument may offer an explanation for the different curve shape of the sugar-free dough. The former argument cannot explain the structure build-up exhibited by the starch dough, since it contained none of the above mentioned components. On the other hand, it has been found that dispersing starch in cold water causes the granule to increase by about 10% in diameter, *i.e.* about 33% in volume, due to swelling⁴⁸. It has also been reported⁴⁹ that the diffusion coefficient of water into the starch granule at room temperature and at water contents found in bread dough is about 10^{-11} m²/s. Then for granules of 40 μ m, the diffusion of water into the starch grain would take approximately 40 s and thus would take place during mixing. However, the rate and/or the extent of starch swelling will be influenced by other factors. In addition to the effect of sucrose, less starch swelling is expected to occur in short doughs since they contain significantly less water than bread doughs. Unambiguous identification of the parameters to which the ageing effects may be ascribed, cannot be obtained from these results. In view of the complexity of the materials tested, it is unlikely that one component is solely responsible. What is observed is a combination of factors exerting an influence on the dough characteristics. The role of water, its amount and its partition should not be underestimated.

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Chapter 3

Rheological Properties of Short Doughs at Large Deformation

Abstract

Large deformation rheological properties of short doughs of various composition were determined in uniaxial compression. Apart from a sugar-free dough, all doughs studied showed pronounced yielding and flow behaviour. Yielding occurred at very small strain, indicating strong strain dependence. At small strain systems were predominantly solid-like; at large strain they behaved more like strain-rate thinning liquids. The doughs showed large differences in apparent biaxial extensional viscosity, depending on fat and sucrose contents. It is concluded that yielding behaviour is strongly influenced by intact flour particles that act as defects. The occurrence of such particles is determined by the presence of sucrose, which delays gluten development through its effect on solvent quality. The sucrose also facilitates formation of a non-fat continuous phase, since it increases the quantity of solvent. Fat content partly determines whether the resulting dough is bicontinuous or not.

3.1 Introduction

Short doughs constitute the intermediate product in the manufacture of short dough biscuits. In general, they contain high amounts of fat and sucrose and a small amount of water. The relatively large quantities of fat and sucrose solution are considered to be the main factor to determine dough plasticity and cohesiveness¹. Moreover, dough consistency is strongly affected by the proportion of solid fat present during dough mixing which, in turn, is a function of temperature¹.

The rheological properties of short doughs greatly influence their handling properties and probably the eating quality of the baked product. In fact, it is dough consistency which determines the method of dough piece formation (rotary moulding, wire cutting, or depositing). Controlling of dough consistency is a prerequisite for automation and mechanisation in the baking industries. Understanding the rheological behaviour of short doughs may provide guidelines for approaching problems in the production process. For instance, short dough biscuits tend to become larger in width and length during baking. Control of this increase in size or "spread" is the biggest single processing problem and it obviously is related to dough consistency¹. Similarly, during depositing, the rate of extrusion is affected by dough consistency.

In recent years consumers are increasingly concerned with nutritional aspects. Low-fat, low-sugar products have become an important issue in the food industry. In developing such products it is essential to objectively assess the desired quality characteristics (*e.g.* mechanical properties) and to know how to modify these characteristics by adjusting composition and/or processing variables.

So far, assessment of short dough consistency is mainly based on trial and error. Often, the sample is squeezed between the fingers. In most of the published work, empirical methods are used to establish a relation between composition and dough consistency²⁻¹⁶. Although empirical methods may provide useful information, the results of such studies are qualitative. Determination of true material properties may yield much better understanding of the relation between added ingredients, process parameters and the characteristics of the intermediate and the final product. However, there is almost no literature on the rheological behaviour of short doughs at large deformation in which fundamental parameters are determined.

During various stages of the manufacturing process (*e.g.* depositing) large deformations are imposed on the dough. Then, dough structure can be affected to an extent depending on its rheological properties and its deformation and deformation rate history. Therefore, to characterise the dough response during such processing steps in a meaningful way, large deformation experiments are needed. Evidently, the tests have to be carried out at relevant time scales. This is particularly true for "time dependent" materials, such as short doughs. In addition, some operations may involve extensional deformation, which means that extensional viscosity may be as important as shear viscosity.

The present study was undertaken to investigate the rheological properties of short doughs with varying composition at large deformation and at various deformation rates.

3.2 Experimental

3.2.1 Materials

Commercial wheat flour was obtained from Meneba Meel BV (Rotterdam, the Netherlands) and native wheat starch (water content 12.32%) from Latenstein Zetmeel BV (Nijmegen, the Netherlands). Granulated sucrose was provided by Suiker Unie (Breda, the Netherlands). Two types of fat were used, namely, a standard fat for short doughs and a firmer one. The doughs are designated as standard dough and firm-fat dough, respectively. The other doughs examined contained the standard fat. Fats were supplied by Bakkerol BV (Rotterdam, the Netherlands). Deionised water was used in

all experiments. The chemical composition of the flour and the fraction of fat solid as a function of temperature were given in Chapter 2.

3.2.2 Preparation of doughs

Dough compositions are given in Table 3.1.

Table 3.1 Composition of the doughs (% w/w) investigated

Ingredient	standard	starch	low	sugar	sucrose
	dough ^a	dough	fat	free	syrup
			dough	dough	dough
Flour ^b	39.8	-	47.7	50	41.5
Starch ^b	-	35.2	-	-	-
Fat	24.7	26.6	9.8	31	25.7
Sucrose	20.4	21.9	24.4	-	17.0
Water	14.4	15.5	17.2	18	15.0
Salt	0.7	0.8	0.9	1	0.8

^a Apart from fat type, the firm-fat dough had the same composition

^b Dry matter

Doughs were prepared in a Farinograph employing a 50 g bowl. Mixing was carried out for 8 min according to the procedure described in Chapter 2. For comparison, both fats were also studied in the worked state using the same mixing procedure. Storage of ingredients, preparation of doughs and determination of mechanical properties were done at 20 °C in a temperature-controlled room. Test-pieces were shaped by punching a tube of 20 mm internal diameter into the dough/fat and cutting the cylinder obtained into pieces of predetermined height by means of a wire.

3.2.3 Uniaxial compression experiments

The large deformation properties of short doughs were investigated by uniaxial compression tests. A Zwick (type 142510/00) or an Instron (table model 1122) tension-compression apparatus was used with a 20, 50 or 2000 N load-cell. In these tests a cylindrical test-piece was compressed between two parallel circular perspex plates. The diameter of the plates was 20 mm. The upper plate was lowered at a constant speed and the force applied on the test-piece was recorded as a function of

time. If a homogeneous deformation is assumed, which implies perfect slip of the test-piece at the surfaces of the plates, the flow is biaxially extensional.

Since the plates had the same diameter as the test-piece, the stress σ ($\text{N}\cdot\text{m}^{-2}$) exerted on the test-piece is defined as¹⁷:

$$\sigma = \frac{F(t)}{\pi R^2} \quad (3.1)$$

where $F(t)$ is the force (N) recorded at time t (s) and R is the radius of the plate (m). For a material with a Poisson ratio μ of 0.5 the biaxial Hencky strain ϵ_b (-) can be calculated from¹⁷:

$$\epsilon_b = -\frac{1}{2} \ln \frac{h(t)}{h_0} \quad (3.2)$$

where $h(t)$ is the height (m) of the test-piece at time t and h_0 is the initial height of the test-piece. The biaxial extensional strain rate $\dot{\epsilon}_b$ (s^{-1}) is given by¹⁷:

$$\dot{\epsilon}_b = \frac{d\epsilon_b}{dt} = -\frac{1}{2} \frac{dh}{h(t) dt} = \frac{v}{2h(t)} = \frac{v}{2(h_0 - vt)} \quad (3.3)$$

where v is the compression speed ($\text{m}\cdot\text{s}^{-1}$). Equation (3.3) shows that $\dot{\epsilon}_b$ will increase with increasing deformation. The apparent biaxial extensional viscosity η_b^* ($\text{Pa}\cdot\text{s}$) is given by¹⁷:

$$\eta_b^* = \frac{\sigma}{\dot{\epsilon}_b} \quad (3.4)$$

All doughs were tested at three crosshead speeds (1, 10 and 100 mm/min); test-pieces had a height of 30 mm. The worked fats were tested at two crosshead speeds (10 and 100 mm/min) on test-pieces of 20 mm height. Excluding preliminary studies, all experiments were carried out in triplicate. Curves shown represent averages of these replicates. Every dough batch was used for 3 experiments. The tests (for each batch) lasted about 4 hours and to minimise drying out the dough was wrapped in plastic film. The sampling scheme was such that, at the same compression speed, every replicate represented a different dough/fat batch with a different ageing time. Whenever η_b^* changed with ageing, the experiments were repeated on freshly prepared doughs.

It should be noted that the stresses calculated with equation (3.1) are somewhat higher than the actual stresses in the sample due to slight bulging of the sample. On the other hand, for plungers of larger diameter than the test-piece, calculation of the stresses would have been hampered by stronger frictional effects.

3.3 Results

3.3.1 Preliminary experiments

3.3.1.1 Standardisation of method

The doughs and the worked fats were very soft and deformable and thus difficult to handle. After cutting the shaped cylinder to the prescribed height, both the upper and the lower surface of the test-piece were not absolutely smooth or parallel to each other; precise measurement of specimen height was, thus, virtually impossible. Besides, placing the test-piece exactly vertical to the compression plates was not feasible, and the sample often was somewhat inclined with respect to the compression plane. As a consequence, there was no full contact with the plates during the early stages of compression. This procedure is designated as technique 1.

To achieve complete contact from the beginning of the measurements the method was modified in such a way that the test-pieces were cut at a height of 22 or 32 mm and slowly (0.5 mm/min) compressed to 20 or 30 mm, respectively (technique 2). This procedure induced stresses to the samples and, to reduce their magnitude prior to the measurement, the samples were allowed to relax. It was observed that most of the stress relaxed within an hour; there was still some residual stress. However, it should be realised that every manipulation induces some stress to the test-piece. This technique had another advantage. Compression expelled a small amount of fat from the sample. This fat on the interface acted as a lubricant, thereby reducing friction. The drawback of this method is that the set deformation rate cannot be achieved instantaneously although the effect is probably small¹⁸.

Therefore, a third procedure was attempted (technique 3). After the sample was precompressed to the desired height the crosshead was raised at a slow speed (0.05 mm/min) to minimise sticking of the dough to the plate. However, the dough then was slightly extended. Therefore, a second perspex plate of identical diameter was placed between the upper plunger and the test-piece so that upon raising the crosshead the dough was not pulled upwards. However, during compression this second plate did slide away.

Figure 3.1 illustrates results obtained for a standard dough with the three techniques. The initial part of the stress-strain curves was clearly different. Not only was the slope higher but also the yielding behaviour was more prominent when using techniques 2 or 3. At larger strains the differences were within the experimental accuracy.

Although all techniques had their limitations as discussed above, the results of the second one were considered the most representative for the behaviour of the materials. This technique also appeared to be the most reproducible and it was decided to adopt this procedure for all subsequent experiments.

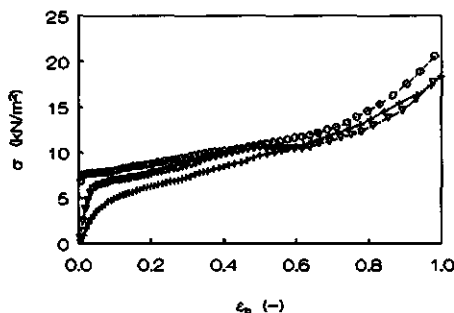


Figure 3.1 Stress-strain curves for a standard dough obtained with different techniques (see text): (+) technique 1; (○) technique 2; (∇) technique 3. Initial $\dot{\epsilon}_b = 2.8 \times 10^{-2} \text{ s}^{-1}$.

Taking the non-uniformity of the materials tested into account, the reproducibility of the measurements was surprisingly good. Regardless of the material tested, the coefficient of variation was, on average, 4% and never higher than 20% even at the largest strain investigated.

3.3.1.2 Effect of height to diameter ratio

The effect of the height to diameter ratio was checked with test-pieces of four heights (4, 10, 20, 30 mm) and constant diameter. Initial $\dot{\epsilon}_b$ was kept constant by adjusting the compression speeds. Results are shown in Figure 3.2. For a given ϵ_b , the stresses were higher for a smaller height. Furthermore, the stress increased stronger with ϵ_b for a smaller height. Most likely these effects are due to friction between the plates and the material. Uniaxial compression tests on gelatin and starch gels¹⁹⁻²¹, various types of cheese²²⁻²⁵, sausages²⁶, bread²⁷ and raw potato flesh²⁸ have shown that friction may considerably affect the results. The effect of friction on the stress naturally increases with increasing strain because the height of the test-piece decreases²⁹. It should be borne in mind that flow was not purely biaxial extensional, as could be observed from the barrel-like shape of the sample, implying that friction was still contributing somewhat to the results.

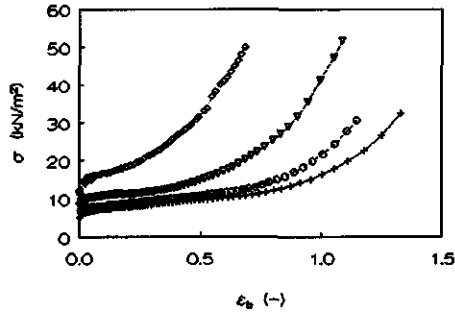


Figure 3.2 Stress-strain curves for standard doughs with different heights: (\diamond) 4 mm; (∇) 10 mm; (\circ) 20 mm; (+) 30 mm. Test-piece diameter = 20 mm, initial $\dot{\epsilon}_b = 2.8 \times 10^{-2} \text{ s}^{-1}$.

Plotting stress (at a biaxial strain of 0.2) versus the reciprocal of initial specimen height reveals that friction contribution would account for about 20% of the stress for a height of 30 mm (Fig. 3.3).

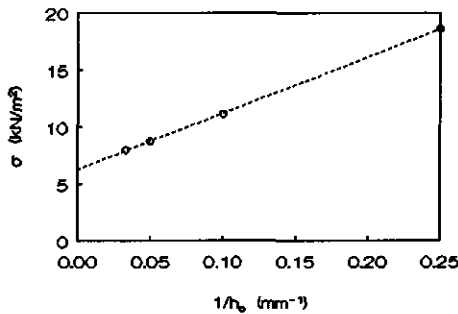


Figure 3.3 Stress versus reciprocal of initial specimen height ($1/h_0$) for standard doughs. Test-piece diameter = 20 mm, initial $\dot{\epsilon}_b = 2.8 \times 10^{-2} \text{ s}^{-1}$, values calculated at $\epsilon_b = 0.2$.

This height was chosen for subsequent experiments; preparation of undeformed specimens of larger height was extremely difficult. In addition, for stiff doughs (*e.g.* low-fat doughs) the test-pieces tended to buckle. In the case of worked fats,

precompression caused fracture of the samples prior to the actual measurement if height was 30 mm; consequently, test-pieces of 20 mm height were used. In attempts to further minimise friction, several lubricating materials were put between compression plates and specimen, viz. parafilm, grease proof paper, baking paper and aluminium foil. Oil could not be used, because it would diffuse into the test-piece. In all cases, the force-time curves (results not shown) showed higher force values for a given deformation, implying greater frictional effects.

3.3.2 Behaviour of various doughs

3.3.2.1 Mode of failure

It was not possible to unequivocally determine the point at which macroscopic fracture occurred from the stress-strain curves. For all doughs tested the biaxial strain at macroscopic fracture, visually determined, was between 0.32 and 0.38, far beyond the "apparent yield strain". Fracture strain did not depend on $\dot{\epsilon}_b$, nor was it affected by dough composition. In other words, the fracture strain was almost constant. All doughs fractured in tension as could be observed from the vertical cracks. It should be noted that cracks were formed at many points at about the same time. Moreover, when compression was stopped, spontaneous crack propagation did not occur. Crack speed appeared to be very slow. This points to strong energy-dissipation mechanisms. In all cases the specimens retained coherence after fracture and, therefore, it is debatable whether the occurrence is to be called fracture or extensive yielding. Consequently, we used the general term failure.

A notable exception was provided by the worked fats. They fractured in shear, since cracks (fewer in number) were visible at an angle of 45° with respect to the direction of compression; also, fracture occurred at lower ϵ_b (very roughly at 0.1). In addition, the cracks appeared to proceed fairly rapidly throughout the whole test-piece. Since all doughs fractured in tension whereas fats fractured in shear, this suggests that, probably, the doughs cannot be considered as fat-continuous.

3.3.2.2 Effect of compositional variation

Stress-strain curves for the standard dough at three deformation rates are given in Figure 3.4. In these curves three regions can be distinguished. Initially the stress ascended sharply with increasing ϵ_b . This was followed by a plateau like region in which the stress increased slightly and sometimes remained constant or even decreased somewhat. With further deformation of the dough ($\epsilon_b > 0.7$) the stress started to increase steeply again.

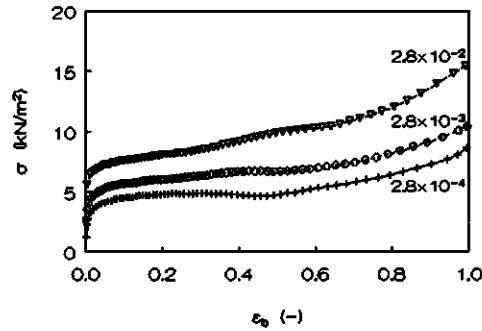


Figure 3.4 Stress-strain curves for a standard dough. Initial $\dot{\epsilon}_b$ indicated.

At the transition from the sharp increase in stress with increasing ϵ_b to the "plateau" region the material yields implying strong energy dissipation³⁰. Yielding occurred at biaxial strains of 0.01-0.05, indicating strong strain sensitivity of the dough. This is typical of particle networks and highly concentrated dispersions. Oscillatory measurements also showed a very limited linear region (Chapter 2). Therefore, values of the Young's modulus could not be derived. The "plateau" region indicates an ongoing rearrangement of structure due to flow and yielding processes. Generally speaking this region constitutes the essential part of the whole curve. The increase of the stress with ϵ_b at high strain (> 0.7) is, at least partly, due to friction between the plates and the material. There also may be an effect of higher $\dot{\epsilon}_b$ on the material properties of the dough.

The slope of the stress-strain curves prior to yielding appeared to increase with increasing $\dot{\epsilon}_b$ implying that the dough is viscoelastic within this strain region. Whereas the "apparent yield stress" increased with $\dot{\epsilon}_b$, no measurable effect of $\dot{\epsilon}_b$ on the "apparent yield strain" was observed within the accuracy of the experiments. In general, the stress needed to obtain a given ϵ_b increased with increasing $\dot{\epsilon}_b$. This is presumably due to energy dissipation processes. The materials tested are very inhomogeneous, even at macroscopic scale (*e.g.* sucrose crystals may be as large as 0.5 mm). This means that deformation will also be locally inhomogeneous. As a result, friction between structural elements may play an important role in energy dissipation (*e.g.* friction between sucrose crystals and particles forming the network such as fat or starch particles)³¹. In addition, viscous flow of the material also causes energy dissipation.

Results for the worked fats are given in Figure 3.5. Even if the difference in initial $\dot{\epsilon}_b$ is taken into account, in comparison with the standard dough, the worked standard

fat had a lower resistance to large deformation. It is obvious that this must be due to the fat being more easily deformed than the non-fat phase. It is also clear that, for each initial $\dot{\epsilon}_b$ applied, the standard fat was more deformable than the firm fat.

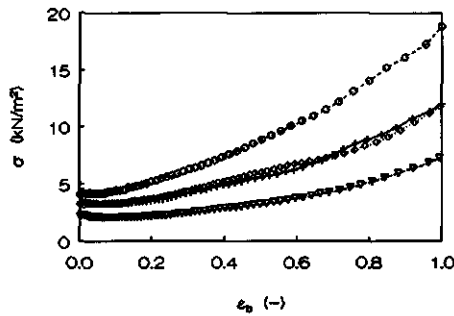


Figure 3.5 Stress-strain curves for (∇), (\diamond) a worked standard fat and ($+$), (\circ) a worked firm fat: (∇), ($+$) initial $\dot{\epsilon}_b = 4.2 \times 10^{-3} \text{ s}^{-1}$; (\diamond), (\circ) initial $\dot{\epsilon}_b = 4.2 \times 10^{-2} \text{ s}^{-1}$.

Stress-strain curves for the other doughs tested are depicted in Figure 3.6 (initial $\dot{\epsilon}_b = 2.8 \times 10^{-4} \text{ s}^{-1}$).

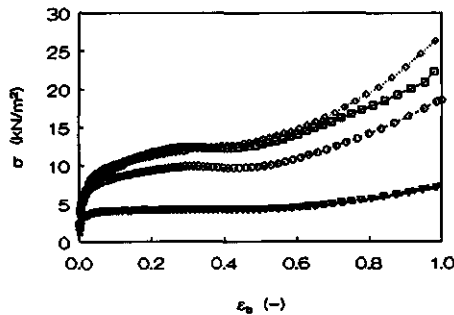


Figure 3.6 Stress-strain curves for various doughs: (\square) sugar-free dough; (Δ) sucrose-syrup dough; (∇) starch dough; (\diamond) low-fat dough; (\circ) firm-fat dough. Initial $\dot{\epsilon}_b = 2.8 \times 10^{-4} \text{ s}^{-1}$.

Similar curves were obtained at higher initial $\dot{\epsilon}_b$. Irrespective of deformation rate, the overall shape of the stress-strain curve for the sugar-free dough was substantially different from that of the standard dough. There was also a kind of "yield point", but the transition was not sharp. The curves did not level off at low ϵ_b but kept rising to reach a region in which more extensive fracture occurred. This demonstrates that at small strains and comparable time scales, the sugar-free dough is subject to less structural breakdown on deformation than the standard dough. In other words, there is a significant elastic contribution to deformation up to larger ϵ_b .

To check whether the different behaviour of the sugar-free dough was due to fewer or smaller inhomogeneities (because of the absence of sucrose crystals), a sucrose-syrup dough was investigated. The sucrose was predissolved in the water to be added. The sucrose content chosen was based on the maximum amount that could be completely dissolved within 1 hour at room temperature. It is noteworthy that, owing to the slow dissolution of crystalline sucrose in water, the amount of undissolved sucrose crystals in the other doughs tested was higher than would follow from the solubility. Stress-strain curves for a sucrose-syrup dough are also presented in Figure 3.6. For all initial $\dot{\epsilon}_b$ applied, the curves were almost identical to those for the standard dough. Therefore, the typical yielding and flow behaviour of the standard dough cannot be explained by the mere presence of sucrose crystals.

Comparison of the curves obtained for the standard dough with those for the starch dough shows no qualitative differences. It should be mentioned that gluten was not separated from the flour. Commercial native starch was substituted for flour. This means that not only gluten but also other components, such as damaged starch and pentosans, were absent in the starch dough.

As can be seen in Figure 3.6 the stress-strain profiles for the firm-fat dough and the low-fat dough were also qualitatively similar. In the low-fat dough, friction between structural elements possibly provided a larger contribution to energy dissipation than in standard dough (less liquid oil acting as a lubricant).

Plots of the apparent biaxial extensional viscosity as a function of biaxial extensional strain rate for the materials investigated are presented in Figure 3.7. In the beginning η_b^* seemingly increased due to elastic effects. It reached a maximum at a certain ϵ_b (about 0.05) beyond which η_b^* decreased steeply. Near the maximum extensive yielding occurs, resulting in biaxial flow at higher ϵ_b . Generally, the apparent biaxial extensional viscosity of all materials tested followed a power law behaviour³²:

$$\eta_b^* = K \dot{\epsilon}_b^{n-1} \quad (3.5)$$

where K (Pa·s^{*n*}) is sometimes called the "consistency index" and n (-) is called the "flow behaviour index". K is a measure of the apparent biaxial extensional viscosity at $\dot{\epsilon}_b = 1 \text{ s}^{-1}$ and n indicates whether a material is Newtonian ($n = 1$), strain-rate thinning ($n < 1$), or strain-rate thickening ($n > 1$).

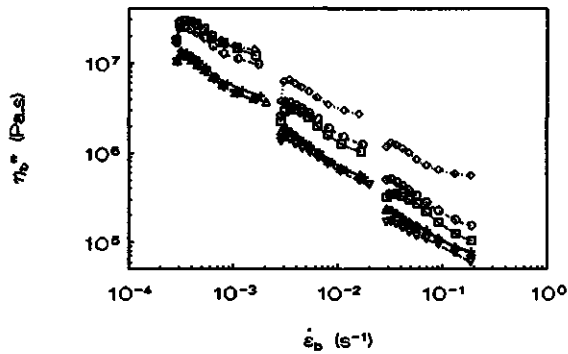


Figure 3.7 η_b^* as a function of $\dot{\epsilon}_b$ for various doughs: (+) standard dough; (□) sugar-free dough; (Δ) sucrose-syrup dough; (∇) starch dough; (◇) low-fat dough; (○) firm-fat dough.

The values of the power law parameters were calculated at a biaxial strain of 0.2 to avoid considerable contribution of either elastic effects (at small ϵ_b) or friction between plates and test-piece (at large ϵ_b). The results are summarised in Table 3.2.

Table 3.2 Consistency index K and flow behaviour index n for η_b^* as a function of $\dot{\epsilon}_b$ for all materials tested. Values calculated at $\epsilon_b = 0.2$

	n	$K \times 10^{-4}$ Pa s ^{n}
Standard dough	0.10	1.1
Firm-fat dough	0.15	2.9
Starch dough	0.09	0.8
Low-fat dough	0.31	12.1
Sugar-free dough	0.03	1.4
Sucrose-syrup dough	0.12	1.2
Worked standard fat	0.21	0.6
Worked firm fat	0.17	0.8

It is evident from Figure 3.7 that, at constant biaxial strain η_b^* fell rapidly with increasing $\dot{\epsilon}_b$, implying a prominent strain-rate thinning character for all materials tested. At every $\dot{\epsilon}_b$ tested, the low-fat dough was less easily deformed than the firm-fat dough, followed by the standard dough and the starch dough. At low $\dot{\epsilon}_b$, the apparent

biaxial extensional viscosity of the sugar-free dough was similar to that of the low-fat dough whereas at higher $\dot{\epsilon}_b$ it was significantly lower and even lower than that of the firm-fat dough. These differences are reflected in the n values shown in Table 3.2. The sugar-free dough and the low-fat dough exhibited the most and the least pronounced strain-rate thinning behaviour, respectively.

The low-fat dough was the only one showing distinct ageing effects within 4 hours after preparation. Drying out of the sample was not the cause. A weight loss of 0.6% was found when a sample was exposed to ambient temperature and relative humidity. This loss may be just significant, but it occurred largely within the first hour. Figure 3.8 depicts η_b^* as a function of $\dot{\epsilon}_b$ for a low-fat dough at different ageing times.

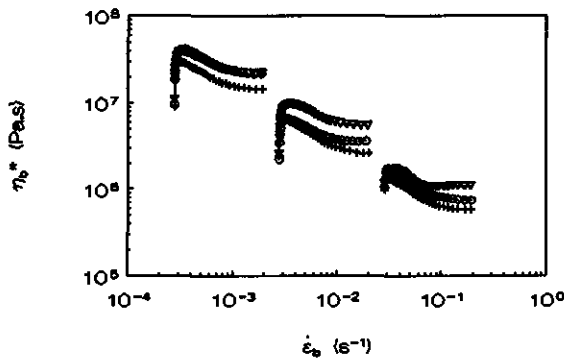


Figure 3.8 η_b^* as a function of $\dot{\epsilon}_b$ and ageing time after completion of mixing for a low-fat dough: (+) 1 hour; (○) 2.5 hours; (∇) 4 hours.

While any effect on the strain-rate thinning behaviour is uncertain, it is clear that, η_b^* (at a given strain rate) increased with ageing time. This suggests that more and/or stronger interactions come into play with time. These effects are unlikely to reside in the fat phase, because, within the experimental accuracy (results not shown), the worked standard fat did not become firmer during ageing (at large $\dot{\epsilon}_b$). If the fat would show firming up, ageing effects would be expected to occur in the other doughs as well.

3.4 Discussion

In Chapter 2 the relation between the small deformation properties of short doughs and their structure was discussed. These results showed that, regardless of the fat content, doughs exhibited a relatively more liquid-like behaviour at low strain rates than at high strain rates. This finding is in accordance with the part of the stress-strain curves prior to yielding, although the time scales are not strictly comparable (Fig. 3.4). In dynamic experiments it was also found that the behaviour of the standard dough changed from solid-like to more liquid-like at a shear strain of about 0.1. This is consistent with observations from Menjivar and Faridi³³, who determined the elasticity of short doughs by measuring the normal stress difference in shear experiments. They reported maximum normal stress difference at shear strains of 0.03 to 0.08, beyond which it fell sharply. This is also in reasonable agreement with the "apparent yield strain" shown in Figure 3.4; it strongly suggests that the properties of the standard dough are markedly altered beyond yielding. Nevertheless, the trends observed at small deformation resemble those at large deformation. For instance, the resistance to deformation followed the order: firm-fat dough > standard dough > starch dough. In addition, these doughs exhibited similar rate-dependent behaviour. The η_b^* values of these three doughs followed the same order (Fig. 3.7) and the strain-rate thinning character was similar (Table 3.2). These qualitative similarities suggest that the overall behaviour is determined by a common factor, which, in turn, may be due to internal structures not varying greatly. Quantitative differences are due to the firm fat being less easily deformed than the standard fat (Fig. 3.5) and possibly to the lower ratio of non-fat solids to water in starch dough as compared to standard dough.

In small deformation experiments (Chapter 2), the low-fat dough was more easily deformed than the standard dough. This shows that there is no direct relationship between small and large deformation properties. This agrees with the observation that the storage modulus curves of the standard and the low-fat doughs (as a function of strain) intersected at strains outside the linear region. The fat being more deformable than the non-fat phase at large deformation (compare Figs. 3.4 and 3.5), would explain the observed differences between the low-fat dough and the standard dough (compare Figs. 3.4 and 3.6). In the previous Chapter it was suggested that the standard dough would be bicontinuous, whereas the low-fat dough would be predominantly fat dispersed; the non-fat phase was described as being composed of a sucrose syrup binding flour/starch particles by means of liquid necks acting as bridges. It seems reasonable to assume that the yielding and flow behaviour exhibited by both doughs, at large deformation, involves primarily the non-fat phase. Since undissolved sucrose crystals did not alter the pattern of the stress-strain curves (Fig. 3.6), flour/starch particles may act as the principal inhomogeneities resulting in stress concentration, and governing the yield behaviour.

Evidence to support these ideas about the principal role of the structure of the non-fat phase is further provided by the stress-strain curves for the sugar-free dough, which point to a different behaviour of the non-fat phase. If fat would dominate the yield behaviour of standard dough, the same should occur in the sugar-free dough, which has a higher fat content.

It may be argued that sucrose can delay the rate of swelling of damaged starch and pentosans¹⁶, which take up water in amounts of 1.5 and 10 times their own dry mass³⁴, respectively. Whether this water uptake is quantitatively the same in a short dough is, however, questionable. Nevertheless, an increased rate of swelling would significantly increase viscosity. However, considering that the experiments were carried out at least 1 hour after the completion of mixing, it is likely that any effect of sucrose on swelling would be small.

The question now arises as to what makes up the non-fat phase in the sugar-free dough. It has been suggested¹⁶ that a sucrose solution acts as an antiplasticiser (compared to water alone) in glassy polymers such as gluten. Since sucrose has a greater molar mass than water, a concentrated sucrose solution would cause less effective plasticising, *i.e.* a smaller depression of the glass transition temperature and a smaller increase in the mobility of solutes in the system. The decreased mobility would indicate a retarded rate of water uptake by gluten, hence a retarded rate of gluten development during mixing.

A matrix with a three-dimensional gluten network can be deformed elastically up to larger strains³³. Bread doughs exhibit strain-hardening behaviour^{35,36} at ϵ_b as large as 1.2. These observations, together with the results shown in Figure 3.6, suggest that gluten affects, to a certain extent, the response of the sugar-free dough at large deformation. Yet, the behaviour of this dough is by no means typical of a three-dimensional gluten network^{37,38}. Corroboration of this idea was further provided by the following experiment. It was attempted to prepare a low-fat dough without sucrose in the Farinograph, while keeping the same flour to water ratio as that in the sugar-free dough. Within the experimental mixing time (8 min), a cohesive dough was not formed, indicating that neither a continuous gluten phase nor a continuous fat phase could be formed, due to insufficient quantities of either water or fat. This means that sucrose would be an essential component of the continuous non-fat phase in the low-fat dough. It also implies that the sugar-free dough would be bicontinuous. The same conclusion was drawn from oscillatory experiments (Chapter 2). It may be mentioned that the dynamic moduli were the same for the standard dough and the sugar-free dough. Also, stress sweeps did not reveal any differences, possibly because the strain region investigated was quite limited ($\gamma < 0.01$). Menjivar and Faridi³³ also reported that small deformation experiments could not resolve differences in gluten development between cracker doughs and short doughs.

It should be realised that the observed differences between the standard dough and the sugar-free dough may not solely be due to solvent quality; sucrose also affects solvent quantity. Sucrose, when dissolved in water, increases the volume of the aqueous phase. One gram of sucrose, when dissolved in 1 g of water, produces 1.63 ml of total solution³⁹.

3.5 References

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Chapter 4

Large Deformation Properties of Short Doughs: Effect of Sucrose in Relation to Mixing Time

Abstract

Large deformation rheological properties of short doughs of various composition prepared under various mixing times were determined in uniaxial compression. Sucrose-syrup doughs exhibited prominent yielding and flow behaviour. Their apparent biaxial extensional viscosity decreased with increasing sucrose content. Compared to the stress-strain curves for the sucrose-syrup doughs, those for the sugar-free doughs indicated a stronger elastic contribution to deformation. The deformability of the latter doughs increased with increasing water content. Regardless of dough type, mixing time had a pronounced effect on dough consistency. In addition, it drastically changed the shape of the stress-strain curve for a sugar-free dough. These results are discussed in terms of the structure of short doughs. It is concluded that sucrose delays, if not inhibits, gluten development and promotes formation of a non-fat continuous phase, whereas mixing promotes formation of a continuous fat phase.

4.1 Introduction

Short doughs encompass the widest range of recipes of any biscuit type; some rough divisions can be made, such as fat-rich, sugar-rich, lean etc., but these are not well-defined. As a result, short doughs are often subdivided on the basis of the method of dough piece formation, *e.g.* rotary-moulded, wire-cut and rout-/bar-press. The methods used depend strongly on the consistency of the dough. For instance, stiff doughs are formed on a rotary moulder. Since the quantities of fat and sucrose vary widely in short dough recipes, these ingredients have a direct impact on the consistency of the dough¹.

Control over dough consistency would be desirable from the manufacturing point of view. This aspect becomes more important in developing new products (*e.g.* low-fat, low-sugar). In this case, modification of the handling characteristics of the dough may require adjustment of the processing variables or even different equipment.

Publications on the effect of sucrose, water and mixing time on the rheological properties of short doughs are limited and sometimes contradictory. Olewnik and Kulp² employed the Brabender Farinograph and found that rotary-moulded doughs

become softer with increasing either sucrose or water contents. Miller³ used the Stevens-LFRA Texture Analyser and found that consistency of rotary-moulded doughs is not affected by sucrose content and decreases with increasing water content. Slade and Levine⁴ reported that sugar-snap dough consistency decreased with increasing amounts of sucrose, as determined by the Stevens-LFRA Texture Analyser. Gaines⁵ evaluated the consistency of sugar-snap doughs by compression-penetration tests. He found that slightly increasing water content significantly affected dough consistency; the test method was insensitive to variations in mixing time. Gaines⁶ employed the "Simon Research water absorption meter" and found that the consistency of sugar-snap doughs immediately after mixing decreased as dough water content increased; the magnitude of the effect was smaller in doughs tested 1 hr after mixing. The changes in dough consistency occurring 1 hr after mixing differ from one flour to another and are a function of water content and initial dough consistency after completion of mixing^{3,6}. Hosney *et al.*⁷ argue that instruments such as the Farinograph and the Mixograph were developed for bread doughs; because the desirable rheological properties of short doughs, albeit not well quantified, are different from those of bread doughs, these techniques are inappropriate for short doughs. Anyway, in the studies cited above²⁻⁶, the parameters measured are not well defined, due to the empirical nature of the test methods.

In commercial operations, assessment of dough consistency is mainly based on the operator's experience; the sample is often squeezed between the fingers⁸. In various processing operations (*e.g.* depositing) the dough is subject to large deformations. This is why determination of "fundamental" rheological properties should involve large deformations at the relevant time scales.

In the previous Chapter it was suggested that sucrose delays gluten development, which implies that mixing time may be an important variable; in addition, sucrose affects both solvent quality and quantity. These aspects merit further investigation. It is the purpose of this Chapter to report the effect of sucrose and mixing time on large deformation properties of short doughs.

4.2 Experimental

4.2.1 Materials

Commercial wheat flour was obtained from Meneba Meel BV (Rotterdam, the Netherlands). The chemical composition of the flour was given in Chapter 2. Granulated sucrose was provided by Suiker Unie (Breda, the Netherlands). Fat was supplied by Bakkerol BV (Rotterdam, the Netherlands). Deionised water was used in all experiments.

4.2.2 Preparation of doughs

Doughs were prepared in a Farinograph employing a 50 g bowl. Unless otherwise mentioned, mixing was carried out for 8 min according to the procedure described in Chapter 2. Sucrose was predissolved in the water to be added. To keep the terminology used consistent with that in the previous Chapter, the terms sucrose-syrup dough and sugar-free dough will be used for compositions with and without sucrose, respectively. Dough compositions are presented in Table 4.1.

Table 4.1 Composition of the doughs (% w/w) investigated^a

Ingredient	sucrose	sucrose	sucrose	sugar	sugar	sugar
	syrup 1	syrup 2	syrup 3	free 1	free 2	free 3
Flour ^b	41.5	45.0	46.5	50.0	46.7	47.8
Fat	25.7	27.9	28.8	31.0	28.9	29.6
Sucrose	17.0	10.1	7.0	-	-	-
Added water	9.3	10.1	10.5	11.2	17.1	15.2
Flour water	5.7	6.1	6.3	6.8	6.4	6.5
Salt	0.8	0.8	0.9	1.0	0.9	0.9

^a For explanation of numbers see text

^b Dry matter

Comparison of doughs with and without sucrose was done in two different ways. The numbers 1, 2 and 3 for the sucrose-syrup doughs denote 64.6%, 50% and 40% sucrose solutions, respectively; the dough called sugar-free 1 is as the one designated sucrose-syrup 1, but without sucrose. These doughs differ in sucrose content, *i.e.* in both solvent quality and quantity. On the other hand, at a constant dry flour content the volumes of water added in the doughs designated as sugar-free 2 and 3 are equal to the volumes of the sucrose solution in the doughs named sucrose-syrup 2 and 3. This was done to separate the effects due to solvent quality and quantity. Calculation of the water added was done through the density of the sucrose solutions⁹; it was assumed that the water in flour does not act as a solvent. Storage of ingredients, preparation of doughs and determination of mechanical properties were done at 20 °C in a temperature-controlled room. Test-piece formation was done by punching a tube of 20 mm internal diameter into the dough and cutting the cylinder obtained into pieces of predetermined height by means of a wire.

4.2.3 Uniaxial compression experiments

Large deformation properties of short doughs were studied by uniaxial compression. A Zwick (type 142510/00) tension-compression apparatus was used with a 50 N load-cell. Cylindrical test-pieces were compressed between two parallel circular perspex plates. To achieve complete contact between the test-piece and the plates from the beginning of the measurements, test-pieces were cut at a height of 32 mm and slowly (0.5 mm/min) compressed to 30 mm. Most of the stress, so induced to the sample, relaxed within an hour, after which the upper plate was lowered at a constant speed and the force applied on the test-piece was recorded as a function of time. The diameter of the plates was the same as that of the test-pieces, *viz.* 20 mm. Doughs were tested at three crosshead speeds (1, 10 and 100 mm/min). Stress σ (N m^{-2}), biaxial Hencky strain ϵ_b (-), biaxial extensional strain rate $\dot{\epsilon}_b$ (s^{-1}) and apparent biaxial extensional viscosity η_b^* ($\text{Pa}\cdot\text{s}$) were calculated as shown in Chapter 3. Experiments were carried out in triplicate on freshly prepared doughs. Curves shown represent averages of these replicates. The reproducibility of the measurements was quite satisfactory. Irrespective of the dough tested, the coefficient of variation was, on average, 9% and never higher than 17% even at the largest strain investigated.

4.3 Results

4.3.1 Stress-strain curves

4.3.1.1 Effect of sucrose content

Stress-strain curves for the sugar-free 1 and all the sucrose-syrup doughs at an initial $\dot{\epsilon}_b$ of $2.8 \times 10^{-4} \text{ s}^{-1}$ are depicted in Figure 4.1. Similar curves were obtained at faster deformation rates. The sucrose-syrup doughs yielded qualitatively similar curves. Initially, the stress increased with increasing ϵ_b . At a certain ϵ_b (< 0.05) pronounced yielding and subsequent flow occurred, as can be seen from the "plateau" region. Obviously, much of the structure of the doughs was broken down. Young's moduli could not be derived because of the limited region of linear elasticity. However, the slope of the stress-strain curves prior to yielding appeared to increase with increasing $\dot{\epsilon}_b$ presumably due to viscoelastic effects. This was accompanied by an increase in the "apparent yield stress", whereas the "apparent yield strain" remained virtually constant. The stress needed to obtain a given ϵ_b increased with increasing $\dot{\epsilon}_b$, implying energy dissipation mainly due to viscous flow. Quantitatively, the doughs were more easily deformed for a higher sucrose content.

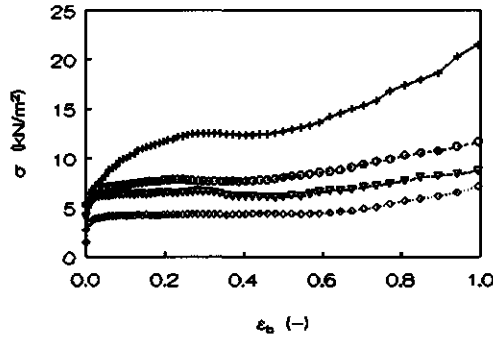


Figure 4.1 Stress-strain curves for various doughs: (\diamond) sucrose-syrup 1; (∇) sucrose-syrup 2; (\circ) sucrose-syrup 3; (+) sugar-free 1; for explanation of numbers see text. Initial $\dot{\epsilon}_b = 2.8 \times 10^{-4} \text{ s}^{-1}$.

As can be seen in Figure 4.1, the sugar-free dough exhibited a different behaviour in comparison with the other doughs; this was also observed at faster deformation rates. Unlike the sucrose-syrup doughs, which change from solid-like to liquid-like, the sugar-free dough did not show such clear yielding.

4.3.1.2 Effect of water content

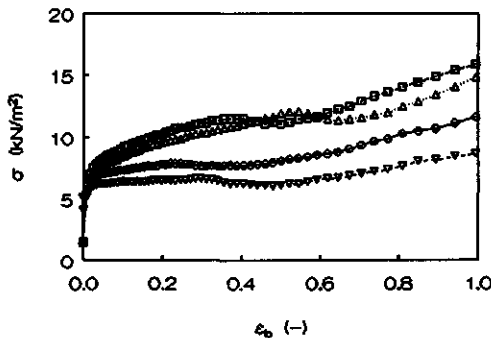


Figure 4.2 Stress-strain curves for various doughs: (∇) sucrose-syrup 2; (\circ) sucrose-syrup 3; (Δ) sugar-free 2; (\square) sugar-free 3; for explanation of numbers see text. Initial $\dot{\epsilon}_b = 2.8 \times 10^{-4} \text{ s}^{-1}$.

To ascertain the effect of solvent quality, sugar-free doughs were prepared in which, for a constant dry flour content, the volumes of solvent (added water) were equal to those (sucrose solution) in the sucrose-syrup doughs. The stress-strain curves for the sugar-free doughs together with the corresponding sucrose-syrup doughs are presented in Figure 4.2. Their shape was markedly different from that for the sucrose-syrup doughs. Instead of the "plateau region" the stress continued to ascend with increasing ϵ_b until a point where a kind of fracture occurred. However, at faster deformation rates (results shown in Figure 4.3) this "fracture region" was not evident; the curves showed an upturn at high ϵ_b , notably different even from that for the sugar-free 1.

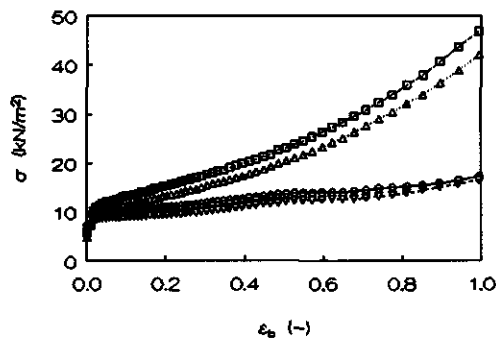


Figure 4.3 Stress-strain curves for various doughs: (∇) sucrose-syrup 2; (\circ) sucrose-syrup 3; (Δ) sugar-free 2; (\square) sugar-free 3; for explanation of numbers see text. Initial $\dot{\epsilon}_b = 2.8 \times 10^{-2} \text{ s}^{-1}$.

Regardless of deformation rate, at high ϵ_b (> 0.6) the stress rose steeply, partly due to the higher $\dot{\epsilon}_b$ and partly due to friction between the plates and the material. As far as solvent quantity is concerned, the deformability of the sugar-free doughs increased with increasing water content.

4.3.1.3 Effect of mixing time

Stress-strain curves for the sucrose-syrup 1 and the sugar-free 1 doughs at various mixing times are given in Figure 4.4. For the former dough the shape of the curves was similar regardless of mixing time. Quantitatively, the dough had a lower resistance to deformation, the more so for a longer mixing time. Mixing time had a profound effect on the rheological properties of the sugar-free dough. The changes induced by short mixing times (between 2 and 8 min) are largely quantitative. Beyond, the character of the dough was also qualitatively altered, the shape of the stress-strain

curve bearing resemblance to that for the sucrose-syrup dough, exhibiting the characteristic yielding.

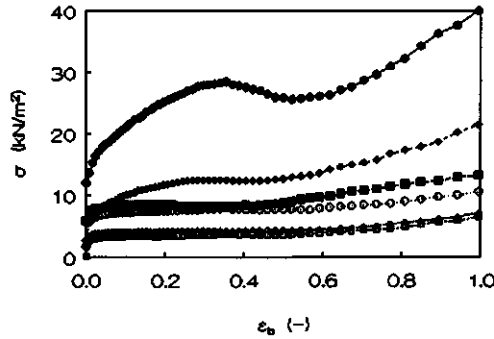


Figure 4.4 Stress-strain curves for various doughs; (open symbols) sucrose-syrup 1, (closed symbols) sugar-free 1: (○), (●) mixing time = 2 min; (◇), (◆) mixing time = 8 min; (□), (■) mixing time = 20 min. Initial $\dot{\epsilon}_b = 2.8 \times 10^{-4} \text{ s}^{-1}$.

It is noteworthy that the curves for the doughs mixed for 8 and 20 min intersected (more clearly observed at slow deformation rates) at small ϵ_b . This implies that at small deformation the dough mixed for 20 min was less easily deformed than that mixed for 8 min. This, in turn, shows that there is no direct relationship between small and large deformation properties.

4.3.2 Apparent biaxial extensional viscosity

The apparent biaxial extensional viscosity as a function of biaxial extensional strain rate for all the doughs tested are plotted in Figures 4.5 - 4.7. The results could be represented by a "power-law model"¹⁰:

$$\eta_b^* = K \dot{\epsilon}_b^{n-1} \quad (4.1)$$

To avoid contribution of either elastic effects present at small ϵ_b or friction between the plates and the test-piece present at large ϵ_b , the "consistency index" K (Pa·sⁿ) and the "flow behaviour index" n (-) were calculated at an ϵ_b of 0.2. The results are tabulated in Tables 4.2 and 4.3.

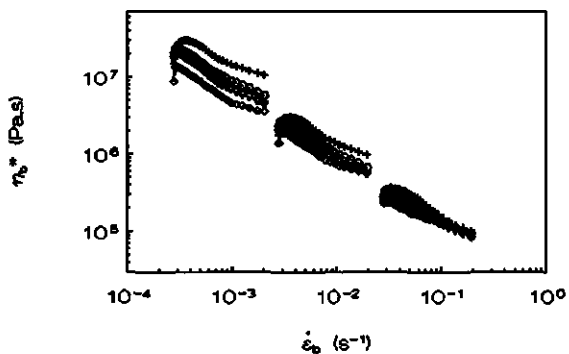


Figure 4.5 η_b^* as a function of $\dot{\epsilon}_b$ for various doughs: (\diamond) sucrose-syrup 1; (∇) sucrose-syrup 2; (\circ) sucrose-syrup 3; (+) sugar-free 1; for explanation of numbers see text.

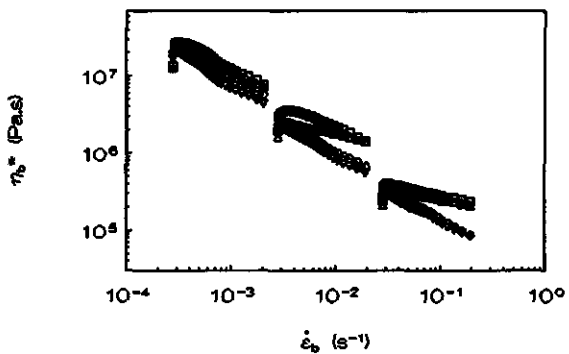


Figure 4.6 η_b^* as a function of $\dot{\epsilon}_b$ for various doughs: (∇) sucrose-syrup 2; (\circ) sucrose-syrup 3; (Δ) sugar-free 2; (\square) sugar-free 3; for explanation of numbers see text.

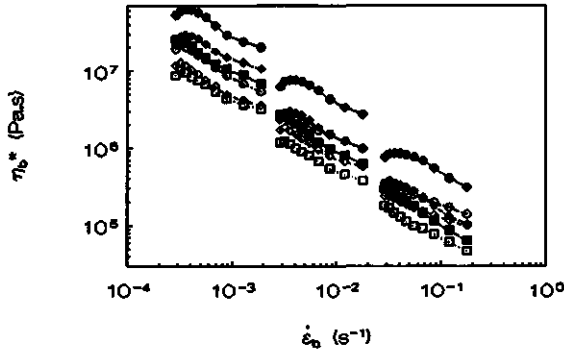


Figure 4.7 η_b^* as a function of $\dot{\epsilon}_b$ for various doughs: (open symbols) sucrose-syrup 1, (closed symbols) sugar-free 1: (○), (●) mixing time = 2 min; (◇), (◆) mixing time = 8 min; (□), (■) mixing time = 20 min.

As can be seen in Figures 4.5 - 4.7, at a given ϵ_b , η_b^* decreased rapidly with increasing $\dot{\epsilon}_b$, suggesting that all doughs exhibited a strong strain-rate thinning behaviour. This is also evident from the n values in Tables 4.2 and 4.3.

Table 4.2 Consistency index K and flow behaviour index n for η_b^* as a function of $\dot{\epsilon}_b$ for various doughs mixed for 8 min. Values calculated at $\epsilon_b = 0.2$

	n	$K \times 10^{-4}$ Pa s ^{n}
Sucrose-syrup 1	0.14	1.3
Sucrose-syrup 2	0.08	1.2
Sucrose-syrup 3	0.08	1.4
Sugar-free 1	0.04	1.6
Sugar-free 2	0.07	1.6
Sugar-free 3	0.09	2.2

Table 4.3 Consistency index K and flow behaviour index n for η_b^* as a function of $\dot{\epsilon}_b$ for two types of doughs and various mixing times. Values calculated at $\epsilon_b = 0.2$

	mixing time min	n	$K \times 10^{-4}$ Pa·s ^{<i>n</i>}
Sucrose-syrup 1	2	0.12	1.8
	8	0.14	1.3
	20	0.09	0.7
Sugar-free 1	2	0.08	4.7
	8	0.04	1.6
	20	0.02	1.0

4.4 Discussion

The results described above (Figs. 4.1 and 4.5) show that the doughs are more easily deformed with increasing sucrose content, particularly at slow deformation rates. The following factors may play a part:

1. The viscosity of a sucrose solution increases with increasing sucrose content⁹. For example, considering the sucrose-syrup doughs 1 and 3, the viscosity of the hypothetical sucrose solution in the former dough would be about 20 times that in the latter⁹.
2. It may be argued that the flour weight fraction increases with decreasing sucrose content and that this could override the effect due to the viscosity of the sucrose solution.
3. In the previous Chapter it was concluded that the fat is more deformable than the non-fat phase at large deformation and that the dough "sucrose-syrup 1" is a bicontinuous dough. Assuming the same fat distribution in the other doughs, the effect of increasing fat weight fraction with decreasing sucrose content will tend to compensate for the effect of increasing flour weight fraction.
4. Sucrose affects the amount of solvent; one gram of sucrose, when dissolved in one gram of water, produces 1.63 ml of total solution⁹. This affects the various volume fractions and it might explain the effect of sucrose content, particularly in view of the results presented in Figs. 4.2 and 4.6. These results show that sugar-free doughs are more deformable with increasing water content, which would be expected from the lower ratio of non-fat solids to water. The density of the flour (including its 12% water content), which can be estimated as shown in Chapter 2, the densities of the fat¹¹ and the sucrose solutions⁹ and their mass fractions (Table

4.1) can be used to calculate their corresponding volume fractions. For the sucrose-syrup 1 these are 40.9, 34.4 and 24.7, respectively; for the sucrose-syrup 3 these are 44.6, 37.6 and 17.8, respectively. Therefore, it seems unlikely that the difference in the volume fraction of the sucrose solution would be solely responsible for overshadowing the effect due to the viscosity of the sucrose solution.

5. It is to be expected that sucrose affects solvent quality, and thereby possibly the gluten properties. For a constant volume of solvent and dry flour content, the elastic contribution to deformation is observed to be more important in the absence of sucrose (Figure 4.2). This difference between the sucrose-syrup and the sugar-free doughs is related to the non-fat phase. The sugar-free dough being deformed elastically to a larger strain and the absence of a prominent yielding, point to some gluten development.

By affecting both solvent quality and quantity, sucrose may also affect the mixing properties of the non-fat phase and thereby the extent of its dispersion. This will be discussed further below. The results presented in this paper support the views discussed in the previous Chapter that sucrose:

- affects gluten development by affecting solvent quality.
- facilitates formation of a non-fat continuous phase via its effect on solvent quantity; this phase contains a sucrose syrup that can "glue" flour/starch particles together by means of liquid necks.

It was also found that mixing time considerably affected the rheological properties of the doughs, particularly those of the sugar-free dough (Figs. 4.4 and 4.7). The doughs were more deformable, the more so for a longer mixing time. This is in contrast to the widely held belief that longer mixing leads to stronger gluten development^{1,8}. Distribution of the fat phase throughout the non-fat phase is primarily determined by the rheological properties of the former relative to those of the latter. During mixing these properties change; as a result, their contribution to the rheological properties of the dough is altered. The shape of the stress-strain curves for the sucrose-syrup dough was similar, regardless of mixing time. Moreover, it was found that fat is more deformable than the non-fat phase at large deformation (Chapter 3). It seems reasonable to conclude that the observed quantitative changes occurring during mixing primarily involve the continuity of the fat phase. From our understanding of the material, it is unlikely that mixing would cause significant changes in the properties of the non-fat phase. So, taking into account our observation that the dough mixed for 8 min is bicontinuous (Chapter 3), the observed changes would be due to an increase in fat continuity, giving rise to a transition from a "more" fat-dispersed dough to a "more" bicontinuous dough with increasing mixing time. This also appears to be true for the sugar-free dough. However, the dramatic change from a "more elastic dough" (mixed for 8 min), which was also found to be bicontinuous (Chapter 3), to a "more plastic dough" (mixed for 20 min) suggests that mixing also exerts a

kind of modification in the rheological properties of the continuous non-fat phase, presumably of the gluten in particular. Regardless of dough type and mixing time, all doughs fractured in tension; given that pure fat fractured in shear⁹, this points to the doughs not being fully fat continuous. If the doughs were fully fat continuous, dough pieces would not retain their shape during baking, but they did.

These findings have important implications for the handling characteristics of the dough. For instance, the intensity of mixing may vary, according to the machinery used. This means that, for a given recipe and mixing time, doughs prepared in different mixers may have different rheological properties. Indeed, it was found (results not shown) that a sugar-free dough mixed in a Hobart mixer for about 2 min yielded a stress-strain curve qualitatively similar to that for a dough mixed in the Farinograph for 8 min. Further mixing (> 2 min) caused the dough to exhibit a behaviour qualitatively similar to that of a dough mixed in the Farinograph for 20 min. These differences have a direct impact on subsequent processing steps, such as depositing, the rate of which depends on dough consistency. For instance, in a bench-scale depositing machine it was not possible to control the rate of extrusion of a sugar-free dough mixed in a Hobart mixer for 2 min. This was due to the changes in dough consistency (discussed above) effected by the rollers; there were virtually "two doughs" in the depositing machine. This would imply that efficient control over the rate of dough extrusion requires a relatively stable dough consistency on further processing. This aspect becomes more important for rotary-moulded doughs; it is common practice to remove the surplus dough, known as "scrap dough", from the roller and return it to the feed hopper¹².

The rheological properties of dough also have important implications for the baked product, as will be shown later. For instance, fat continuity in the biscuit is related to fat continuity in the dough which, in turn, is affected by mixing time. Gas bubble expansion during baking would involve factors such as the viscosity of the dough and the extent of gluten development.

4.5 References

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Chapter 5

Method for Determining Fracture Properties of Short-dough Biscuits

Abstract

Fracture properties of short-dough biscuits were determined in three-point bending tests. Biscuits exhibited an essentially elastic behaviour and fractured at small strain. Test conditions were varied. Span length did not influence the parameters measured, in accordance with theory. Biscuits fractured with the bottom side upwards had slightly higher fracture stress and fracture strain than those in the inverted position. Presumably, air cells at the bottom side acted as defects. Fracture strain was strain-rate dependent over the range studied possibly due to local plastic deformation. Reducing the fat content increased the fracture stress of the biscuits, which indicates that the method provides a useful tool for discriminating among short-dough biscuits of various composition.

5.1 Introduction

Short-dough biscuits comprise widely diverse products. Generally, they are made from short doughs relatively rich in both fat and sucrose with small amounts of water¹. Short-dough biscuits can be further classified according to the method of dough piece formation, e.g. rotary-moulded, wire-cut, rout-press, etc. Differences in these methods are related to variation in dough consistency which, in turn, reflects variation in dough composition¹. The terminology used varies in different countries. Owing to this, a rigorous definition of the term short-dough biscuit does not exist. However, a basic difference between short-dough biscuits and other types is that the former increase in size (so-called "spread") during the early stages of the baking process².

It is widely recognised, that fracture properties constitute an important quality characteristic of biscuits. Fracture behaviour affects the perception of the sensory properties during mastication. Fracture properties may also be an important issue, for instance, when choosing proper storage conditions and/or packaging materials. Fracture of a material starts when the stress imposed, which will be locally higher near inhomogeneities, exceeds the adhesive or cohesive forces in the material at that place. When the material fractures, strain energy is released. If the differential energy released becomes available at the tip of the crack and is at least equal to the

differential energy needed to create more new surfaces, fracture propagates spontaneously³.

Various instruments have been employed to assess mechanical properties of baked products. Apart from the FMBRA Biscuit Texture Meter⁴⁻⁶, the principle is to deform the sample and monitor the resistance offered by the sample. Such instruments include the General Foods Texturometer⁷, the Stevens-LFRA Texture Analyser, the Kramer Shear Press and the Instron. The latter three are very versatile since numerous types of tests can be performed depending on the attachments utilised. Examples are:

- shear tests⁸⁻¹⁵.
- puncture tests¹⁶⁻²³.
- texture profile analysis²⁴.

In the above mentioned tests the types of forces exerted on the test-piece are complex and the results depend on the configuration of the load-cells and probes besides on specimen properties.

Another approach adopted involves more "fundamental" methods. Doescher *et al.*²⁵ employed compression tests to evaluate sugar-snap biscuits. Jowitt and Mohamed²⁶ and Mohamed *et al.*²⁷ carried out compression tests on biscuits and reported that no meaningful information could be extracted from the force-time curves. This was attributed to the constant deformation rate applied by the Instron, so they designed the Constant Loading Rate instrument which recorded deformation-time curves. This method could characterise fracture properties better than could be done with deformation at constant rate, because the deformation-time trace exhibited a discernible fracture point. Romano *et al.*²⁸ evaluated the application of impact tests to various bakery products. They stated that impact tests could provide a promising alternative. However, Stinson and Huck²⁹ found that, for certain products, the results of sensory evaluation did not agree with those of impact tests. Tensile tests are not popular due to difficulties in gripping the sample³. The most frequently used method is the three-point bending test³⁰⁻³⁸. Apart from the work of Brennan *et al.*³⁹ and Vickers and Christensen⁴⁰, in the foregoing studies the results are expressed in force and deformation units, e.g. slope of force-deformation curve (wrongly referred to as Young's modulus), fracture force, etc. This does not allow comparison of results because the force-deformation relationship is strongly affected by specimen dimensions. Mechanical properties, such as Young's modulus, should be independent of specimen size. In addition, fracture properties depend on the size and shape of defects³. This aspect becomes extremely important for foods, such as biscuits, that invariably have an inhomogeneous structure. Therefore, before examining the effect of compositional variables, one has to ascertain effects due to test conditions and effects due to inherent variability of the material.

This Chapter is concerned with developing a procedure for the determination of fracture properties of short-dough biscuits. To identify possible effects due to

differences in air volume fraction of the biscuits specific volumes of test-pieces were also determined.

5.2 Experimental

5.2.1 Materials

Commercial wheat flour was obtained from Meneba Meel BV (Rotterdam, the Netherlands). Granulated sucrose was provided by Suiker Unie (Breda, the Netherlands). Fat was supplied by Bakkerol BV (Rotterdam, the Netherlands). The chemical composition of the flour was given in Chapter 2.

5.2.2 Preparation of biscuits

All ingredients were stored at room temperature. Doughs were prepared in a N-50 Hobart mixer according to the compositions given in Table 5.1. Three mixing speeds are available with this mixer. Mixing was carried out for 8 min at room temperature. Initially fat, sucrose, water and salt were premixed for half a minute at the lowest speed, flour was then added followed by mixing for half a minute at the same speed. The medium speed was then employed for 7 min, during which mixing was stopped twice to scrape the dough from the bowl sides to ensure uniform mixing.

Table 5.1 Dough composition (% w/w)

Ingredient	standard	low-fat
	dough	dough
Flour ^a	39.8	43.4
Fat	24.7	18.0
Sucrose	20.4	22.2
Water	14.4	15.6
Salt	0.7	0.8

^a Dry matter

The dough was rolled to oblong strips (20 cm length × 6 cm width × 0.3 cm thickness) and baked at 160 °C for 20 min in an electric oven. Immediately after baking

the strips were cut into test-pieces by means of blades. The overbaked edges of the strips were discarded. Moreover, then the upper surface of the biscuits was flatter. The dimensions of each biscuit were measured with a micrometer screw. Length and width were, on average, 8 and 4 cm, respectively. Thickness was determined midway across the test-piece length being, on average, 0.48 and 0.54 cm for standard and low-fat biscuits, respectively. The test-pieces were held at room temperature, for about 15 min, to cool down and subsequently placed in a desiccator, under vacuum, over a saturated solution of lithium chloride⁴¹ ($a_w = 0.11$). It was found that a conditioning period of one week, at 20 °C, was sufficient for the biscuits to reach an "equilibrium" water content (about 2%). Every dough batch yielded 16 biscuits.

5.2.3 Determination of fracture properties

Preliminary uniaxial compression experiments were not successful. A clear fracture point (force dropping to low levels) was not evident from the force-deformation trace, possibly because the ratio of thickness to length was very low. Therefore, fracture properties of short-dough biscuits were investigated by three-point bending tests. According to the theory for flexure of straight beams, the concave side is in compression and the convex side is in tension during bending; halfway between them is a plane, neither in compression nor in tension, known as the neutral plane⁴². Maximum strains and stresses are exerted on the middle of the upper and lower surface of the test-piece. Following Young⁴² for a beam of rectangular cross-section, the tensile or compressive stress σ (N m^{-2}), is given by:

$$\sigma(F) = \frac{3FL}{2db^2} \quad (5.1)$$

where F is the force (N), L the distance between supports also known as span length (m), d the width of the test-piece (m) and b the thickness of the test-piece (often referred to as depth) (m). The tensile or compressive strain ϵ (-), can be calculated from:

$$\epsilon(y) = \frac{6by}{L^2} \quad (5.2)$$

where y is the deflection (m). For a rectangular beam, Young's modulus E (N m^{-2}) is equal to:

$$E = \frac{L^3}{4db^3} \left(\frac{dF}{dy} \right)_{y=0} \quad (5.3)$$

Equations (5.1) and (5.3) show that, for a given material, small variations in thickness will result in large variations in force values. This emphasises the need to quote the results in size-independent parameters. The strain rate $\dot{\epsilon}$ (s^{-1}) is given by:

$$\dot{\epsilon} = \frac{d\epsilon}{dt} = \frac{6b}{L^2} \frac{dy}{dt} = \frac{6bv}{L^2} \quad (5.4)$$

where v is the crosshead speed (m s^{-1}).

The previous equations are based on the assumptions that:

- the material is homogeneous and isotropic and has the same modulus of elasticity in tension and compression.
- the beam is straight or nearly so and the cross section is uniform.
- all loads and reactions are perpendicular to the axis of the beam, implying that the maximum strain should be small.
- the stress is linearly related to the strain.
- bending due to the weight of the beam itself is negligible. Assuming that force is uniformly distributed over the entire span, the strain ϵ_w (-) due to the weight of a rectangular test-piece would be:

$$\epsilon_w = \frac{3\rho gL^2}{4Eb} \quad (5.5)$$

where ρ is the density of the test-piece (kg m^{-3}) and g the acceleration due to gravity (9.81 m s^{-2}).

Other factors which must be taken into consideration are associated with orientation and dimensions of specimen and strain rate. Elaborate standards, depending on the material tested, have been reported⁴²⁻⁴⁴. However, the conditions described were intended to be used for plastics and may not be strictly applicable to biscuits.

An Instron (table model 1122) material testing instrument was used with a 20 N load-cell. Tests were carried out at room temperature. The specimen was centred on two parallel perspex supports, a known distance apart, with its long axis at right angles to the supports. The supports were fitted into a base with parallel grooves to facilitate adjustment of the distance between them. The perspex loading nose, parallel to the supports, was lowered at a constant speed and the force, applied on the specimen equidistant from either support, was recorded as a function of time. The supports (6.2 mm in diameter and 11.5 cm long) and the loading nose (6.2 mm in diameter and 6 cm long) had cylindrical surfaces to avoid excessive stress concentration directly near the supports or loading nose. This aspect becomes more important for materials exhibiting low compressive strength⁴⁴. Results shown represent the average of, at least, 7 biscuits. Data were statistically analysed by using Student's t -tests. A typical force-deformation curve for standard biscuits is shown in Figure 5.1. Fracture stress σ_f was calculated from the maximum in force-deformation curves, using equation (5.1). For calculation of the fracture strain ϵ_f the initial "toe region" was ignored as an artefact caused by unevenness of the surface of the specimen and its alignment with the supports/loading nose. A tangent to the initial slope was drawn and the point at which it intersected the deformation axis was taken as the zero point of deformation. ϵ_f was then calculated from the deformation corresponding to σ_f , using equation (5.2). Often, the initial slope was identical to the steepest slope. Therefore, Young's modulus was calculated from the steepest slope of the curve, because it was the most reproducible.

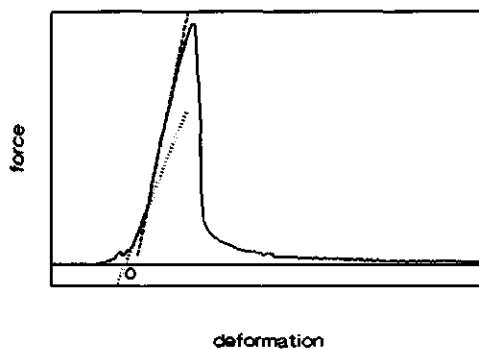


Figure 5.1 Typical force-deformation curve; gradients for assigning zero deformation (····) and for calculation of Young's modulus (----).

5.2.4 Specific volume measurements

Specific volumes of biscuits were determined by the solid displacement technique using glass beads. A container of known weight and volume was filled with glass beads and tapped. Excess glass beads were trimmed off slowly. The specific volume of the glass beads was calculated from the average of three determinations. The bottom of the container was covered with glass beads and a biscuit of measured weight was placed in the container with the flattest surface downwards. The container was filled with glass beads, tapped and the total weight measured. The specific volume v_b ($\text{m}^3 \cdot \text{kg}^{-1}$) of the biscuit was calculated from⁴⁵:

$$v_b = \frac{V - (m_t - m_b) v_g}{m_b} \quad (5.6)$$

where V is the volume of the container (m^3), m_t the weight of the glass beads and the biscuit (kg), m_b the weight of the biscuit (kg) and v_g the specific volume of the glass beads ($\text{m}^3 \cdot \text{kg}^{-1}$). For every biscuit, the mean of two determinations was taken as the specific volume.

5.3 Results and discussion

5.3.1 General characteristics

The curve shown in Figure 5.1 bears resemblance to those described for materials exhibiting brittle fracture. They are characterised by an essentially elastic response and

a small fracture strain⁴⁶. Some curves had a jagged appearance prior to fracture. These small peaks may be attributed to fracture of material between air cells and reflect the highly inhomogeneous structure of the biscuit. Occasionally (not shown), there was a shoulder in the part of the curve just before fracture. Perhaps, this implies slight plastic deformation confined to the vicinity of the crack⁴⁶. In this case a slight error will be introduced when equations (5.1) - (5.4) are applied, yet, they will be valid for comparison purposes⁴⁴. A notable feature of all biscuits was the sound emitted and the concomitant abrupt drop in force upon fracture. This demonstrates that, once sufficient strain energy was stored, the crack propagated spontaneously at a high velocity. For the curve given in Figure 5.1, the shortest time taken for the force to drop to zero would be about 0.14 s due to pen response limitations. The actual time was approximately 0.53 s, possibly due to the compressed parts of the biscuit being hooked into one another.

5.3.2 Effect of span length

Generally, the specimen must be long in proportion to its thickness to avoid shear deformation at the neutral plane. This is particularly so for highly anisotropic (e.g. laminated) materials when the ratio of Young's modulus to shear modulus is high, since otherwise the computed E values are substantially underestimated⁴². On the other hand, if the ratio of span length to thickness is large, excessive deflection may occur, leading to lower calculated maximum stress values^{43,44}. Specimens of relatively great width are observed to be stiffer than equation (5.3) indicates⁴². This implies that the specimen width should be adjusted in relation to specimen thickness. Regardless of the conditions relating to beam theory, the smallest dimension of the test-piece must be large compared to the size of fracture inducing defects³.

In these experiments, the span length was set at either 6 or 4 cm. $\dot{\epsilon}$ was approximately kept constant by setting the crosshead speed at 5 or 10 mm/min for a span length of 4 or 6 cm, respectively. Biscuits were placed on the supports with the upside up. Results obtained with different span lengths are tabulated in Table 5.2. All parameters determined did not vary significantly with L . Furthermore, all biscuits fractured in tension relatively close to the middle region of the biscuit. This suggests that the condition with respect to span length was satisfied. Applying the results of Table 5.2 to equation (5.5) shows that ϵ_w due to the weight of the biscuit was indeed negligible (0.6% of the fracture strain ϵ_f). The validity of the beam theory was also addressed by Bruns and Bourne⁴⁷, who obtained similar results for sugar-snap biscuits, although crosshead speed was not adjusted. Equation (5.3) shows that force is inversely proportional to the cube of the span length. Therefore, to enable measurements to be made well within the sensitivity of the testing machine a span length of 4 cm was chosen for subsequent experiments.

Table 5.2 Influence of span length L on Young's modulus E , fracture stress σ_f and fracture strain ϵ_f of standard biscuits. Specific volumes v_b are also indicated. Measurements made at room temperature, $\dot{\epsilon} \approx 1.5 \times 10^{-3} \text{ s}^{-1}$, biscuits placed on supports with top surface up

Span length (cm)		E (MN·m ⁻²)	σ_f (kN·m ⁻²)	$\epsilon_f \times 10^3$ (-)	$v_b \times 10^3$ (m ³ ·kg ⁻¹)
4	mean	84.1	324	6.32	1.48
	s.e. ^a	5.5	11	0.48	0.05
6	mean	85.3	336	6.64	1.56
	s.e. ^a	5.0	12	0.48	0.05

^a Standard error of the mean

5.3.3 Effect of sample orientation

Two batches of biscuits were tested; one with the top surface up and the other in the inverted position. Crosshead speed was 5 mm/min. Table 5.3 summarises results obtained. It can be seen that fracture stress σ_f and fracture strain ϵ_f were somewhat higher when biscuits were tested upside-down. A highly inhomogeneous structure was observed when the cross-section of a biscuit was visually examined. The slightly curved top showed a thin crust. The flat bottom showed numerous, almost semi-spherical, air cells up to 2 mm in diameter. These air cells will act as defects, and almost like notches when they are at the bottom side. Fracture properties strongly depend on the size of defects³. Large defects may severely decrease the fracture stress and fracture strain of a material. The shape of defects is also important. Long and narrow cracks cause more intense stress concentration than round holes⁴⁶. Moreover, the orientation of the inhomogeneities relative to the direction of the applied stress affects the magnitude of stress amplification. To surmount these effects artificial notches are, sometimes, applied to the test-piece³. However, it would be very difficult, if at all possible, to standardise the dimensions of notches on biscuits due to their brittle character. According to beam theory stresses are higher at the surface of the specimen. This means that, all other factors being identical, outer cracks will be more effective in intensifying stress than inner cracks. Therefore, it was decided not to flatten the hemispherical top side of biscuits by sawing. Doing so would expose the inner porous region. Besides, the error in values calculated from equation (5.1) when the cross-section of the test-pieces is slightly hemispherical, i.e. when the radius of curvature is more than about eight times the depth of the beam⁴², is only about 5%.

For further experiments biscuits were tested with their upper surface facing downwards.

Table 5.3 Influence of biscuit orientation on Young's modulus E , fracture stress σ_f and fracture strain ϵ_f of standard biscuits. Specific volumes v_b are also indicated. Measurements made at room temperature, $v = 5$ mm/min, $L = 4$ cm

Orientation of top surface		E (MN·m ⁻²)	σ_f (kN·m ⁻²)	$\epsilon_f \times 10^3$ (-)	$v_b \times 10^3$ (m ³ ·kg ⁻¹)
up	mean	80.2	338 ^b	6.18 ^c	1.60
	s.e. ^a	5.0	14	0.34	0.03
down	mean	86.1	445 ^b	7.38 ^c	1.61
	s.e. ^a	5.7	20	0.42	0.05

^a Standard error of the mean

^b Means are significantly different ($P < 0.001$)

^c Means are significantly different ($P < 0.05$)

Gormley⁴⁸ carried out three-point bending tests on crackers. He noticed lower fracture force values when crackers were tested upside-down; the effect was accounted for by the upper surface being overbaked. This explanation would hold if crackers fractured in compression. However, the imprinted lettering on the upper side of the crackers may have caused stress concentration and this may have been more important for the effects described.

5.3.4 Effect of strain rate

The effect of strain rate on E is expected to be negligible for almost ideally elastic materials, particularly at high compression speeds. However, fracture characteristics of concentrated starch gels, which were ideal-elastic at small deformation, were strain-rate dependent³. Therefore, it was decided to also apply a higher deformation rate. Crosshead speed was set at 50 mm/min. Results are presented in Table 5.4 together with those from the previous section. In terms of variability, the results at the lowest crosshead speed were more satisfactory. It appears that, in the range investigated, strain rate influenced the fracture strain ϵ_f . This is possibly due to local permanent deformation occurring at low strain rates. However, it should be realised that precise determination of fracture strain from the chart paper was not feasible particularly when

curves exhibited multiple peaks prior to fracture. In view of the small fracture strain this difficulty was greater at higher crosshead speeds.

Table 5.4 Influence of strain rate $\dot{\epsilon}$ on Young's modulus E , fracture stress σ_f and fracture strain ϵ_f of standard biscuits. Specific volumes v_b are also indicated. Measurements made at room temperature, $L = 4$ cm, biscuits placed on supports with top surface down

$\dot{\epsilon}$ (s ⁻¹)		E (MN·m ⁻²)	σ_f (kN·m ⁻²)	$\epsilon_f \times 10^3$ (-)	$v_b \times 10^3$ (m ³ ·kg ⁻¹)
1.5×10^{-3}	mean	86.1	445	7.38 ^b	1.61
	s.e. ^a	5.7	20	0.42	0.05
1.5×10^{-2}	mean	87.7	412	5.21 ^b	1.53
	s.e. ^a	11.9	45	0.55	0.06

^a Standard error of the mean

^b Means are significantly different ($P < 0.01$)

Vickers and Christensen⁴⁰ noticed that crosshead speed had minimal effects on deformation at fracture and peak force for ginger-snap biscuits. They also found that deformation rate influenced Young's modulus but no consistent pattern was observed. However, since E values differed by less than 25% and given that standard deviations were not reported, it is not clear whether or not the observed effect was significant. Boyd and Sherman⁴⁹ found that the effect of crosshead speed for biscuits was insignificant as studied by compression tests. They also observed that the response time of the recording device imposed limitations upon using high crosshead speeds. In a subsequent paper, Sherman and Deghaidy⁵⁰ employed three-point bending tests in conjunction with a fast response recorder. They studied the effect of crosshead speed in the range from 5 to 1000 mm/min for a number of selected commodities, among them various commercial biscuits. They found that the maximum force and the deformation at fracture depended on the type of biscuit concerned and the crosshead speed. Differences between the fracture force for different samples were minimal at the highest and maximal at the lowest crosshead speed. The opposite was found for the deformation at fracture. However, the results were not corrected for sample dimensions. As can be seen from equation (5.4) $\dot{\epsilon}$ is inversely proportional to the square of the span length for a given thickness and crosshead speed. Consequently, different span lengths cause different $\dot{\epsilon}$.

5.3.5 Applicability of method

As a means of testing the applicability of the method to short-dough biscuits of various composition, a low-fat type was also studied. Results are presented in Table 5.5.

Table 5.5 Influence of fat content on Young's modulus E , fracture stress σ_f and fracture strain ϵ_f . Specific volumes v_b are also indicated. Measurements made at room temperature, $v = 5$ mm/min, $L = 4$ cm, biscuits placed on supports with top surface down

Type of biscuit		E (MN·m ⁻²)	σ_f (kN·m ⁻²)	$\epsilon_f \times 10^3$ (-)	$v_b \times 10^3$ (m ³ ·kg ⁻¹)
standard	mean	86.1	445 ^b	7.38	1.61 ^c
	s.e. ^a	5.7	20	0.42	0.05
low-fat	mean	92.0	624 ^b	7.97	1.46 ^c
	s.e. ^a	8.0	24	0.55	0.03

^a Standard error of the mean

^b Means are significantly different ($P < 0.001$)

^c Means are significantly different ($P < 0.05$)

The fracture stress of the low-fat biscuits was higher compared to the standard ones. More extensive results on effects of composition will be given in the following Chapter. Presumably, the method is useful for determining differences among biscuits of various composition.

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Chapter 6

Fracture Properties of Short-dough Biscuits: Effect of Composition

Abstract

Mechanical properties of short-dough biscuits of various composition were determined in three-point bending tests. In general, reduction of fat content increased the modulus and the fracture stress of biscuits. The relative magnitude of this effect depended on fat type. Sucrose syrup slightly enhanced the brittle character of biscuits, compared to crystalline sucrose, indicating that sucrose crystals were not the fracture inducing defects. Substituting starch for part of the flour did not practically affect the mechanical properties. Sugar-free biscuits had a significantly lower modulus and fracture stress. Differential scanning calorimetry showed that, irrespective of composition, starch gelatinisation was slight, if not absent, presumably due to the limited water content coupled with the low baking temperature. It is concluded that biscuits are composed of a glassy matrix; their mechanical properties are mainly determined by air and fat volume fraction as well as the size of inhomogeneities.

6.1 Introduction

Nowadays, automation and mechanisation is a primary goal for the baking industries. As a corollary, the need for deeper understanding of variables that influence the characteristics of the finished product has increased. For instance, control of biscuit thickness and density are prevalent problems germane to commercial packaging of short-dough biscuits¹.

Presently, consumers attention is directed towards low-calorie products owing to increasing emphasis on health issues. Innovation of new products and exploitation of alternative raw materials to reduce production costs are important aspects for biscuit manufacturers. Consequently, knowledge of the function of the ingredients in question is critical in order to manipulate the palatability of the product.

Short-dough biscuits widely vary in composition. For example, fat content ranges between 20 and 60% and sucrose content between 25 and 55% (expressed as percentage of weight of flour)². This gives rise to products with distinctive properties as evaluated by instrumental methods³⁻⁶. In the afore-mentioned studies not only did processing conditions vary but also composition differed in more than one ingredient; therefore, a cause-and-effect relationship cannot be established.

Mechanical properties of biscuits are salient quality attributes as they have a direct impact on sensory perception and thereby important implications for consumer acceptance of such products. Despite that, it appears that research on the effect of compositional factors is rather scarce and fragmentary; the role of basic ingredients is still not adequately understood.

Curley and Hosoney⁷ carried out three-point bending tests to investigate the effect of corn sweeteners on sugar-snap biscuits. They reported that deformation to fracture decreased between one and three days after baking, beyond which no change was observed. When sucrose was partly replaced by high-fructose corn syrup, deformation to fracture was higher, the more so for a higher level of substitution. Moreover, at high levels of corn syrup, deformation to fracture increased with time after baking. These effects were attributed to sucrose recrystallisation upon storage and its inhibition by fructose and glucose. Similar conclusions were drawn by Arndt and Wehling⁸. Gaines *et al.*^{6,9} employed puncture tests on various biscuits and observed that fracture force increased with increasing protein content of the flour. From fractionation and reconstitution experiments, these authors concluded that the fractions which contributed to product hardness were, in decreasing order, water-solubles, tailings and gluten. It was postulated that the effect is related to the amount of water that these fractions absorb, which followed the same order. Bullock *et al.*¹⁰ found that, when sucrose was replaced by polydextrose, the resulting biscuits (either fresh or after one week's storage) required a smaller force to break as measured by three-point bending tests. Regardless of biscuit type, fracture force increased with storage time. On the other hand, Campbell *et al.*¹¹ reported that polydextrose had no significant effect on the mechanical properties of biscuits as measured by puncture tests. Hutchinson *et al.*¹² performed "Kramer shear tests" to evaluate the effect of emulsifiers on sugar-snap biscuits. The results depended on the type of emulsifier, the amount used and the storage time. Similar findings were reported by other researchers¹³⁻¹⁵. Burt and Thacker¹⁵ also found that decreasing fat content increased the hardness of short-dough biscuits, as measured by the Biscuit Texture Meter. Wade¹⁶ studied the effect of fat type on short-dough biscuits by using the Biscuit Texture Meter. Biscuits made with butter fat were harder than those made with either commercial fat or lard. However, the findings of Hornstein *et al.*¹⁷, who examined shortbread biscuits by three-point bending tests, suggest that, for a given fat type, the physical properties of the fat may affect the results.

In some of the studies cited above mechanical properties were determined in an empirical manner. In others, more fundamental methods were employed, but the results were not expressed in size-independent parameters. Whatever the approach, interpretation of the results is far from easy. This is especially so for biscuits which contain large inhomogeneities that strongly influence fracture properties. A procedure for the determination of fracture properties was described in the previous Chapter. The

objective of this Chapter is to determine mechanical properties of short-dough biscuits in relation to composition. The physical structure of short-dough biscuits will be the subject of the following Chapter.

6.2 Experimental

6.2.1 Materials

Commercial wheat flour was obtained from Meneba Meel BV (Rotterdam, the Netherlands) and native wheat starch (water content 12.32%) from Latenstein Zetmeel BV (Nijmegen, the Netherlands). The chemical composition of the flour was given in Chapter 2. Granulated sucrose was provided by Suiker Unie (Breda, the Netherlands). Three fat types were used: a standard fat for short doughs, a firmer one and sunflower oil. Fats were supplied by Bakkerol BV (Rotterdam, the Netherlands) and commercial sunflower oil was purchased from a local retailer.

6.2.2 Preparation of biscuits

Two sets of experiments were carried out. In the first set, biscuits of various fat types and fat contents were studied. Their composition is presented in Table 6.1.

Table 6.1 Composition of short-dough biscuits of various fat type and fat content. For calculation of % starch and % gluten the amount of dry flour should be multiplied by 0.83 and 0.14, respectively

Fat type	w/w %				
	flour ^a	fat	sucrose	water	salt
standard ^b	40.3	37.0	20.6	1.3	0.8
	45.8	28.4	23.4	1.5	0.9
	50.5	20.9	25.9	1.8	0.9
	53.1	16.4	27.1	2.4	1.0
firm	45.9	28.5	23.4	1.3	0.9
	50.5	20.9	25.8	1.9	0.9
liquid oil	50.2	20.5	25.7	2.6	1.0
	52.6	16.3	26.9	3.2	1.0

^a Dry matter

^b Reference biscuits were those with 28.4% standard fat

The term reference biscuits will be used throughout to identify biscuits with 28.4% standard fat. In the second set, biscuits with variation in composition other than fat type were studied. These biscuits contained standard fat. Table 6.2 gives their composition. A short dough composition typical of commercial operations was used as the reference material. The compositions of the other doughs were chosen in such a way that, when omitting an ingredient or reducing its amount, the weight ratios of the other components were constant. All ingredients were stored at room temperature.

Table 6.2 Short-dough biscuits with variation in composition other than fat type (standard fat). For calculation of % starch and % gluten the amount of dry flour should be multiplied by 0.83 and 0.14, respectively

Biscuit type	w/w %					
	flour ^a	starch ^a	fat	sucrose	water	salt
sucrose-syrup	48.1	-	29.8	19.7	1.5	0.9
sugar-free	59.7	-	37.0	-	2.2	1.1
flour/starch	32.0	13.7	28.3	23.3	1.8	0.9

^a Dry matter

Doughs were prepared in a D-300 Hobart mixer. Mixing was carried out for 8 min at room temperature according to the procedure described in Chapter 5, whereas the method of dough piece formation was modified as follows: Instead of a wooden roller, a bench-scale depositing machine was used. The dough was extruded through a die as a continuous oblong strip. The strip could yield 4 biscuits and, to ensure uniform heat treatment, it was cut to pieces (10 cm length x 6 cm width x 0.3 cm thickness) which were subsequently baked at 160 °C for 20 min in a small-scale electric oven. Since the method of dough piece formation, the size of the dough pieces and the oven used were different, results presented in the next section may not be strictly comparable, in terms of absolute values, to those reported in Chapter 5; the use of different oven types may affect biscuit sensory characteristics¹⁸. Test-pieces were placed in a desiccator, under vacuum, over a saturated solution of lithium chloride¹⁹ ($a_w = 0.11$) and stored for one week at 20 °C. Water content of the biscuits was determined in triplicate after conditioning by a Netherlands Standardisation Institute (NNI) method²⁰; values reported represent averages of these replicates.

6.2.3 Differential scanning calorimetry

Differential scanning calorimetry was carried out using a TA Instruments DSC 2910 calorimeter. Samples were heated from 10 °C to 140 °C at a rate of 10 °C/min in Perkin-Elmer stainless steel hermetically sealed pans. Calibration of the calorimeter was previously done with indium. An empty pan was used as a reference. Reference and sugar-free biscuits were treated identically to those used for bending tests. In addition, samples of the same biscuits were mixed with excess distilled water (20% biscuit, 80% water) and allowed to stand for 1 hour at room temperature in sealed pans prior to heating.

6.2.4 Determination of fracture properties

Fracture properties of biscuits were studied by three-point bending tests; they were carried out at room temperature. An Instron (table model 1122) materials testing instrument was used with a 20 or 1000 N load-cell. Span length was 4 cm and compression speed was set at 5 mm/min; biscuits were placed on supports with their top surface down. Justification for this experimental set-up was discussed in the previous Chapter. At least 8 biscuits were tested for each batch. Where applicable, results were subjected to one-way analysis of variance²¹ (*i.e.* effects of fat type and fat content were treated separately). Differences in means were evaluated by the least significant difference (LSD) or by Student's *t*-tests. Force-time values were recorded through a Daqbook/100 data acquisition system. Young's modulus E (N m^{-2}), fracture stress σ_f (N m^{-2}) and fracture strain ϵ_f (-) were calculated as shown in Chapter 5.

6.2.5 Density measurements

Bulk densities of doughs and biscuits were determined according to the methods described in Chapters 2 and 5, respectively. Air volume fraction ϕ_{void} was estimated from the solid densities of biscuits as shown in Chapter 2. Fat volume fraction ϕ_{fat} was derived from²²

$$\phi_{\text{fat}} = \frac{m_f \rho}{\rho_f} \quad (6.1)$$

where m_f is the mass fraction of the fat, ρ the bulk density of the biscuit (kg m^{-3}) and ρ_f the density of the fat, *i.e.* 900 kg m^{-3} .

6.3 Results

6.3.1 DSC measurements

DSC experiments were performed in order to evaluate whether starch gelatinisation occurred during baking. Figure 6.1 depicts thermograms for the reference biscuits. Only one endotherm, corresponding to fat melting, was observed. Upon addition of water, a second endotherm occurred; it may be assigned to starch gelatinisation for the following reasons:

- first, it cannot be due to starch retrogradation during storage by virtue of the water content of biscuits being very low²³.
- second, it cannot be due to sucrose melting because, even if the water uptake by the other hydrophilic components (starch, gluten, pentosans) is taken into account, the ratio of water to sucrose is very high.

The finding that starch would be gelatinised to a limited extent, if at all, is in line with published studies on biscuits²⁴⁻²⁷. Values reported in the literature for the amount of starch gelatinised are 4% and 11%, as determined enzymatically^{28,29}. It is well established that sucrose raises the gelatinisation temperature of starch³⁰⁻⁴². Therefore, sugar-free biscuits were tested to check whether or not sucrose inhibited starch gelatinisation.

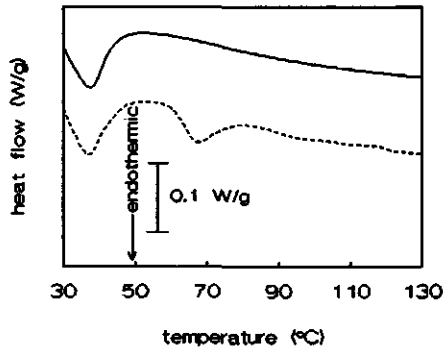


Figure 6.1 DSC thermograms of: (solid line) reference biscuits; (dashed line) same biscuits with excess water (see text).

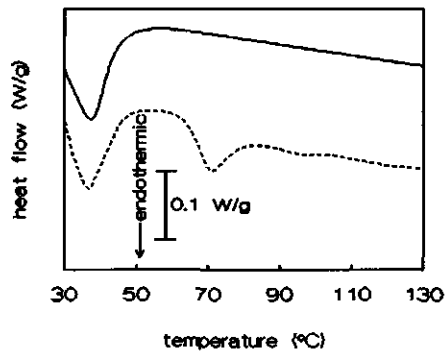


Figure 6.2 DSC thermograms of: (solid line) sugar-free biscuits; (dashed line) same biscuits with excess water (see text).

Figure 6.2 illustrates thermograms of sugar-free biscuits with and without added water. Apparently, it was the deficiency of water in the corresponding doughs in conjunction with a relatively low baking temperature that depressed, if not prevented, starch gelatinisation^{43,44}.

6.3.2 Mechanical properties

6.3.2.1 Effect of fat type and fat content

Preliminary measurements on reference biscuits (results not shown) were quite reproducible, as far as batch to batch variation is concerned; regardless of the parameter determined, means of different batches were not significantly different at the 0.05 level.

The effects of fat type and fat content are shown in Table 6.3. Biscuits with firm fat at low fat contents, and biscuits with liquid oil at high fat contents, could not be prepared, because the corresponding doughs were either too stiff or too soft to be deposited. Reduction of fat content increased the modulus of biscuits made with standard fat. This was accompanied by a rather similar increase in fracture stress. A notable exception was observed at the lowest fat content. Comparison of these biscuits to those with 20.9% fat content shows similar modulus values. However, a twofold increase in both fracture stress and fracture strain was observed.

The moduli and the fracture stress of biscuits made with either firm fat or liquid oil also increased with decreasing fat content though the relative magnitude of the effect was different for each fat type.

Table 6.3 Influence of fat content and fat type on Young's modulus E , fracture stress σ_f and fracture strain ϵ_f . Air volume fraction ϕ_{void} and fat volume fraction ϕ_{fat} are also indicated

Fat type	fat w/w %	ϕ_{fat} %		E (MN·m ⁻²)	σ_f (kN·m ⁻²)	$\epsilon_f \times 10^3$ (-)	ϕ_{void} %
standard	37.0	18.4	mean	33	151	6.2 ^c	63 ^c
			s.e. ^a	1.8	3.5	0.3	0.8
	28.4 ^b	15.0	mean	47	240	6.5 ^c	62 ^{c,d}
			s.e. ^a	2.9	10.1	0.5	0.8
	20.9	16.2	mean	97 ^{c,d}	446	5.8 ^c	47 ^e
			s.e. ^a	2.4	8.7	0.3	0.9
16.4	13.2	mean	100 ^c	807	11.6	46 ^c	
		s.e. ^a	8.0	33.1	1.1	1.1	
firm	28.5	15.4	mean	65	404	9.0 ^d	62 ^d
			s.e. ^a	5.2	18.9	0.8	0.6
	20.9	19.3	mean	92 ^d	671	8.3 ^{d,e}	36
			s.e. ^a	5.4	22.0	0.4	1.2
liquid oil	20.5	13.1	mean	125	829	8.6 ^{e,f}	56
			s.e. ^a	4.1	23.4	0.3	0.9
	16.3	11.7	mean	149	961	7.9 ^f	52
			s.e. ^a	5.3	35.9	0.3	1.0

^a Standard error of the mean^b Reference biscuits^{c-f} For a given fat content or fat type, pairs of means, within each column, with the same letter are not significantly different at the 0.05 level

As far as ϕ_{void} is concerned the emerging trend is towards a decrease with decreasing fat content. Although at high fat contents absolute values of ϕ_{void} may vary (reasons for this will be given in the following Chapter), this tendency is by no means altered. An interesting point is that, at a given fat content, for instance 20.9%, biscuits with liquid oil had the highest ϕ_{void} . The densities of the corresponding doughs at this fat content were 1135, 1165 and 1186 kg·m⁻³ for the doughs with standard, firm fat and liquid oil, respectively. This shows that there is no direct relationship between dough and biscuit air volume fraction. It may be partly related to the rheological properties of the corresponding doughs in impeding gas bubble expansion during baking. At 20 °C apparent dough viscosities followed the order: dough with firm fat > dough with standard fat > dough with liquid oil (Chapters 2 and 3). If this notion is correct, and

since the rate of water loss during baking was comparable (results not shown), then the solid fat content and the fat distribution may come into play.

6.3.2.2 Effect of variation in composition other than fat type and fat content

It has been advocated that the proportion of sucrose that is in the crystalline state and the size of the sucrose crystals affect its rate and/or extent of dissolution in water; depending on the temperature-water loss profile during baking this may influence the ratio of glassy to crystalline sucrose in the baked product²³. This, in turn, would affect the partition of water between other hydrophilic components because glassy sucrose has a higher affinity for water than the crystalline form^{45,46}. Even if water distribution remains unchanged the ratio of glassy sucrose to water may exert an influence on the mechanical properties of the product. In addition, the presence of crystals may alter the importance of other inhomogeneities. For instance, considering sucrose crystals as large as 0.5 mm, the film between gas cells may be expected to become thinner than crystal size at high ϕ_{void} ; this would mean that the film is inhomogeneous, which will affect its fracture stress. Therefore, the effect of predissolving the sucrose was studied.

Table 6.4 Influence of various ingredients other than fat type (standard fat) on Young's modulus E , fracture stress σ_f and fracture strain ϵ_f . Air volume fraction ϕ_{void} and fat volume fraction ϕ_{fat} are also indicated

Biscuit type	fat w/w %	ϕ_{fat} %		E (MN m ⁻²)	σ_f (kN m ⁻²)	$\epsilon_f \times 10^3$ (-)	ϕ_{void} %
sucrose-syrup	29.8	16.0	mean	65	271 ^b	5.4	61 ^b
			s.e. ^a	5.2	20.8	0.2	0.7
sugar-free	37.0	22.0	mean	16	71	6.5 ^b	54
			s.e. ^a	1.1	1.9	0.4	1.7
flour/starch	28.3	14.6	mean	41 ^b	191	8.0 ^b	63 ^b
			s.e. ^a	3.9	10.5	0.8	0.7

^a Standard error of the mean

^b Compared to biscuits with same fat type and similar fat contents (Table 6.3), means are not significantly different at the 0.05 level

Compared to the reference biscuits, the composition of sucrose-syrup biscuits was somewhat different (Tables 6.1 and 6.2) because the sucrose content in the reference dough exceeded the saturation limit at 20 °C. It should be mentioned that a few

sucrose crystals were clearly visible in all biscuits, but essentially absent in sucrose-syrup ones. Results are presented in Table 6.4. Compared to the reference biscuits, the sucrose-syrup biscuits had a somewhat higher modulus and a slightly smaller fracture strain, suggesting that the latter biscuits had an enhanced brittle character. This indicates that sucrose crystals were not the fracture inducing defects.

Omitting sucrose from the recipe markedly influenced the modulus and the fracture stress which were lower than those for biscuits of the same fat content by a factor of 2 (Tables 6.3 and 6.4). This indicates that sucrose is an important component of the non-fat phase. Figure 6.3 shows that the stress-strain curves for the sugar-free biscuits did not have the typical spike-like shape but instead bent towards the strain axis prior to fracture. This means that, in comparison with all other biscuits, more energy was dissipated due to plastic deformation. Moreover, once fracture stress was reached, the subsequent drop in stress was not as sharp as for all other biscuits, which implies that cracks propagated at a smaller velocity.

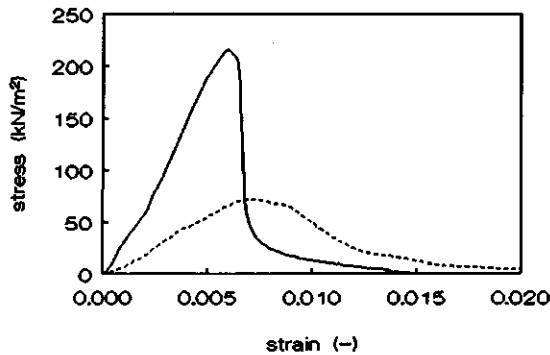


Figure 6.3 Stress-strain curves of: (solid line) reference biscuits; (dashed line) sugar-free biscuits.

To quantify possible effects of gluten, biscuits (28.4% fat content) were baked in which flour was replaced by starch. The amount of starch was such that the ratio of starch to water in the dough was constant. The attempt was unsuccessful; biscuits "spread" extensively during baking and had a very inhomogeneous structure. In fact, air cells extended throughout the whole thickness of the biscuit (like an open honeycomb). Therefore, it was decided to replace part (30%) of the flour by starch (Table 6.2). Results in Table 6.4 for these biscuits and comparison with those for reference biscuits indicate a slightly lower fracture stress, possibly due to stronger local permanent deformation as a result of a somewhat different fat distribution, whereas the other parameters remained nearly unaffected.

6.4 Discussion

The DSC results described earlier (Figs. 6.1 and 6.2) show that starch would be hardly gelatinised, regardless of the biscuit type. This means that the continuous non-fat phase in a sugar-free biscuit will be composed of gluten. It has been reported that absorption of water vapour at an a_w between 0.8 and 0.9, at 27 °C, caused considerable caking in flour and gluten, but not in starch⁴⁷. From their sorption isotherm of flour, water content would be about 16% at an a_w of 0.85. Similar observations were made by Pratap *et al.*⁴⁸ at 20 and 30 °C. In that case the water contents of flour at an a_w of 0.85 were calculated at about 19% and 17%, respectively. These findings, together with the absence of caking in starch, strongly suggest that gluten in flour underwent a glass transition on water uptake. Indeed, the above calculated values agree with results of Doescher *et al.*⁴⁹, who reported glass transition temperatures T_g of gluten in flour as a function of flour water content for different wheat cultivars. Considering that sugar-free biscuits were stored at an a_w of 0.11 (or their water content for that matter; Table 6.2), in combination with the results of Doescher *et al.*⁴⁹, T_g of gluten would be > 96 °C, *i.e.* gluten would be in a glassy state. One may express reservations as to the tenability of the above premise, because biscuits are not only subject to heat treatment but also contain fat. However, fat was found to have a negligible effect on T_g of gluten⁵⁰. Several researchers have published glass transition temperatures T_g as a function of water content for native gluten⁵¹⁻⁵³. These slightly vary depending on method of determination, wheat cultivar and residual starch and water contents. Nevertheless, they agree reasonably well with results of Nicholls *et al.*⁵⁴ on heat-set gluten. On the other hand, Nicholls and Davies⁵⁵ found that increasing the temperature of heat setting from 90 °C to 150 °C lowered T_g of gluten by 5 to 8 °C possibly due to thermal degradation. At 150 °C, an endothermic transition was observed⁵⁶ by DSC. It is unlikely that biscuits reached a temperature as high as 150 °C during baking, but if so, it would be even more unlikely for T_g of gluten to drop to room temperature.

As for the biscuits containing sucrose, there is a lack of information with respect to T_g of gluten-sucrose mixtures. Cherian *et al.*⁵⁷ noted a broader transition in the presence of sucrose (about 10% water content). Kalichevsky *et al.*⁵¹ found that, in general, at low water contents (about 10%), sugars had little plasticising effect on gluten, though the effect increased with increasing sugar content. This was probably due to incomplete miscibility, as observed by DMTA (dynamic mechanical thermal analysis) and shown by a broadening of the transition region, especially at low sugar contents. At higher water contents (about 20%) incompatibility was more pronounced; sugars increased T_g of gluten and this was possibly caused by water redistribution in favour of sugar or by increased hydrophobic bonding in gluten. It should be mentioned that interpretation of results depends on whether comparison between ternary and

binary systems is drawn at constant water content or constant weight ratio of gluten to water. For instance, in the latter case, the results of Kalichevsky *et al.*⁵¹ at low water and high sugar contents would signify absence of plasticising, if not antiplasticising, effects. Slade and Levine⁵⁸ assert that sugars act as co-solvents with water at high water contents, whereas the polymer acts as the sole solute and its T_g is elevated in the presence of sugars at the same weight ratio of polymer to water, *i.e.* sugars would behave as antiplasticisers. In contrast, sugars act as co-solutes with the polymer in low-water systems, whereas water acts as the sole solvent and the polymer's T_g is depressed in the presence of sugars at the same weight ratio of polymer to water, *i.e.* sugars would behave as plasticisers. Nevertheless, the above studies involved gluten-sucrose ratios much higher than those relevant to biscuits. Since starch is not gelatinised, four possibilities exist for the continuous non-fat phase:

- sucrose in the rubbery state
- gluten in the rubbery state
- sucrose in the glassy state
- gluten in the glassy state

The first possibility is ruled out, because in that case one would expect a strain-rate dependent modulus. Such a dependence was not found (Chapter 5). For the second possibility one cannot rely on the strain-rate independence of the modulus, because gluten may be in the rubbery plateau region. However, biscuits would then exhibit ductile fracture, *i.e.* a prominent yield point would precede fracture stress. The stress-strain curves pointed to little and no permanent deformation, at high and low fat contents, respectively. Likewise, the stress dropped abruptly to low levels on fracture, the more so for a lower fat content. In all cases the broken pieces could be fitted together to form the original biscuit. It may be concluded that, irrespective of biscuit composition, the matrix (be it gluten, sucrose or both) would be in a glassy state.

A biscuit may be considered as a cellular solid *i.e.* as a structure of connected beams or plates. A model to describe the mechanical properties of such materials has been proposed by Gibson and Ashby⁵⁹. For an isotropic cellular solid of uniform size of gas cells and thickness and density of cell wall material, linear elasticity is controlled by cell wall bending at uniaxial compression. Following the theory for a beam loaded at its midpoint Gibson and Ashby⁵⁹ give the following expression for an open-cell foam

$$\frac{E}{E_w} = C_1 \left(\frac{\rho}{\rho_w} \right)^2 = C_1 (1 - \phi_{\text{void}})^2 \quad (6.2)$$

and for a closed-cell foam

$$\frac{E}{E_w} = C_2 \left(\frac{\rho}{\rho_w} \right)^3 = C_2 (1 - \phi_{\text{void}})^3 \quad (6.3)$$

where E , E_w are Young's moduli of foam and cell-wall material, respectively ($\text{N}\cdot\text{m}^{-2}$), ρ , ρ_w their corresponding densities ($\text{kg}\cdot\text{m}^{-3}$) and C_1 , C_2 dimensionless constants, with values near unity, that depend on cell shape. This model is theoretically applicable to

materials with low relative densities; at $\rho/\rho_w > 0.3$ the beams are too short and bulky to bend them, implying that shear and axial deformations become important; at $\rho/\rho_w > 0.8$ the material can be thought of as a solid containing small holes. In spite of these limitations, experimental data for foamed ceramics, metals and plastics followed the model over a wide range of relative densities. This model was also applied to foamed foods⁶⁰⁻⁶³.

Another model was proposed by Kerner⁶⁴. For an isotropic and macroscopically homogeneous composite consisting of homogeneous and isotropic filler particles which are firmly attached to and randomly dispersed in a homogeneous and isotropic matrix, the relation would be

$$\frac{G_c}{G_m} = \frac{\frac{\phi G_f}{(7 - 5\mu) G_m + (8 - 10\mu) G_f} + \frac{(1 - \phi)}{15(1 - \mu)}}{\frac{\phi G_m}{(7 - 5\mu) G_m + (8 - 10\mu) G_f} + \frac{(1 - \phi)}{15(1 - \mu)}} \quad (6.4)$$

where G_c , G_m , G_f are the shear moduli of composite, matrix and filler, respectively ($\text{N}\cdot\text{m}^{-2}$), μ is the matrix Poisson ratio and ϕ is the filler volume fraction. Young's modulus of the composite can be calculated if Poisson ratios of the matrix and the filler are known, so that the corresponding shear moduli can be converted to Young's moduli. The model applies to spherical inclusions of approximately the same size at values of ϕ not exceeding 0.4.

In applying the above models to the results obtained for biscuits, the latter may be conceived as a gluten or sucrose glassy matrix, if not both, in which ungelatinised starch granules, fat crystals and globules, sucrose crystals and gas bubbles are embedded.

Ollett *et al.*⁶⁵ have demonstrated that glucose has a plasticising effect on mixtures with gelatinised starch as measured by three-point bending tests. Kalichevsky *et al.*⁶⁶ studied mixtures of amylopectin with various sugars by DMTA. At high water contents (about 20%) and low ratios of amylopectin to sugar (2:1) they found some degree of phase separation; at low water contents (about 10%) and high ratios of amylopectin to sugar (10:1) plasticising occurred. Since native starch has a higher T_g than gelatinised one⁶⁷, the results of the above studies, together with the low water content of the biscuits (see Tables 6.1 and 6.2) suggest that starch granules would be a glassy filler.

Published values of Young's modulus for a gluten glass vary^{51,54,55,68}; for native starch they are not available. Nevertheless, amorphous materials are generally characterised as glassy⁶⁵ if the modulus is of the order of $1 \text{ GN}\cdot\text{m}^{-2}$. It has been reported⁶⁹ that the densities of crystalline and glassy sucrose are 1588.4 and $1507.7 \text{ kg}\cdot\text{m}^{-3}$, respectively. Because of this small difference, similar modulus values may be expected for crystalline and glassy sucrose. Poisson ratios of the matrix (be it gluten or sucrose glass) are not known. However, as can be seen from equation (6.4),

calculated modulus values are insensitive to small variations in Poisson ratio (e.g. between 0.3 and 0.5); a value of 0.5 was used.

Young's moduli of the fats are estimated to be of the order of $1 \text{ MN}\cdot\text{m}^{-2}$, assuming a Poisson ratio of 0.5 (Chapter 2). Since the modulus of fat is significantly smaller than that of the matrix material, ϕ_{void} was replaced by the sum of ϕ_{void} and ϕ_{fat} in applying equations 6.2 - 6.3. This would approximate the contribution of fat to the modulus of the biscuit. Similarly, in equation 6.4, ϕ was replaced by the sum of ϕ_{void} and ϕ_{fat} and G_f was assumed to be zero.

Modulus values calculated according to equations 6.2 - 6.4 together with the experimental data are plotted in Figure 6.4. As can be seen, the curve for Kerner's equation is higher than the results for biscuits. As discussed above, the model applies to low filler volume fraction and for closed-cell foams. Although the biscuits did not have uniform gas cell size, the results were better represented by the model of Gibson and Ashby, falling almost all between the curves for open- and closed-cell foams. This is in agreement with results of Hiçşasmaz and Clayton⁷⁰ who characterised the pore structure of biscuits and found both open and closed cells.

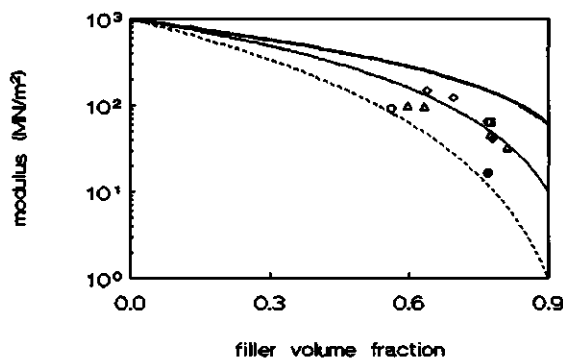


Figure 6.4 Modulus values of various biscuits as a function of filler volume fraction: (Δ) standard-fat biscuits; (\circ) firm-fat biscuits; (\diamond) liquid-oil biscuits; (\square) sucrose-syrup biscuits; (\bullet) sugar-free biscuits; (\blacklozenge) flour/starch biscuits. Lines represent theoretical values calculated according to: (heavy solid) Kerner; (solid) Gibson and Ashby open-cell foam; (dashed) Gibson and Ashby closed-cell foam (see text).

In short, it appears that the effect of compositional factors on the modulus can be rationalised on the basis of ϕ_{void} , ϕ_{fat} and the geometrical characteristics of the foam (open- or closed-cell). It is possible that, at high fat contents, where plastic deformation was stronger as seen from the stress-strain curves, fat forms a partly continuous phase. This will be discussed further in the following Chapter.

The results for the biscuits made with standard fat (see Table 6.3) show an increase in moduli (excluding the lowest fat content) and almost the same increase in fracture stress, with decreasing fat content. Since fracture was essentially elastic and fracture strain remained practically constant, a higher fracture stress seems to ensue from a higher modulus. In other words, it appears that there was no change in size of inhomogeneities. In contrast, biscuits with 16.4% fat content had similar moduli, yet significantly higher fracture stress and fracture strain than those with 20.9% fat content. Defects in a material may cause both stress and strain concentration as a result of which both fracture stress and fracture strain are strongly decreased⁷¹. For biscuits, which exhibit almost brittle fracture, the relative decrease in stress would be the same as the relative decrease in strain⁷². In an ideally elastic isotropic material fracture propagates if half the length of an internal crack or the length of a crack proceeding inwards from a surface is equal to⁷³

$$l_c = \frac{2 R_s E}{\pi \sigma_f^2} \quad (6.5)$$

where l_c is the critical crack length (m), and R_s the specific fracture energy (J m^{-2}). In view of the comparable ϕ_{void} and ϕ_{fat} , R_s may be considered as being virtually invariant; therefore, the higher fracture stress and fracture strain of the biscuits with the lowest fat content is a clear manifestation of a different scale of inhomogeneities (gas cells).

At the same fat content the effect of fat type on fracture stress and fracture strain can be explained by a changed modulus (*e.g.* compare biscuits with firm fat and liquid oil at 20.9% fat content), a changed scale of inhomogeneities (*e.g.* compare biscuits with standard and firm fat at 20.9% fat content) or a change in both of the above (*e.g.* compare biscuits with standard and firm fat at 28.5% fat content).

Overall, the mechanical properties of biscuits are governed by

- ϕ_{void} , ϕ_{fat} and geometrical characteristics of the cellular materials
- size of inhomogeneities

The former influence the modulus which, in turn, together with the latter influence the fracture stress and fracture strain. However, there are other factors, such as the state of the matrix (glassy or rubbery) and fat continuity, that come into play. These will be discussed in the ensuing Chapter.

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Chapter 7

Properties of Short-dough Biscuits in Relation to Structure

Abstract

Mechanical properties of short-dough biscuits of various composition were determined in three-point bending tests. Temperature during dough preparation and dough water content affected air volume fraction and mechanical properties to an extent which depended on fat content. These results suggest that air volume fraction of the biscuits is likely to be related to the rheological properties of the doughs. Measuring temperature affected the mechanical properties of biscuits, the more so for a higher fat content. Diffusion of Sudan III into the biscuits also depended on fat content. Mechanical properties were markedly influenced by water content; as a result of the plasticising effect of water, the matrix underwent a glass transition. The effect of sugar type on mechanical properties depended on the type of biscuit. It is concluded that low-fat biscuits are fat-dispersed systems and high-fat biscuits are bicontinuous. Whether biscuits are in a glassy or rubbery state cannot be predicted from their water contents in relation to the state diagrams of either gluten or sucrose.

7.1 Introduction

The mechanical properties of a material derive from its structure at macroscopic, microscopic and molecular scale which, in turn, is determined by processing conditions and composition. In a composite food, mechanical properties are especially affected by volume fraction and distribution of each phase and by the nature and extent of interaction between the various phases. In addition, fracture behaviour depends on size and distribution of defects in the material¹.

A biscuit may be described as a matrix in which gas bubbles of various size and shape are embedded. However, there is rudimentary understanding of the factors that affect the mechanical properties of short-dough biscuits. Part of the work reported in literature on quality characteristics of short-dough biscuits, has aimed at establishing relationships between sensory properties and either instrumental measurements ("empirical"^{2,4}, or "fundamental"⁵) or measurements from ultrasonic techniques⁶. Little research has aimed at elucidating how the mechanical properties of the biscuits are related to their internal structure. Brennan *et al.*⁷ investigated the effect of water content on mechanical properties of biscuits by three-point bending tests. They found

a drop in fracture stress and modulus accompanied by an increase in deformation to fracture at a water content of about 4 to 5%. Slade and Levine⁸ reported a sharp drop in the modulus of wire-cut biscuits at a water content of about 4%, as measured by three-point bending tests. Zabik *et al.*⁹ carried out "Kramer shear tests" and noted that relative humidity significantly affected the mechanical properties of sugar-snap biscuits. These results suggest that water acts as a plasticiser, thereby decreasing the glass transition temperature and causing biscuits to exhibit rubbery behaviour. This raises the question of what forms the amorphous matrix in a short-dough biscuit. Slade and Levine¹⁰ propose that gluten goes through a glass transition, either during mixing or during baking. At baking temperatures above T_g , gluten from soft wheat flour may form a continuous network in water or very low sucrose concentrations, but not in high-sucrose environments. Contrarily, gluten from hard wheat flour may develop a network both in water and at high sucrose contents. They speculate that this difference may involve the relative potential for permanent disulphide cross-link formation. The theory is based on time-lapse pictures of biscuits baked from soft and hard flours. The former exhibit macroscopic structural collapse, whereas the latter show contraction due to the elastic properties of the gluten network. However, both biscuits are described as a sucrose glass matrix filled with starch, gluten and fat; depending on the ratio of sucrose to water, biscuits would be in a glassy or rubbery state at room temperature. Doescher *et al.*¹¹ postulate that, during preparation of short doughs, gluten is in a glassy state. During heating in the presence of a sucrose solution, gluten undergoes a glass transition, as a result of which its molecules become mobile; the gluten then expands to form a continuous matrix. Biscuits baked from hard flour show less extensive "spreading" than those from soft flour, because the gluten protein in the former flour has a lower T_g , although T_g values of gluten material isolated from either flour are similar. Based on microscopic results, Flint *et al.*¹² concluded that in short-dough biscuits no continuous protein network is present; the structure consists of a mixture of protein and starch with fat being the filler. In the previous Chapter, we suggested that short-dough biscuits are in a glassy state under certain storage conditions; fat was claimed to act as a filler at low concentrations or form a partly continuous phase at high concentrations. The aim of this Chapter is to examine the validity of this hypothesis and to obtain additional information with respect to the continuous amorphous matrix.

7.2 Experimental

7.2.1 Materials

Commercial wheat flour was obtained from Meneba Meel BV (Rotterdam, the Netherlands) and native wheat starch (water content 12.32%) from Latenstein Zetmeel BV (Nijmegen, the Netherlands). The chemical composition of the flour was given in Chapter 2. Granulated sucrose was provided by Suiker Unie (Breda, the Netherlands) and fructose was purchased from Merck. Fat was supplied by Bakkerol BV (Rotterdam, the Netherlands).

7.2.2 Preparation of biscuits

Biscuit compositions are presented in Table 7.1. The term standard biscuits will be used for compositions in which neither gluten nor sucrose was omitted. In particular, the term reference biscuits will be used throughout to identify standard biscuits with 28.4% fat. A short dough composition typical of commercial operations was used as the reference material. The compositions of the other doughs were chosen in such a way that, when omitting an ingredient or reducing its amount, the weight ratios of the other components were constant. All ingredients were stored at room temperature, unless mentioned otherwise.

Table 7.1 Composition of various biscuits stored at an a_w of 0.11. For calculation of % starch and % gluten the amount of dry flour should be multiplied by 0.83 and 0.14, respectively

Biscuit type	w/w %					
	flour ^a	starch ^a	fat	sucrose	water	salt
standard ^b	45.8	-	28.4	23.4	1.5	0.9
	50.3	-	20.8	25.8	2.2	0.9
	53.2	-	16.4	27.2	2.2	1.0
sugar-free	59.4	-	36.8	-	2.7	1.1
starch	-	50.9	12.8	31.6	3.5	1.2

^a Dry matter

^b Reference biscuits were those with 28.4% fat

Doughs were prepared in a D-300 Hobart mixer. Unless mentioned otherwise, mixing and dough piece formation were carried out at room temperature according to

the procedure outlined in Chapter 6. Dough pieces were baked at 160 °C for 20 min in a small-scale electric oven. The biscuits were held at room temperature, for about 15 min, to cool down. For three-point bending tests they were subsequently placed in desiccators, under vacuum, over one of the following saturated salt solutions¹³: lithium chloride ($a_w = 0.11$), magnesium chloride ($a_w = 0.33$), potassium carbonate ($a_w = 0.44$) and sodium bromide ($a_w = 0.57$). It was observed that biscuits stored at 20 °C reached an "equilibrium" water content after a conditioning period of one week for the first treatment humidity, or ten days for the remaining three treatment humidities. Water content of the biscuits was determined in triplicate after conditioning by a Netherlands Standardisation Institute (NNI) method¹⁴; values reported represent averages of these replicates.

For diffusion experiments, Sudan III was sprinkled on the surface of biscuits, which were then placed in Petri dishes, sealed with parafilm and stored at 20 °C for seven months.

7.2.3 Determination of fracture properties

Fracture properties of biscuits were studied by three-point bending tests; they were carried out at room temperature, unless mentioned otherwise. An Overload Dynamics (table model S 100) or an Instron (table model 1122) materials testing instrument was used with a 20, 50 or 1000 N load-cell. Span length was 4 cm and compression speed was set at 5 mm/min; biscuits were placed on supports with their top surface down. At least 8 biscuits were tested for each batch. Where applicable, results were subjected to one-way analysis of variance¹⁵. Differences in means were evaluated by the least significant difference (LSD) or by Student's *t*-tests. Force-time values were recorded either on the chart paper or through a Daqbook/100 data acquisition system. Young's modulus E ($\text{N}\cdot\text{m}^{-2}$), fracture stress σ_f ($\text{N}\cdot\text{m}^{-2}$) and fracture strain ϵ_f (-) were calculated as shown in Chapter 5.

7.2.4 Density measurements

Bulk densities of doughs and biscuits were determined according to the methods described in Chapters 2 and 5, respectively. Air volume fraction ϕ_{void} and fat volume fraction ϕ_{fat} were estimated from equations given in Chapters 2 and 6, respectively.

7.3 Results

7.3.1 Mechanical properties

7.3.1.1 Preliminary experiments

As regards batch to batch variation, results for reference biscuits were satisfactory in terms of reproducibility. For all the parameters determined, means of different batches were not significantly different at the 0.05 level. However, results presented in Table 7.2 were significantly different from earlier results (Chapter 6), apart from fracture strain. In particular, previously found ϕ_{void} and modulus values were higher. The discrepancy between the two sets of results was smaller at low fat contents. Nevertheless, despite this quantitative discrepancy, qualitatively the results are in agreement with those reported in Chapter 6; in other words, the trends found with respect to fat content still hold. It is noteworthy that water content of biscuits as well as dough densities and dough temperatures after mixing and during depositing were comparable to those from previous experiments.

Table 7.2 Young's modulus E , fracture stress σ_f and fracture strain ϵ_f for biscuits of various fat contents. Air volume fraction ϕ_{void} and fat volume fraction ϕ_{fat} are also indicated

fat w/w %	ϕ_{fat} %		E (MN·m ⁻²)	σ_f (kN·m ⁻²)	$\epsilon_f \times 10^3$ (-)	ϕ_{void} %
28.4 ^a	19.2	mean	38	184	7.5 ^d	52
		s.e. ^b	1.1	4.3	0.4	0.5
20.8	15.8	mean	74 ^c	466	8.9 ^d	48
		s.e. ^b	5.1	9.7	1.0	0.7
16.4	14.4	mean	78 ^c	803	14.2	42
		s.e. ^b	5.5	25.3	1.2	0.7

^a Reference biscuits

^b Standard error of the mean

^{c,d} Difference not significant at the 0.05 level

As shown in Chapter 6, for given geometrical characteristics of the cellular materials, the mechanical properties of biscuits would depend on ϕ_{void} and ϕ_{fat} . Changing ϕ_{fat} can be achieved by altering the fat weight fraction, ϕ_{void} , or both. Therefore, control over ϕ_{void} would be an important facet for biscuit manufacturers.

It was considered that the cause of the afore-mentioned quantitative discrepancy in ϕ_{void} was related to a slight change, either in the processing or in the compositional variables, and since such changes may occur in typical commercial operations, testing this hypothesis would also be important from the manufacturing point of view.

To this end, experiments were conducted in which doughs of various fat content were prepared at 25 °C. Ingredients were also stored at 25 °C for a week prior to dough preparation. For a given fat content, biscuits had similar water contents to those baked from doughs prepared at room temperature. It should be mentioned that temperature during dough preparation did not materially affect dough density at the low fat level (1080 and 1120 kg m⁻³ at room temperature and 25 °C, respectively), whereas at the high fat level it had a pronounced effect (980 and 800 kg m⁻³ at room temperature and 25 °C, respectively). Results are shown in Table 7.3.

Table 7.3 Young's modulus E , fracture stress σ_f and fracture strain ϵ_f for biscuits of various fat contents baked from doughs prepared at 25 °C. Air volume fraction ϕ_{void} and fat volume fraction ϕ_{fat} are also indicated

fat w/w %	ϕ_{fat} %		E (MN m ⁻²)	σ_f (kN m ⁻²)	$\epsilon_f \times 10^3$ (-)	ϕ_{void} %
28.4	16.5	mean	26	156	8.7 ^b	58
		s.e. ^a	0.8	6.9	0.4	0.5
16.4	13.7	mean	76 ^b	809 ^b	14.3 ^b	44
		s.e. ^a	1.9	18.8	0.5	0.6

^a Standard error of the mean

^b Compared to biscuits with same fat contents (Table 7.2) means are not significantly different at the 0.05 level

It can be seen that, at the low fat level, mechanical properties remained unaffected, though ϕ_{void} of biscuits was somewhat higher when doughs were prepared at 25 °C (compare Tables 7.2 and 7.3); at the high fat level, all parameters determined, apart from fracture strain, were significantly different. However, if the reason for the inconsistency in ϕ_{void} values with earlier findings (Chapter 6) were a higher temperature then dough densities would have been significantly different.

To check whether slight drying out of flour during storage would affect ϕ_{void} , biscuits were baked from doughs prepared with 1% extra added water. Doughs with and without extra water had similar densities; the corresponding biscuits had similar water contents. Results are shown in Table 7.4.

Table 7.4 Young's modulus E , fracture stress σ_f and fracture strain ϵ_f for biscuits with a fat content of 28.4% prepared from doughs with and without extra water. Air volume fraction ϕ_{void} and fat volume fraction ϕ_{fat} are also indicated

% extra water ^a	ϕ_{fat} %		E (MN·m ⁻²)	σ_f (kN·m ⁻²)	$\epsilon_f \times 10^3$ (-)	ϕ_{void} %
0	19.2	mean	38	184 ^c	7.5	52
		s.e. ^b	1.1	4.3	0.4	0.5
1	15.6	mean	30	179 ^c	9.7	61
		s.e. ^b	1.9	7.6	0.8	0.5

^a W/W on dough basis

^b Standard error of the mean

^c Means are not significantly different at the 0.05 level

Apart from fracture stress, the other parameters were significantly altered when doughs were prepared with extra water. This is particularly so for ϕ_{void} the increase of which was striking.

Unlike temperature during dough preparation and dough water content, baking powder would provide a means to manipulate ϕ_{void} of biscuits without altering the mixing properties of the corresponding doughs.

Table 7.5 Influence of baking powder on Young's modulus E , fracture stress σ_f and fracture strain ϵ_f for biscuits with 20.8% fat content. Air volume fraction ϕ_{void} and fat volume fraction ϕ_{fat} are also indicated

% baking powder ^a	ϕ_{fat} %		E (MN·m ⁻²)	σ_f (kN·m ⁻²)	$\epsilon_f \times 10^3$ (-)	ϕ_{void} %
0	15.8	mean	74 ^c	466 ^d	8.9 ^e	48
		s.e. ^b	5.1	9.7	1.0	0.7
1	14.9	mean	78 ^c	432 ^d	7.6 ^e	51
		s.e. ^b	3.3	14.6	0.4	1.1

^a W/W on dough basis

^b Standard error of the mean

^{c-e} Difference not significant at the 0.05 level

The amount of baking powder used was typical of commercial operations (0.5% sodium bicarbonate and 0.5% sodium acid pyrophosphate both expressed in dough weight). Results for biscuits with 20.9% fat content are displayed in Table 7.5. Apparently, baking powder had negligible leavening action.

7.3.1.2 Effect of temperature during bending tests

Reference and sugar-free biscuits were tested. Bending tests were performed at 15 °C, 20 °C and 25 °C. To minimise any effects due to fat and/or sucrose recrystallisation, biscuits were stored at an a_w of 0.11 for six days at 20 °C and overnight at the measuring temperature. The a_w of the saturated salt solution was found to be independent of temperature within this range¹³. Results are presented in Table 7.6.

Table 7.6 Influence of measuring temperature on Young's modulus E , fracture stress σ_f and fracture strain ϵ_f for various biscuits. Air volume fraction ϕ_{void} and fat volume fraction ϕ_{fat} are also indicated

Biscuit type	T (°C)	ϕ_{fat} %		E (MN m ⁻²)	σ_f (kN m ⁻²)	$\epsilon_f \times 10^3$ (-)	ϕ_{void} %
reference	15	18.5	mean	55 ^b	296	7.9 ^b	54 ^b
			s.e. ^a	3.9	12.3	0.4	0.6
	20	19.3	mean	38	179	7.2 ^{b,c}	51 ^b
			s.e. ^a	0.9	4.3	0.3	0.6
	25	19.2	mean	47 ^b	228	6.2 ^c	52 ^b
			s.e. ^a	2.5	4.9	0.3	0.9
sugar-free	15	22.4	mean	24	116	6.7 ^d	54 ^c
			s.e. ^a	1.9	4.6	0.4	0.9
	20	22.5	mean	14	66	8.1 ^c	54 ^c
			s.e. ^a	0.6	2.8	0.6	0.6
	25	22.9	mean	9.6	46	7.3 ^{d,e}	53 ^c
			s.e. ^a	0.9	3.1	0.3	0.5

^a Standard error of the mean

^{b-c} For a given biscuit type, pairs of means, within each column, with the same letter are not significantly different at the 0.05 level

For the sugar-free biscuits there was a clear decrease in modulus and fracture stress with increasing temperature. The values at 20 °C were in good agreement with

previously reported results (Chapter 6). For the reference biscuits, all parameters showed little temperature dependence.

7.3.1.3 Effect of storage humidity

Since in biscuits with high fat contents fat may partly form a continuous phase, it was felt that the effect of storage humidity would be better quantified in biscuits with a fat content of 16.4%. Results are shown in Table 7.7.

Table 7.7 Influence of storage humidity on Young's modulus E , fracture stress σ_f and fracture strain ϵ_f for biscuits with 16.4% fat content. Air volume fraction ϕ_{void} and fat volume fraction ϕ_{fat} are also indicated

a_w	water w/w %	ϕ_{fat} %		E (MN m ⁻²)	σ_f (kN m ⁻²)	$\epsilon_f \times 10^3$ (-)	ϕ_{void} %
0.11	2.2	14.4	mean	78	803	14.2 ^b	42 ^b
			s.e. ^a	5.5	25.3	1.2	0.7
0.33	3.4	12.9	mean	99	702	10.9 ^b	47
			s.e. ^a	8.3	22.6	1.3	0.7
0.42	6.0	13.2	mean	25	386	25.2 ^c	44 ^c
			s.e. ^a	2.0	12.5	1.3	0.8
0.57	6.9	13.3	mean	15	173	23.0 ^c	43 ^{b,c}
			s.e. ^a	1.8	6.8	1.8	0.4

^a Standard error of the mean

^{b,c} Within each column, pairs of means with the same letter are not significantly different at the 0.05 level

Up to an a_w of 0.33 the mechanical properties were virtually unaffected; beyond, considerable reduction in modulus and fracture stress occurred, accompanied by an increase in fracture strain. It is clear that, owing to the plasticising action of water, the biscuits underwent a glass transition as a result of which their mechanical properties were profoundly affected. This is further supported by the stress-strain curves given in Fig. 7.1. At low a_w they exhibited a sharp drop in stress upon fracture, typical of a brittle material. Conversely, at high a_w there was no clear point of fracture; the shape of the curves points to a yielding phenomenon, indicative of stronger energy dissipation due to plastic deformation. It should be mentioned that the equations used to calculate E , σ_f and ϵ_f are only applicable to ideally elastic, isotropic materials. Nevertheless, the data so calculated are useful for comparison purposes.

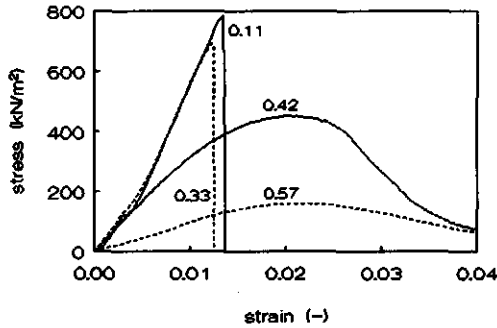


Figure 7.1 Stress-strain curves for biscuits with 16.4% fat content stored at various a_w . For corresponding water contents see Table 7.7.

Sorption isotherms of different types of biscuits vary, depending on their structure, which, in turn, is related to their composition^{16,17}. As a result different biscuits stored at a given a_w may not necessarily have identical water contents and, therefore, their mechanical properties may vary. This aspect is important when choosing storage conditions. In view of the above, it is difficult to precisely determine the water content corresponding to the onset of plasticisation. This difficulty is greater above T_g where the time-scale of the measurement may be an important variable. Nevertheless, our results are both in qualitative and quantitative agreement with those reported in the literature^{7,8}. Wade¹⁸ employed the FMBRA Biscuit Texture Meter and observed no changes in short-dough biscuits as the water content increased from 2.4 to 5.8%. However, in empirical methods the results depend on the equipment design in addition to the specimen properties. In general, water content of fat-free and sucrose crystal-free material would be more relevant.

7.3.1.4 Effect of fructose

The theory of Doescher *et al.*¹¹ does not predict any effect of type of sugar on gluten continuity. To obtain additional information on the continuity of the phases, biscuits with fructose were baked in the hope to manipulate the mechanical properties of one phase ("sugar") without affecting the other ("gluten"). Apart from reference biscuits, starch biscuits with either sucrose or fructose were also baked. Starch was used instead of flour such that the ratio of starch to water in the dough was constant. Starch biscuits had a lower fat content than reference biscuits to avoid test-pieces becoming irregularly shaped. Regardless of the type of biscuit, the type of sugar had no effect

on dough densities; biscuits with fructose had a somewhat higher water content (2.6% and 3.9% for reference and starch biscuits, respectively) compared to those with sucrose (see Table 7.1). Results are shown in Table 7.8.

Table 7.8 Influence of sugar type on Young's modulus E , fracture stress σ_f and fracture strain ϵ_f for reference (28.4% fat content) and starch biscuits (12.8% fat content). Air volume fraction ϕ_{void} and fat volume fraction ϕ_{fat} are also indicated

Biscuit type	Sugar type	ϕ_{fat} %		E (MN m ⁻²)	σ_f (kN m ⁻²)	$\epsilon_f \times 10^3$ (-)	ϕ_{void} %
reference	sucrose	19.2	mean	38	184	7.5 ^b	52
			s.e. ^a	1.1	4.3	0.4	0.5
	fructose	20.8	mean	74	402	7.3 ^b	47
			s.e. ^a	2.3	10.7	0.3	0.5
starch	sucrose	10.3	mean	147 ^b	680	5.7	47
			s.e. ^a	7.9	48.1	0.3	1.7
	fructose	7.4	mean	164 ^b	967	7.3	62
			s.e. ^a	4.4	20.1	0.2	0.9

^a Standard error of the mean

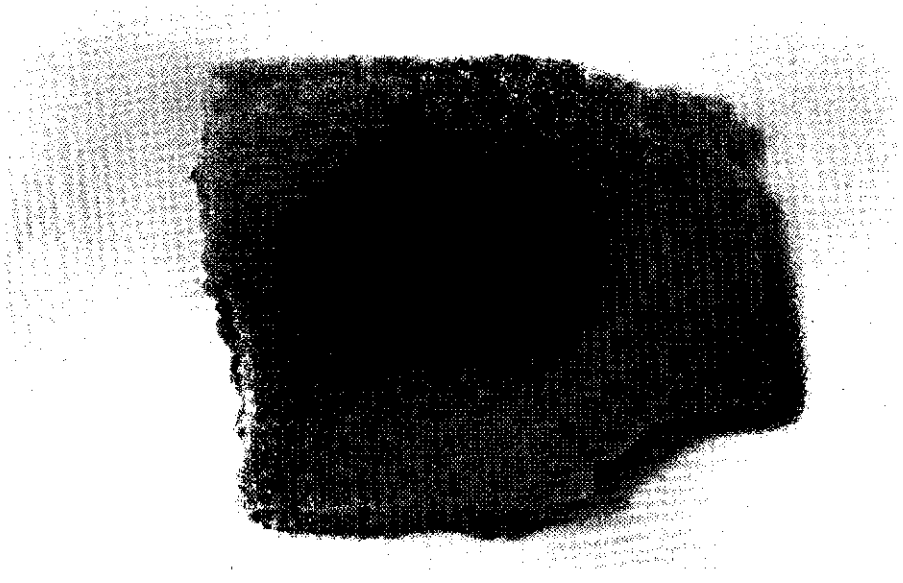
^b For a given biscuit type and within each column, means are not significantly different at the 0.05 level

For reference biscuits, replacing sucrose by fructose clearly increased the modulus and fracture stress by a factor of 2, whereas fracture strain was constant. In materials exhibiting elastic fracture and of the same fracture strain, a higher modulus results in a higher fracture stress, provided that there is no change in scale of inhomogeneities (Chapter 6). In starch biscuits the type of sugar had no clear effect on the moduli. However, starch biscuits with fructose had a higher fracture stress and a larger fracture strain than those with sucrose. As explained in Chapter 6, this is an indication of a different size of the inhomogeneities involved in fracture. It may be mentioned that, as could be seen visually, starch biscuits with fructose had a more open structure in comparison with their sucrose counterparts.

7.3.2 Diffusion experiments

Diffusion experiments were carried out using Sudan III, a fat-soluble stain, to get an indication on fat continuity.

A



B



Figure 7.2 (A) Standard biscuits (16.4% fat) and (B) sugar-free biscuits (37% fat) stained with Sudan III and kept for 7 months at 20 °C.

Biscuits with 16.4% fat content and sugar-free biscuits (37% fat content) were investigated, because they represent extremes in fat content. Figure 7.2 shows that the stain diffused throughout the entire sugar-free biscuit. Limited diffusion occurred in the biscuit with 16.4% fat content, as can be seen from the small diffusion band. In addition, the stain did not reach the bottom of the biscuit. This implies that this biscuit is a fat-dispersed system whereas the sugar-free biscuit is bicontinuous. The Fourier number for mass transfer F_o (-) is given by¹⁹

$$F_o = \frac{D t}{l^2} \quad (7.1)$$

where D is the diffusion coefficient (m^2s^{-1}), t the time (s) and l a characteristic dimension of the body. If we consider a biscuit as an isotropic body, on the surface of which the solute concentration C is constant with time, the relative concentration difference at time t and depth l is given by¹⁹

$$\frac{\Delta C}{\Delta C_o} = \frac{C - C_f}{C - C_o} \quad (7.2)$$

where C_o is the initial solute concentration and C_f the final solute concentration. Assuming that the concentration of Sudan III at the bottom of the biscuit with 16.4% fat content after 7 months is $\leq 1\%$, the relative concentration difference would be > 0.99 . For bodies of well-defined shapes, charts have been published relating the Fourier number and the relative concentration difference¹⁹. For the biscuit with 16.4% fat content the Fourier number would be around 0.1; applying equation (7.1) for a thickness of 5 mm yields a diffusion coefficient of $\leq 10^{-13} \text{ m}^2\text{s}^{-1}$.

7.4 Discussion

In the previous Chapter it was shown that, under certain storage conditions, biscuits are composed of a glassy matrix; their moduli could be represented by the Gibson and Ashby²⁰ model for open- and closed-cell foams assuming that:

- the modulus of the matrix material is $1 \text{ GN}\cdot\text{m}^{-2}$.
- starch granules and sucrose crystals act as fillers with a modulus of $1 \text{ GN}\cdot\text{m}^{-2}$.
- fat is a filler, the modulus of which is significantly smaller than that of the matrix material. Thus, the ratio of the foam density to the density of the matrix was calculated by substituting the sum of ϕ_{void} and ϕ_{fat} for ϕ_{void} .

Figure 7.3 illustrates theoretical modulus values calculated according to the above model together with the results obtained for various biscuits. It can be seen that the fit of the model to the results is quite satisfactory. As mentioned earlier, control over ϕ_{void} would be desirable from the processing point of view. The results given in Table 7.2 show that ϕ_{void} increased with increasing fat content. This is in qualitative agreement with previously reported results (Chapter 6). However, it is evident, from

a comparison of Table 7.2 with Table 7.3, that ϕ_{void} is not solely a function of the fat content. With respect to doughs prepared at room temperature, those prepared at 25 °C gave rise to biscuits of higher (at the high fat level) or similar (at the low fat level) ϕ_{void} . This might originate from differences in density of the corresponding doughs. However, doughs with and without 1% extra water had similar densities; yet, the corresponding biscuits had significantly different ϕ_{void} (Table 7.4). This implies that ϕ_{void} is not directly related to the dough density. The same observation was made for biscuits with various fat types (Chapter 6). It may be argued that, when doughs were prepared with 1% extra water, the corresponding biscuits had higher ϕ_{void} due to the higher amount of leavening agent, *i.e.* water vapour. However, this cannot explain why baking powder had virtually no effect on ϕ_{void} (Table 7.5). Combining these observations with those on the effect of fat type (Chapter 6), it may be concluded that ϕ_{void} is likely to be related to the rheological properties of the doughs.

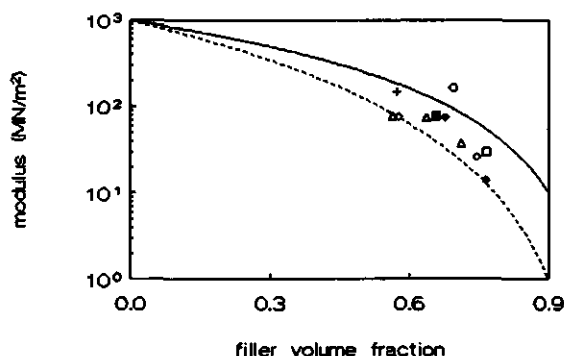


Figure 7.3 Modulus values of various biscuits (stored at an a_w of 0.11 and at 20 °C) as a function of filler volume fraction: (Δ) standard biscuits; (\diamond) biscuits baked from doughs prepared at 25 °C; (\square) biscuits prepared from doughs with 1% extra water; (\blacksquare) biscuits with 1% baking powder; (\bullet) sugar-free biscuits; (\blacklozenge) reference biscuits with fructose; (+) starch biscuits with sucrose; (\circ) starch biscuits with fructose. Lines represent theoretical values calculated according to: (solid) Gibson and Ashby open-cell foam; (dashed) Gibson and Ashby closed-cell foam.

As noted above, the values for the mechanical properties of reference biscuits did not quantitatively agree with previously reported results (Chapter 6). By applying the model described above, this discrepancy may be explained by changed geometrical characteristics of the cellular materials, *i.e.* a transition from an open-cell foam to a more closed-cell foam. Since the pore structure of the biscuits was not characterised, it is not clear whether this reasoning is, at least partly, valid.

Table 7.6 shows that the mechanical properties of sugar-free biscuits strongly depend on the measuring temperature. As discussed in Chapter 6 the continuous non-fat phase in a sugar-free biscuit will be composed of gluten, the glass transition temperature T_g of which is expected to be well above room temperature at an a_w of 0.11. Therefore, this temperature dependence must be associated with the fat phase. In other words, fat forms a continuous phase in sugar-free biscuits. Only a slight temperature dependence was observed for the reference biscuits. This suggests that the continuous non-fat phase is in a glassy state. Moreover, the slight temperature dependence must be attributed to the fat phase, which implies that these biscuits are not entirely fat-dispersed systems. Further evidence to support this assumption is provided by the diffusion experiments which clearly show that, regardless of the nature of the non-fat phase (gluten or sucrose glass or both), low-fat biscuits are fat-dispersed systems and high-fat biscuits are bicontinuous. So, it is possible that the discrepancy between the data presented in this Chapter and those reported in the previous one, though its origin is as yet unidentified, is merely a manifestation of an altered fat continuity in the reference biscuits. Such a gradual transition from a matrix continuous to a bicontinuous system would explain why the results for low-fat biscuits (fat dispersed) and sugar-free biscuits (fat already forming a continuous phase) were consistent with those in the previous Chapter. This argument may also offer an explanation for the results shown in Table 7.3. It was found that the effect of temperature during dough preparation on the mechanical properties of biscuits is more pronounced at high fat contents. High temperatures may lower the viscosity of the fat phase bringing about changes in the mixing properties. This could have caused a different distribution, hence an altered contribution of the fat phase to the dough properties (Chapter 4) and perhaps to the biscuit properties. By the same reasoning 1% extra water in the dough could have changed the viscosity of the non-fat phase with similar outcome.

Amorphous sucrose melts are much more susceptible to thermal decomposition than the crystalline material at temperatures well below the crystal melting point; addition of less than 1% sodium chloride accelerates the degradation, the main products of which include glucose, fructose and trisaccharides²¹. The latter may increase the compatibility of solutes in mixtures, thus diminishing or preventing the tendency to form two separate glasses²². The presence of sucrose during the extrusion of cornmeal proteins enhanced their solubility in sodium chloride; it was suggested that this is due to a hydrogen bond complex formed between amino acid residues and glucose released during the thermal degradation of sucrose²³. However, such a reaction depends on the type of sugar and the nature of the protein²⁴. The above studies would suggest that gluten-sucrose mixtures may be miscible. On the other hand, it has been reported that sugars stabilise protein-protein interactions through a solvent structure effect which excludes sugar from the protein molecules²⁵⁻²⁷. This would imply that gluten-sucrose mixtures may be incompatible. Information on gluten-sucrose mixtures is meagre; it

was found that such systems may or may not be compatible, depending on sucrose and water contents^{28,29}. In these studies, however, the ratios of gluten to sucrose were higher than those in biscuits. In general, miscible blends have a single T_g between the glass transition temperatures of the two components; phase separated systems have two glass transition temperatures corresponding to the two components²⁹. From the above discussion, it is obvious that the observed glass transition in biscuits (Table 7.7) can neither support nor discount the hypothesis of Slade and Levine¹⁰ that the matrix in short-dough biscuits is composed of a sucrose glass. These authors also suggest that the mechanical properties of such biscuits are determined by the state of sucrose (glassy or rubbery), hence by the ratio of sucrose to water. Numerous studies have been published on glass transition temperatures T_g of sucrose as a function of water content^{8,30-41}. These so-called state diagrams, vary considerably according to method of determination, and previous thermal history of the sample; moreover, traces of water are important, whenever T_g of pure substances is determined. For instance, values reported for the amount of water necessary to drop T_g to 20 °C, ranged between 5 and 13%. Regardless of the state diagram, the ratio of sucrose to water is not a sufficient criterion for predicting the mechanical properties of biscuits because it is assumed that all sucrose is in a glassy state, whereas part of the sucrose may be crystalline. In addition, any distribution of water over the various hydrophilic components is neglected.

Several researchers have published glass transition temperatures T_g of anhydrous fructose^{22,29,39-48}. Reported values vary, yet almost all of them are below room temperature. Therefore, if sucrose were the continuous phase, biscuits with fructose would be in a rubbery state. However, results given in Table 7.8 together with the stress-strain curves (not shown) indicate that this is not true. The most plausible explanation for the effect of fructose would appear to be gluten continuity. Considering that starch is not gelatinised (Chapter 6), together with the results for starch biscuits (Table 7.8), it may be argued that, in the absence of gluten, the amorphous matrix is composed of sucrose. Then, for reasons explained above, one would expect that starch biscuits with fructose would be in a rubbery state. However, the reverse was true. In a quenched, amorphous melt of pure crystalline β -D-fructose, Finegold *et al.*⁴³ observed a low T_g at 13 °C and a high one at 100 °C. They also noted that annealing (repeated heating and recooling of the initial fructose melt) eventually "removed" the high-temperature glass transition and made the low temperature one more prominent. Slade and Levine²² also reported two glass transition temperatures for fructose. They suggest that, depending on the time the melt is held above the melting temperature T_m and the quenching rate, a melt of fructose may be heterogeneous, with the appearance of two or more structural relaxations. Their argument is based on the fact that the principal thermal reaction of a reducing sugar (*e.g.* α -D-xylose⁴⁹) in the melting-point region is anomerization and equilibration of α - and β -

forms⁵⁰. However, Fernandez-Martin *et al.*⁵¹ found that α -lactose monohydrate underwent, when pure enough, the anomeric equilibrium (mutarotation to the β -anomer) when still in the solid state, much before its melting point. At equilibrium, mutarotation of fructose gives rise to a mixture containing the β -furanose form and both the α - and β -pyranose forms; the first two increase in concentration while the latter decreases with increasing temperature⁵². Slade and Levine²² theorise that xylose exhibits simple mutarotation and the two anomers so formed are compatible resulting in a single value of T_g ; on the other hand, fructose and galactose are subject to complex mutarotation leading to two conformationally different populations which are chemically and mechanically incompatible, hence two glass transition temperatures. However, it has been stated that all glass-forming liquids and polymers exhibit both a primary and a secondary relaxation process; the former is held responsible for T_g , while the latter is more local in nature, still occurs at temperatures much lower than T_g and in certain cases may be the dominant process⁵³. If the idea of Slade and Levine²² is correct, other factors would come into play. For instance, the rate of mutarotation of α -lactose monohydrate was found to be affected by the purity of the α -form, the size and conditioning of the sample and the heating rate⁵¹. Other factors, such as nature of the solvent and sugar concentration may also affect the equilibrium composition⁵⁴. Finegold *et al.*⁴³ found that in a two-component glass with another small sugar the higher T_g of fructose is no longer detectable. As a result, in fructose-glucose blends, fructose takes on the role of plasticiser, since it depresses the T_g of glucose. It was concluded that the lower T_g of dry fructose controls its thermomechanical behaviour. Slade and Levine²² suggest that the higher T_g value is the one of overriding thermomechanical importance in aqueous fructose glasses and in aqueous binary glasses, whereas the lower T_g value is the predominant species in dry binary glasses. This argument may offer an explanation for the results given in Table 7.8. Fructose melts undergo thermal decomposition much more rapidly than sucrose and glucose melts. For example, only 27% of fructose remains after heating a melt at 150 °C for 30 min; the disaccharides formed are assumed to be dianhydrides of fructose²¹. Then, an alternative explanation for the results of Table 7.8 may be that such disaccharides could raise the low T_g value of fructose above room temperature.

Summarising, with increasing fat content, there is a progressive transition from fat-dispersed to bicontinuous systems. No definitive conclusion can be reached as to the components making up the glassy continuous phase. State diagrams of individual components (gluten, sucrose, etc.) in conjunction with water content of biscuits do not provide a sufficient criterion for predicting the behaviour (glassy or rubbery) of biscuits.

7.5 References

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Chapter 8

General Discussion

This Chapter discusses the structure and mechanical properties of short doughs and short dough-biscuits. Where possible, implications for the handling and eating characteristics of the intermediate and finished products together with suggestions for further research will be given.

8.1 Structure and mechanical properties of short doughs

Mixing involves the blending of ingredients to form a dough as homogeneous as possible at a macroscopic scale. The type and amount of fat have significant effects on the structure and rheological behaviour of short doughs. A gradual transition from a fat-continuous to a fat-dispersed dough was observed with decreasing fat content. The effect of fat type appeared to be small at high and low fat contents. At intermediate fat levels, however, the continuity of the fat phase was influenced by the physical state of the fat incorporated; a dough with liquid oil was found to be fat-dispersed, whereas doughs with solid fat appeared to be bicontinuous (Chapter 1). Fat continuity increased with longer mixing time. Replacing flour by starch (*i.e.* omitting gluten, pentosans, damaged starch etc.), or omitting sucrose, resulted also in bicontinuous doughs. Sucrose has a dual functional role. On one hand it facilitates formation of a non-fat continuous phase via its effect on solvent quantity; on the other hand it retards, if not inhibits, gluten development by affecting solvent quality. The continuous non-fat phase comprises a sucrose syrup that "glues" intact flour particles together. The above mentioned differences in fat continuity and nature of the non-fat phase are reflected on small (Chapter 2) and, more clearly, on large deformation properties of short doughs (Chapters 3 and 4). The relationship between composition and structure and mechanical properties of short doughs can be described as follows:

Distribution of the fat phase throughout the non-fat phase is primarily determined by the rheological properties of the former relative to those of the latter and their corresponding volume fractions. During mixing, liquid oil will be divided into about spherical, fine droplets. By contrast, the properties of the fat and also, if present in sufficient quantities, its continuity will change on mixing. As a result, its contribution to the rheological properties of the dough is altered. By affecting both solvent quality and quantity, sucrose may affect the mixing properties of the non-fat phase and thereby the extent of its dispersion and its contribution to the dough properties. Likewise, the absence of hydrophilic components, such as gluten, may change the

distribution of water with similar outcome. From the processing point of view the desirable characteristics of a short-dough can be summarised as follows:

- Considering the machinery used for dough piece formation, gluten development should be avoided to allow proper dough handling properties. This is also important in product innovation; for instance, in choosing an alternative sweetener as a substitute to sucrose its effect on gluten development should be taken into account.
- A dough which is fat-continuous, with the other components dispersed in it, would have poor handling characteristics; moreover and more important, dough pieces would then not retain their shape during baking. Therefore, it is essential that the dough has a non-fat continuous phase. This can be achieved by increasing the water content, by which the potential for gluten development is enhanced. Alternatively, this can be done by increasing the sucrose content.
- To meet the requirements of the particular method of dough piece formation, the consistency of the dough may be manipulated by changing fat content or fat type, or by adjusting the mixing time. However, mixing should always proceed until further processing would cause relatively small changes in dough consistency. This aspect is important for efficient control of the dough piece formation process, and also in cases where the surplus dough, known as "scrap dough", is removed from the roller and returned to the feed hopper.

8.2 Structure and mechanical properties of short-dough biscuits

From the consumer standpoint the desirable characteristics of a short-dough biscuit are not evident. The main factors that influence the mechanical properties of biscuits include:

1. Biscuit air volume fraction.

In general, the modulus is lower for a higher air volume fraction. Regardless of fat type, reduction of fat content increased dough and biscuit density. Temperature during dough preparation did not materially affect dough and biscuit density at low fat contents; at high fat contents a higher temperature resulted in a pronounced decrease in both dough and biscuit density. However, substituting liquid oil for fat increased dough density, yet decreased biscuit density. Slightly increasing dough water content did not affect dough density but it decreased biscuit density for the same biscuit water content. Baking powder had negligible leavening action in doughs with low fat content; as a result biscuit density was practically unaffected. These results show that there is no direct relationship between dough and biscuit air volume fraction (Chapters 6 and 7).

2. Geometrical characteristics of the cellular material.

In the investigations described in this thesis the pore structure of the biscuits was not characterised. The model of Gibson and Ashby was applied assuming that fat is a filler. However, fat continuity increases with increasing fat content (Chapter 7) and, therefore, it is not clear whether changes in mechanical properties are associated with changes from open- to closed-cell systems (Chapter 6). A possible exception was the starch biscuits; those with fructose had a more open structure in comparison with their sucrose counterparts, as could be seen visually.

3. State of the continuous non-fat phase.

Under certain storage conditions, the non-fat phase of short-dough biscuits is in a glassy state. Obviously, this is an important quality characteristic. The mechanical properties of biscuits were markedly influenced by water content; as a result of the plasticising effect of water, the matrix underwent a glass-rubber transition with increasing water content (Chapter 7). Regardless of biscuit composition, starch gelatinisation was slight, if not absent, presumably due to the limited water content coupled with the low baking temperature (Chapter 6). Therefore, the continuous non-fat phase must consist of a sucrose and/or a gluten glass. No definitive conclusion could be drawn as to the components making up the glassy continuous phase. Issues such as compatibility or incompatibility of mixtures of gluten with various sugars, remain a subject for future research. These aspects are important for developing new products. For instance, the use of alternative sweeteners may call for a modification of baking or storage conditions to adjust biscuit water content. This is particularly true in view of the different crystallisation kinetics, melting points and extents of dissolution of various sugars, which may affect the ratio of glassy to crystalline sugar in the baked product.

4. Fat continuity.

Measuring temperature affected the mechanical properties of biscuits, the more so at a higher fat content. At low fat contents, biscuits exhibit elastic fracture; at high fat contents, plastic deformation was more pronounced and, thereby, fracture behaviour was also qualitatively changed. These results, together with those from diffusion experiments, indicate that low-fat biscuits are fat-dispersed systems and high-fat biscuits are bicontinuous, regardless of the nature of the non-fat phase (Chapter 7).

5. Size of inhomogeneities.

It was found that for a given fat content, the effect of fat type can be explained, in addition to the changed air volume fraction, by a changed scale of inhomogeneities. The same was also observed in starch biscuits with different sugars. The invariably inhomogeneous structure of the biscuits and the importance of inhomogeneities is more clearly demonstrated in biscuits tested with the top surface up as opposed to the inverted position (Chapters 5, 6 and 7).

The relationship between dough and biscuit properties is incompletely understood. A precise account of the events occurring during baking is not yet possible; however some clues will be given below.

During baking, fat will melt, water will evaporate and gas will be liberated from the leavening agent. The difference in thermal expansion between the various phases will impose a stress on the non-fat continuous phase; moreover, during drying, pressure gradients may develop within the dough piece, providing an additional source of stress¹. In that respect, the size and the distribution of gas cells in the dough may be important. Stresses may lead to crack formation, which may cause more intense stress amplification than spherical holes would do². The factors affecting the mechanical properties of biscuits (discussed earlier) are probably associated with these stresses in relation to the rheological properties of the dough. For instance, fat globules may be deformed and coalesce, giving rise to a continuous fat phase in the biscuit. A high dough viscosity may impede gas bubble expansion and may thereby play a role, not only in biscuit air volume fraction, but also in the size of gas cells. In addition, the large deformation properties of the non-fat phase may also come into play; during baking, a point may be reached where the film between gas cells ruptures, thus affecting the pore structure and the shape of the inhomogeneities. Therefore, apart from factors, such as composition, which directly affect the rheological properties of the dough, other variables may be equally important. As mentioned in the Introduction, the rate of gas liberation depends on the type of leavening acid used; the rate of water loss, which will affect the stiffness of the non-fat phase, may be adjusted by baking temperature. It is also relevant to note that the water content of the finished product will be determined by the glass-rubber transition, if it indeed occurs during baking³.

8.3 References

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Summary

Nowadays, biscuit manufacture is increasingly automated and mechanised. Nevertheless, the characteristics of the intermediate and finished products rely to a large extent upon the experience of the operators; this hinders efficient quality control. The main reason for this is the lack of knowledge with respect to:

- the desirable properties of the various types of doughs and biscuits.
- the critical parameters, be it composition or processing conditions, affecting these properties.

Studies on short doughs and their corresponding biscuits are fragmentary; interpretation of the results is hindered by the absence of universally accepted terminology to describe products and their properties. This difficulty is aggravated by the empirical nature of the tests performed. The aim of this thesis is to study the effects of composition and processing variables on the mechanical properties of short doughs and biscuits and relate these properties to their internal structure.

Chapter 2 deals with the rheological properties of short doughs of various composition. These were determined in dynamic shear experiments at small deformation. Regardless of composition, the linear region was very limited; beyond that, significant structure breakdown occurred. This suggests that the elastic properties stem from a network of particles rather than a macromolecular one.

Reduction of fat content caused a considerable decrease in the stiffness of the dough. Curves of the modulus as a function of strain of the standard and the low-fat doughs crossed over at strains outside the linear region. This difference in strain dependence can be explained by the fact that the response of the doughs at small deformation is primarily governed by fat or by flour particles bridged by sucrose syrup, respectively.

The modulus of pure fats was strain-rate independent; but, a positive frequency dependence was found for all doughs. These results indicate that doughs are not fully fat-continuous with the other components dispersed in it, but bicontinuous. Further evidence was obtained from conductivity studies, which showed a transition from a fat-continuous to a fat-dispersed system with decreasing fat content. At intermediate fat levels, also the physical state of the added fat altered its distribution in the dough. *The most important factor determining the distribution of the fat phase throughout the non-fat phase after mixing, is the rheological properties of the former relative to those of the latter.* Replacing solid fat by liquid oil drastically decreased the modulus of the dough. This signifies that the non-fat phase must have a low modulus, in agreement with the lower modulus of a low-fat dough.

Substituting starch for flour, or omitting sucrose from the recipe, decreased the liquid-like character of the dough. The absence of gluten or sucrose may affect the

Summary

distribution of water, hence the rheological properties of the non-fat phase and so the mixing properties. As a result the extent of dispersion of the various components may be affected, giving rise to a changed contribution of the starch and/or the fat to the dough properties.

Chapter 3 discusses the large deformation properties of short doughs of various composition as determined in uniaxial compression. Apart from a sugar-free dough, all doughs studied showed pronounced yielding and flow behaviour; their behaviour showed a strong strain sensitivity, changing from solid-like to more liquid-like at very small strain. All doughs yielded in tension, whereas pure fats yielded in shear, providing further evidence that the doughs cannot be considered as fully fat-continuous. Replacing flour by starch decreased and replacing standard fat by a firmer fat increased the apparent biaxial extensional viscosity of the doughs. However, the similar rate-dependent behaviour suggests that the structure of these doughs does not vary greatly.

A dough with a low fat content had a higher resistance to large deformation than a standard dough, due to the fat being more deformable than the non-fat matrix at large deformation; in addition, the strain-rate thinning character was less pronounced.

Stress-strain curves for the sugar-free dough showed a substantially different rheological behaviour, which was assigned to the non-fat phase. In the presence of sucrose, the non-fat phase is made up by a sucrose syrup binding flour particles by means of liquid necks. By contrast, the response of the sugar-free dough at large deformation is affected, to a certain extent, by gluten.

The effect of sucrose on the large deformation properties of short doughs was studied in more detail with particular reference to mixing time. Results presented in Chapter 4 show that the doughs are more easily deformed with increasing sucrose content and, in the absence of sucrose, with increasing water content. It is concluded that sucrose retards gluten development by affecting solvent quality, and facilitates formation of a non-fat continuous phase through its effect on solvent quantity. Mixing time considerably affected the rheological properties of a dough with sucrose. The observed changes were quantitative in nature and were attributed to an increase in fat continuity with increasing mixing time. This was also true for a sugar-free dough. However, the elastic contribution to deformation was more prominent, the more so for a shorter mixing time, which is not due to a change in fat continuity.

Chapter 5 describes the development of a procedure for the determination of fracture properties of short-dough biscuits. This was considered necessary in order to ascertain effects due to test conditions and those due to the invariably inhomogeneous structure of the biscuits. Three-point bending tests were chosen as the measuring method. Biscuits exhibited brittle fracture, *i.e.* an essentially elastic response and a small fracture strain. A notable characteristic was the abrupt drop in force upon fracture. Span length did not influence the parameters measured, in accordance with the theory

for flexure of straight beams. Biscuits fractured with the bottom side upwards had slightly higher fracture stress and fracture strain than those in the inverted position. Presumably, air cells at the bottom side acted as fracture inducing defects. Fracture strain was strain-rate dependent over the range studied, possibly due to local plastic deformation.

Mechanical properties of short-dough biscuits of various composition were determined in three-point bending tests. The results are reported in Chapter 6. Differential scanning calorimetry showed that starch was hardly gelatinised, regardless of the biscuit type. Because of the strain-rate independence of the modulus and the brittle fracture of the biscuits, it was concluded that the continuous phase is in a glassy state under certain storage conditions. This phase must be composed of a gluten and/or sucrose glass.

In general, reduction of fat content increased the modulus and the fracture stress of biscuits. The relative magnitude of this effect was dependent on fat type. Air volume fraction of biscuits increased with increasing fat content. However, there was no direct relationship between dough and biscuit air volume fraction; the latter may be partly due to the rheological properties of the dough in impeding gas bubble expansion during baking.

Predissolving the sucrose resulted in biscuits with mechanical properties similar to those with crystalline sucrose. This indicates that sucrose crystals were not the fracture inducing defects. Omitting sucrose from the recipe markedly decreased the modulus and the fracture stress of biscuits. In addition, their stress-strain curves pointed to more energy dissipation due to plastic deformation; once fracture stress was reached, the subsequent drop in stress was not as sharp as for all other biscuits.

The modulus values for all biscuits studied were satisfactorily represented by the model of Gibson and Ashby for open- and closed-cell foams. The effect of compositional factors on the modulus could roughly be explained on the basis of air and fat volume fractions and the geometrical characteristics of the foam. In certain cases the effect of fat type on fracture stress and fracture strain can be explained by a different scale of inhomogeneities.

Chapter 7 discusses the relationship between the mechanical properties of short-dough biscuits and their internal structure. The mechanical properties of sugar-free biscuits strongly depended on the measuring temperature. Given that the continuous non-fat phase, *i.e.* gluten, is expected to be in a glassy state, this temperature dependence is due to the fat forming a continuous phase. Only a slight temperature dependence was observed for biscuits with sucrose, which suggests that the continuous non-fat phase is in glassy state and that the biscuits are not entirely fat-dispersed systems. Diffusion experiments showed that, regardless of the nature of the non-fat phase (gluten and/or sucrose glass), low-fat biscuits are fat-dispersed systems and high-fat biscuits are bicontinuous.

Summary

Temperature during dough preparation did not affect the mechanical properties of low-fat biscuits; for high-fat biscuits most of the parameters determined, including air volume fraction, were significantly different. Unlike baking powder, dough water content significantly altered the air volume fraction and the mechanical properties of biscuits. These results suggest that factors affecting the rheological properties of the dough may bring about changes in the mixing properties causing a different distribution of the components, hence an altered fat continuity, in the biscuit.

Increasing biscuit water content considerably reduced the modulus and the fracture stress and increased the fracture strain. At low water contents the stress-strain curves were typical of brittle fracture; at high water contents there was a kind of yielding rather than a discernible fracture point. Apparently, the biscuits underwent a glass-rubber transition owing to the plasticising action of water.

The effect of sugar type on mechanical properties depended on the type of biscuit. Replacing sucrose by fructose increased the modulus and the fracture stress of reference biscuits. By applying the model of Gibson and Ashby, the effect of sugar type may be explained by changed geometrical characteristics of the cellular materials. However, it is not clear whether this reasoning is valid, in view of a possible change in fat continuity. On the other hand, the effect of sugar type on starch biscuits, which had a low fat content, may be explained by a changed scale of inhomogeneities as well as a transition from an open-cell foam to a more closed-cell foam. Although the pore structure was not characterised, visual examination suggested that starch biscuits with fructose had a more open structure, as compared to their sucrose containing counterparts. No definitive conclusion can be reached as to the components making up the glassy continuous phase.

Chapter 8 summarises the main findings of this thesis. Some implications for the handling properties of doughs and the eating properties of biscuits are also given.

Samenvatting

De bereiding van koekjes wordt tegenwoordig steeds verder geautomatiseerd en gemechaniseerd. Desondanks hangen de eigenschappen van de tussen- en eindprodukten nog sterk af van de ervaring van de operator. Dit belemmert een effectieve beheersing van de kwaliteit. De voornaamste oorzaak van deze situatie is het gebrek aan kennis op het gebied van:

- de gewenste eigenschappen van de verschillende typen degen en koekjes,
- de kritieke kengetallen op het gebied van samenstelling en bewerkingscondities, die deze eigenschappen beïnvloeden.

Onderzoek aan spritsdegen en de daaruit bereide koekjes is fragmentarisch. Interpretatie van de verkregen resultaten is lastig door de afwezigheid van algemeen geaccepteerde termen voor het beschrijven van de produkten en hun eigenschappen. Dit probleem wordt nog versterkt, doordat gewerkt wordt met niet goed gedefinieerde testmethoden. Het doel van de studie beschreven in dit proefschrift is het bestuderen van het effect van bepaalde samenstellings- en bewerkingsvariabelen op de mechanische eigenschappen van spritsdegen en de daaruit bereide koekjes en het leggen van een verband tussen deze eigenschappen en de structuur beide.

In hoofdstuk 2 worden de reologische eigenschappen van spritsdegen behandeld, waarbij de samenstelling gevarieerd is. De reologische eigenschappen werden bepaald met behulp van dynamische experimenten in afschuiving bij kleine vervormingen. Het lineaire gebied was erg klein, onafhankelijk van de samenstelling. Bij metingen buiten het lineaire gebied vond een duidelijke afbraak van de structuur plaats. Dit suggereert dat de elastische eigenschappen van het deeg afkomstig zijn van een deeltjesnetwerk en niet zo zeer van een macromoleculair netwerk.

Verlaging van het vetgehalte resulteerde in een aanzienlijke lagere stijfheid van het deeg bij kleine vervormingen. De moduli van een standaarddeeg en van deeg met een verlaagd vetgehalte uitgezet tegen de relatieve vervorming, kruisten elkaar echter bij relatieve vervormingen buiten het lineaire gebied. Dit verschil in afhankelijkheid van de modulus van de relatieve vervorming kan verklaard worden doordat bij kleine vervormingen het gedrag van de degen voornamelijk bepaald wordt door respectievelijk het vet of de bloemdeeltjes, die aan elkaar verbonden zijn door vloeistofnekken van suikerstroop.

De modulus van de zuivere vetten was onafhankelijk van de vervormingssnelheid, maar die van degen nam toe met de oscillatiefrekwentie. Deze gegevens wijzen erop dat de degen niet zuiver vetcontinu zijn, met de andere deeltjes daarin verdeeld, maar bicontinu. Verder bewijsmateriaal hiervoor werd verkregen uit metingen van de geleidbaarheid, welke met afnemend vetgehalte een overgang lieten zien van een vet-

continu naar een vet gedispergeerd systeem. Bij tussenliggende vetgehalten, vergelijkbaar met die van een typisch spritsdeeg, beïnvloeden de natuurkundige eigenschappen van het toegevoegde vet ook de verdeling van het vet in het deeg. De allerbelangrijkste factor die de uiteindelijke verdeling bepaalt van de vetfase door de niet-vetfase is de verhouding van de reologische eigenschappen van beide. Het vervangen van vast vet door vloeibare olie leidt tot een drastische daling van de modulus van het deeg. Dit betekent dat de niet-vetfase een lage modulus moet hebben, wat in overeenstemming is met de lagere modulus van een deeg met een laag vetgehalte.

Het vervangen van bloem door zetmeel, of het weglaten van de sacharose uit het recept, verlaagt het vloeistofkarakter van een deeg. De afwezigheid van gluten of sacharose kan de verdeling van het water beïnvloeden en zo de reologische eigenschappen van de niet-vetfase en daarmee de mengeigenschappen. Als gevolg daarvan kan de mate van verdeling van de verschillende componenten beïnvloed worden, wat tot een veranderde bijdrage van het zetmeel en/of vet tot de deegeigenschappen leidt.

In hoofdstuk 3 worden de eigenschappen bij grote vervormingen bediscussieerd van spritsdegen met variërende samenstelling, zoals bepaald in eenassige (uniaxiale) compressie. Behalve het suikervrije deeg vertoonden alle bestudeerde degen een uitgesproken zwicht- en stromingsgedrag. Hun mechanisch gedrag was erg gevoelig voor vervorming; het veranderde van een meer vaste stofachtig tot een meer vloeistofachtig gedrag bij zeer kleine vervormingen. Alle degen zwichtten in rek, terwijl zuivere vetten in afschuiving zwichtten, wat een extra bewijs is dat de degen niet beschouwd mogen worden als zuiver vet continu. Het vervangen van bloem door zetmeel of van het standaard vet door een harder vet leidde, respectievelijk, tot een lagere en een hogere schijnbare biaxiale rekviscositeit van de degen. De overeenkomende afhankelijkheid van de vervormingssnelheid suggereert echter dat de structuur van de degen niet al te sterk verschilt.

Een spritsdeeg met een laag vetgehalte heeft een hogere weerstand tegen een grote vervorming dan het standaard deeg, omdat vet makkelijker sterk vervormbaar is dan de niet-vetmatrix. Daarenboven is het afschuifsnellheid verdunnende (strain rate thinning) karakter van deze degen minder uitgesproken.

Spanning versus relatieve vervorming curven van de suikervrije deeg lieten een duidelijk ander reologisch gedrag zien, wat toegeschreven werd aan de niet-vetfase. In de aanwezigheid van sacharose bestaat de niet-vetfase uit bloemdeeltjes, die door vloeistofnekjes van een sacharosestroop met elkaar verbonden zijn. Daarentegen wordt het gedrag van een suikervrij deeg bij grote vervormingen tot op zekere hoogte bepaald door het gluten.

Het effect van sacharose op de grote vervormingseigenschappen van spritsdegen is in meer detail bestudeerd in samenhang met de mengtijden. Resultaten, zoals weergegeven in hoofdstuk 4, laten zien dat degen met een hoger sacharosegehalte gemakkelijker vervormd kunnen worden en, in de afwezigheid van sacharose, bij een

hoger watergehalte. Geconcludeerd werd dat sacharose de glutenontwikkeling remt door beïnvloeding van de oplosmiddelkwaliteit en de vorming van een continue niet-vetfase vergemakkelijkt door het effect ervan op de hoeveelheid oplosmiddel. De mengtijd beïnvloedde in hoge mate de reologische eigenschappen van een deeg met suiker. De waargenomen veranderingen waren kwantitatief van aard en werden toegeschreven aan het meer continu worden van de vetfase bij langere mengtijden. Dit was ook het geval voor de suikervrije degen. Echter de elastische bijdrage aan de vervorming trad dan meer naar voren en wel des te sterker voor een kortere mengtijd. Dit was niet het gevolg van een verandering in de mate van continuïteit van de vetfase.

In hoofdstuk 5 wordt de ontwikkeling beschreven van een procedure voor het bepalen van de breukeigenschappen van spritskoekjes. Dit werd noodzakelijk geacht ten einde effecten als gevolg van de testomstandigheden en als gevolg van de onvermijdelijke inhomogene structuur van de spritsen vast te kunnen stellen. Als meetmethode werd voor de driepuntsbuigtest gekozen. De spritsen vertoonden brosse breuk; dat wil zeggen in essentie elastisch gedrag en breuk bij een kleine relatieve vervorming. Een opmerkelijke karakteristiek was de abrupte daling in de voor de vervorming benodigde kracht bij breuk. De afstand tussen de leggers beïnvloedde de gemeten kengetallen niet, in overstemming met de theorie voor buiging van rechte balken. Spritsen die gebroken werden met de onderkant naar boven hadden een iets hogere breukspanning en relatieve vervorming bij breuk dan bij breken in de omgekeerde positie. Waarschijnlijk werken luchtbellen aan de onderkant als defecten. In het bestudeerde gebied was de relatieve vervorming bij breuk, mogelijk als gevolg van locale plastische vervorming, afhankelijk van de vervormingssnelheid.

De mechanische eigenschappen van spritskoekjes met variërende samenstelling werden bepaald in driepuntsbuigtesten. De resultaten zijn vermeld in hoofdstuk 6. "Differential scanning calorimetrie" liet zien dat het zetmeel, onafhankelijk van het spritstype, nauwelijks verstijfseld was. Aangezien de modulus niet van de vervormingssnelheid afhing en gezien het brosse breukkarakter van de spritsen, werd geconcludeerd dat de continue fase zich onder bepaalde opslagcondities in de glastoe-stand bevond. Deze fase moet bestaan uit een gluten en/of een sacharose glas.

In zijn algemeenheid leidde een verlaging van het vetgehalte tot een hogere modulus en breukspanning van de spritsen. De relatieve grootte van dit effect was afhankelijk van het type vet. De volumefractie lucht in de spritsen was hoger voor hogere vetgehaltes. Er was echter geen direct verband tussen de volumefractie lucht in een deeg en in de spritsen. Dit laatste kan gedeeltelijk samenhangen met het gegeven dat de reologische eigenschappen van het deeg de expansie van een gasbel tijdens het bakken kan beïnvloeden.

Het vooraf oplossen van de sacharose resulteerde in spritsen met gelijke mechanische eigenschappen als van die gemaakt met kristallijn sacharose. Dit wijst er op dat de sa-

charosekristallen niet de breuk initiërende defecten vormden. Weglaten van de sacharose uit het recept verlaagde de modulus en de breuksterkte van de biscuits aanzienlijk. Bovendien wees de vorm van de spanning versus relatieve vervorming curven dan op meer energiedissipatie als gevolg van plastische vervorming. De daling in de spanning op het moment dat de breuksterkte was bereikt, was niet zo scherp als voor alle andere biscuits.

De waarde van de modulus van alle onderzochte biscuits kon op bevredigende wijze worden weergegeven door het model van Gibson en Ashby voor "schuimen" met open en gesloten bellen. Het effect van de samenstelling op de modulus kon globaal verklaard worden op basis van de volumefractie lucht en vet en op basis van de geometrische karakteristieken van het schuim. In bepaalde gevallen kan het effect van het type vet op de breukspanning en de relatieve vervorming bij breuk verklaard worden door een verschillende schaal van de inhomogeniteiten.

In hoofdstuk 7 wordt het verband tussen de mechanische eigenschappen van spritskoekjes en hun interne structuur bediscussieerd. De mechanische eigenschappen van suikervrije spritsen hingen sterk af van de meettemperatuur. Gezien het gegeven dat de continue niet-vetfase, dat is de gluten, naar verwachting in de glastoestand is, betekent dit dat de temperatuurafhankelijkheid het gevolg moet zijn van het vet, dat ook een continue fase vormt. Voor spritsen met sacharose werd slechts een geringe temperatuursafhankelijkheid waargenomen, wat suggereert dat de continue niet-vetfase in de glastoestand is en dat de spritsen niet als zuiver vet gedispergeerde systemen beschouwd kunnen worden. Diffusie-experimenten lieten zien dat Onafhankelijk van de aard van de niet-vetfase (gluten en/of een sacharose glas) lieten diffusie-experimenten zien dat spritsen met een laag vetgehalte een vetgedispergeerd systeem zijn; en spritsen met een hoog vetgehalte bicontinu.

De mechanische eigenschappen van spritsen met een laag vetgehalte werden niet beïnvloed door de temperatuur tijdens de deegbereiding. Bij spritsen met een hoog vetgehalte werden de meeste van de bepaalde kengetallen, inclusief de volumefractie lucht, significant beïnvloed door de deegbereidingstemperatuur. In tegenstelling tot de hoeveelheid bakpoeder beïnvloedde het watergehalte van het deeg de volumefractie lucht en de mechanische eigenschappen van de biscuits significant. Deze resultaten suggeren dat factoren, die de reologische eigenschappen van het deeg beïnvloeden, veranderingen in de menigeenschappen te weeg brengen, wat een verschillende verdeling van de samenstellende bestanddelen veroorzaakt en aldus een veranderde mate van het continu zijn van het vet in de spritsen.

Een toename van het watergehalte van de spritsen verlaagde de modulus en de breuksterkte aanzienlijk en resulteerde in een grotere relatieve vervorming bij breuk. Bij een laag watergehalte was de vorm van de spanning versus relatieve vervorming curven typisch voor brosse breuk; bij hoge watergehalten was er eerder een soort zwichtgedrag te zien dan een duidelijk te onderscheiden breukpunt. De spritsen ondergingen

klaarblijkelijk een soort glas-rubber overgang als gevolg van de plasticerende werking van het water.

Het effect van het type suiker op de mechanische eigenschappen hing af van het type sprits. Vervanging van sacharose door fructose gaf een hogere modulus en breukspanning van de referentie sprits. Door gebruik te maken van het model van Gibson en Ashby kan het effect van het type suiker in principe verklaard worden door het effect ervan op de geometrische karakteristieken van het cellulaire materiaal. Het is echter niet duidelijk of deze redenering houdbaar is, gezien de mogelijkheid van een verandering in de mate waarin het vet continu is. Het effect van het type suiker op biscuits met zetmeel en een laag vetgehalte kan ook verklaard worden door een veranderde schaal van de inhomogeniteiten of door een overgang van een "schuim" met open cellen naar een "schuim" met meer gesloten cellen. Hoewel de poriestructuur niet precies bepaald werd, was het op grond van visuele waarnemingen aannemelijk dat spritsen met fructose een meer open structuur hadden in vergelijking met dezelfde spritsen met sacharose. Geen definitieve conclusie kon verkregen worden over de componenten die de continue glasfase vormen.

In hoofdstuk 8 worden de voornaamste resultaten van het proefschrift samengevat. Bovendien worden enkele implicaties voor de bewerkingseigenschappen van de degen en de eeteigenschappen van spritsen gegeven.

List of symbols

a_w	water activity	-
b	thickness of test-piece	m
C_1	constant in equation 6.2	-
C_2	constant in equation 6.3	-
C	solute concentration	w/w %
C_o	initial solute concentration	w/w %
C_f	final solute concentration	w/w %
D	diffusion coefficient	$m^2 s^{-1}$
d	width of test-piece	m
E	Young's modulus	$N m^{-2}$
E_w	Young's modulus of cell-wall material of a cellular solid	$N m^{-2}$
F	force	N
F_o	Fourier number	-
G'	storage modulus	$N m^{-2}$
G''	loss modulus	$N m^{-2}$
G_c	composite shear modulus	$N m^{-2}$
G_m	matrix shear modulus	$N m^{-2}$
G_f	filler shear modulus	$N m^{-2}$
h	height of test-piece	m
h_o	initial height of test-piece	m
k	electrical conductivity of dispersion	$\Omega^{-1} m^{-1}$
k_p	electrical conductivity of disperse phase	$\Omega^{-1} m^{-1}$
k_m	electrical conductivity of continuous medium	$\Omega^{-1} m^{-1}$
K	consistency index	$Pa s^n$
L	span length	m
l	characteristic dimension of test-piece	m
l_c	critical crack length	m
m	mass	kg
m_f	mass fraction of fat	-
m_i	mass fraction of constituent i	-
m_t	weight of glass beads and test-piece	kg
m_b	weight of test-piece	kg
n	flow behaviour index	-
R	radius of test-piece	m
R_s	specific fracture energy	$J m^{-2}$
t	time	s

List of symbols

$\tan \delta$	loss tangent	-
T	temperature	$^{\circ}\text{C}$
T_g	glass transition temperature	$^{\circ}\text{C}$
T_m	melting temperature	$^{\circ}\text{C}$
V	volume	m^3
v_g	specific volume of glass beads	$\text{m}^3 \cdot \text{kg}^{-1}$
v_b	specific volume of test-piece	$\text{m}^3 \cdot \text{kg}^{-1}$
v	crosshead speed	m s^{-1}
y	deflection	m
γ	shear strain	-
δ	loss angle	-
ϵ_b	biaxial Hencky strain	-
$\dot{\epsilon}_b$	biaxial extensional strain rate	s^{-1}
ϵ_f	fracture strain	-
$\dot{\epsilon}$	compressive or tensile strain rate	s^{-1}
μ	Poisson ratio	-
η_b^*	apparent biaxial extensional viscosity	Pa s
ρ	bulk density	kg m^{-3}
ρ_s	solid density	kg m^{-3}
ρ_w	density of cell-wall material of a cellular solid	kg m^{-3}
ρ_i	solid density of constituent i	kg m^{-3}
σ	stress	N m^{-2}
σ_f	fracture stress	N m^{-2}
ϕ	filler volume fraction	-
ϕ_{void}	air volume fraction	-
ϕ_{fat}	fat volume fraction	-
ω	angular frequency	rad s^{-1}

Abbreviations

AMCP	anhydrous monocalcium phosphate
CAEM	citric acid esters of monoglycerides
DATEM	diacetyl tartaric acid esters of monoglycerides
DCP·H ₂ O	dicalcium phosphate dihydrate
DRR	dough rate of reaction
DMTA	dynamic mechanical thermal analysis
DSC	differential scanning calorimetry
DTE	dithioerythritol
EMG	ethoxylated monoglycerides
LAEM	lactic acid esters of monoglycerides
LSD	least significant difference
MCP·H ₂ O	monocalcium phosphate monohydrate
MDG	mono-diglycerides
NEMI	<i>N</i> -ethylmaleimide
NMR	nuclear magnetic resonance
NV	neutralising value
PGE	polyglycerol esters
SAPP	sodium acid pyrophosphate
SDS	sucrose distearate
SMDS	sucrose mono- and distearate
SMP	sucrose monopalmitate
SSF	sodium stearyl fumarate
SSL	sodium stearyl lactylate
ST	sucrose tallowate

Curriculum vitae

Alexios Baltasvias was born on the 9th of February 1965 in Athens, Greece. In 1987 he obtained the B.Sc. degree at the Technological Educational Institute, Department of Food Technology and Nutrition, Athens. In 1988 he obtained the M.Sc. degree at the University of Reading, Department of Food Technology, England. After completing his national service in early 1991, he worked in the sales department of a company involved with trading in chemicals, providing technical assistance. Between 1992 and 1996 he carried out his research work at TNO, Wageningen, while being registered as a Ph.D. student at the Wageningen Agricultural University.