



ALMA MATER STUDIORUM – UNIVERSITÀ DI BOLOGNA  
Facoltà di Agraria  
Dipartimento di Scienze degli Alimenti

**Dottorato di Ricerca in Scienze degli Alimenti  
(AGR/15)**

# **Empirical and fundamental mechanical tests in the evaluation of dough and bread rheological properties**

***Presentata da: Dr.ssa Federica Balestra***

*Coordinatore:*

Prof. Claudio Cavani

*Relatore:*

Prof. Gian Gaetano Pinnavaia

**XXI Ciclo - Esame finale anno 2009**

## **ABSTRACT**

Bread dough and particularly wheat dough, due to its viscoelastic behaviour, is probably the most dynamic and complicated rheological system and its characteristics are very important since they highly affect final products' textural and sensorial properties. The study of dough rheology has been a very challenging task for many researchers since it can provide numerous information about dough formulation, structure and processing. This explains why dough rheology has been a matter of investigation for several decades.

In this research rheological assessment of doughs and breads was performed by using empirical and fundamental methods at both small and large deformation, in order to characterize different types of doughs and final products such as bread. In order to study the structural aspects of food products, image analysis techniques was used for the integration of the information coming from empirical and fundamental rheological measurements.

Evaluation of dough properties was carried out by texture profile analysis (TPA), dough stickiness (Chen and Hosney cell) and uniaxial extensibility determination (Kieffer test) by using a Texture Analyser; small deformation rheological measurements, were performed on a controlled stress-strain rheometer; moreover the structure of different doughs was observed by using the image analysis; while bread characteristics were studied by using texture profile analysis (TPA) and image analysis.

The objective of this research was to understand if the different rheological measurements were able to characterize and differentiate the different samples analysed. This in order to investigate the effect of different formulation and processing conditions on dough and final product from a structural point of view.

For this aim the following different materials were performed and analysed:

- frozen dough realized without yeast;
- frozen dough and bread made with frozen dough;
- doughs obtained by using different fermentation method;
- doughs made by Kamut<sup>®</sup> flour;
- dough and bread realized with the addition of ginger powder;
- final products coming from different bakeries.

The influence of sub-zero storage time on non-fermented and fermented dough viscoelastic performance and on final product (bread) was evaluated by using small deformation and large deformation methods. In general, the longer the sub-zero storage time the lower the positive viscoelastic attributes.

The effect of fermentation time and of different type of fermentation (straight-dough method; sponge-and-dough procedure and poolish method) on rheological properties of doughs were investigated using empirical and fundamental analysis and image analysis was used to integrate this information throughout the evaluation of the dough's structure. The results of fundamental rheological test showed that the incorporation of sourdough (poolish method) provoked changes that were different from those seen in the others type of fermentation.

The affirmative action of some ingredients (extra-virgin olive oil and a liposomic lecithin emulsifier) to improve rheological characteristics of Kamut<sup>®</sup> dough has been confirmed also when subjected to low temperatures (24 hours and 48 hours at 4°C).

Small deformation oscillatory measurements and large deformation mechanical tests performed provided useful information on the rheological properties of samples realized by using different amounts of ginger powder, showing that the sample with the highest amount of ginger powder (6%) had worse rheological characteristics compared to the other samples.

Moisture content, specific volume, texture and crumb grain characteristics are the major quality attributes of bread products. The different sample analyzed, "Coppia Ferrarese", "Pane Comune Romagnolo" and "Filone Terra di San Marino", showed a decrease of crumb moisture and an increase in hardness over the storage time. Parameters such as cohesiveness and springiness, evaluated by TPA that are indicator of quality of fresh bread, decreased during the storage. By using empirical rheological tests we found several differences among the samples, due to the different ingredients used in formulation and the different process adopted to prepare the sample, but since these products are handmade, the differences could be account as a surplus value.

In conclusion small deformation (in fundamental units) and large deformation methods showed a significant role in monitoring the influence of different ingredients used in formulation, different processing and storage conditions on dough viscoelastic performance and on final product.

Finally the knowledge of formulation, processing and storage conditions together with the evaluation of structural and rheological characteristics is fundamental for the study of complex matrices like bakery products, where numerous variable can influence their final quality (e.g. raw material, bread-making procedure, time and temperature of the fermentation and baking).

# CONTENTS

INTRODUCTION .....	1
1. BREAD MAKING PROCESS .....	3
1.1 Ingredients .....	5
1.2 Processing .....	7
1.2.1 The functions of mixing .....	7
1.2.2 Fermentation .....	8
1.2.3 Baking .....	11
1.3 Types of dough-making processes .....	13
1.3.1 Straight dough method .....	13
1.3.2 Sponge-and-dough procedure .....	15
1.3.3 Sourdough method .....	16
1.3.4 Mechanical dough development .....	18
Continuous bread-making procedure .....	18
Chorleywood bread process .....	18
Brimec process .....	21
1.4 Textural changes in bread during storage .....	22
1.5 Frozen bakery products .....	24
1.5.1 Refrigeration applied to bread dough .....	25
1.5.2 Technological problems, solutions, and requirements .....	27
2. RHEOLOGICAL TESTS .....	38
2.1 Rheological test methods .....	40
2.1.1 Descriptive empirical measurements .....	40
Texture Profile Analysis (TPA test) .....	43
Dough stickiness .....	55
Dough extensibility .....	64
2.1.2 Fundamental rheological tests .....	67
Dynamic oscillation measurements .....	67

3. MATERIALS AND METHODS.....	72
3.1 Formulation and samples preparation.....	72
3.1.1 Frozen dough without yeast.....	72
3.1.2 Frozen dough and bread made with frozen dough.....	75
3.1.3 Doughs obtained by using different fermentation methods.....	79
3.1.4 Doughs made by Kamut <sup>®</sup> flour.....	83
3.1.5 Dough and bread with ginger powder.....	85
3.1.6 Final products coming from different bakeries.....	87
3.2 Analyses.....	90
3.2.1 Dough and bread Texture Profile Analysis (TPA).....	90
TPA on dough.....	91
TPA on bread.....	93
3.2.2 Dough uni-axial extensibility (Kieffer method).....	97
3.2.3 Dough stickiness.....	105
3.2.4 Fundamental rheological tests.....	107
3.2.5 Image analysis.....	109
Image Analysis on bread.....	112
Image Analysis on dough.....	114
3.3 Statistical analysis.....	115
4. RESULTS AND DISCUSSION.....	116
4.1 Frozen dough without yeast.....	116
4.2 Frozen dough and bread made with frozen dough.....	120
4.3 Doughs obtained by using different fermentation methods.....	125
4.4 Doughs made by Kamut <sup>®</sup> flour.....	135
4.5 Dough and bread with ginger powder.....	143
4.6 Final products coming from different bakeries.....	149
CONCLUSIONS.....	167
REFERENCES.....	169

## INTRODUCTION

Rheology principles and theory can be used as an aid in process control and design, and as a tool in the simulation and prediction of the materials response to the complex flows and deformation conditions often found in practical processing situations which can be inaccessible to normal rheological measurement. For instance, it is difficult to access dough during mixing, sheeting, proving and baking without interrupting the process or disturbing the structure of the material.

The general aims of rheological measurements are: to obtain a quantitative description of the materials' mechanical properties; to obtain information related to the molecular structure and composition of the material; to characterise and simulate the material's performance during processing and for quality control.

Rheology can be also related to product functionality: many rheological tests have been used to attempt to predict final product quality such as mixing behaviour, baking performance.

This is based on the structural engineering analysis of materials, where small-scale laboratory measurements of mechanical properties have successfully been extrapolated to the behaviour of large engineered structures such as bridges, buildings, pressure vessels etc. resulting in the idea that controlled tests on well-defined small samples of food in the laboratory can be related to the larger, more complex multi-component situations found in practical processing conditions (Dobraszczyk, B.J. and Morgenster, M.P., 2003).

People often intuitively assess the quality of solid foods by gently squeezing them, or liquid viscosity is assessed by gently rotating the liquid in its container, and indeed these sort of tests are often applied on the factory floor as a crude measure of quality. These intuitive assessments gradually became formalised into quantitative descriptions of material properties by scientists such as Newton, Boyle, Pascal, Hooke, Young and Cauchy.

Within the cereal science community, there is a widespread conviction that the rheological properties of dough are related to baking quality, mainly due to a long tradition of subjective manual assessments of dough rheology prior to baking; for example the practice among bakers of kneading and stretching the dough by hand to assess its quality. Although this is a very subjective method of measuring rheology, it gives us an indication of the sort of rheological measurements we should be making in order to predict baking performance.

Since then rheology has grown rapidly as a science and contributed to a number of applications such as colloids, suspensions and emulsions, polymer processing, extrusion and polymer modelling. Recent developments in polymer rheology have established a quantitative link between the molecular size and structure of polymers to their rheology and end-use performance. Rheological measurements are increasingly being used as rapid, sensitive indicators of polymer molecular structure and predictors of end-use performance and are being applied to bread doughs as indicators of the gluten polymer molecular structure and predictors of its functional behaviour in breadmaking (Dobraszczyk, B.J., 2003).

Full understanding of the rheological behaviour of flour dough is of great importance from the practical point of view. Dough rheology directly affects the baking performance of flours, and rheological analyses have been made in order to optimize dough formulation. Although dough rheology has long been investigated, there remains a significant lack of understanding. This lack of progress is due to the complexity of this biological system (Masi, P. *et al.*, 2001).

Wheat flour and water mixtures, doughs, are used in the manufacture of many different food products. A wheat flour and water mixture when subjected to input of mechanical energy such as mixing will allow for the formation of dough. Even a simple wheat flour and water based dough is a complex system. Thus, the complexity of dough is not restricted to its chemical composition, but also includes physical properties. The rheological properties of dough reflect its machining properties during processing and the quality of the final product. Effective quality control of dough based products should therefore include its characterization during all stages of processing. Additionally the rheological properties of dough at many stages in processing can be indicative of the quality of the finished product. Thus, knowledge or characterization of the rheological properties of dough can be effective in predicting its behaviour during processing and controlling its quality (Ross, K.A. *et al.*, 2004).



## **1.BREAD MAKING PROCESS**

The aim of the bread making process is quite simple: namely to convert wheat flour and other ingredients into a light, aerated and palatable food. Bread is probably the oldest “processed” food. We are unlikely to ever identify the moment when bread was “discovered” though it is probable that the place of discovery was in the Middle East where the origins of cereal farming also lie in antiquity. Early forms of bread would have been very different from how we see it in industrialized countries today and it would probably be closest in character to the modern flat breads of the Middle East. We will probably never know whether the gathering and cooking of wild grass seeds provided the spur to arable farming or whether the ability to grow and harvest the forerunners of modern wheats provided the impetus for breadmaking. Whichever way round the two events occurred there is no doubt that one depends on the other and this simple relationship is the foundation of all modern breadmaking. The move to improve the digestibility of the wild grass seed forerunners of early wheat types through fermentation and baking represents a major step in the evolution of human food production. To make this step requires an appreciation, but not necessarily a scientific understanding, of the unique properties of the proteins in wheat with their ability to form a cohesive mass of dough once the flour has been wetted (hydrated) and subjected to the energy of mixing, even by hand. This cohesive mass is the one bakers call ‘gluten’ and once it has formed into a dough it has the ability to trap gases during resting (fermentation and proof) and baking and this allows the mass to expand to become a softer, lighter and even more palatable food after the final heat processing. The discovery that dough left for long periods of time would increase in volume without being subjected to the high temperatures of baking identified the basis of fermentation (gas production). There is no doubt that the changes in the rheological character of the dough would have been observed by those in charge of food production. The combined effect of these changes is for the subsequent baked mass to further increase in volume and give a product with an even softer, more digestible character and different flavour. Gradually the appreciation of the actions of wild yeasts and portions of old dough (e.g. starter dough) were to lead to the transfer of fermentation technology from the brewing industry and eventually to the production of specialised bakers’ yeast. There are a few basic steps which form the basis of all bread making. They can be listed as follows:

- The mixing of wheat flour and water, together with yeast and salt, and other specified ingredients in appropriate ratios.
- The development of a gluten structure in the dough through the application of energy during mixing, often referred to as “kneading”.
- The incorporation of air bubbles within the dough during mixing.
- The continued “development” of the gluten structure created as the result of kneading in order to modify the rheological properties of the dough and to improve its ability to expand when gas pressures increase because of the generation of carbon dioxide gas in the fermenting dough. This stage of dough development may also be referred to as “ripening” or “maturing” of the dough.
- The creation and modification of particular flavour compounds in the dough.
- The sub-division of the dough mass into unit pieces.
- A preliminary modification of the shape of the divided dough pieces.
- A short delay in processing to further modify physical and rheological properties of the dough pieces.
- The shaping of the dough pieces to achieve their required configurations.
- The fermentation and expansion of the shaped dough pieces during “proof”.
- Further expansion of the dough pieces and fixation of the final bread structure during baking (Cauvain, S.P., 2001).

## 1.1 Ingredients

The minimum formula for bread is flour, yeast, salt and water. If any one of these ingredients is missing, the product is not bread. Other ingredients that are often found in the formula are fat, sugar, milk or milk solids, oxidants, various enzyme preparations (included malted grain), surfactants, and additives to protect against moulds. Each of the components in the formula performs a function in producing the finished loaf.

The flour, of course, is the major component and is responsible for the structure of the bread. It allows the formation of a viscoelastic dough that retain gas (Hoseney, R.C.,1994).

Since the formation of gluten the flour is an essential component of breadmaking processes and wheat is the contributor of the proteins necessary for its formation, it follows that a significant factor that determines final bread quality comes from the wheat via the flour from the mill. The level and quality of the gluten-forming proteins depend heavily on the wheat variety, agricultural practices and environmental effects. The protein content of wheat flour varies according to the wheats that are used by the millers and adjustments they may make in the mill. In general, the higher the protein content in the wheat, the higher the protein content of the flours produced from it. The higher the protein content of a flour, the better is its ability to trap and retain carbon dioxide gas and the larger can be the bread volume.

Protein quality also influences final product quality. It is most often judged by some form of dough rheological test though the prediction of final product quality is less certain because most dough rheological testing methods are carried out using conditions that have a limited relationship to the breadmaking process in which the flour will be used. Protein quality testing relies heavily on the interpretation of the rheological data by experts.

The grade colour figure (GCF), ash or Branscan values of flour are measures of the amount of bran that is present in a white flour. The higher the GCF, ash or Branscan value, the lower will be bread volume, in part because of the dilution effect on the functional protein content.

During the growing cycle for the wheat plant there are a large number of enzymes at work. Of interest to us are the ones known collectively as amylases, and especially alpha-amylase. The term alpha-amylase is used to describe a range of enzymes capable of breaking down damaged starch granules into dextrins and, in combination with beta-amylase, they will produce maltose. Alpha-amylase is produced during the growing cycle and can achieve quite high levels if the period around harvesting is wet. Large numbers of the starch granules are

damaged during milling. These damaged starch granules absorb more water than the undamaged granules, so that the larger the proportion of damaged starch the higher the water absorption of the flour (Cauvain, S.P., 2003).

Yeast is one of the fundamental ingredients; its major role is to convert fermentable carbohydrates into carbon dioxide and ethanol. The gases that result from that conversion provide the lift that produces a light, or leavened, loaf of bread. In addition to its gas production, the yeast has a very marked effect on the rheological properties of the dough.

Salt is generally used at levels of about 1-2% based on the flour weight. It appears to have two major functions. First is taste; bread made with no salt is quite tasteless. The second is to affect the dough's rheological properties, salt makes dough stronger, presumably by shielding charges on the dough proteins (Hoseney, R.C., 1994). Moreover the salt has an inhibiting effect on the formation of gluten during mixing (Cauvain, S.P., 2003).

The last fundamental ingredient is water, which is a plasticizer and solvent. Without water, we have no dough and therefore no viscous flow properties, and many of the reactions that take place during fermentation cannot occur because there is no solvent (Hoseney, R.C., 1994).

The term improvers covers any ingredient added to "improve" the breadmaking potential of a given flour. Different breadmaking processes use different flours and different improver formulations. The functional ingredients used in improvers vary but typically contain one or more of the following ingredients:

- Oxidising agents to improve the gas retention abilities of the dough. The functions of the oxidant are complex and at the protein molecule level are currently thought to be mostly related to "cross-linking" of proteins. By improving dough development we will get larger product volume and improved crumb softness.
- Reducing agents such as L-cysteine may be added to "weaken" the dough structure. It will be used only at low levels in improvers but by reducing dough resistance to deformation it helps in moulding and shape forming without structural damage.
- Emulsifiers may be added to bread to improve its quality, each one acting slightly differently and having its own special effects. There are four commonly used emulsifiers: DATA (diacetyl tartaric acid esters of mono- and di-glycerides) esters, sodium stearoyl lactylate, distilled monoglycerides and lecithins.
- Enzyme-active materials have become important to many sectors of the baking industry following the limitations placed on the use of oxidants. Those most commonly used

are the alpha-amylases (fungal and cereal) and the hemicellulases. Proteolytic enzymes may be used in the USA .

- Full-fat, enzyme-active soya flour may be used as a functional dough ingredient. It has two principal beneficial functions, both arising from its lipoxygenase enzyme system. They are to bleach the flour and assist in dough oxidation (Cauvain, S.P., 2003).

## **1.2 Processing**

The processing of bread can be divided into three basic operations: mixing or dough formation, fermentation, and baking.

### ***1.2.1 The functions of mixing***

In the breadmaking processes mixing plays a major role on forming and developing the gluten structure in the dough and incorporating the necessary gas bubbles for cell structure formation in the baked product. It is the latter which makes bread a light, aerated and palatable food.

In essence mixing is the homogenisation of the ingredients, whereas kneading is the development of the dough (gluten) structure by “work done” after the initial mixing. However, in the context of modern breadmaking both processes take place within the mixing machine and so can be considered as one rather than two processes.

The sub-processes taking place during mixing can be summarised as follows:

1. The uniform dispersion of the recipe ingredients.
2. Dissolution and hydration of those ingredients, in particular the flour proteins and the damaged starch.
3. The development of a gluten (hydrated flour protein) structure in the dough arising from the input of mechanical energy by the mixing action.
4. The incorporation of air bubbles within the dough to provide the gas bubble nuclei for the carbon dioxide which will be generated by yeast fermentation and oxygen for oxidation and yeast activity.
5. The formation of a dough with suitable rheological properties for subsequent processing.

The production of a defined cellular structure in the baked bread depends entirely on the creation of gas bubbles in the dough during mixing and their retention during subsequent

processing. After mixing has been completed the only “new” gas which becomes available is the carbon dioxide gas generated by the yeast fermentation. Carbon dioxide gas has high solubility relative to other gases and in bread dough cannot form gas bubbles (Baker, J.C. and Mize, M.D., 1941). As the yeast produces carbon dioxide gas the latter goes into solution in the aqueous phase within the dough until saturation is achieved. Thereafter continued fermentation causes dough expansion as the gas is retained within the dough structure. The two other gases present in the dough after mixing are oxygen and nitrogen. The residence time for oxygen is relatively short since it is quickly used up by the yeast cells within the dough. Indeed so successful is yeast at scavenging oxygen that no oxygen remains in the dough by the end of the mixing cycle. With the removal of oxygen the only gas which remains entrapped is nitrogen and this plays a major role by providing bubble nuclei into which the carbon dioxide gas can diffuse as the latter comes out of solution.

The numbers and sizes of gas bubbles in the dough at the end of mixing are strongly influenced by the mechanism of dough formation and the mixing conditions in a particular machine (Cauvain, S.P., 2001).

### ***1.2.2 Fermentation***

Yeast is a living organism that is inactive during storage. The inactivity is caused either by drying, in the case of active dry yeast, or by low temperature, in the case of compressed or crumbled yeast.

When yeast is incorporated into a dough, conditions are suitable for it to become active. Yeast is a versatile organism; it can ferment under either aerobic or anaerobic conditions. The production of yeast and the early stages of brewing are aerobic processes, whereas bread fermentation is an anaerobic process. Thus, little growth of yeast occurs during dough fermentation. The oxygen in a dough is rapidly consumed by the yeast and bacteria as fermentation starts. Thereafter, the fermentation is anaerobic unless we add oxygen to the system (i.e. by remixing). The major products of yeast fermentation are carbon dioxide and ethanol. As carbon dioxide is produced, the pH decreases and the aqueous phase becomes saturated with carbon dioxide. The initial lag that is found in a gas “production” curve for bread dough is because the dough’s aqueous phase must become saturated with carbon dioxide before the evolution or loss  $\text{CO}_2$  can be measured. Only after the aqueous phase has become saturated is the carbon dioxide available to leave the system.

As fermentation proceeds, it is customary to punch or remix the dough, depending upon which baking system is being used. Why is this done and what does it accomplish? The gas cells in the dough become larger and larger as more gas is produced. Punching or remixing subdivides the gas cells to produce many more smaller cells. To be sure, a large amount of carbon dioxide is lost in the atmosphere, but the important aspect of the process is the creation of the new gas cells.

Another important benefit of punching or remixing is the mixing of the dough ingredients. Yeast cells do not have mobility in dough. Therefore, they depend upon the sugar diffusing to them. As fermentation proceeds, the diffusion distances become large, so the concentration of sugar diminishes, along with the rate of fermentation. Punching or remixing brings the yeast cells and fermentables together again. In zero- or short-time baking systems, punching is not practical, as the dough is not given sufficient time to expand. The net result is usually a coarser grain (fewer cells) in the bread. A partial solution to this problem is to mix under partial vacuum, which expands the dough and allows the gas cells to be subdivided without the need for waiting for the dough to expand.

In addition to its gas-producing capabilities, yeast also affects dough rheology. The effects of yeast on dough rheology can best be shown by a simple *spread test*. The logic of the test is shown in Figure 1.1.

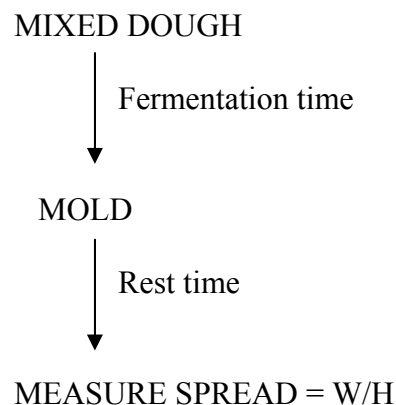


Figure 1.1 Experimental scheme for determining the “spread” of a wheat flour dough.

W, width; H, height.

One can consider a dough to have both viscous-flow properties and elastic properties. A dough that has more viscous-flow properties has a large *spread ratio* (width divided by height), whereas a dough that is more elastic has a smaller one.

As can be seen in Figure 1.2, a flour-water dough gives a large spread ratio after 3 hours. This indicates that the viscous-flow properties are large in a flour-water dough. When yeast is added to such dough, the spread ratio is quite different. This shows that yeast influences the rheological properties of dough. The addition of yeast to the formula causes a dough to go from one with a large viscous-flow component to one that is elastic, as a result of 3 hours of fermentation.

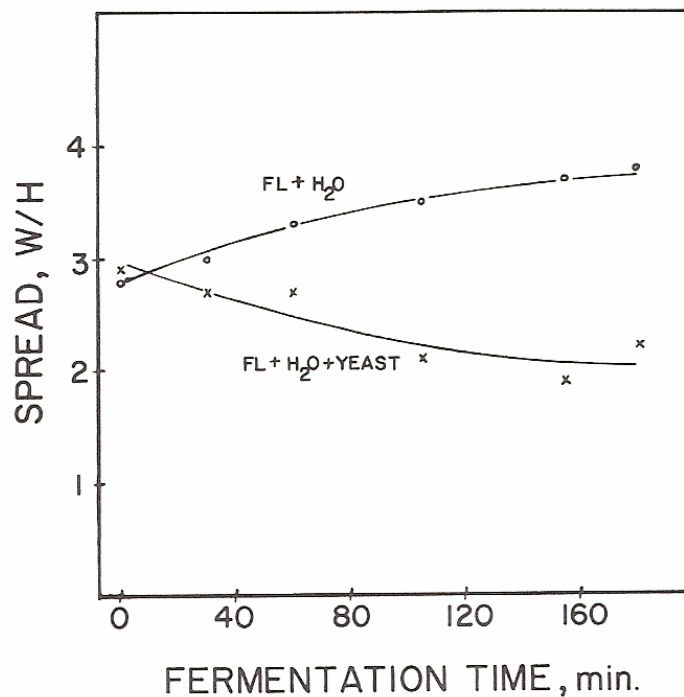


Figure 1.2 Effect of yeast on the spread ratio of a fermenting dough. FL, flour (modified from Hoseney, R.C., 1994).

The trend toward a dough with more elastic properties is the same trend that we find when we add oxidants to a dough. Thus, yeast clearly has an oxidizing effect.

This raises an obvious question. Do the product of fermentation produce the rheological change or is this a property of the yeast itself? The question can be easily answered by running a preferment containing no flour and centrifuging the system to separate yeast cells from the products of fermentation. When this is done and each is added to separate flour-water doughs, it is clear that the products of fermentation do not change dough rheology. The yeast itself appears to be the entity that changes dough rheology. How the yeast cell changes dough rheology is not clear.



The chemical oxidants that are added to the bread formula also affect dough rheology. Certain of the oxidants (potassium iodate and azodicarbonamide, for example) are fast-acting oxidants that have their effect during mixing. Potassium bromate, on the other hand, has essentially no effect during mixing but does effect dough rheology during fermentation. At least part of potassium bromate's time-dependent effect may be because of the change in dough pH during fermentation; potassium bromate reacts faster at lower pH. Ascorbic acid has both a rapid and a time-dependent reaction on dough rheology.

An optimally fermented and oxidized dough has no viscous-flow properties under the force of gravity at the proof stage. Thus, the dough in the pan expands to fill the pan rather than flows to fill the pan. After fermentation, presumably because of the mechanical punching, the gluten fibrils appear to be aligned.

The change in pH associated with fermentation time is also important to the dough's rheological properties. Dough just out the mixer usually has a pH of about 6.0. During fermentation, the pH drops to 5.0. A first rapid drop is caused at first by carbon dioxide dissolving in water to produce carbonic acid. A second factor is the slow production of organic acids by the bacteria in the dough. The flour itself and either milk or soy proteins in the formula are good buffers and therefore help to control pH. The lower pH decreases the mixing time of dough. This is at least in part, the reason for the shorter mixing time in a sponge-and-dough or preferment system than in a straight-dough system. However, the change in pH has a little effect on the dough's spread ratio (Hoseney, R.C., 1994).

### ***1.2.3 Baking***

Baking temperatures will vary from oven to oven and with product but typically they lie in the region of 220–250°C. A key parameter of loaf quality is to achieve a core temperature of about 92–96°C by the end of baking to ensure that the product structure is fully set. For the centre of the dough piece, the move from prover to the oven has little impact because it is so well insulated by surrounding dough. This means that the centre of the dough gets additional proof. The driving force for heat transfer is the temperature gradient from regions near the crusts, where the temperature is limited to the boiling point of water, to the centre. The heat transfer mechanism is conduction along the cell walls and the centre temperature will rise independently of the oven temperature and approach boiling point asymptotically. There is no significant movement of moisture and the moisture content will be the same at the end of

baking as at the beginning. As dough warms up it goes through a complex progression of physical, chemical and biochemical changes. Yeast activity decreases from 43°C and ceases by 55°C. Structural stability is maintained by the expansion of the trapped gases. Gelatinization of the starch starts at about 60°C and initially the starch granules absorb any free water in the dough.  $\alpha$ -amylase activity converts the starch into dextrins and then sugars and reaches its maximum activity between 60 and 70°C. Too little amylase activity restricts loaf volume, because the starch structure becomes rigid too soon, while too much may cause the dough structure to become so fluid that the loaf collapses completely. The formation of a crust provides much of the strength of the finished loaf and the greater part of the flavour. Condensation on the surface of the loaf at the start of baking is essential for the formation of gloss, but quite soon the temperature of the surface rises above the local dew point temperature and evaporation starts. Soon after that the surface reaches the boiling point of the free liquid and the rate of moisture loss accelerates. The heat transfer mechanisms at the evaporation front are complex. There is conduction within the cell walls and water evaporates at the hot end of the cell. Some is lost to the outside but the rest moves across the cell towards the centre and condenses at the cold end of the cell. In doing so it transfers its latent heat before diffusing along the cell wall to evaporate again at the hot end. The evaporation front will develop at different rates depending on the bread types. The crust is outside the evaporation front and here the temperature rises towards the air temperature in the oven. As water is driven off and the crust acquires its characteristic crispness and colour, flavour and aroma develop from the Maillard reactions, which start at temperatures above 150°C. The other contributor to crust formation is the continuing expansion of the inside of the dough piece from the final burst of carbon dioxide production from yeast fermentation and the thermal expansion of the gases trapped in the cellular structure of the dough. If the dough is contained in a pan then it can only expand upwards. This effect is most obvious at the top edges of the loaf, where the displacement is greatest and where a split develops as the top crust lifts, exposing a band of elongated inner crust cells, called the 'oven break', 'oven spring' or 'shred'. Some types of bread are characterised by the crispness of their crust, e.g. baguette. The first few moments in the oven are vital for the formation of a glossy crust. To obtain gloss, it is essential that vapour condenses on the surface to form a starch paste that will gelatinize, form dextrins and eventually caramelize to give both colour and shine. If there is excess water, paste-type gelation takes place while with insufficient water crumb-type gelation occurs. To deliver the necessary water steam is introduced into the oven (Cauvain, S.P., 2001).

## 1.3 Types of dough-making processes

### 1.3.1 *Straight dough method*

The straight dough method is the simplest mixing method, consisting of only one step (Figure 1.3).

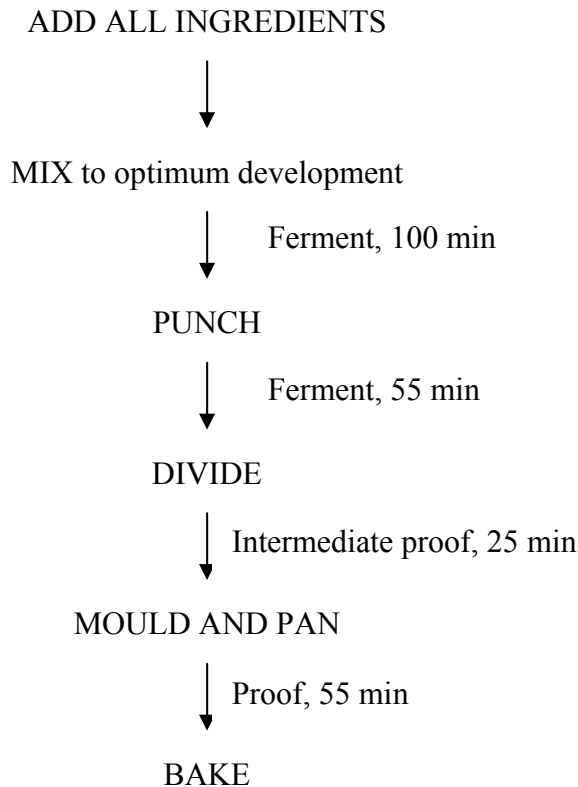


Figure 1.3 Outline of a straight-dough process.

All ingredients are mixed in one operation, and then given a bulk fermentation time (that is, up until molding and proofing) of 1 to 2<sup>1/2</sup> hours. This is called short fermentation straight dough. For rich sweet doughs, the straight dough method is modified to ensure even distribution of the fat and sugar. The fat, sugar, salt, milk solids, and flavorings are mixed first until well combined. Then, the flour and yeast are added and mixed to a smooth dough.

A no-time straight dough is made with a large quantity of yeast, taken from the mixer at a higher temperature (up to 32°C 90°F), and given only a few minutes rest before being scaled and made up. The dough is generally transferred to the dough divider within 10 to 20 minutes after mixing. All further processing is the same as for other doughs. Since no-time doughs are

not subjected to bulk fermentation, they do not require degassing prior to dividing. However, since the fermentation process does not condition no-time doughs, this must be done through the addition of extra maturing (oxidizing) agents, such as 60–120 ppm ascorbic acid. Although shortening the dough processing time by eliminating the bulk fermentation period is a significant advantage to the baker, the lack of fermentation has an adverse effect on the flavor and shelf-life of the baked product. This process is usually used only in emergencies.

Long-fermentation doughs are fermented for 5 or 6 hours or longer, sometimes overnight, at a temperature of 24°C (75°F) or lower (retarded fermentation). Retarding means slowing down the fermentation or proof of yeast doughs by refrigeration. This may be done in regular refrigerators or in special retarders that maintain a high humidity. The amount of yeast should be adjusted depending on the fermentation temperature and time for good control of fermentation. The advantage of this method is that the long, slow fermentation greatly enhances the flavour of the product. The major disadvantage of the straight dough method is that the fermentation is hard to control because of fluctuations in temperature and other factors. Doughs often become over-fermented. Therefore, the straight dough method is usually used in small-scale productions (Hsi-Mei, L. and Tze-Ching, L., 2006).

In general, straight-dough bread is chewier than bread made by other techniques; it has a coarser cell structure; and it is generally considered to have less flavour (Hoseney, R.C., 1994).

### 1.3.2 Sponge-and-dough procedure

The most popular baking process in the United States is the *sponge- and -dough* procedure (Figure 1.4) .

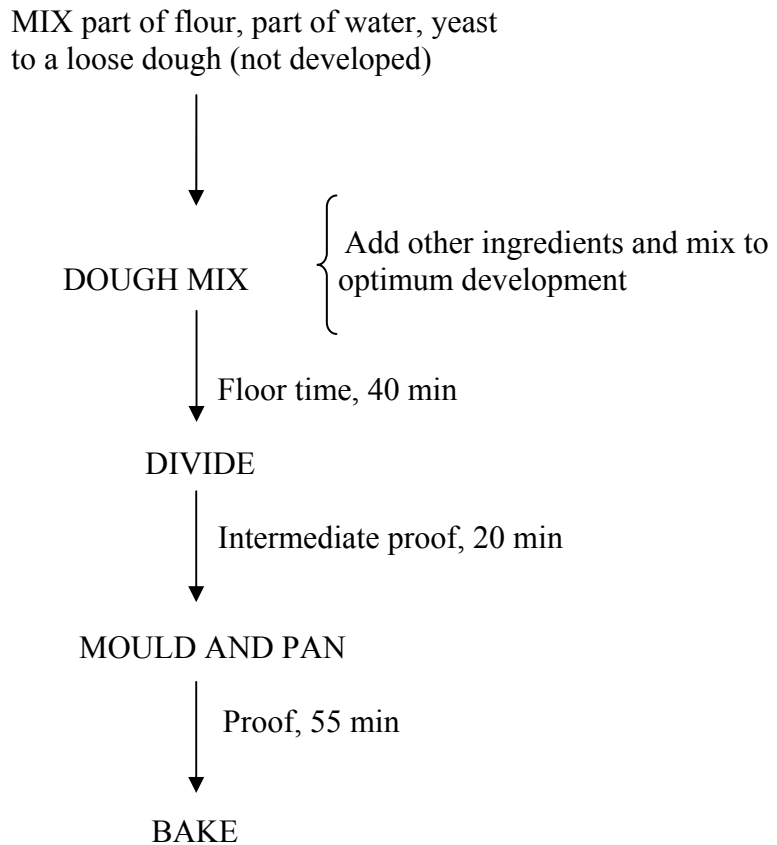


Figure 1.4 Outline of a sponge-and-dough baking process.

In this procedure, part of the flour (approximately two thirds), part of the water, and the yeast are mixed just enough to form a loose dough (*sponge*). The sponge is allowed to ferment for up to 5 hrs. Then it is combined with the rest of the formula ingredients and mixed into a developed dough. After being mixed, the dough is given an intermediate proof (referred to a “floor time”) of 20-30 min so that it can relax, and then it is divided, moulded, and proofed as is done in the straight-dough procedure.

The sponge-and-dough procedure gives a soft bread with a fine cell structure. It is generally considered to have well-developed flavour (Hoseney, R.C.,1994).

The key features of sponge and dough processes are:

- A two-stage process in which part of the total quantity of flour, water and other ingredients from the formulation are mixed to form an homogeneous soft dough – the sponge.
- The resting of the sponge so formed, in bulk for a prescribed time (floortime), mainly depending on flavour requirements.
- Mixing of the sponge with the remainder of the ingredients to form an homogeneous dough.
- Immediate processing of the final dough, although a short period of bulk fermentation may be given.

The sponge contributes to flavour modification and the development of the final dough. The process of flavour development in the sponge, though complex, is observed as an increase in the acidic flavour notes arising from the fermentation by the added yeast and other microorganisms naturally present in the flour. To maintain the right flavour profile in the finished product the sponge fermentation conditions are closely controlled and care is taken to avoid a build-up of unwanted flavours. During the sponge fermentation period there will be a decrease in sponge pH with increasing fermentation. Under these conditions the rheological character of the gluten formed during initial sponge mixing changes and the sponge becomes soft and loses much of its elasticity. The low pH of the sponge and its unique rheological characters are carried through to the dough where they have the effect of producing a softer and more extensible gluten network after the second mixing. In many cases the addition of the sponge changes the rheological character of the final dough sufficiently to warrant further bulk resting time unnecessary so that dividing and moulding can proceed without further delay. Improver additions are commonly made in the dough rather than the sponge. Flours used in typical sponge and dough production will be at least as strong as those used in bulk fermented doughs with protein contents not less than 12% and high Falling Numbers. High  $\alpha$ -amylase activity could be a problem in the sponge because of excessive softening but is less likely to be a problem in the dough (Cauvain, S.P., 2001).

### **1.3.3 Sourdough method**

Finally there is a *sourdough (levain) method*.

Utilization of sour dough is an old-time leavening method in bread making. During sour dough fermentation a typical microflora develops that includes lactic acid bacteria (lactobacilli) and yeasts. The yeasts are generally responsible for the leavening action via carbon dioxide production and for flavor precursor formation. Lactic acid bacteria lower the pH by producing lactic and acetic acid. A typical ratio is about 20% acetic and the remainder

lactic acid. The acidity protects against spoilage by inhibiting mold growth. The growth of rope bacteria is increasingly inhibited as the pH drops below 6.0 and the acids formed preserve the dough. The desired pH of the dough is in the range of 4.0 to 4.6. However, the pH must not become too low, because below about pH 3.7, putrefaction bacteria take over, developing bad odors. Generally this is not a problem, as flour is a relatively good buffer. In addition, lactic acid bacteria produce enzymes ( $\alpha$ -amylase, phytase and proteolytic enzymes) that form low molecular mass carbohydrates, peptides, and amino acids that can act as flavor precursors. For the production of rye bread an acidification is required. The acidity affects the swelling properties of rye flour constituents (proteins, arabinoxylans), controls the enzyme activity in the dough and improves the bread making performance (i.e., improved crumb grain, elasticity, slicing properties, flavor and taste perception, crumb/crust color, and shelf life). Compared to wheat bread rye bread prepared with sour dough has a denser loaf with lower volume, sour-aromatic taste and prolonged shelf life. The sour dough process is a very complex biological system. Factors affecting this system include process variables such as fermentation temperature and time, and ingredient parameters such as flour type, flour ash content, media sources (availability of carbon, nitrogen sources, vitamins), pH, water concentration and the presence of antimicrobial compounds. These parameters affect the multiplication of lactobacilli and yeast, the amounts of acids formed, gas ( $\text{CO}_2$ ,  $\text{O}_2$ ) and ethanol concentrations, the proteolytic activity of lactobacilli, and thereby, the organoleptic properties of the baked product such as taste, flavor, texture, and crumb color. Sour dough production traditionally includes three stages of fermentation: a fresh sour, a basic sour and a full sour (final dough for breadbaking). To initiate sour dough fermentation and to prepare the fresh sour, a spontaneous sour (spontaneously fermented flour-water mixture), a portion of an already developed, old sour dough or a commercial starter culture derived from natural sour dough fermentations can be used. As dough becomes old, the yeast is inactivated, as the pH is low. At the lower pH, bacteria are still quite active. With time, the food available for the organisms becomes limited; however, as new flour is added and the “starter” is fed or rebuilt, the bacteria become more predominant. In general, the gas-producing ability of sour-dough organisms is lower than that of commercial yeast. Consequently proof time of sour dough bread is often long, of the order of several hours. Various sour dough processes (multi-, two-, single stage varying from 2 h up to 24 h) were designed to increase the growth of yeast and lactic acid bacteria to give the final sour dough (full sour) proper acidity (especially the lactic acid/acetic acid ratio) and dough consistency. Often, baker’s yeast (*Saccharomyces cerevisiae*) is added to accelerate the leavening process. The use of organic acids (dough

acidifiers) including lactic, acetic, tartaric, and citric acids alone or in combination with other additives such as sour dough concentrates can replace sour dough in single-stage processes or partly replace it in multistage processes. Apart from the usual batch process, technologies for continuous preparation of sour doughs have been developed (Sievert, D., 2007).

### **1.3.4 Mechanical dough development**

The common attribute of all *mechanical dough development* methods is that there is no fermentation period, when dough is largely, if not entirely developed in the mixing machine. The physico-chemical changes, which normally occur during bulk fermentation periods, are achieved in the mixer through the addition of improvers, extra water, and a specifically planned level of mechanical energy. Several systems have been developed and some of the most popular are: the continuous system in the U.S., the Chorleywood process in the U.K., and the Brimec process in Australia (Giannou, V. *et al.*, 2006).

### **Continuous bread-making procedure**

The *continuous bread-making procedure* that became popular (and was used for > 40% of production) in the United States a few years ago was, in part, such as a procedure. It used a preferment, after which the dough was mixed into a developed dough and extruded into the pan, proofed and baked. The procedure was economical; fewer personnel and less time were required to produce the same amount of bread. However, the bread produced was different from sponge- and dough bread and not well accepted by consumers. The procedure is essentially no longer used (Hoseney, R.C., 1994).

### **Chorleywood bread process**

The CBP may be broadly described as “a no-time dough-making process which uses mechanical development” (Figure 1.5). The basic principles involved in the production of bread and fermented goods by the CBP remain the same as those first published by the Chorleywood team in 1961 (Chamberlain, N. *et al.*, 1961) although the practices have changed slightly with developments in ingredients and mixing equipment.



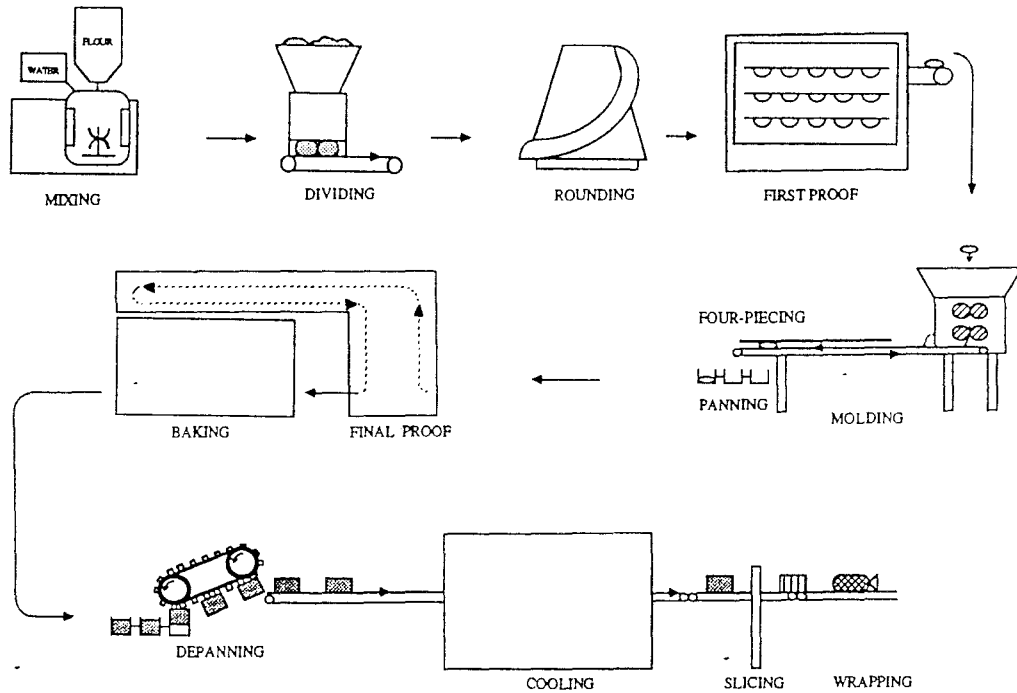


Figure 1.5 Steps in the Chorleywood Bread Process ( modified from Dobraszczyk, B.J. *et al.*, 2001)

The essential features of the CBP may be described as follows:

1. Mixing and dough development in a single operation lasting between 2 and 5 min to a defined energy input. Originally this was considered to be a fixed value equivalent to 0.4hpm/lb, 5Wh/lb, 11Wh/kg or 42kJ/kg dough in the mixer. While later work has shown that higher energy levels are required for some flours, optimum results (i.e. greatest product volume and finest cell structure) are obtained when the total required energy is delivered within the originally specified time. In the case of North American flours the upper limit of mixing time was extended to 7 min.
2. One of the basic principles of the CBP is the addition of an oxidising agent to improve the gas retention abilities of the dough. The functions of the oxidant are complex and depend on the particular oxidising agent that is being used. However, the main effect of any oxidant is to increase dough gas retention, generating greater oven spring and increasing loaf volume. Now legal restrictions only allow the use of ascorbic acid as the sole oxidising agent in many parts of the world.
3. The inclusion of a high-melting-point fat, emulsifier or fat-emulsifier combination. It is important that a proportion of the fat should remain solid in bread dough at the end of final proof, typically at around 40–45°C. The CBP is a no-time dough-making process and during

its development it was recognised that the addition of fat improved the gas retention of the dough and thereby increased bread volume and softness. The level will vary according to the type of flour being used and so it is common to recommend a “blanket” level in order to ensure that sufficient solid fat is always present. With white flours this is usually 0.7% flour weight or higher. Wholemeal (wholewheat) flours commonly require higher levels of fat addition, often twice to three times that of white flour to achieve maximum bread volume .

4 The addition of extra water to adjust dough consistency to be comparable with that obtained with doughs produced by bulk fermentation. This extra water yields doughs with similar machinabilities which can be processed on the same plant as bulk fermented doughs. While some aspects of plant design have changed since the CBP was introduced, the same principle related to dough consistency remains true.

5. The addition of extra yeast to maintain final proof times comparable with those seen with bulk fermentation doughs. The extra yeast is needed in CBP doughs because of the lower gas levels in the dough compared with bulk fermented doughs when they reach the start of proof.

6. The control of mixer headspace atmosphere to achieve given bread cell structures. When the CBP was first introduced, this was restricted to the application of partial vacuum for the whole or part of the mixing cycle but more recently this has been extended to include pressures greater than atmospheric and sequential changes during the mixing cycle. The main difference between the CBP and bulk fermentation processes lies in the rapid development (“maturing”) of the dough in the mixer rather than through a prolonged resting period.

The advantages gained by changing from a bulk fermentation system to the CBP include the following:

1. A reduction in total processing time by at least the fermentation time of the dough in bulk.
2. Space savings from the elimination of the need to keep bowls of dough at different stages of bulk fermentation. These savings have been estimated as being a reduction in the mixing room area of 75% (Chamberlain, N. *et al.*, 1961).
3. A reduction in the need for temperature-controlled areas for the bulk doughs with consequent energy savings. Although CBP doughs typically have a higher dough temperature than bulk fermented doughs, the shorter processing times and poor heat conductivity of dough mean that variations in ambient bakery temperature will have less impact on the dough temperature before reaching the prover.
4. Improved process control and reduced wastage in the event of plant breakdowns because there will be less dough at an intermediate processing stage, e.g. in bulk fermentation.

5. More consistent dough and final product quality through the elimination of the potential variability of dealing with doughs at different stages of fermentation.
6. More consistent dough and final product quality arising from the reduced variability in dough piece weights coming from the divider. This arises because CBP doughs have less gas in them at this stage than those made by bulk fermentation and the yeast is less active.
7. Financial savings from higher dough yield through the addition of extra water and retention of flour solids normally fermented away.

The disadvantages include the following:

1. The need to process the dough at a faster rate because of the higher dough temperatures used compared with those used with bulk fermentation.
2. A need for larger quantities of refrigerated water to control final dough temperature during mixing.
3. A second mixing is required for the incorporation of fruit into fruited breads and buns.
4. A possible reduction in breadcrumb (but not crust) flavour because of the shorter fermentation times involved in processing the dough to bread (Cauvain, S.P. and Young, L.S., 2006).

## **Brimec process**

Mechanical dough development in Australia was first seen as the Brimec process developed by the then Bread Research Institute (BRI) of Australia and launched in 1962. This process featured dough development in the mixer with some crumb cell structure control by varying the position of a ram which restricted the free space in the mixing chamber and exerted some pressure on the dough. The dough could be shaped and placed directly in a pan.

A no-time dough process using extra mixing on low-speed mixers was launched in 1964 (Collins, T.H. *et al.*, 1968). In 1965 an extended proof version of mechanical dough development was launched by the BRI with low yeast level and long proof times (typically 16–18 h).

Today mechanical dough development in Australian bakeries is very similar to the CBP, other than the tendency to use higher work levels in the dough because of the generally stronger wheat varieties which are available in Australia and New Zealand (Cauvain, S.P. and Young, L.S., 2006).

## 1.4 Textural changes in bread during storage

“All bread fresh from the oven is good bread”. There is a lot of truth in that saying. Bread loses its desirability progressively with the time it is out of the oven. Those undesirable changes that occur with time are collectively called “staling”. They include toughening of the crust, firming of the crumb, a loss of flavour, an increase in the opaqueness of the crumb, and a decrease in soluble starch (Hoseney, R.C., 1994).

Freshly baked bread has very different characteristics to that which has been stored for short periods of time. The nature and magnitude of the changes depends on the conditions under which the product has been kept. If held unwrapped then the products in most cases will dry out as moisture evaporates from the product to the surrounding atmosphere. The rate at which moisture is lost from the product depends in part on the differential in moisture content between product and atmosphere, and it proceeds faster when the moisture content of the atmosphere is lower.

A further factor controlling moisture loss from baked products is the water activity ( $a_w$ ); the lower the  $a_w$  the lower the rate at which the product will lose moisture.

Wrapping bread will cause it to lose moisture more slowly; however, in this case the shelf life of the product will be limited by the occurrence of mould growth. The appearance of mould on the surface of the bread product is possible because the  $a_w$  is high enough to permit its growth, typically 0.90–0.98. At the end of baking the moisture content and  $a_w$  of bread crust is usually too low to permit mould growth. During storage, moisture moves from the moist crumb zone to the drier crust. In unwrapped bread the moisture evaporates to the atmosphere, but for wrapped bread an equilibrium is reached between the crumb, crust and atmosphere in the wrapper surrounding the bread. Collectively the changes result in a reduction of the crumb moisture content and an increase in that of the crust. In addition to creating the potential for mould growth, the absorption of moisture by the bread crust causes it to lose its crispness and go “soft”. This change reduces the sensory pleasure experienced by the consumer, especially if the expectation is that the crust should be hard (e.g. as with baguette), and the product is seen as being “stale”. It is common practice to reduce the loss of crispness of bread crust by wrapping the product in a perforated film. The small holes in the wrapper allow some of the moisture that migrates from the moist crumb to evaporate from the crust which allows the latter to remain hard and crisp. However, the overall effect of the moisture loss is for the

crumb to quickly dry out and become hard. In composite products there is considerable potential for moisture migration to and from the bread crumb and the other materials which may be used. Bread staling may be described as the loss of “oven-freshness”. It encompasses a number of different changes:

- loss of crumb and crust moisture, especially if the product is unwrapped;
- loss of crust crispness, more likely to occur if the product is wrapped;
- increases in product crumbliness, commonly related to moisture content; increases in crumb firmness;
- changes in taste, usually a loss of;
- changes in aroma, usually a loss of.

Even when bread products are wrapped to prevent moisture losses during storage there is progressive increase in the firmness of the crumb with increasing storage time. This intrinsic firming is the change most commonly referred to as “staling” in the scientific literature and arises because of changes in the crystalline structures of the starch component of the product (Cauvain, S.P., 2004).

The changes that occur in the crumb appear to be much more complex. It was shown, almost 150 years ago, that the firming of bread crumb is not a drying phenomenon. Firming occurs even though no moisture is lost. Occurring over the same general time span as the firming the recrystallization of the starch. This is referred to as “retrogradation”. Over the last 20 years ago or so, there has been a general consensus that firming and retrogradation are the same phenomenon. However, no firm proof that the two are causatively linked has been offered. Recently, a number of reports have shown that the rate of firming and rate of retrogradation are not the same.

The firming of bread crumb can be reversed by heating. This is one of the advantages to toasting bread; it is said to be “refreshed”. The amylopectin crystal melts at about 60°C, but bread crumb continues to lose firmness as it is heated above 60°C to about 100°C. This strongly argues that firming is not related to retrogradation of amylopectin.

A number of factors are known to alter the rate of staling and to produce a bread that retains its softness over time. First, surfactants that complex with amylose are known for their ability to produce bread, presumably because starch, in the presence of surfactants, does not swell as much as starch alone. Second, inclusion of shortening in the bread formula alters the staling rate. Finally, the use of relatively heat-stable  $\alpha$ -amylase in the bread formula retards staling. The temperature at which bread is stored also appears to be important, with higher

temperatures retarding staling and cooler temperatures (above freezing) increasing the rate of firming (Hoseney, R.C., 1994).

## **1.5 Frozen bakery products**

During the last decades, breadmaking has been slowly but significantly captured by the industry. The use of refrigeration or freezing in the food industry, which started in the late 19th century, is becoming increasingly popular to the breadmaking industry as it provides bakery products with extended shelf-life, postpones the proofing-baking phase, and allows the benefits of producing freshly baked products while saving on equipment and labor costs.

The first kind of product developed in the 1970s was the frozen fully baked bread. Earlier in the 1960s, the frozen part baked bread called “brown and serve” was already proposed. This product was the main frozen bakery product in France. Frozen fully baked products had a limited success due to crust-flaking problems. At that time, yeasted frozen dough was introduced, which has now become the leading product in terms of the market share of frozen bakery products. This technology consists of preparing a dough and freezing it before fermentation starts or after limited fermentation prior to freezing. The final transformation of frozen dough is a three-step process requiring thawing, fermentation, and finally baking. Approximately a decade later, frozen partially baked bread (or frozen part baked bread) developed significantly in the industry. At industrial level, this technology consists of preparing bread with partial baking usually done at moderate temperature and bread being thereafter chilled and frozen. This frozen product can be placed directly into the oven and perform thawing–baking in a single unit operation. One could mention a French patent related to this type of product (LeDuff, L., 1985). In the end of the 1990s, the idea of producing fermented frozen products came up and this technology, which was considered as a rather ascending technique until few years ago, seems to attract more the breadmaking industry. These products are also called “frozen ready to bake.”

Nowadays, frozen bakery products occupy an important share of the market. Researchers believe that in 2006, 17% of “fresh bread” will be done from frozen products (13% in 2001). In Europe, the overall consumption of bread and viennoiserie increases by 1% per year; at the same time, the production of frozen bread and viennoiseries should increase by 7% by 2006.

This expansion of the frozen bakery products in Europe is driven by two patterns:

- The research for convenient products that can be quickly prepared and proposed “as fresh” to the consumer.

- The consumers demand for a large variety of bakery products that is unprofitable to be prepared by retailers.

The market of frozen bakery products is therefore expected to increase in the coming years.

### ***1.5.1 Refrigeration applied to bread dough***

Frozen bakery products can be mainly divided into two categories: frozen yeasted dough and part baked products. These are two complementary products although they produce two different qualities of bread. Frozen bread dough products are especially formulated to survive freezing and thawing. They present quality similar to conventional bread but require a minimum preparation of 2–3 hours. They are normally allowed to thaw and rise (proof) at temperatures slightly above ambient to provide an expanded open grain dough structure and then baked to produce a suitable finished product. The time required for thawed dough proofing is usually determined as “slack time” in the baking industry.

On the contrary, frozen partially baked or part-baked products exhibit shorter preparation time, as they can be ready in less than 20 min, but give a bread with slightly lower sensorial quality. The freshly made dough is allowed to rise and then is partially baked, usually at milder temperature than in the case of conventional breadmaking (i.e., 180°C vs. around 230°C for a French baguette).

Baking must be interrupted before Maillard reactions take place; a sufficient baking is required to achieve a rigid product center at the end of the post baking chilling. Afterward, the product is frozen and then distributed. Thawing is sometimes recommended before final baking, which mainly consists of reheating the product for a short baking time; it is recommended to bake products for up to two thirds of the time required for full baking, until the color change of the crust due to Maillard reactions is achieved.

Retailers using refrigerated bakery products are very often combining the use of frozen dough to cover customary needs and part-baked products to deal with increased consumer demand during peak periods.

Frozen part-baked bread is dragging the innovation and its market share is continuously growing in Europe due to its convenience and the reduced requirements in equipment and labor as it requires a very limited know-how for the final transformation before retailing.

It also allows the production of more elaborated products at industrial level, whereas frozen dough is usually applied for the mass production of conventional products. In 2002, the

industrial production of frozen bakery products in France was 65% for yeasted frozen dough and 35% for frozen part-baked, whereas 40% of the frozen part-baked breads is exported abroad.

Figure 1.6 and Figure 1.7 illustrate the process flow diagrams for the production of frozen dough and part baked products as well as the modifications from conventional breadmaking for yeasted and fermented bakery products, respectively.

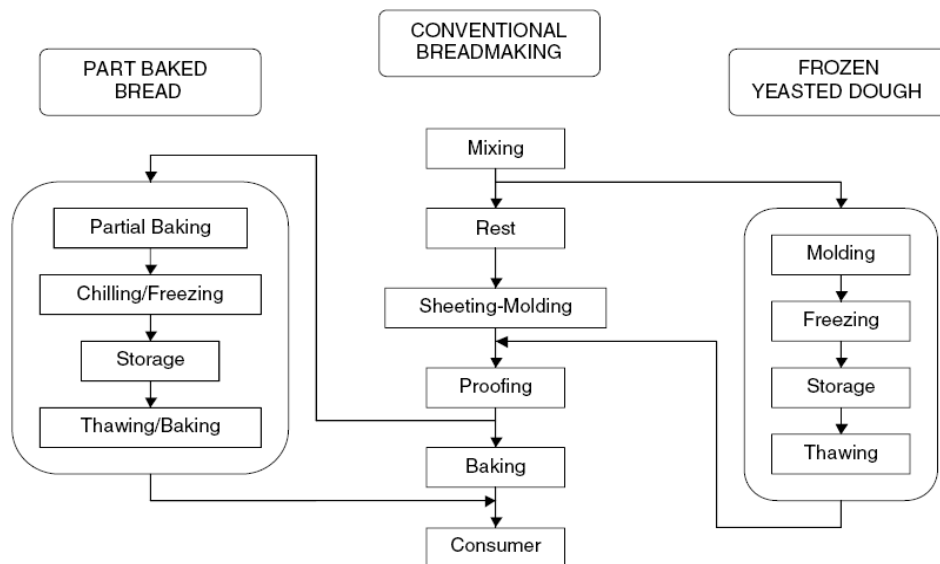


Figure 1.6 Flowsheet for the application of freezing in breadmaking: frozen yeasted dough and part baked bread (modified from Giannou, V. *et al.*, 2006).

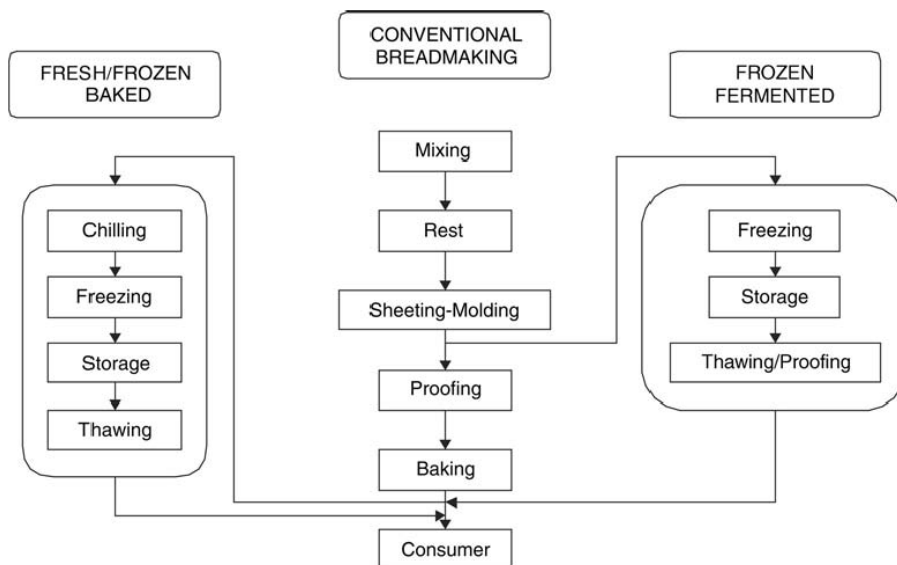


Figure 1.7 Flowsheet for the application of freezing in breadmaking: frozen fermented and baked bread (modified from Giannou, V. *et al.*, 2006).



### ***1.5.2 Technological problems, solutions, and requirements***

#### **Problems associated with frozen dough products freezing**

During food freezing, a number of serious physical changes occur such as the uneven growth of ice crystals within products or moisture migration due to water vapor pressure variance. This results in the accumulation of moisture particularly at the surface of the products and can be detrimental to their textural or sensory characteristics.

The quality of the bread made out of frozen dough in specific is influenced by dough formulation as well as by processing parameters such as dough mixing time, freezing rate, frozen storage temperature, storage duration, and thawing rate.

It appears that these factors may act either independently or synergistically to reduce yeast activity, which results in reduced CO<sub>2</sub> production or weakening or damage to the gluten network and entails in poor retention of CO<sub>2</sub> and poor baking performance.

The main consequences of these phenomena include longer proof or fermentation times, increased extensibility, decreased loaf volume, textural characteristics deterioration, and variable performance.

The rheological characteristics of frozen dough bread have been studied extensively. The presence of dead yeast cells in the dough has been implicated in poor bread quality but some researchers (Autio, K. and Sinda, E., 1992) did not observe significant modification in the rheology of the dough with or without yeast. Others (Varriano-Marston, E.K. *et al.*, 1980) showed that the gluten structure in frozen dough could be damaged by the formation of ice crystals. Researchers (Ribotta, P.D. *et al.*, 2001; Varriano-Marston, E.K. *et al.*, 1980) who worked with extensigraph observed the strengthening of dough (increase of extensigraph resistance and decrease of extensibility) submitted to freeze–thaw cycles because of the reducing substances leached out from yeast cells (mainly glutathione), which cause depolymerization by cleaving disulfide bonds and subsequently weaken the gluten matrix, or the redistribution of water caused by a change in water-binding capacity of flour constituents. The opposite effect was observed by others (Inoue, Y. and Bushuk, W., 1991). The use of different oxidants may explain this result (potassium bromate for Ribotta, P.D. *et al.*, 2001 vs. ascorbic acid for Inoue, Y. and Bushuk, W., 1991).

## **Problems associated with partially baked products**

There is a quite limited amount of literature concerning this product. One of the main problems concerning the quality of the crust is that the undergoing intense heating and cooling phases result in a risk of excessive surface dehydration. In some extreme cases, crust flaking might occur. Crust is the result of successive dehydration of the surface area of the dough during proofing and baking. Even though most of the literature recommends the use of moist air during proofing, there is no clear evidence about this allegation. Neither the effect of post-baking chilling nor the effect of the freezing conditions has been studied. One evidence is that crust flaking is visible at the end of the freezing process. Poor storage conditions may magnify the problem but cannot be considered as solely responsible for the flaking phenomena.

## **Solutions proposed for confrontation of problems**

Different ways to minimize the effect of freezing on doughs and prevent loss of dough quality are suggested in the literature: maintaining yeast viability during freezing and thawing, improving parts of the breadmaking process, or using suitable ingredients, additives, and cryoprotectants for frozen doughs. All these parameters are individually developed and discussed subsequently.

## **Freezing Effect on Yeast Performance — Requirements and Suggestions**

Yeast cells in bulk are regarded cryoresistant and their ability to produce CO<sub>2</sub> is not affected considerably by successive freeze–thaw cycles. However, when the cells are dispersed in a dough, and especially when unfavorable processes such as freezing intervene, this resistance is seriously restricted. The loss of cell viability in the dough during freezing has been attributed to intracellular freezing and increased internal solute concentrations, which may result in pH lowering, dehydration, ionic toxicity, damage to essential membrane processes, impairment of cytoskeletal elements, and decreased glycolytic enzymes activity.

Yeast survival and gassing power are strongly affected by freezing rate, frozen storage temperatures, and duration of frozen storage. From previous studies, it appears that a slow freezing rate is preferable to preserve yeast activity (Bhattacharya, M. *et al.*, 2003). Yeast strain, age of cells, protein content, as well as nature and concentration of cryoprotectants (e.g., trehalose) also influence the yeast activity. In addition, processing conditions such as fermentation prior to freezing may reduce yeast cryoresistance.

To minimize the freezing effect on product stability, several suggestions have been proposed. Some researchers support that dry yeast may be superior to compressed yeast in preserving the shelf-life of frozen dough as it presents longer lag period and consequently more restrained fermentation before freezing, providing a more stable dough. However, reports also show that doughs made with dried yeast exhibit slightly longer proof times and could contain more broken cells that might release glutathione, which is known to affect the gluten network (Ribotta P.D. *et al.*, 2001 ). Another approach for the maintenance of yeast viability is the commercial production of new yeast strains that are more resistant to freeze damage. Finally, it is suggested that yeast content in the dough formula should normally be higher than in conventional breadmaking to overcome the prospective loss of activity during freezing and storage and any inadequacy in proofing conditions.

## Processing Parameters — Requirements and Suggestions

The poor baking performance of frozen dough can be overcome to a great extent through the use of appropriate processing conditions, which aim at the restraint of yeast damage and the enhancement of gluten network ability to retain gas. Mixing duration, dough temperature, and resting after mixing are very important parameters. If dough is undermixed, starch and proteins are unevenly distributed, and when it is overmixed gluten proteins become stressed and partially depolymerised. To minimize yeast activity before freezing, dough temperature after mixing should be slightly lower than conventional (usually between 24 and 26°C) breadmaking, and range between 19 and 22°C. Several researchers suggest that in frozen bakery products, dough resting after mixing should be completely avoided to minimize fermentation before freezing, whereas others consider short rest times (8–10 min) to be beneficial (Kulp, K. *et al.*, 1995).

The influence of sheeting and molding conditions on the stability of frozen dough was not found to be very significant. However, as far as dough shape is concerned, it is believed that round-shaped dough pieces produce less satisfactory bread than slabs and cylinders. Packaging is also very important as it performs a number of functions: it contains, protects, identifies, and merchandizes food products. It should provide an effective barrier to contamination and variable moisture conditions, compressive strength to withstand stresses, and perform satisfactorily during storage and transport. The packaging materials and their shapes may vary according to product specifications but the most popular materials applied to frozen bakery product are plastic (films, membranes, etc.) and aluminium. Films used for frozen dough products should present good oxygen and moisture barrier characteristics, physical strength against brittleness and breakage at low temperature, stiffness to work on automatic machinery, and good heat sealability.

As far as freezing is concerned, reports show that slow rates ( $<2^{\circ}\text{C}/\text{min}$ ) provide higher yeast survival levels and bread scores (Gélinas, P. *et al.*, 1995). It has also been shown that slow freezing at  $-20^{\circ}\text{C}$  is better than freezing at  $-40^{\circ}\text{C}$  (Inoue, Y. and Bushuk, W., 1991). At relatively slow freezing rates, ice is formed outside the yeast cell and can lead to a relative increase in external solute concentration and hence hyperosmotic conditions that cause efflux of intracellular water and relative dehydration. On the contrary, high freezing rates result in the formation of small ice crystals inside yeast cells. This can eventually lead to the rupture of

cell membranes if recrystallization into larger crystals occurs especially during prolonged frozen storage and slow thawing at low temperatures.

However, studies on the influence of both freezing rate and dough geometry show that a high freezing rate and a low yeast activity were observed at the core of cylindrical dough pieces whereas lower freezing rate and a higher yeast activity were noticed at the surface of these samples (Le Bail, A. *et al.*, 1998; Havet, H. and Le Bail, A., 1999). These observations on the freezing rate are in agreement with the heat transfer theory in cylindrical geometry, which demonstrates that the velocity of the freezing front is the lowest at the surface and in the bulk zone and tends to become infinite at the center.

Another factor that should be considered is the influence of storage time and conditions on the gluten structure, which appears to be disrupted during extended storage resulting in dough weakening, loss of gas retention ability, and deterioration of product quality. Temperature fluctuations during storage were shown to be as important as the storage duration.

Thawing should preferably be performed under stepwise temperature increase to avoid the formation of an excessive temperature differential between dough surface and surrounding air, which can cause condensation in the crust and generate crust spotting and blistering. Rapid thawing also results in temperature rising only to the outer surface of the dough, which becomes ready for proofing, whereas the center of the dough still remains frozen. This can lead to unconformable gas cell structure and significantly affects frozen storage stability because when dough comprises a large number of small bubbles with narrow size distribution and thick walls, it appears to be more stable than a dough that contains bubbles with less uniform size distribution (Giannou, V. *et al.*, 2006).

## **Raw Material — Requirements and Suggestions**

The ability of dough to withstand harsh freezing and thawing conditions significantly depends on flour type and protein quality, which are important variables for the stability of frozen dough. A medium to strong flour is recommended for frozen doughs to maintain their ovenspring potential even after losing some intrinsic strength over storage period. Doughs made from strong flours are generally more resistant to freeze damage, and hard red spring (HRS) wheat is preferred for frozen dough because of its superior gluten strength. However, flour protein content is found to be less important than flour protein strength for optimum frozen dough performance. Dough rheology and consequently dough machinability are strongly affected by water content. It is recommended to incorporate less water in the formula

of frozen bakery products to minimize free water in the dough. This is important because free water is responsible for water migration and ice crystal formation, which can be detrimental for yeast cells and gluten proteins and may result in reduced gas retention. The use of chilled water is also preferable as it retards yeast activity and accelerates freezing of dough pieces.

Reports indicate that higher levels of sugars should be used as well because due to their hygroscopic properties they can reduce the amount of free water in frozen dough products and therefore constrain its undesirable effects on dough stability. Besides, sugars are responsible for the osmotic stress of yeast cells during freezing and therefore, specific yeast strains with increased “osmotolerance” are proposed by yeast companies. Salt addition is also important, as it has been found to retard the production of carbon dioxide by the yeast thus delaying dough fermentation. Finally, shortening is considered to improve dough processing and freezing tolerance. More preferably, saturated or partially saturated shortening should be used in frozen bakery products (Giannou, V. *et al.*, 2006).

## **Additives — Requirements and Suggestions**

Additives are used in breadmaking to facilitate processing, confront raw material variations, ensure stability in quality characteristics, and sustain freshness. Especially when incorporated in the frozen dough formula, they are able to counteract with several of the changes occurring during freezing, frozen storage, and thawing. More specifically, they can decrease final proof time, improve dough rheological properties, and increase loaf volume and bread softness. The most frequently used additives in frozen dough products are: oxidative substances (L-ascorbic acid, azidocarbonamide, and potassium bromate), emulsifiers (monoglyceride, sodium or calcium stearoyl-2-lactylate, diacetyl tartaric acid esters of monoglycerides, etc.), enzymes and hydrocolloids (xanthan gum, guar gum, hydroxypropylmethylcellulose (HPMC), k-Carrageenan, etc.).

The use of optimum levels of oxidant ingredients, whether from natural or chemical origin, exerts an improving effect on dough rheology and handling and on the overall quality of the finished product. During mixing, oxidizing agents convert sulfhydryl (SH) groups of the gluten protein to disulfide (SS) linkages between adjacent molecules, building up the gluten matrix and providing a stronger dough. An oxidant also exhibits its improving effect by increasing the loaf volume during the first few minutes of the baking process.

Ascorbic acid is probably the most popular oxidizing agent used in frozen bakery products. It is reported to significantly reduce dough stickiness, decrease extensibility, and increase its

elasticity by inducing intermolecular interactions between dough protein molecules, and consequently increase ovenrise and bread score. The use of potassium bromate is prohibited in most countries even though it shows better performance than ascorbic acid.

Emulsifiers may also be used in the formulation of yeasted frozen dough products. They are commonly added to commercial bread products to improve bread quality and dough handling characteristics and usually result in a foamy crumb with fine and uniformly dispersed cells. Such a foamy crumb is easy to recognize and is more or less accepted by the consumer. The effect of emulsifiers has been extensively studied and numerous references can be found in the literature on this aspect. They are reported to improve mixing tolerance, gas retention, and dough resistance, to increase the loaf volume of the final product and to endow it with resilient texture, fine grain as well as slicing properties.

Monoglyceride (MG) was the first industrial emulsifier; since then other emulsifiers have been developed, some of them requiring the combined presence of “conventional” emulsifiers such as MG. For example propylene glycol monostearate (PGMS), which is an additive typically used in the United States and enhances the efficiency of MG. Polyglycerol esters of fatty acids (PGEs, HLB 3-13) has been patented by “PURATOS” and must be used with MG as well. Diacetyl tartaric acid esters of monoglycerides (DATEMs), acetic acid esters of monoglycerides (AMGs), and lactic acid esters of monoglycerides (LMGs) can be used alone or with other emulsifiers. These emulsifiers can be used as crumb softeners or antistaling agents as they interact with amylose by forming complexes that are known to delay the retrogradation, resulting in the staling of bread. They also interact with lipids and shortening, resulting in a foamy structure with refined cells (i.e., sandwich bread). Sodium stearoyl lactylate (SSL) has also been shown to be effective in maintaining both volume and crumb softness and provide longer shelf-life stability in fresh and frozen dough products subjected to extended storage.

Enzymes such as amylases, proteases, hemicellulases, lipases, and oxidases can be naturally present in foods such as wheat and soy products (as long as they are not removed or inactivated) or incorporated in them as additives. They have been shown to influence the entire breadmaking process by decolorizing (bleaching) dough, improving dough volume and texture, and maintaining shelf-life by extending product freshness during storage. Their effects on frozen dough products can be seriously enhanced when used in combination with selected oxidants such as ascorbic acid. Transglutaminase is also reported to significantly improve the cohesion of the gluten network and seems to be well adapted in the puffing pastry.

The supplementation of vital gluten to relatively weak doughs improves the mixing tolerance and stability of dough, resulting in increased loaf volume, improved crumb texture and softness, and prolonged shelf-life of bakery products. As gluten is a protein, it also enhances nutritional value of the products. The embedment of pentosans is particularly important as well in breadmaking because of their physical properties. They improve dough machinability, increase bread volume, and decrease the rate of starch recrystallization, and therefore reduce staling rate of baked bread.

The incorporation of cryoprotective materials in frozen bakery products is increasingly becoming appealing. Cryoprotectants are substances, which have the ability to protect protein cells from chemical changes and loss of functionality during freezing or thawing and consequently improve quality and extend the shelf-life of frozen foods. Cryoprotective agents may include sugars, amino acids, polyols, methyl amines, carbohydrates, some proteins, and inorganic salts such as potassium phosphate and ammonium sulfate (Giannou, V. *et al.*, 2006).

One group of the most extensively used additives in the food industry is the hydrocolloids. These compounds are capable of controlling both the rheology and texture of aqueous systems through stabilization of emulsions, suspensions and foams. In the baking industry, hydrocolloids are of increasing importance as bread improvers as they can induce structural changes in the main components of wheat flour systems along the breadmaking steps and bread storage.

Such structural changes modify the selectivity of some enzymes and change the technological quality of dough and bread. Hydrocolloids affect the baking performance of dough and also the shelf-life of stored bread. The presence of hydrocolloids influences melting, gelatinization, fragmentation and retrogradation processes of starch. These effects were shown to affect the pasting properties and rheological behaviour of dough. The synergism between hydrocolloids and starch may be due to the formation of complexes between the starch polymers, i.e. amylose and/or amylopectin, and the hydrocolloids during pasting.

When used in small quantities (< 1% w/w in flour) in dough, hydrocolloids are expected to increase water retention and loaf volume, as well as to decrease firmness and starch retrogradation. The addition of hydrocolloids into frozen products can provide stability during freeze-thaw cycles and help to minimize the negative effects of freezing and frozen storage on starch-based products .

It also decreases water activity due to the competition for water by the hydrocolloids with the bread polymers like protein and starch. The overall effects on the functional performance of



dough and the subsequent bread quality, however, depend on the nature, origin and particle size of the principal components, dosage of the hydrocolloids incorporated into dough, as well as the formulation, processing condition and other ingredients (Selemulyo, V.O. and Zhou, W., 2007).

## Quality and safety control

The most common quality defects, which appear in frozen bakery products, the cause of their existence are presented in Table 1.1 (Giannou, V. *et al.*, 2006).

Cause	Defect
<b>Raw materials</b>	
<i>Flour</i>	
Very weak	Coarse crumb Small volume Inadequate shape Unsatisfactory crumb color
Very strong	Small volume Inadequate shape Unsatisfactory crumb color Lechy crumb
<i>Yeast</i>	
Smaller amount	Small volume Excessively dark crust color Lechy crumb
Larger amount	Small volume Unsatisfactory crust/crumb color Inadequate shape Excessive crumb brittleness Skinning Fissures on crust White spots or blisters
<i>Salt</i>	
Smaller amount	Poor crust/crumb color Excessive crumb brittleness Excessive volume
Larger amount	Lechy crumb Excessively dark crust color Small volume
<i>Water</i>	
Smaller amount	Small volume Inadequate shape Crust-crumb separation Excessive crumb brittleness Lechy crumb Unsatisfactory crumb color
Larger amount	Coarse crumb Unsatisfactory crumb color Small volume Inadequate shape
<i>Sugar</i> (larger amount)	Dark crust color

---

*Continued*

**Processing**

*Mixing*

Restricted

Crumb fissures  
Lechy crumb  
Unsatisfactory crumb color

Excessive

Lechy crumb  
Excessive crumb brittleness

---

*Molding (inadequate)*

Lechy crumb  
Large blisters either on the surface causing shape  
distorsion or under the top crust

---

*Freezing (very slow)*

Crust fissures  
Uneven or open cell structure

---

*Storage (very long)*

White spots and blisters  
Patches of uneven color on the side  
and bottom crusts of breads

---

*Proofing/thawing*

Very slow

White spots and blisters  
Small volume

At higher temperatures

Inadequate shape  
Areas of dense crumb  
Excessive volume  
Ragged crust breaks  
Overlapping top  
Thick and hard crust

---

*Baking*

At lower temperatures

Coarse/Lechy crumb  
Unsatisfactory crumb/crust color  
Excessive dough volume

At higher temperature

Coarse/Lechy crumb  
Small volume  
Excessively dark crust color  
Inadequate shape

---

Table 1.1 Quality defects in frozen bakery products (modified from Giannou, V. *et al.*, 2006).

## 2. RHEOLOGICAL TESTS

*Rheology* is the study of how materials deform, flow, or fail when force is applied (Hoseney, R.C., 1994).

The name is derivated from Greek: rheos- the river, flowing, streaming, e.g. the Greek philosopher Heraklit of Ephesos (about 540 to 480 BC) thaught: “phanta rei”- everything is flowing.

Therefore rheology means “flow science” rheological investigations not only include flow behaviour of liquids, but also deformation behaviour of solids.

The first Rheological Society was founded in 1929 in the USA by Eugen C. Bingham (1878 to 1945) and M. Reiner.

Therefore only since the 20 century rheology has been an independent science, but earlier already many scientist worked more or less scientifically on the behaviour of liquids and solids. The targets of rheologists are measurement, characterization and interpretation of the flow and deformation behaviour of materials.

A controlled, well-defined deformation or strain is applied to a material over a given time and the resulting force response is measured (or vice versa) to give an indication of material parameters such as stiffness, modulus, viscosity, hardness, strength or toughness of the material (Dobraszczyk, B.J., 2003).

The rheological properties of some materials can be described by a single value. For example, the flow of water is defined by its viscosity. The deformability of a steel spring is defined by Hooke’s constant (a modulus of elasticity). However, most materials, and certainly doughs and butters, are not that simple in their properties or behaviours but show a more complex rheological behaviour.

If a material’s viscosity is constant regardless of shear rate (the rate of stirring or flowing through the pipe), the material is said to show *Newtonian* or *ideal viscosity*. Its behaviour then can be defined by a single viscosity value. However, in many systems, including most flour-water systems, the viscosity changes (decrease) as the shear rate is increased. Thus, the system shows more complicated *non-Newtonian* behaviour, and we cannot define the system by a single viscosity value but must give a viscosity at each shear rate. In addition, viscosity can also be affected by the time involved in making the measurement.

There are many different kinds of moduli. In general, modulus refers to the stiffness of the material and is a proportionality constant relating stress or strain. In lay terms, it tells how much force is required to produce a specific deformation of the material under test.

In the cereal chemistry literature, we often see measurements taken with a farinograph or mixograph referred to as rheological measurements. Certainly, these instruments measure how doughs deform and flow. The problem with the use of these instrument for rheological studies is that we cannot define the stress on the sample at any moment of time during the test. For example, in a mixograph bowl, only a small part of the dough is in contact with a pin at any given time, and the shape of the sample (dough) changes in a very complicated and unpredictable ways. Thus, it is impossible to determine the stress on the dough, as we do not know the geometry of our test piece. As a result, the measurement made using a mixograph are valid only for the mixograph, and measurements made using the farinograph are relevant only to the farinograph.

This is not to say that the above instruments are not useful. They have stood the test of time and can give much useful information. They are particularly useful when used to characterise or “fingerprint” a flour. We often want to know whether the mixing properties of the flour we are using today are similar to or different from those of the flour we used yesterday. The mixograph or farinograph can easily and rapidly answer that question for us.

Instead from fundamental measurements we can learn more about our dough or batter. We can see the effect of various interactions and how the properties of the dough or batter change as a function of time or temperature. In addition, the measurement can often be made on the complete dough or batter system so the results are relatively easy to interpret. Another reason is that, with the advent of minicomputers and their related equipment, it has now become much easier to obtain good rheological data (Hoseney, R.C., 1994).

Besides the rheological properties should be independent of size, shape and how they are measured; in other words, they are universal, rather like the speed of light or density of water, which do not depend on how much light or water is being measured or how it is being measured. It would be comforting to know that the stiffness of bread or viscosity of dough measured in a laboratory in Reading will be the same measured in any laboratory in the world, even if they are measured using different tests, sample sizes or shapes. The whole point of the rheological approach is that the properties that are measured are reproducible and can be compared between different samples, test sizes and shapes, and test methods (Dobraszczyk, B.J., 2003).

## **2.1 Rheological test methods**

There are many test methods used to measure rheological properties. It is not possible to describe all the available testing geometries here, and the reader is referred to general reviews of rheology (Ferry, J.D., 1980; Barnes, H.A. *et al.*, 1989; Whorlow, R.W., 1992), rheological testing of foods (Sherman, P., 1970; Carter, R.E., 1990; Rao, M.A. and Steffe, J.F., 1992; Dobraszczyk, B.J. and Vincent, J.F.V., 1999; Van Vliet, T., 1999) and cereal products (Bloksma, A.H. and Bushuk, W., 1988; Faridi, H. and Faubion, J.M., 1986, 1990; Muller, H.G., 1975). It is common to categorise rheological techniques according to the type of strain imposed: e.g. compression, extension, shear, torsion, and also the relative magnitude of the imposed deformation, e.g. small or large deformation.

The main techniques used for measuring cereal properties have traditionally been divided into descriptive empirical techniques and fundamental measurements (Dobraszczyk, B.J., 2003).

### ***2.1.1 Descriptive empirical measurements***

Within the cereals industry there has been a long history of using descriptive empirical measurements of rheological properties, with an impressive array of ingenious devices such as the Penetrometer, Texturometer, Consistometer, Amylograph, Farinograph, Mixograph, Extensigraph, Alveograph, various flow viscometers and fermentation recording devices, reviewed by Muller (1975) and Shuey (1975) (Table 2.1).

Method	Products	Property measured
<b>Empirical Methods</b>		
Mixers:	Dough	Mixing time/torque
Farinograph		Apparent Viscosity
Mixograph		
Reomixer		
Extensigraph	Dough	Extensibility
TAXT2/Kieffer rig	Dough/gluten	Extensibility
Alveograph	Dough/gluten	Biaxial extensibility
Amylograph RVA	Pastes, suspensions	Apparent Viscosity, Gelatinisation temperature
Consistometer	Sauces, fillings	Apparent Viscosity
Flow cup	Fluids, sauces, batters	Apparent Viscosity
Falling ball	Fluids	Apparent Viscosity
Flow viscosimeters	Fluids, pastes	Apparent Viscosity
Fermentometers	Dough	Height, volume
Penetrometers	Semi-solid foods, gels	Firmness, hardness
Texturometer, TPA		Texture, firmness
<b>Fundamental methods</b>		
Dynamic oscillation		
Concentric cylinders	Fluids, pastes, batters	Dynamic shear moduli
Parallel plates	Doughs	dynamic viscosity
Tube viscometers:		
Capillary	Fluids	Viscosity
Pressure, extrusion	Sauces, pastes, dough	Viscosity
Pipe flow		In-line viscosity
Transient flow:		
Concentric cylinders	Semi-solid viscoelastic	Creep relaxation
Parallel plates	material	moduli and time
Extrusion:		
Uniaxial, biaxial		Extensional viscosity
TAXT2 dough		strain hardening
Inflation system		
Lubricated compression		

RVA= Rapid Visco-Analyser, Newport Scientific Pty., Australia.

TPA= Texture Profile Analysis.

Table 2.1 Rheological methods used for cereal products (modified from Dobraszczyk, B.J., 2003).

Empirical tests are easy to perform and are often used in practical factory situations, providing data that are useful in evaluating performance during processing and for quality control. The instruments are often robust and capable of withstanding demanding factory environments, and do not require highly skilled or technically trained personnel. Simply because they do not provide data in fundamental units does not mean that these tests are worthless: indeed, they have provided a great deal of information on the quality and performance of cereal products such as consistency, hardness, texture, viscosity, etc. However, these measurements are not strictly “rheological” tests since:

- the sample geometry is variable and not well defined;
- the stress and strain states are uncontrolled, complex and non-uniform and
- it is not possible to define any rheological parameters such as stress, strain, strain rate, modulus or viscosity.

Therefore, these tests are purely descriptive and dependent on the type of instrument, size and geometry of the test sample and the specific conditions under which the test was performed. For example, empirical tests have been used to characterise the behaviour of bread doughs during processing, such as the Farinograph and Mixograph. Many of these are used as “single point” tests, where a single parameter is often arbitrarily selected from a whole range of data acquired during the test as, for example, in selecting the peak torque from a mixing trace and then using this to correlate with performance. This neglects a large part of the recorded data, and is appropriate only to the set of conditions under which that test was performed and is generally not applicable to any other deformation conditions. Since dough experiences a wide range of conditions of stress states and strain rates during processing and baking, and the rheological properties of dough are dependent both on time and strain, there is often a discrepancy between such single point type tests and actual performance on the plant, where conditions of strain and strain rate may be poorly defined and very different from those in the laboratory test. While this may give the illusion of a “scientific” test by being performed on a machine (frequently with a computer attached), and may give satisfactory correlations with a textural or processing parameter, it is impossible to compare results between different testing machines, or to extrapolate the results to other deformation conditions.

Most food materials are viscoelastic and therefore their properties depend on how quickly the test is performed (the strain rate or frequency). This is important in many aspects of dough processing: if the dough is deformed quickly, such as in mixing or sheeting, then the rheological properties of the dough will be very different if measured at the typically slower rates of deformation found in conventional testing machines. Alternatively, during processing



dough will experience strains very different in magnitude and nature than those generally available in a rheological test. Many food processes operate under extensional flow, while most rheological tests on foods are performed in shear. Tests under only one particular set of conditions of rate, temperature and strain will almost certainly not be applicable to another set of deformation conditions. What is necessary is to define the set of deformation conditions that the food endures in practice and perform tests under similar conditions (Dobraszczyk, B.J., 2003).

### **Texture Profile Analysis (TPA test)**

Instrumental Texture Profile Analysis (TPA) was developed about 30 years ago, constituting an interesting way of analyzing a series of textural parameters in only one test.

Together with texture, appearance and flavour are the principal sensory factors that determine food acceptability for consumers (Bourne, M.C., 1978). Thus, the development of objective, precise experimental methods that allow the study of mechanical properties of foods related to texture has been an important target for food technology in recent decades.

In recent decades, much work has been done on the development of mechanical tests that tried to imitate the sensory evaluation of food texture (Peleg, M., 1983).

Among these one must mention instrumental Texture Profile Analysis (TPA), which has been applied as a useful method for a wide range of foods. However, it is often applied without knowing the correct definition of its parameters or selecting suitable experimental conditions.

### **Historical background**

The origin of TPA was Szczesniak's classification of textural characteristics (Szczesniak, A.S., 1963) the key principle of which was to serve as a bridge between the instrumental and sensory evaluation of texture. The use of the General Foods (GF) Texturometer by Szczesniak's research team was the major breakthrough in instrumental Texture Profile Analysis. This equipment, which was a modification of the original MIT Denture Tenderometer (Proctor, B.E. *et al.*, 1956a,b) was designed to simulate the masticating action of the human mouth and can be described as a unit composed of a plate supported by a flexible arm attached to a strain gauge, and a plunger which acted upon the food sample, usually a bite-size piece, compressing it twice in a reciprocating motion that imitated the

action of the jaw. The strain gauge detected the force generated, which was recorded on a strip chart recorder.

The curve generated by the GF Texturometer is a plot of force as a function of time. The curve provided seven textural parameters, five measured and two calculated from the measured one (Figure 2.1). These parameters were named hardness, cohesiveness, elasticity, adhesiveness, brittleness, chewiness, gumminess and viscosity (Friedman, H.H. *et al.*, 1963). Szczesniak (1963) evaluated selected foods for GF TPA parameters, reporting that correlation was good between the sensory and instrumental evaluations. Cohesiveness and elasticity values can be obtained from Texturometer curves, but these parameters were not included in the early organoleptic evaluations because they were very difficult to perceive as such.

$$\text{Cohesiveness} = \frac{A_2}{A_1}$$

$$\text{Elasticity} = C - B$$

C = Time constant for clay

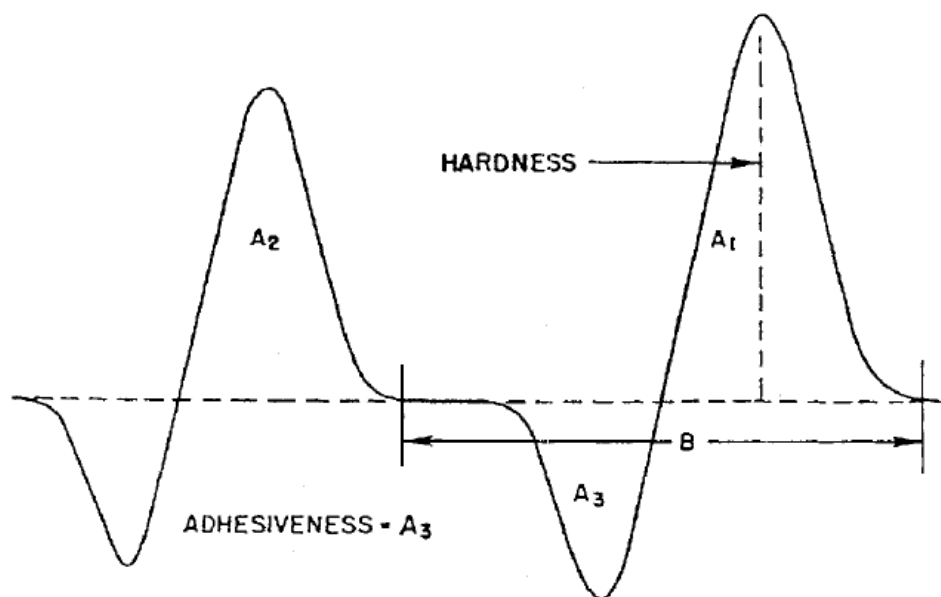


Figure 2.1 A typical GF texturometer curve, which should be read from right to left, with peak  $A_1$  being the first chew, and peak  $A_2$  the second chew (modified from Friedman, H.H. *et al.*, 1963).

A very important milestone was the adaptation of the Instron Universal Testing Machine to food studies described by Bourne (1966). In a later paper (Bourne, M.C., 1968), this author demonstrated the first application of the Instron to objective Texture Profile Analysis of a food (ripening pears). He argued that the Instron was a better tool for determining TPA parameters than the GF Texturometer for several main reasons:

- (1) The speed of Instron compression is constant at all times during the downstroke. This and the immediate reversal of the compression stroke at the end of the first bite result in sharp peaks in the curves.
- (2) The plunger moves in a strictly rectilinear manner, so the plunger area in contact with the food is constant throughout the compression, assuming sample surface is flat and there is no slippage at the interface.
- (3) The Instron chart also gives a force-time curve but, in contrast to the Texturometer, it is also a force-distance curve. Therefore, the Instron curve areas are force-time and also force-distance integrals, so that these areas are true measurements of work.

Bourne followed the individual parameters of Friedman (1963) fairly closely, except in the definition of cohesiveness. Drake (1966), who worked with the Masticometer instrument, established that the area recorded when the plunger moves upwards represents work delivered by the sample. From a physical point of view it should be taken with a negative sign, i.e., it should be subtracted from the area recorded during the downward movement of the plunger. Bourne (1968) also noticed that there was no distinction for work done by the sample to the instrument during decompression, so he proposed that this work should be separated from the work performed on the sample by the instrument, ignoring this part of the area for the calculation of cohesiveness. Olkku and Rha (1975) and Peleg (1976) followed Drake's suggestion, subtracting the decompression area from the compression area to give the net work done in the two compression cycles. Some years later, Bourne (1978) again listed the seven textural parameters, with the modifications introduced since they were first published (Szczesniak, A.S., 1975a), as follows (Figure 2.2):

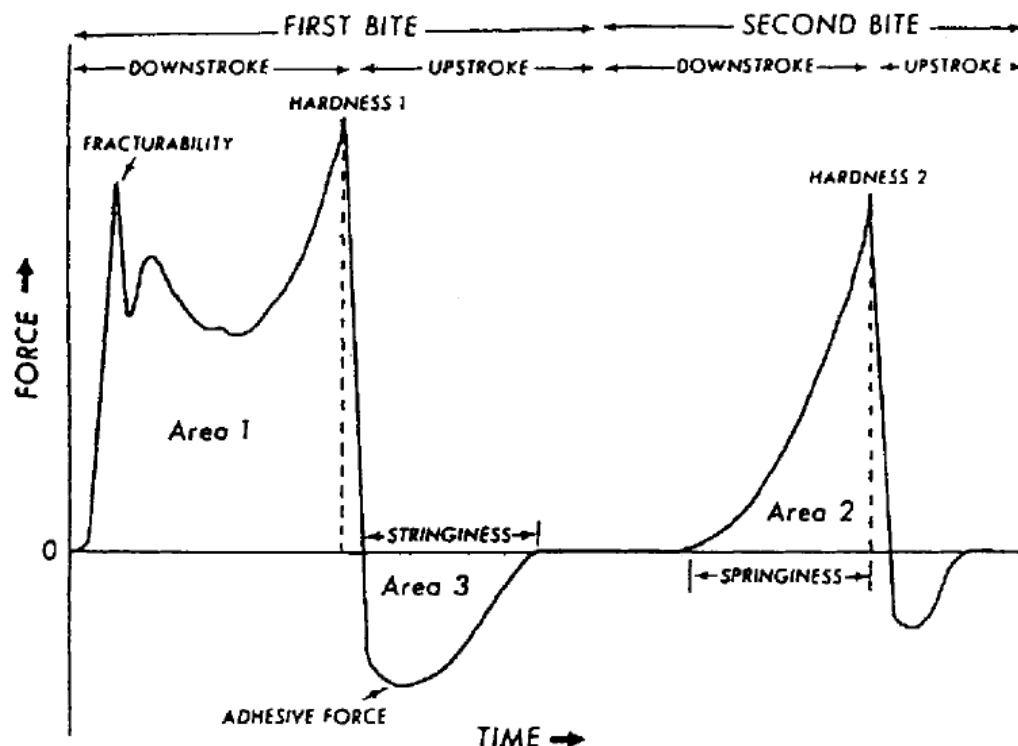


Figure 2.2 A generalized TPA curve from the Instron universal testing machine (modified from Bourne, M.C., 1978).

- (1) Fracturability (originally called brittleness) is defined as “the force at the first significant break in the curve”. As a break is a visible phenomenon related to the macrostructure of the sample, it must be identified as a change in the inflection of the curve whose magnitude must be defined.
- (2) Hardness is defined as “the peak force during the first compression cycle” (“first bite”).
- (3) Cohesiveness is defined as “the ratio of the positive force area during the second compression portion to that during the first compression ( $\text{Area}_2/\text{Area}_1$ ), excluding the areas under the decompression portion in each cycle”.
- (4) Adhesiveness is defined as “the negative force area for the first bite, representing the work necessary to pull the plunger away from the food sample”.
- (5) Springiness (originally called elasticity) is defined as “the height that the food recovers during the time that elapses between the end of the first bite and the start of the second bite”.
- (6) Gumminess is defined as “the product of hardness  $\times$  cohesiveness”.
- (7) Chewiness is defined as “the product of gumminess  $\times$  springiness” (which is equivalent to hardness  $\times$  cohesiveness  $\times$  springiness).

Table 2.2 shows the units of the seven texture profile parameters.

<b>Mechanical parametr</b>	<b>Measured variable</b>	<b>Units</b>
Hardness	Force	$\text{m.l.t}^{-2}$
Cohesiveness	Ratio	dimensionless
Springiness	Distance	l
Adhesiveness	Work	$\text{m.l.}^2\text{t}^{-2}$
Fracturability	Force	$\text{m.l.t}^{-2}$
Gumminess	Force	$\text{m.l.t}^{-2}$
Chewiness	Work	$\text{m.l.}^2\text{t}^{-2}$

Table 2.2 Parameter units of instrumental Textural Profile Analysis (modified from Pons, M. and Fiszman, S.M., 1996).

Bourne's procedure has been the basis for practically all subsequent instrumental TPA studies using the Instron, which has been one of the most popular choices of instrumentation for objective Texture Profile Analysis in further studies. Nowadays, in TPA records fully automated computer readout of data from force-distance point pairs is used, allowing selection of predetermined forces, distances, and areas to be used by the computer in subsequent calculations of TPA parameters.

In modern computer-assisted texturometers which provide great versatility, e.g. the TA-XT2 Texture Analyser (SMS), it is possible to perform TPA tests and directly obtain all TPA parameters by means of its software, without any previous selection of curve values for calculations. However, depending on the type of sample, such a degree of automation is not always advisable. In all cases, a profound knowledge of the physical basis of each parameter will avoid misleading results.

## **Testing conditions**

In most cases, the conditions reported for obtaining TPA parameters are very different for different workers, making a comparison of results impossible. The differences are due to experimental conditions such as sample size and shape, ratio of compressing probe size versus sample, extent of deformation, crosshead speed, number of bites, and replicates per mean value.

### **Sample Size and Shape.**

In general, the samples were sampled by cutting standard-sized cubes or cylinders, or by allowing the samples to set into moulds of standard diameter and height.

### **Size of Compressing Unit Versus Sample.**

The size of the compressing unit (sometimes called “punch,” “plunger” or “probe”) is also very important, especially in relation to sample size. This ratio certainly determines the magnitude of the results. Thus, when the probe is larger than the sample, the forces registered are largely due to uniaxial compression. However, when the opposite is true, the forces derive largely from puncture, a combination of compression and shear. Bourne (1968) was the first to adopt true uniaxial compression to perform TPA with an Instron. This experimental condition was subsequently used by other authors working with the same instrument.

Generally speaking, most recent work done on TPA normally uses compression devices larger than the sample size, so that the forces registered in TPA tests are largely due to uniaxial compression forces.

### **Extent of Deformation (Percent deformation)**

The General Foods team used up to 88% deformation in TPA tests on strawberries (Szczesniak, A.S. and Smith, B.J., 1969), precooked freeze-dried beef (Szczesniak, A.S., 1971), cheese and dessert gel systems (Szczesniak, A.S. and Hall, B.J., 1975). In general, 75-80% deformation has been the choice of most authors who have worked on fruits, vegetables and meats. This may be because they had to compress to a larger degree in order to obtain the brittle fracture peak. The choice of extent of deformation will depend upon the purpose of the

test. If it is to imitate the highly destructive process of mastication in the mouth, as in the TPA's origins, deformation values to break the gel system must be reached. Usually, for example in gel type systems, compressions greater than 70-80% completely break the gel sample. The second compression cycle does not usually find a weakened sample with just the first internal cracks (like natural turgent vegetables), but portions or small pieces of the initial sample. Deformation levels between 20-50% have commonly been applied in recent works on gel food systems. At these levels, samples do not break (a brittleness peak does not appear in the curve) but it is still possible to obtain valuable information on important parameters such as hardness (force at a given deformation), springiness, cohesiveness and their derivatives gumminess or chewiness.

### **Cross-head Speed**

Shama and Sherman (1973) were the first to show that, as the cross-head speed increases, the force required to achieve a particular compression also increases. They stated that since humans use different forces and chewing rates depending on the textural characteristics of the food evaluated, both the magnitude of the force applied and the rate at which it is applied must be taken into account when testing conditions are established in a texturometer, aiming to reproduce those associated with the sensory evaluation of the specific type of food, as also stated by Szczesniak and Hall (1975).

### **Time Elapsed Between Bites**

Little information is available on this topic. Current machines such as the TA.XT2 (Stable Micro Systems-SMS) give the option of selecting a variable time period to elapse between bites. In any case, if a time period is selected, it should be specified in the testing conditions, since the amount of time between bites clearly determines TPA parameters such as springiness, cohesiveness, gumminess and chewiness, mainly in those systems with a high viscous component.

## **Effect of Lubrication**

To obtain real information on the mechanical properties of foods, the results should be reproducible, and the parameters describing the materials, as determined experimentally, should be independent of testing conditions or sample dimensions. However, use of unlubricated plates in compression tests, can produce frictional phenomena at the sample-plate interface, resulting in non reproducible data. Also, compression data with unlubricated plates will yield parameters that depend both on material bulk properties and on the frictional characteristics of the sample-plate interface. This means that the force-deformation curves will depend on sample geometry and, in particular, on the area of contact between sample and plates.

## **TPA terminology**

As has been said, the pioneering work of Szczesniak (1963) named the TPA parameters as hardness, cohesiveness, elasticity, adhesiveness, brittleness, chewiness and gumminess (Figure 2.1). Nevertheless, several authors have examined them critically and proposed several modifications to their names or definitions.

### **Hardness**

This parameter was originally defined by Szczesniak (1963) as “the force needed to attain a given deformation”. Sherman (1969) criticized the use of the term “hardness” and proposed “firmness” in relation to a given deformation. This new term was adopted by a few authors performing instrumental TPA (Henry, W.F. *et al.*, 1971; Shama, F. and Sherman, P., 1973). It should be mentioned that the term firmness was already part of the soft ↔ hard scale in Szczesniak’s sensorial classification.

### **Springiness**

This parameter was originally named “elasticity” by Szczesniak (1963) and defined as “the rate at which a deformed material goes back to its undeformed condition after the deforming force is removed”. Due to sharp edges and relative insensitivity of the teeth, difficulties in the sensory evaluation of this parameter were found and no standard scale was constructed



(Szczesniak, A.S. *et al.*, 1963). This definition was adapted in the GF Texturometer as “the difference between the distance B, measured from the initial sample contact to the contact on the second chew, and the distance C, the same measurement made on a completely inelastic standard material such as clay” (Figure 2.1).

Afterwards, Szczesniak replaced the term “elasticity” with “springiness.” This substitution was made to avoid confusion with the well defined rheological term “modulus of elasticity,” and in fact this new term is widely accepted today (Szczesniak, A.S., 1975a). Springiness is measured several ways, but most typically, by the distance of the detected height of the product on the second compression (length 2 ), as divided by the original compression distance (length 1). The original definition of springiness used the length 2 only and the units were in mm or other units of distance.

### **Cohesiveness**

Szczesniak (1963) defined this parameter as “the strength of the internal bonds making up the body of the product”. In the TPA curve, it is measured as “the ratio of the area under the second peak and the area under the first peak ( $A_2/A_1$ )” (Figure 2.1) (Friedman, H.H. *et al.*, 1963). It is worth noting that these authors had also indicated that if the food sample exhibits adhesiveness, it is necessary to coat the material with talcum powder before recording the cohesiveness profile, because of the distortion created by the negative adhesiveness peak on the start of the second curve. As already stated, the first paper in which Bourne (1968) adapted the TPA methodology to an Instron suggested the use in both compression cycles of the areas corresponding to the downstroke for calculating cohesiveness. This modification has since been adopted by most authors. Some authors subtracted the decompression area from the compression area to give net work done in the two compression cycles (Drake, B., 1966; Olkku, J. and Rha, C.K., 1975; Peleg, M., 1976) (Figure 2.3 II).

It is interesting to note that cohesiveness was originally found difficult to perceive and evaluate as such by a sensory panel (Szczesniak, A.S. *et al.*, 1963). This is probably because this parameter does not correspond to a single sensation such as hardness, brittleness, etc., but to a complex one.

### **Fracturability**

Szczesniak (1963) originally named this parameter brittleness and defined it as “the force with which the material fractures,” related to the primary parameters hardness and cohesiveness. In

brittle materials, cohesiveness is low whereas hardness can vary. This parameter was measured in instrumental TPA as “the height of the first significant break in the first peak” (Friedman, H.H. *et al.*, 1963), which implies multippeak recording. Later, the term “brittleness” was changed by Szczesniak (1975a) to “fracturability,” which is now accepted almost universally. This parameter is principally obtained in turgid vegetable tissues, such as fruits and vegetables, and also in low moisture farinaceous products (e.g., raw noodles, crisp breads, toast).

### **Adhesiveness**

Szczesniak (1963) defined this term as “the work necessary to overcome the attractive forces between the surface of the food and the surface of other materials with which the food comes in contact (e.g., tongue, teeth, palate, etc.), so, adhesiveness is related to surface properties. Analyses of some results indicated that the presence of saliva during human mastication could be a decisive factor, which obviously modifies the magnitudes of the mechanical parameter. Bourne (1968) noted that adhesiveness could be considered as zero in pears, as in most fresh fruits and vegetables, and in low moisture farinaceous products

### **Gumminess and Chewiness**

Szczesniak (1963) stated that gumminess is “the energy required to disintegrate a semisolid food product to a state ready for swallowing, and it is related to the primary parameters of hardness and cohesiveness”. As can be seen, this secondary parameter only refers to semisolid materials in which hardness is low. Szczesniak (1963) stated that chewiness is “the energy required to masticate a solid food product to a state ready for swallowing, and it is related to the primary parameters of hardness, cohesiveness, and elasticity”. In the TPA curve produced by the GF Texturometer, this parameter was expressed mathematically as “the product of hardness, cohesiveness and elasticity” (Friedman, H.H. *et al.*, 1963). This research group at General Foods also found a high level of correlation for this parameter between instrumental and sensory evaluation.

Chewiness was judged sensorially as the length of time in seconds required to masticate a sample, at the rate of one chew per second, in order to reduce it to a satisfactory consistency for swallowing (Szczesniak, A.S. *et al.*, 1963). In his key paper describing instrumental TPA, Bourne (1978) failed to make clear that gumminess and chewiness are mutually exclusive and should not both be reported for the same product.

## Modulus of Deformability

Even though no modulus was defined by Szczesniak's research team for TPA, in some of the papers reviewed a parameter called "modulus" is measured, being defined as the initial slope of the force-deformation curve before failure point in the first bite. This is a measure of how the sample behaves when it is compressed to a small degree of deformation (Breene, W.M., 1975). In food texture evaluation, instrumental methods such as TPA subject food samples to large deformations (10-80%), so that the parameters obtained are not in conformity with well-defined fundamental rheological parameters, such as the "modulus of elasticity" (also known as Young's modulus), a fundamental rheological parameter which is the ratio of stress to strain within the elastic range of the material.

Since this fundamental parameter is concerned with the elastic range of the material, Mohsenin and Mittal (1977) suggested that the term "modulus of deformability" should be used instead of modulus of elasticity, avoiding the confusion of taking it as an indication of the degree of elasticity of the food material. Thus, the modulus of deformability is actually an indication of the rigidity or stiffness of the material, where total deformation at selected points on the stress-strain curve is taken into account for its calculation.

Recent papers on TPA indicate that it is better to derive the true stress-strain data from the force-deformation curve during the initial compression cycle to calculate this "modulus of deformability" (Konstance, R.P., 1993; Tang, Q. *et al.*, 1995), employing the following equations:

$$\text{True strain} = \varepsilon_T = \ln(h_0/h_0 - \Delta h)$$

and

$$\text{True stress} = \sigma_T = F_t/A_0 * h_0 - \Delta h/h_0$$

where  $h_0$  = original height of the sample,  $\Delta h$  = change in height during compression (e.g. deformation),  $F_t$  = compressive force at time  $t$ , and  $A_0$  = original cross-sectional area of the sample.

The use of true strain (also called Hencky's strain) for large deformations is appropriate for most food applications, since the cross-sectional area is considerably expanded during compression and therefore cannot even be approximated by the original area (Peleg, M., 1987). True stress represents an adjustment of engineering stress ( $F_t/A_0$ ) to account for the

expansion of the deformed specimen cross-sectional area. The ratio of true stress to true strain gives the “modulus of deformability” for compression,  $E_d = \sigma_T / \epsilon_T$ . In this context, Tang *et al.* (1995) studied the effects of pH on the properties of gels prepared with whey protein concentrate by compression (TPA) in an Instron. They evaluated hardness, cohesiveness and springiness from the double compression curves obtained, and also the parameter which the authors called the “modulus of elasticity,” using the true compressive stress and strain at 10% compression during the first compression cycle. They concluded that the obtained “modulus of elasticity” correlated quite well with  $G'$ , the storage modulus obtained from oscillatory shear testing with a Bohlin Rheometer. Reinforcing what was stated by Calzada and Peleg (1978), the present paper emphasizes that true stress-strain curves will provide a better indication of the true nature of the food’s mechanical properties than apparent curves (force-deformation).

In this way it has been shown that different solid foods can have characteristic “rheological fingerprints”, which are expressed in the form of the true stress-strain relationship. These forms, although affected to a certain degree by the test conditions, are mainly determined by the deformation mechanism of the food material. It will be an interesting topic for future research to associate sensory parameters with these types of fundamental mechanical properties (e.g. compressibility), as a result of which correct interpretation of the true stress-strain relationships would become an essential step in food texture evaluation (Pons M. and Fiszman, S.M., 1996).

## **Dough stickiness**

The measurement of adhesiveness in foods is a matter that remains unresolved. This situation is partly due to the fact that both the manifestation of this characteristic and its sensory perception vary according to the food in question, with the consequence that there is no universally accepted standard system for measuring stickiness within the food industry. In some cases the adhesiveness of foods presents a problem; for example, in relation to a disagreeable sensation in the mouth when they are consumed, or the equipment with which they are handled, or the materials in which they are packaged. Remarkably few of the works published study adhesiveness to packaging materials. In some foods, a certain degree of adhesiveness is desirable and is what the consumer expects of them, depending on cultural factors and different forms of presentation and consumption, for example, in products such as cooked rice, pasta, certain cheeses, or sweets. Kilcast and Roberts (1998) published an interesting paper in which they reviewed numerous questions, such as: (1) definitions of the texture terms adhesiveness, stickiness, and tack, including information drawn from the field of adhesives and international regulatory organizations, (2) how consumers perceive stickiness, in terms of tactile as well as oral sensation, and (3) physical aspects of stickiness, including surface energetics, sample rheology and failure mechanisms. Of the two most important instrumental methods for measuring stickiness in foods, these authors found that probe tests are used more frequently, while peel tests have not been widely used. Hoskeney and Smewing (1999) recognized that in most food systems, the adhesion force is a combination of an adhesive force and a cohesive force. A food material is perceived as being sticky when the adhesive force is high and the cohesive force is low. The authors emphasized that in order to measure adhesive properties it is imperative to have a procedure that forces a clean separation at the probe-material interface. Many authors measure adhesiveness in foods by applying probe tests and calculating the negative area of a force-time curve, either in Texture Profile Analysis (TPA) tests, with a double cycle, or in single-cycle trials. A certain concern about differences in results when different experimental conditions are used in the application of TPA was expressed by Walstra and Peleg (1991) in a paper on the texture of cheeses. The authors declared that this method can detect the existence of textural differences between samples, particularly when these differences are large; this, however, does not mean that the magnitude of the observed differences is of any significance, or that identical or similar readings are an indication that the corresponding samples have the same or similar texture.

With the increasing precision and versatility of modern instrumentation, TPA is a very useful tool for determining instrumental texture parameters. Naturally, attention must be paid to proper selection of testing and sampling conditions in order to obtain reliable data; a knowledge of the philosophy of the method, its terminology, and how the parameters are calculated permit good interpretation of results. It is clear that not all foods possess adhesiveness, hence it is not worth measuring adhesiveness in all cases. The foods in which this property is important and in which it has been studied most can be classified into four main groups: bakery and cereal-derived products, cheese, gelled system, and meat protein products (Fiszman S.M. and Damasio, M.H., 2000).

Before the problems associated with measuring stickiness can be discussed it is necessary to first define what stickiness means. Stickiness is the force of adhesion that results when two surfaces are contacted with each other. Given that simple definition the measurement of stickiness would appear straight forward. Only the area of contact would have to be controlled. Unfortunately, it is often not that simple. An example of one problem is the case of two polished surfaces: there is no adhesion if the surfaces are dry but the force is large if the surfaces are wet. To experience this phenomenon try to pull apart two microscope slides after a drop of water has been added to the surfaces. The surface tension of the water is responsible for the adhesive force. Thus, it appears obvious that factors that affect surface tension of water will have a large effect on the stickiness of aqueous systems. It is also obvious that if the test material is an aqueous system it is necessary to guard against drying of the surface prior to or during the test for stickiness (Hoseney, R.C. and Smewing, J.O., 1999).

Food stickiness is particularly important during food processing. The high speed mechanized processing of food requires that the material does not stick to machine surfaces. A small amount of adhesion can lead to major problems that can result in the line being stopped. The second important item in food stickiness is in consumer acceptance of the product. Finger foods that are sticky and that stick to the fingers are, in general, not acceptable. On the other hand, some confectioneries, salad dressings, and barbecue sauces should be sticky or at least possess cling or adhesion to surfaces. Some foods should be sticky or nonsticky depending upon the culture of the intended consumer. An example of this is rice. Ideally in the Western world rice is dry, fluffy, and nonsticky while in the Orient sticky rice is preferred. In the United States 3 types of rice are grown; long, medium, and short grain. In general, the long grain types give dry, flaky grains when cooked, while the short and medium grains tend to give a sticky product. As with all cereal grains, rice undergoes an after-ripening phenomena after it is harvested. As a result of this after-ripening, the stickiness of rice decreases with

storage time. Whether this is good or bad depends upon your preference in the stickiness of rice. Mossman *et al.* (1983) developed an Instron test to measure the stickiness of cooked rice. The test was sufficient to easily determine the difference between sticky and dry rice. The stickiness of rice has been negatively correlated with amylose content of the rice starch (Juliano, B.O. *et al.*, 1981). Deshpande and Bhattacharya (1982) have shown that stickiness was not affected by the water-to-rice ratio used in cooking but was markedly reduced by storage of cooked rice. The General Foods Texturometer was used (Szczesniak, A.S. and Hall, B.J., 1975) to detect adhesiveness in cooked rice. Okabe (1979) related this technique to eating quality of rice in Japan. He concluded that hardness was most important but high hardness was acceptable if stickiness was also high. In her classic work on Texture Profile Analysis, Szczesniak (1963) used the term adhesiveness in relation to the stickiness of food. The term was defined as “the work necessary to overcome the attractive forces between the surface of the food and the surface of the other materials with which the food comes in contact (e.g., tongue, teeth, palate, etc.)”. Food stickiness is also important in the mouth. In fact, a definition of stickiness given by Jowitt (1974) is “possessing the textural property manifested by a tendency to adhere to contacting surfaces, especially the palate, teeth, and tongue during mastication”. The importance of stickiness in the mouth not only relates to consumer acceptance where, for instance, excessive stickiness in e.g. cookie or caramel products, may receive an extremely negative consumer response but also to adhering of food to teeth. Consequently this textural property accounts for a great deal of work on dental research.

Another undesirable example of stickiness is when the food sticks to the packaging material. This results in distortion and disfigurement of the product and generally adverse consumer reaction.

### **Instrumental Measurement of Food Stickiness**

In the first of a series of papers Claassens (1958) studied the stickiness of butters. He coined the term hesion (analogous to sorption) in preference to stickiness because he felt in measuring stickiness the components of force due to adhesion and cohesion were inseparably linked. He developed a balance type apparatus to measure the force with which butter adheres to solid material. Claassens (1959a) using the balance developed above found that the hesion force was dependent of the contact time, load and temperature of the butter and the material contacting the butter. The highest forces were found with metals (stainless, steel, brass,

aluminum) and glass, with intermediate forces with plastics and the lowest with beech wood. In related work, Claassens (1959b, c) developed a method to measure the apparent static friction of butter. Using this procedure, he found a reasonable correlation ( $r = 0.76$ ) betweenhesion values and the apparent static friction. As pointed out above, the force required to separate a probe from a surface depends on the material from which the probe is made. This occurs because low-energy materials absorb strongly to high-energy surfaces. As a result the surface energy of the system is decreased. In general, inorganic materials have high surface energies while polymer materials have low surface energies. Thus, probes made from metal or glass will give higher tack readings than similar probes made from plastics. The phenomena is illustrated in the data given in Table 2.3.

<b>Material</b>	<b>Force(g)</b>
Polytetrafluoroethylene	180
Polyethylene	450
Nylon	650
Polystrene	850
Stainless Steel	1000

Table 2.3 Influence of probe composition on tack measurement (modified from Hammond, JR, F.R., 1989).

The problem of dough stickiness was neatly summed up by the rheologist Scott Blair (1936) over 60 years ago when he stated that “stickiness appears to be the property of dough to which the baker’s hand is most sensitive”. Today, when dough is handled more by machine and less by the baker’s hand the importance of sticky doughs is even greater (Hoseney, R.C. and Smewing, JO., 1999). The dough with high adhesiveness can create serious problems in modern mechanized factories by causing production stoppages, product losses or contamination (Fizsman S.M. and Damasio, M.H., 2000).

Doughs made with wheat flour (for bread or biscuits), corn flour (for arepa or tortilla), or legumes (for papad) are products for which the measurements of adhesiveness is of vital importance, and there is an extensive literature on the subject (Table 2.4).



Reference	Food system	Method	Parameters Measured
Armero and Collar (1997)	white and whole flour bread dough	TA-XT2 TPA Chen and Hosney cell	named "apparent adhesiveness" (as negative area). three stickiness related parameters
Chen and Hosney (1995a)	bread dough	TA-XT2 Chen and Hosney cell	three stickiness related parameters
Dhaliwal <i>et al.</i> (1990)	bread dough	Digital Gram Gauge Single compression	three parameters from the tensile curve
Dobraszczyk (1997)	bread dough	Instron Peel test	stickiness (average peel force)
Haridas-Rao <i>et al.</i> (1989)	whole wheat <i>chapati</i> dough	GF texturometer TPA	adhesiveness ( negative area)
Heddleson <i>et al.</i> (1993) Heddleson <i>et al.</i> (1994)	flour water dough	Instron with Wetzel modified probe Single compression	probe tack and tack energy
Noguchi <i>et al.</i> (1976)	bread dough	GF texturometer TPA	stickiness measured as the change of slope of the negative area.
Padua and Padua-Maroun (1984)	arepas corn dough	Instron Single compression	adhesiveness ( negative area)
Ram and Nigam (1983)	bread dough	GF texturometer TPA	adhesiveness ( negative area)
Ramirez-Wong <i>et al.</i> (1993)	<i>tortilla corn masa</i>	Stickiness Device Test Special "cling" test: Instron Single compression	adhesiveness ( special index) maximum tensile force tensile work
Ramirez-Wong <i>et al.</i> (1994)	<i>tortilla corn masa</i>	Instron Single compression	adhesiveness ( negative area)
Sai-Manohar and Haridas-Rao (1997)	biscuit dough	Instron TPA	dough adhesion (negative area) and stickiness (slope of negative area)
Sila-Battacharya and Narasimha (1997)	blackgram <i>papad</i>	GF texturometer TPA	stickiness measured as the change of slope of the negative curve
Wang <i>et al.</i> (1996)	bread dough	Lloyd with specially designed cell Double cycle compression	24 texture parameters obtained many of which highly correlated with sensorial stickiness scores

Table 2.4. Determination of adhesiveness of dough methods, parameters and references (modified from Fiszman S.M. and Damasio, M.H., 2000).

In about half the works consulted the term “stickiness” is used, while the remainder speak of “adhesiveness.” In the bakery world the term “stickiness” recalls the negative property associated with the stages of manipulation during manufacture rather than a sensory quality characteristic perceived by the consumer in the final cooked product. There are works in which the two terms are used as synonyms without distinction (Ramírez-Wong, B. *et al.*, 1994) while some authors distinguish the terms as different concepts.

Noguchi *et al.* (1976) noted that doughs made by the continuous process tended to stick to the handling machines much more than conventionally made doughs. They studied various flour streams in an attempt to determine the cause of stickiness in doughs. A texturometer that inserted a cylindrical plunger into dough and measured the forces of insertion and withdrawal was used to evaluate stickiness. The slope of the force-displacement curve on withdrawal correlated well with subjective estimates of dough stickiness. Their conclusions were that low grade flours gave doughs that were more sticky than did higher grade flours. In addition, they found that ash content, SH content, and proteolytic activity were most useful in predicting stickiness. Of course, those three factors may be a reflection of lower grade flours. Many other attempts to measure dough stickiness have been reported. Hosney *et al.* (1990) measured dough stickiness by compressing a dough ball using a flat probe and measuring the force needed to pull away the probe. Nonsticky doughs separated cleanly from the probe, whilst a sticky dough stuck to the probe and stretched into a long filament, usually breaking in the center after a large amount of extension. Dhaliwal *et al.* (1988) measured stickiness by bringing a flat probe into contact with a thin layer of dough spread over a perforated metal plate under standardized conditions of compression force, contact time, and temperature, and measuring the force required to remove the probe. They reported that the area under the resulting force-distance curve produced the best indicator of stickiness. Strong correlations between the viscoelastic properties of doughs measured in compression, relaxation, and tension and sensory stickiness scores were reported by Wang *et al.* (1996). Studies have linked a number of factors to enhanced dough stickiness. These include flour extraction (Gore, P.J., 1991); amount of water soluble pentosans (Zeller, F.J. *et al.*, 1982); differences in protein composition (Dhaliwal, A.S. *et al.*, 1988);  $\alpha$ -amylase activity (Ranhotra, G.S. *et al.*, 1977; Ibrahim, Y. and D'Appolonia, B.L., 1979); and proteolytic activity (Hwang, P. and Bushuk, W., 1973). However, those studies of factors affecting dough stickiness were all hampered by lack of an objective test to measure stickiness. The baker's classic dough stickiness test is to touch the dough with his or her hand. If the dough sticks to the hand, the

dough is sticky. Whether the dough sticks to the hand depends on both the adhesive and cohesive properties of the dough. In this regard dough is no different than any other viscoelastic material. Heddleson *et al.* (1993, 1994) studied wheat flour dough as a pressure sensitive adhesive. They measured the relationship between the adhesive behavior of doughs using a probe tack test and dynamic rheological properties. They found a maximum in loss tangent corresponded with a maximum in tack energy, suggesting that tack is primarily controlled by a rheological mechanism. There is a large volume of literature dealing with the rheological properties of dough. It appears obvious that anything that affects dough rheology could potentially affect dough stickiness. Given a constant adhesive force between the dough and probe, anything that gives a weaker dough (more viscous and less elastic) will be measured as a sticky dough. The dough will adhere to the probe and it will neck (Figure 2.3).

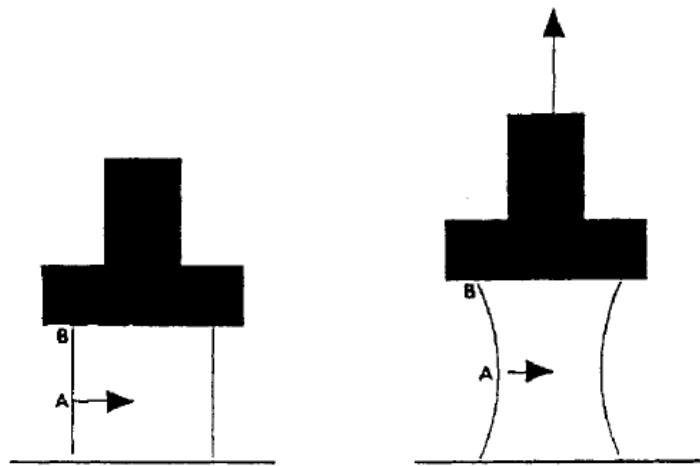


Figure 2.3 Necking of a dough piece (modified from Hoskeney, R.C. and Smewing, JO., 1999).

The force measured is a function of the dough rheology and not truly related to the stickiness of dough. All that can be concluded is that the adhesive force is greater than the cohesive force. With the same adhesive force, if the dough is strengthened (made more elastic and less viscous) at some point the cohesive force will exceed the adhesive force and the dough will separate at the probe interface. Heddleson *et al.* (1994) showed the great effect of temperature, separation rate, and moisture content on that adhesive-cohesive failure transition point. To study dough stickiness it appears imperative to separate the adhesive and cohesive properties. To measure the adhesive properties it is necessary that there is a clean separation at the probe-dough interface. To accomplish this Chen and Hoskeney (1995a) selected a probe

material with low surface energy (Plexiglas), and developed a cell to produce a flat dough surface and maximize the dough strength. They used a texture analyzer (TA.XT2) and chose the maximum reverse speed of the probe to minimize the measurement of viscous flow. This leads to a clean separation at the probe-dough interface (Hoseney R.C. and Chen, W.Z., 1994).

The cell is designed to extrude a thin layer of dough through a fine screen before compressing it, and the prepared dough surface is then allowed to rest 30s to release the stress produced by extrusion. Compression is effected with a 25 mm probe up to a compression force of 40g applied at a constant velocity and then the probe is raised at a very high velocity in order to encourage separation of the probe from the dough surface.

This cell is commercially available from Stable Micro Systems Ltd. (Godalming, U.K.)/Texture Technologies (Scarsdale, NY) (Fizman S.M. and Damasio, M.H., 2000).

Using this objective method to measure the adhesive properties they found that many things can affect the results. These include the surface tension of the water solubles from flour, added  $\alpha$ -amylase or protease, amount of water added to the dough, and the degree to which the dough is mixed. While these were shown to affect adhesive properties it is also true that many of the same factors can, and do, affect the dough rheology (cohesive properties). However, none of the above appeared to explain the difference between flours that gave sticky and nonsticky doughs. In subsequent work Chen and Hoseney (1995b) used the above procedure together with fractionation and reconstitution techniques to show that the entity that caused the dough to be sticky (adhesive) was water soluble and of relatively small size. The compound was identified as ferulic acid esterified to a hexose chain (Figure 2.4).

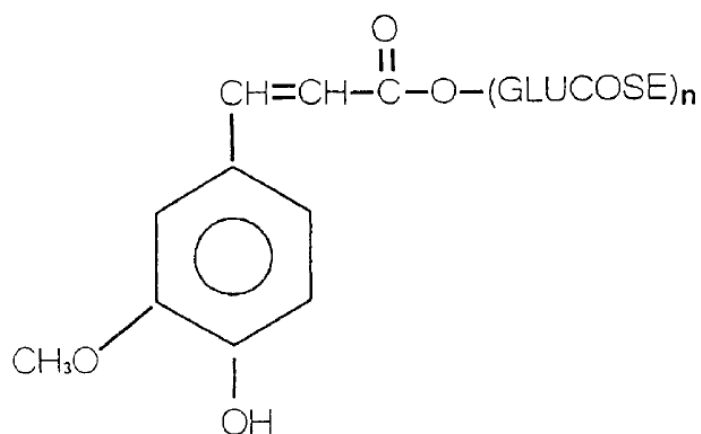


Figure 2.4 Proposed structure of the compound causing dough stickiness . The glucose polymer is a 1-3,1-4 beta glucan with n=70 (modified from Hoseney, R.C. and Smewing, JO., 1999).

Recently Huang and Hoseney (1998) have shown the hexose chain to be a (1-3,1-4) mixed linkage  $\beta$ -glucan with a DP of about 70. It was also shown that saponification of the ester bond destroyed the entities ability to cause a sticky dough (Hoseney, R.C. and Smewing, JO., 1999).

## Dough extensibility

Currently wheat is one of the most abundant crops in the world. From wheat flour a large variety of food products can be made, like breakfast cereals, breads, crackers, cakes, biscuits and pasta. By mixing, flour and water are transformed into a cohesive dough with viscoelastic properties. The rheological properties of wheat flour dough affect its behaviour during processing and consequently the quality of the finished loaf of bread. This conviction has led to the design of instruments with which rheological properties can be determined. Such measurements are widely used for the selection of new cultivars in breeding, for quality control in mills and bakeries and to study the effects of ingredients and adaptations of processes in the milling and baking industry.. Therefore tests determining large-deformation and fracture properties are relevant. Information on the resistance to extension and the extensibility of dough can be obtained by load-extension instruments such as the Brabender extensograph (Dunnewind B. *et al.*, 2004).

The extensograph has been widely used in both quality control and research laboratories for studying flour quality and the effect of certain additives in bread baking. This load-extension instrument was designed in the 1930s to provide empirical measures of stress- strain relationships in doughs. The result is a load (resistance) vs extension curve called an extensigram (Figure 2.5). Interpreting extensigram measurements in terms of basic physical or rheological terms is difficult because the dough geometry changes constantly as the test is being performed. However, several useful measurements can be made.

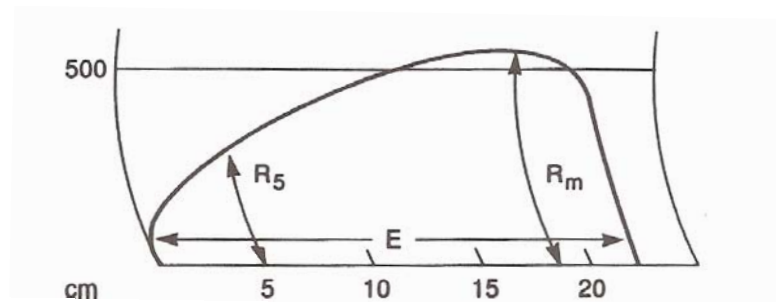


Figure 2.5 Extensigram, showing extensibility (total curve length in centimetres) ( $E$ ), resistance at a constant extension of 5 cm ( $R_5$ ), and maximum resistance (the maximum height of the curve) ( $R_m$ ). The  $R$  values are often given in arbitrary units of resistance called Brabender units. Total area under the curve ( $\text{cm}^2$ ) (modified from Hosney, R.C., 1994).

For most practical applications, the curve height and area under the curve are taken as measures of the flour's strength, with larger values indicating greater strength. The overall shape of the curve, or the ratio of curve height ( $h$ ) to extensibility ( $E$ ), gives an estimation of dough's viscoelastic balance. Obviously, long, low curves produce low  $h/E$  ratios and a predominance of viscosity over elasticity.

By using these simple relationships, extensigrams can classify flours according to their strength: weak, medium, strong and very strong. The instrument is also quite useful in studying anything that alters the strength of the dough. Examples of this are proteolytic enzymes and various oxidizing or reducing agents (Hoseney, R.C., 1994).

Although Bloksma (1962) has related the data obtained by this test to more fundamental parameters, the test still remains very empirical. The force and extension are not expressed in Newtons and strain, respectively. Major practical disadvantages of this test are that the position of the cradle (the clamp) depends on the force and that the amount of dough deformed increases with extension (Bloksma, A.H. and Bushuk, W., 1988). The conversion of extensograph load-extension curves into stress-strain curves is therefore seriously hampered. Under fermentation conditions, the rate of deformation of dough is three orders of magnitude smaller than the maximum rate of deformation of the test-piece in load-extension instruments like the Brabender extensograph (Bloksma, A.H., 1990). Moreover, the dominant type of deformation during fermentation and oven rise is biaxial extension and not uniaxial. Finally, a large amount of flour (300g) is needed to perform the test.

An apparatus similar to the Brabender extensograph was developed by Kieffer: the "Kieffer dough and gluten extensibility rig", also called a microextensograph (Kieffer, R. *et al.*, 1981a, b). This rig has been developed by Stable Micro Systems (SMS) for use exclusively with the TA.XT2 texture analyser. Based on research at the German Food Chemistry Research Institute (Deutsche Forschungsanstalt für Lebensmittelchemie, Garching) and named after its inventor dr. Rolf Kieffer, offers a simple, efficient and cost-effective alternative to traditional dough and gluten extensibility measurement techniques. It can produce accurate and reproducible results from very small samples, as little as 10g flour or 1-2g gluten respectively. Such micro methods are of great benefit to manageability, cost and time savings and are of particular value to wheat breeders, in determining the processing qualities of new wheat strains at an early stage, when only limited grain quantities may be available.

Using the SMS/Kieffer rig it is possible to compare gluten and dough extensibility scientifically for the first time. Previously the common method of measuring the extension

behaviour of gluten was to extent the gluten between the fingers. Using the SMS/Kieffer rig, the direct comparison is the objective and accurate and provides results in a graphical format. The parallel dough and gluten tests can hence be useful for selecting suitable dough additives, such as salts, emulsifiers, oxidants or enzymes, according to different wheat qualities to control optimum dough processing (Dunnewind, B. *et al.*, 2004).

In this apparatus a test can be performed with only about 0.4 g of dough and the apparatus can be fitted on any materials testing machine, which gives us the possibility to measure the force in Newtons, to adjust the test speed and temperature and to store the data in computer files for further calculations. Recently, Grausgruber (2002) comparing both instruments, concluded that the micro-extensograph method is valuable in early generation selection for wheat quality where the amount of available sample does not allow testing by the standard extensograph. The test method itself, however, has not been described extensively and formulas to calculate the stress and relative deformation rate are not given. Also only very little has been reported about the reproducibility of the test. Hence, to test this uniaxial extension instrument the rheological properties of a weak and a strong dough were evaluated with special attention to the correct determination of the type of deformation, the starting point of the actual extension and the reproducibility of the test (Dunnewind B. *et al.*, 2004).



### **2.1.2 Fundamental rheological tests**

Fundamental rheological tests measure well-defined physical properties independent of size, shape and how they are measured and can be used for process design calculations and to model complex processing situations not amenable to direct measurement. Problems encountered with such fundamental tests are: complex instrumentation which is expensive, time consuming, difficult to maintain in an industrial environment and requires high levels of technical skill; often inappropriate deformation conditions, difficulty in interpretation of results and slip and edge effects during testing.

The main types of fundamental rheological tests used in cereal testing are: (i) dynamic oscillation, (ii) creep and stress relaxation, (iii) extensional measurements and (iv) flow viscometry (Dobraszczyk, B.J., 2003).

#### **Dynamic oscillation measurements**

Adapted from techniques developed for measuring viscoelastic properties of polymer melts and concentrated solutions (Ferry, J.D., 1980), this is one of the most popular and widely used fundamental rheological techniques for measuring cereal doughs and batters. It has the advantage of a well-developed theoretical background, readily available instrumentation and simultaneous measurement of elastic and viscous moduli, while the non-destructive nature of the test enables multiple measurements to be performed as temperature, strain or frequency are varied (Dobraszczyk, B.J., 2003).

In these measurements, a sample is placed between two parallel plates or between a plate and a cone of very small angle and is subjected to sinusoidal displacement. The force generated by the motion of one plate is transmitted through the sample to the other plate and recorded. If the material between the plates is perfectly elastic material, Hook's law governs the stress-strain relationship and consequently stress and strain wave functions will have a  $0^\circ$  lag (in phase). Conversely, if the material is a viscous liquid, Newton's law applies, and the lag between stress and strain waves will be  $90^\circ$  (out of phase). A viscoelastic material will have a lag angle,  $\delta$ , between  $0^\circ$  and  $90^\circ$  (Masi., P. *et al.*, 2001).

Disadvantages of the dynamic oscillation method are that the deformation conditions are often inappropriate to practical processing situations, because they are carried out at rates and

conditions very different from those experienced by the dough during processing or baking expansion. For example, rates of expansion during proof and oven rise in bread doughs have been calculated between  $5 \times 10^{-3} \text{ s}^{-1}$  to  $5 \times 10^{-4} \text{ s}^{-1}$ , compared with measuring rates in rheological tests several orders of magnitude greater (Dobraszczyk, B.J., 2003).

Dynamic testing instruments may be divided into two general categories: controlled rate instruments where the deformation (strain) is fixed and stress is measured, and controlled stress instruments where the stress amplitude is fixed and the deformation is measured. Both produce similar results (Steffe, J.F., 1996).

Stress is defined as a force acting over a unit area, often in the units called Pascals ( $\text{N/m}^2$ ). It is usually referred to by the symbol  $\sigma$ . When sufficient force is applied to the dough, the dough deforms and the deformation produces a strain in the dough. Here the strain is the amount of deformation divided by the height or thickness of the dough. Strain is usually referred to by the symbol  $\gamma$  and is measured as a percentage. For example, if the deformation is 1% of the thickness of the dough, the dough is under a 1% strain.

The top plate, which is in contact with the sample (it must not slip), is caused to oscillate sinusoidally at some frequency ( $\omega$ , in radians per second) and with an amplitude ( $d$ ) measured in millimetres. The bottom plate remains stationary and is attached to a force transducer, if not slippage occurs at either plate, a deformation gradient is created across the thickness of the sample ( $h$ ). The force transducer thus measure the force ( $f$ ) transmitted through the sample in Newtons. The force is distributed over the sample area ( $l \times w$ ) and is uniform over the sample thickness. The output of such a dynamic rheometer is given in Figure 2.6. A deviation from sinusoidal behaviour would indicate the slippage occurring.

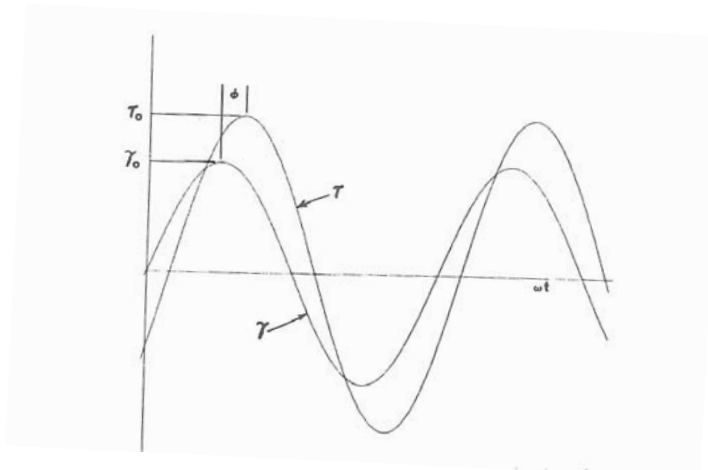


Figure 2.6 Sinusoidal signal output from the force and deformation transducers.  $\tau_0$  = shear stress amplitude;  $\gamma_0$  = shear strain amplitude,  $\Phi$  = phase angle,  $\omega t$  = radians/sec (modified from Hosney, R.C., 1994).

The  $\tau$  curve is output of the force transducer and represents the shear stress. The  $\gamma$  curve is from the moveable plate and is a measure of the amplitude or the shear strain. If the sample were completely elastic, the two curve would peak together, and if the sample were completely viscous, the two curves would be out of phase by  $90^\circ$ . The *phase angle*  $\phi$  ( $\Phi$ ) in radians, gives a measure of how much the system is out of phase. The *complex modulus* ( $G^*$ ) is equal to  $\tau_0/\gamma_0$ . As the name implies, the complex modulus is made up of the *storage modulus* ( $G'$ ) and the *loss modulus* ( $G''$ ). The storage modulus and the loss modulus are given by the following equations:

$$G' = (\tau_0/\gamma_0) \times \cos \Phi$$

$$G'' = (\tau_0/\gamma_0) \times \sin \Phi$$

In lay terms,  $G'$  is the part of the energy that is stored during a cycle, and  $G''$  is the part that is lost. Another term that is often used is  $\tan \delta$  or  $\tan \Phi$ , which is the ratio  $G''/G'$ . This is a simple index of the relative elastic or viscous nature of the material under test (Hoseney, R.C., 1994).

### Typical Operating Modes of an Oscillatory Testing instrument.

Commercially available oscillatory instruments will operate in numerous modes. A strain or stress sweep, conducted by varying the amplitude of the input signal at a constant frequency (Figure 2.7) , is used to determine the limits of linear viscoelastic behaviour by identifying a critical value of the sweep parameter.

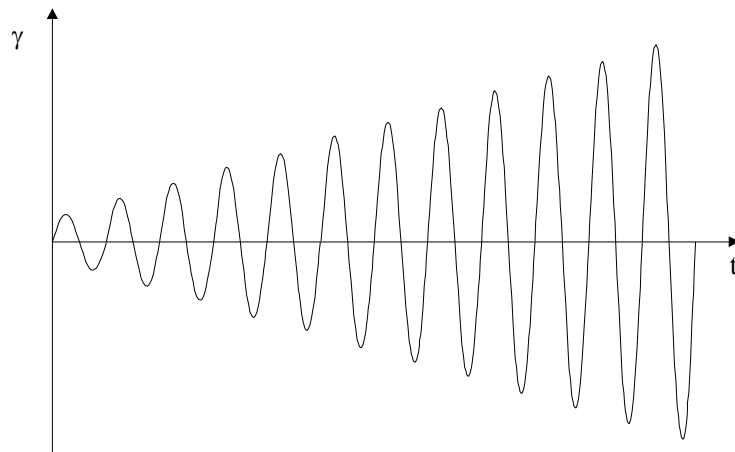


Figure 2.7 Strain or stress sweep mode in oscillatory testing.

In the linear region, rheological properties are not strain and stress dependent. Storage and loss moduli versus the sweep parameter are plotted in Figure 2.8. Some experimenters prefer to plot combined material functions such as the complex modulus or the complex viscosity.

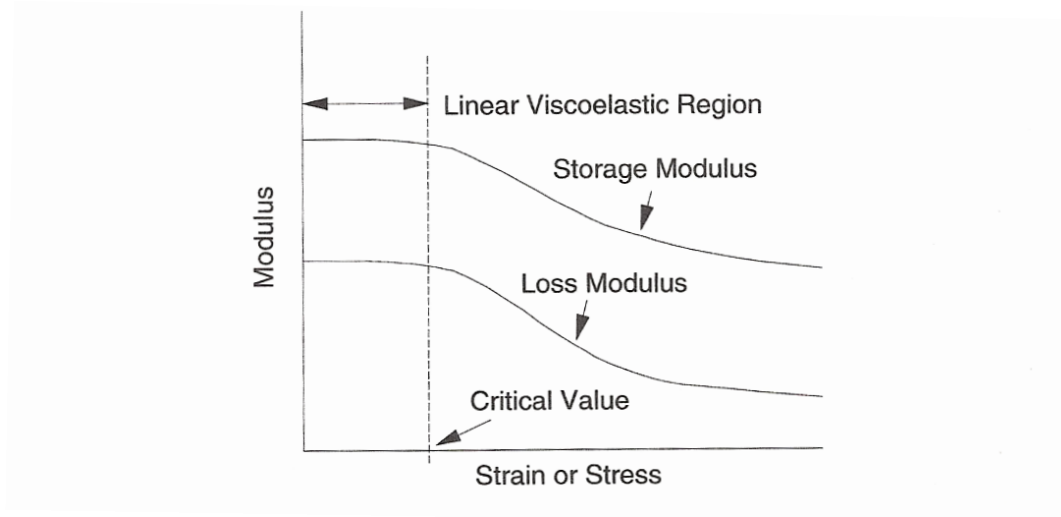


Figure 2.8 Typical response to a strain or stress sweep showing the linear viscoelastic region defined by the critical value of the sweep parameter (modified from Steffe, J.F., 1996).

Stress and strain sweeps are conducted on controlled rate and controlled stress instruments, respectively. It has been suggested that stress sweeps produce superior results (Holland, D., 1994); however, both strain and stress sweeps are known to provide an excellent basis for comparing the viscoelastic nature food products. In addition to establishing the linear viscoelastic range of the sweep parameter, strain and stress sweeps have been used to differentiate weak and strong gels: strong gels may remain in the linear viscoelatsic region over greater strains than weak gels.

The frequency sweep is probably the most common mode of oscillatory testing because it shows how the viscous and elastic behaviour of the material changes with the rate of application of strain and stress. In this test the frequency is increased while the amplitude of the input signal (stress or strain) is held constant (Figure 2.9).

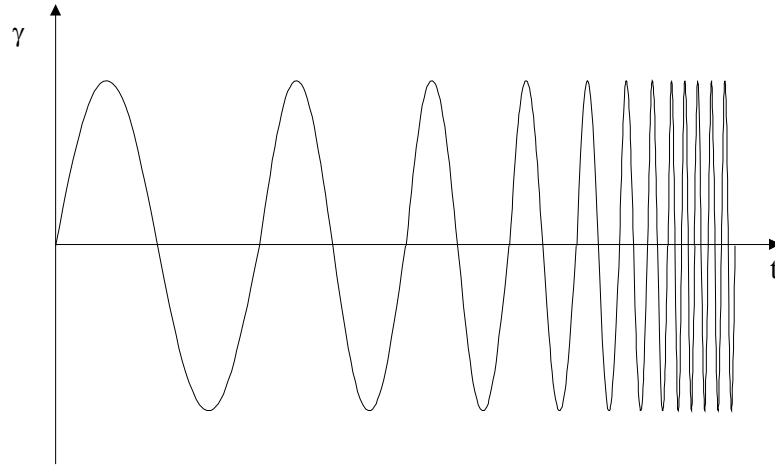


Figure 2.9 Frequency sweep mode in oscillatory testing.

Frequency sweeps are very useful in comparing, sometimes called “finger printing”, different food products or in comparing the effects of various ingredients and processing treatments on viscoelasticity. Materials usually exhibit more solid like characteristics at higher frequencies (Steffe, J.F., 1996).

### 3. MATERIALS AND METHODS

#### 3.1 Formulation and samples preparation

##### 3.1.1 Frozen dough without yeast

The materials used for this experimentation were commercial strong bread-making (sample A) and weak biscuit-making (sample B) wheat flours, supplied by Molino Naldoni (RA-Italy). Flour analysis (moisture 44-19, ash 08-01, proteins 46-10, gluten content 38-12, Zeleny index 56-61A and Falling Number 56-81B), Alveograph 45-30A and Farinograph 45-21 characteristics were determined by using AACC (1995) methods. The physicochemical characteristics of the used flours are reported in Table 3.1.

	Flour A	Flour B
Protein <sup>a</sup> (%)	14.50 ± 0.14	13.31 ± 0.21
Gluten (%)	13.40 ± 0.13	10 ± 0.09
Ash <sup>a</sup> (%)	0.60 ± 0.03	0.57 ± 0.09
Moisture (%)	15.50 ± 0.21	14.50 ± 0.13
Falling Number(s)	> 300	> 300
Zeleny (cc) <sup>b</sup>	64 ± 1.14	44 ± 1.22
Water absorption (%)	59.20 ± 0.13	52.8 ± 0.11
W (x 10 <sup>-4</sup> J)	345 ± 11	119 ± 13
P/L	0.40 ± 0.12	0.26 ± 0.14

<sup>a</sup> Corrected to 14% moisture content

<sup>b</sup> Values expressed and derived from 5g flour.

Values represent mean of three replicates ± standard deviation

Table 3.1 Flour analytical properties (modified from Angioloni, A. *et al.*, 2008).

Optimal water absorptions obtained by using Farinograph of 59.20 and 52.80 (based on 14% moisture in the flour) were used to prepare A and B samples, respectively. Mixing was carried out by using a spiral mixer (Kenwood Major, Hampshire – UK) for 10 and 5 min for samples A and B, respectively. As soon as doughs were formed, they were separated in samples of 50 g and then placed in plastic pots where they rested for 30 min at room temperature. After this

step the samples were frozen at -18 °C for 60 days. A thermocouple, GG-30-KK (Tersid, Milano, Italy) and a digital multimeter (Keithley, Cleveland, OH) were used to measure the temperature changes during freezing at the centre of the sample (Figure 3.1).

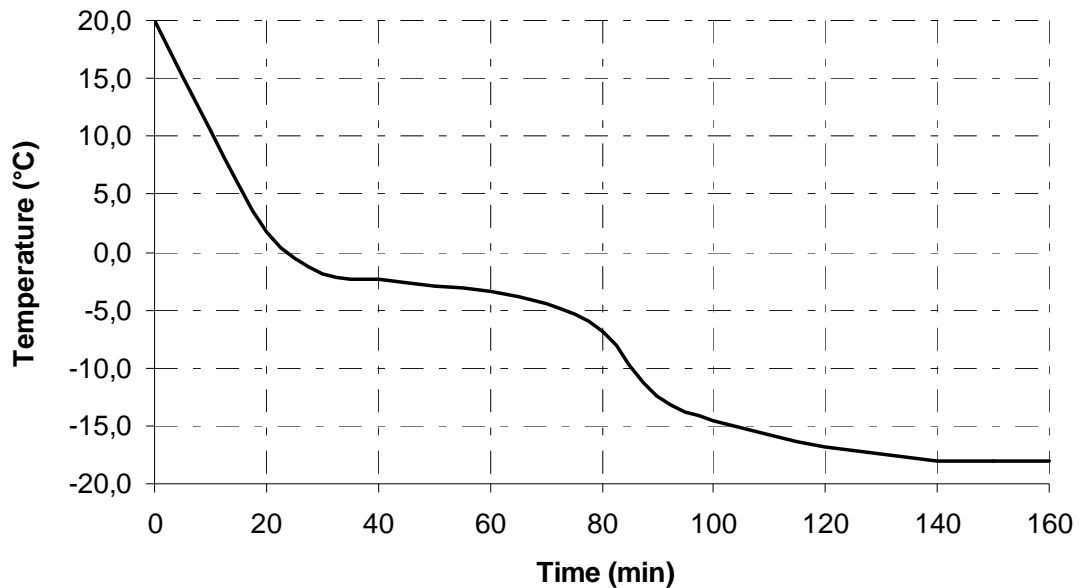


Figure 3.1 Evolution of dough temperature during freezing (modified from Angioloni, A. *et al.*, 2008).

After 15, 30, 45, 60 days of storage and before rheological analysis, thawing was carried out at room temperature for 90 min. Dough viscoelastic properties of fresh and freeze-thaw samples were evaluated by using empirical and fundamental rheological measurements (Figure 3.2).

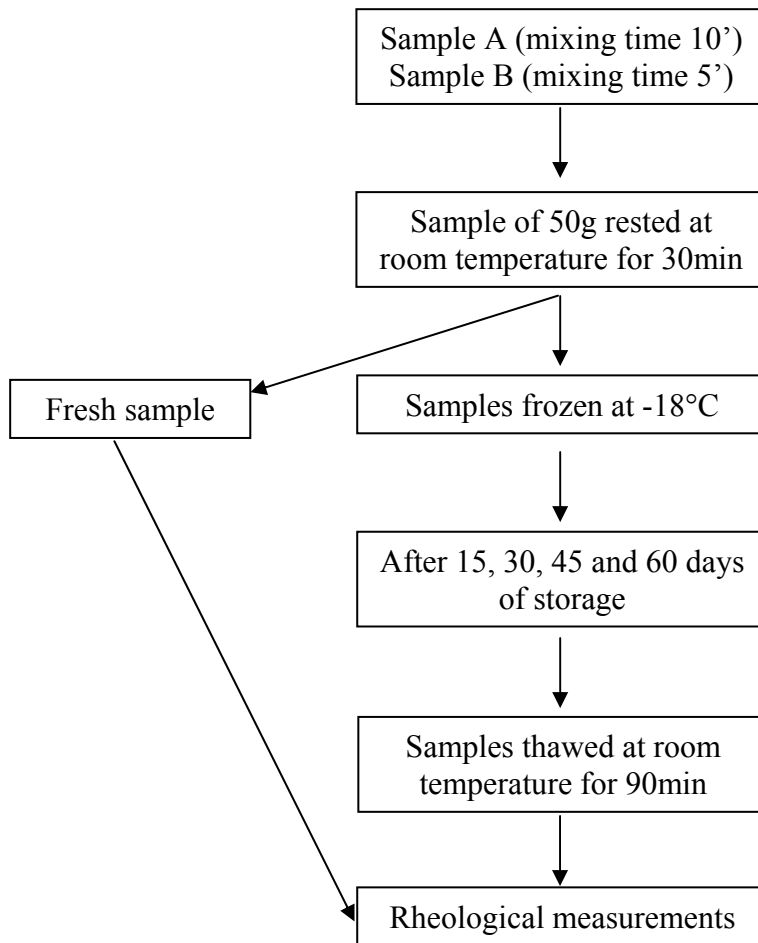


Figure 3.2 Process flow diagram for the production of dough samples.



### 3.1.2 Frozen dough and bread made with frozen dough

Bread dough recipe included flour (moisture 14,5%; alveograph characteristics: W=394, P/L=0.67; G=25.57), mineral water, salt, bakery yeast, malt and a technological additive “Fine Frozen” supplied by Pan Technology (Table 3.2).

	Flour (g)	Water (%)	Bakery Yeast (%)	Technological additive (%)	Salt (%)	Malt (%)
Sponge	1000	45	1			
Dough	1000*	70	2.77	2.5	2	1

\*Total flour = sponge's flour + dough's flour.

Table 3.2 Formulation of experimental doughs.

The sponge was prepared the day before by using a spiral mixer at the first speed for 7 min.

The final dough was obtained by using a mixer at the first speed for 3 min and at the second speed for 18 min.

After a rest of 30 min at 30°C and relative humidity of 75% , portion of dough (250g) were put in wicker basket and allow to ferment for 30 min at 30°C and 80% relative humidity. The use of these rigid boxes during the fermentation allows to obtain a more regular sample's shape.

After the fermentation period two samples were baked for 28 min at 235°C without being subjected to any frozen treatment and analysed after 1 hour of cooling at room temperature (fresh sample).

Some samples were frozen at -20C° for 6 hour (Figure 3.3) and some were deep-frozen for 3 hour.



Figure 3.3 Samples inside the freezer.

After this period the samples were removed and were put into a paper bag and stored at  $-18/20^{\circ}\text{C}$  for 1, 7 and 14 days.

A thermocouple, Testo 445 (Vac measuring instrument, Milano, Italy) were used to measure the temperature changes every 5 min at the centre of the sample during freezing and deep-freezing (Figure 3.4).

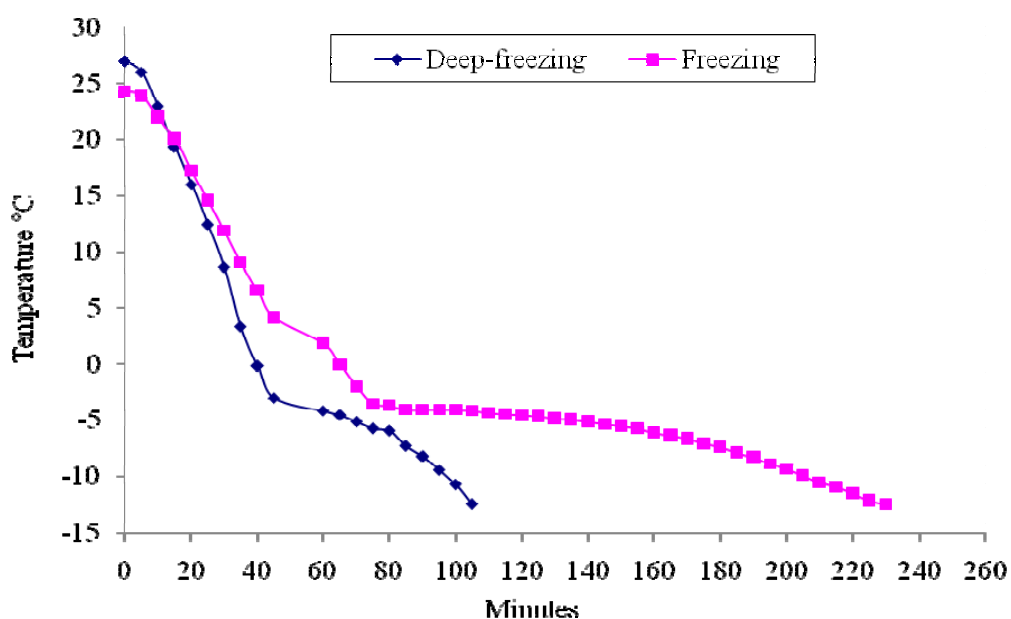


Figure 3.4 Evolution of dough temperature during freezing and deep-freezing.

As we can see from the Figure 3.4 the two different method allow the doughs to reach the temperature of  $-12.5^{\circ}\text{C}$  at the centre of the sample (the optimal temperature at which there is a stabilisation of the dough, according to the information of the company that supplied the technological additive). In order to reach the optimal temperature at the centre of the samples, 230 min were necessary when freezing process was adopted, while when deep-freezing process was chosen 100 min were enough.

Besides we can see that during the freezing process there was a longer period of time (65 min) where the temperature remain at around  $4.5^{\circ}\text{C}$  than in the deep-freezing process (20 min).

After the storage period (1, 7 or 14 days), thawing were carried out at room temperature for two hours before carrying out dough analysis.

The analysis were performed on thawed dough samples and on the final products obtained after the baking process at  $235^{\circ}\text{C}$  for 28 min.

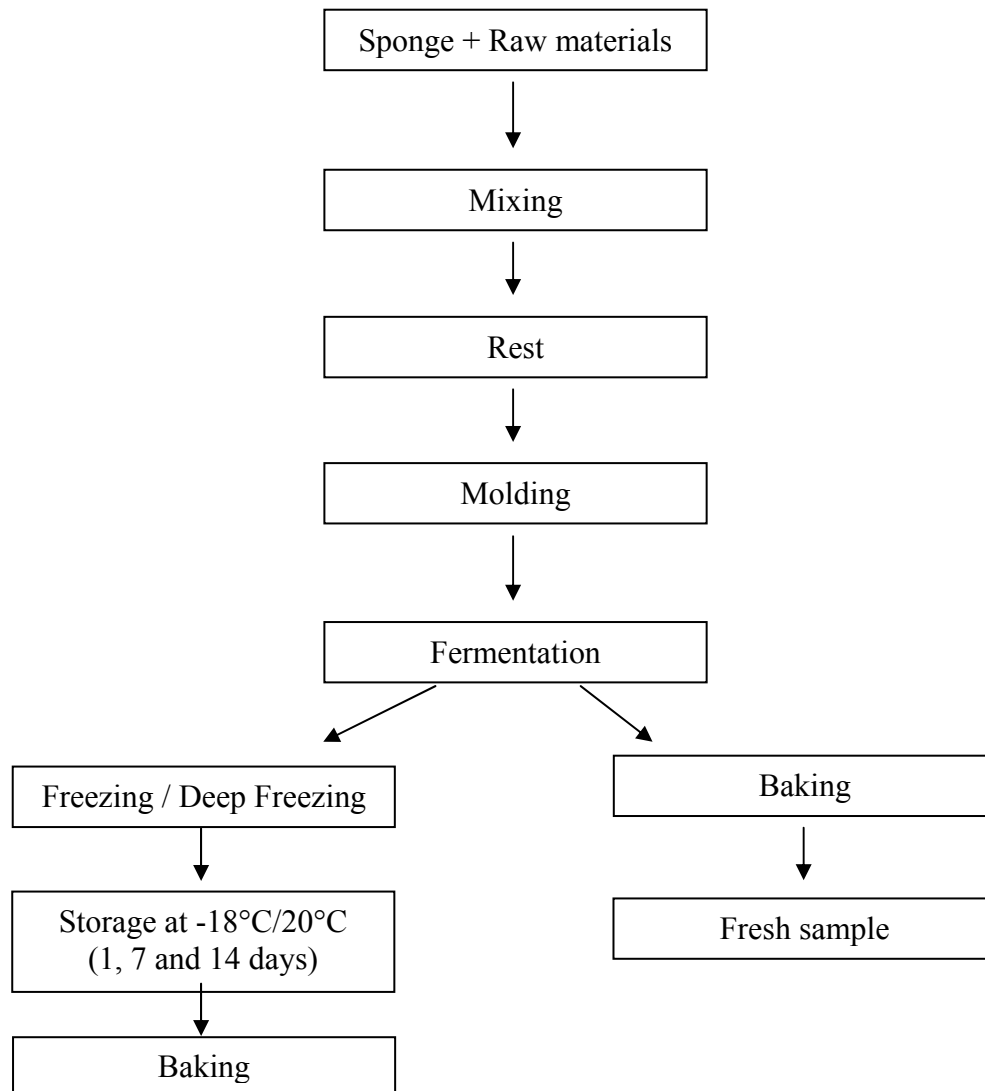


Figure 3.5 Process flow diagram.

### 3.1.3 Doughs obtained by using different fermentation methods

Doughs were realized by using three different fermentation methods:

- Straight-dough system;
- Sponge and dough procedure;
- Poolish and dough method.

	Flour (g)	Water (%)	Bakery yeast (%)	Salt (%)	Sourdough (g)	Sponge (%)	Poolish (g)
Straight-dough system	2000	60	2.5	1			
Sponge and dough							
Sponge	1000	50	1				
Dough	2000	60	1	1		20	
Poolish and dough							
Poolish	500	660g			500		
Dough	2000	60	2	1			525

Ingredients added in % on flour basis

Table 3.3 Formulation of experimental doughs.

The physicochemical characteristics of the flours used to realize the doughs were:

Moisture (%) : 14.5; Ash (%) : 0.53; Falling Number (s) : 298; Alveograph characteristics :  $W(\times 10^{-4} \text{ J})$ : 183, P : 73, P/L : 0.6.

The physicochemical characteristics of the flours used for the sponge and the sourdough were:

Moisture (%) : 14.3; Ash (%) : 0.50; Falling Number (s) : 295; Alveograph characteristics :  $W(\times 10^{-4} \text{ J})$  : 20, P : 78, P/L : 0.56.

In the *straight-dough system* all the formula ingredients are mixed into a developed dough that is then allowed to ferment.

All the ingredients were mixed in a spiral mixer (Sigma, Brescia, Italy) for 11 min and after 10 min of rest at 25°C the samples were moulded and then allowed to ferment for 1 hour at 30°C.

In the *sponge and dough procedure*, part of the flour, part of the water and the yeast were mixed for 4 min just enough to form a loose dough (*sponge*). The sponge was allowed to ferment 24 hour at 18°C. Then it was combined with the rest of the formula ingredients and mixed into a developed dough. After being mixed, an intermediate proof (referred to a “floor time”) of 10 min at 25°C was given to the dough so that it could relax. Then it was divided, moulded, and proofed for 1 hour at 30°C.

In the fermentation method called *poolish and dough method* a pre-ferment with sourdough, water and flour was realized (Figure 3.6).

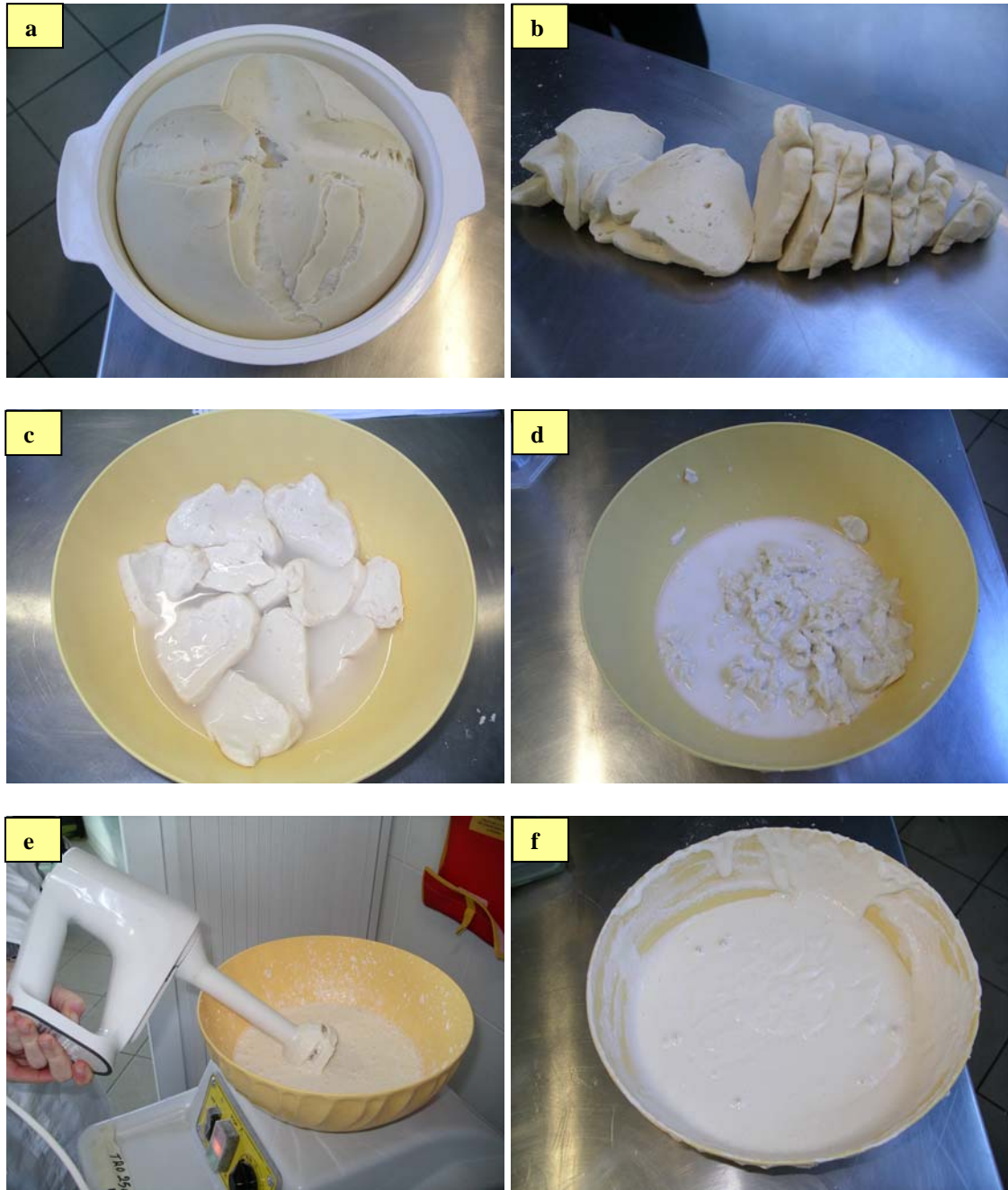


Figure 3.6 Polish's preparation flow diagram.

**a:** sourdough; **b:** slice's sourdough; **c** and **d:** slices' s sourdough in a bowl with water and flour; **e:** polish was prepared by mixing the flour, water and yeast together until the mixture had the consistency of a smooth, thick batter; **f:** final polish before fermentation.

The poolish was allowed to ferment 4 hours at 24°C. Then it was combined with the rest of the formula ingredients and mixed into a developed dough. After being mixed, the dough was given an intermediate proof (referred to a “floor time”) of 10 min at 25°C so that it could relax, and then it was divided, moulded, and proofed for 1 hour at 30°C.

The rheological measurements were carried out on the doughs immediately after mixing (after 10 min of rest), and after 1 hour of fermentation; image analysis was performed also after 30 min of fermentation.

The different types of dough were realised in triplicate.



### 3.1.4 Doughs made by Kamut® flour

Kamut® flour was characterised by moisture of 14.60%, ash 1.20%, protein contents 13% on dry matter, gluten 14.87%, W( $\times 10^{-4}$  J) 138 and P/L 1.78. The ingredients used in this study to prepare the different dough formulations were: mineral water, salt, bakery yeast, extra-virgin olive oil and a liposomic lecithin emulsifier (Lipos B 20 Activ ; TNA, Cava Manara, Italy).

In order to study rheological properties three different doughs were prepared: dough CTR was the control dough, while formulation OIL and EMU contained emulsifier (30g, corresponding to 2% on flour basis) and extra-virgin olive oil (54g corresponding to 3.6% on flour basis) respectively (Table 3.4).

Dough	Flour (g)	Water (ml)	Extra- Virgin Olive Oil (g)	Emulsifier (Liposomic lecithin) (g)	Salt (g)	Bakery yeast (g)
CTR	1500	825	—	—	37.5	3
EMU	1500	825	—	30	37.5	3
OIL	1500	825	54	—	37.5	3

Table 3.4 Formulation of experimental doughs.

After oxygenating the flour for 1 min at speed “min” using a mixer mod. Major mixer (Kenwood, Treviso, Italy) all the ingredients were mixed with the same mixer at the first speed for 6 min. Water was added at the temperature of 4°C to initially inhibit yeast activity. Different types of dough were realised in triplicate.

Each dough was submitted to:

- 1) a standard fermentation process of 4 hours at 25°C;
- 2) a storage period of 24 and 48 hours at 4°C.

Rheological analyses were carried out immediately after mixing (T0), at the end of fermentation process time (L4) and after 24 and 48 hours of storage at 4°C (S24 and S48 respectively), after 2 hours of resting at room temperature (Figure 3.7).

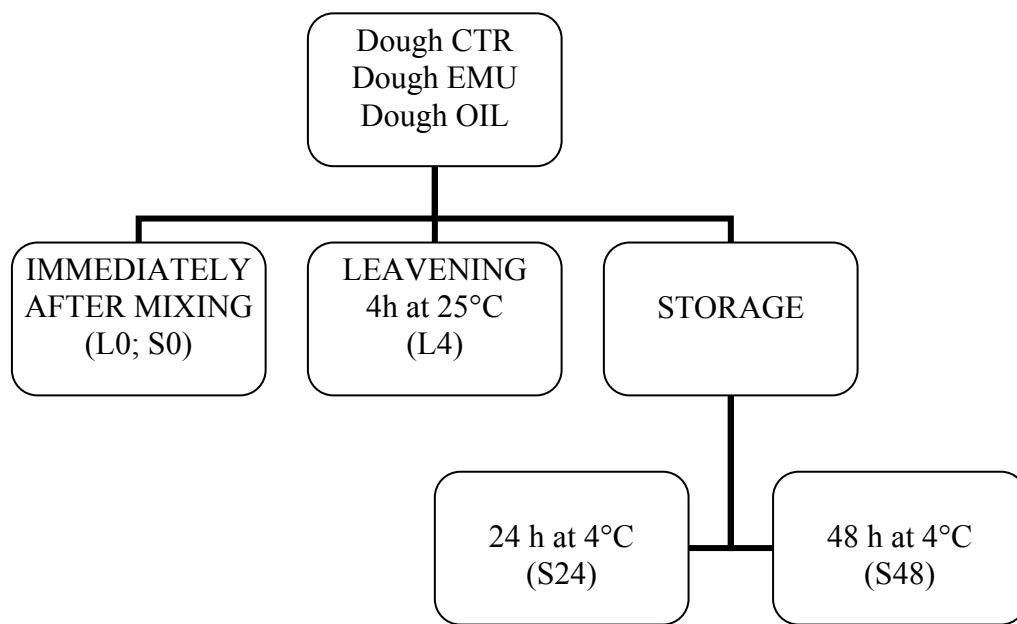


Figure 3.7 Process flow diagram.

### 3.1.5 Dough and bread with ginger powder

Basic dough formula consisted of fermented sponge, flour, water, salt, bakery yeast, additive and ginger powder. A control dough, without ginger powder, and samples with different amount of ginger powder were realized: 3%, 4.5% and 6% on flour basis.

Sponge (sponge dough process) was prepared by mixing flour, water and bakery yeast in a mixer for 8 min and was left to ferment 24 hour at 13°C before addition into bread doughs (Table 3.5).

	Sponge* (g)	Flour (W 190) (g)	Water (g)	Bakery yeast (g)	Salt (g)	Additive (g)	Ginger powder (g)
Control	1136	773	510	39	37	5	
3% of ginger powder	1136	726,62	510	39	37	5	46,38
4.5% of ginger powder	1136	703,43	510	39	37	5	69,57
6% of ginger powder	1136	680,24	510	39	37	5	92,76

**\*Sponge: Flour (W=240) 2000g; Water 910g; Bakery yeast 20g.**

Table 3.5 Formulation of experimental doughs.

Mixing was carried out by using a Kenwood Major mixer (Kenwood, Treviso, Italy) for 11 min and the analysis were carried out, in triple, on the doughs after 10 min of rest. After 20 min of rest at room temperature the samples were shaped (Figure 3.8).



Figure 3.8: Shaping of dough.

The samples were then allowed to ferment for 1 hour at a temperature of 32°C and relative humidity of about 70%. Fermented dough were then baked at 210°C for 23 min (Figure 3.9). After baking, bread samples were allowed to cool down for about 2 hour at room temperature before the analyses.



Figure 3.9 Samples of bread made with different amount of ginger powder.

### ***3.1.6 Final products coming from different bakeries***

Different samples of different regional bread coming from different bakeries were analysed and compared.

The samples were:

- a. “Coppia Ferrarese”, a typical regional bread of Ferrara;
- b. “Pane Comune Romagnolo”, a typical regional bread of Cesena area and
- c. “Filone Terra di San Marino”, a typical regional bread of San Marino.

9 different samples of “Coppia Ferrarese” were analysed about 2 hours after baking (fresh sample) and after 1 day of storage at room temperature (Figure 3.10).



Figure 3.10 Samples of “Coppia Ferrarese”.

11 different samples of “Pane Comune Romagnolo” were analysed at the same sampling time of Coppia Ferrarese (the fresh sample and the sample after 1 day of storage at room temperature) (Figure 3.11).

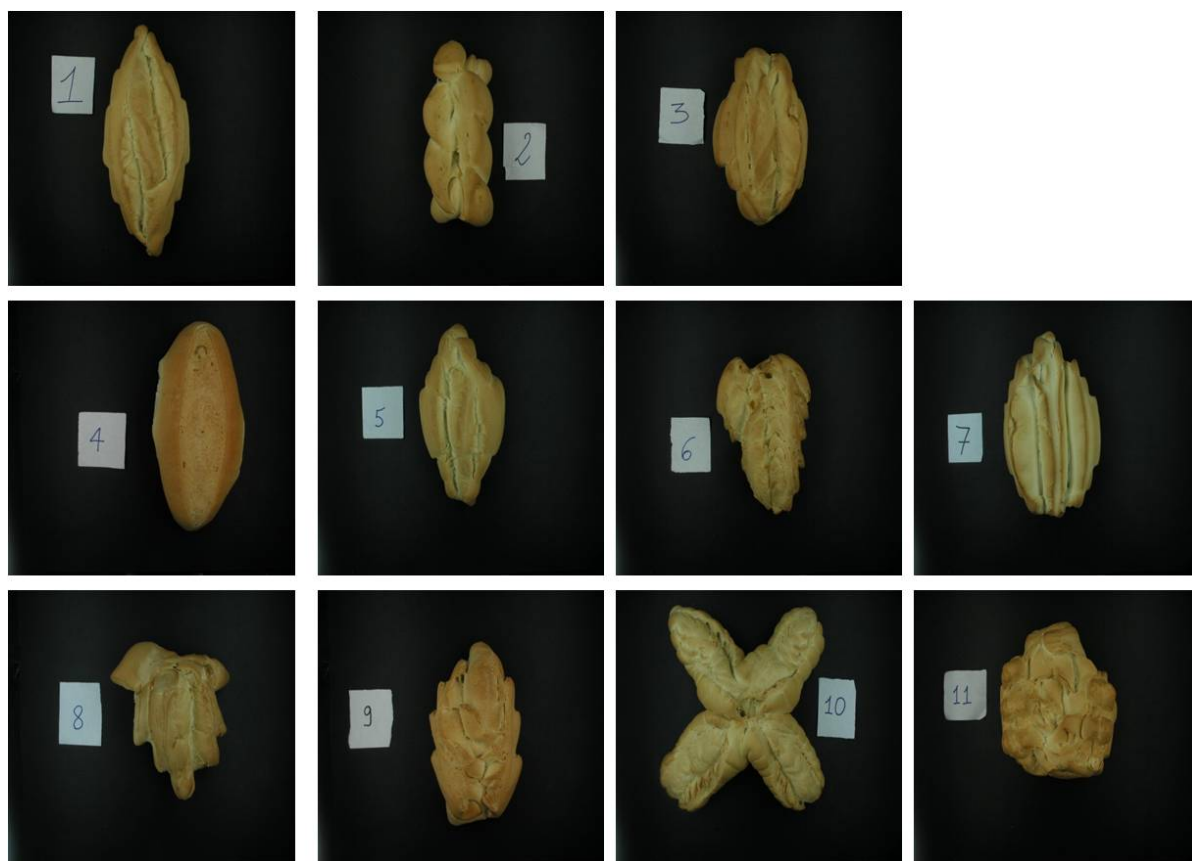


Figure 3.11 Samples of “Pane Comune Romagnolo”.

3 different samples of “Filone Terra di San Marino” were analysed. Analyses were performed on the fresh sample (2hours after the baking) and the samples after 1, 2, 3, 4 days of storage at room temperature to evaluate the effects of the staling process (Figure 3.12).

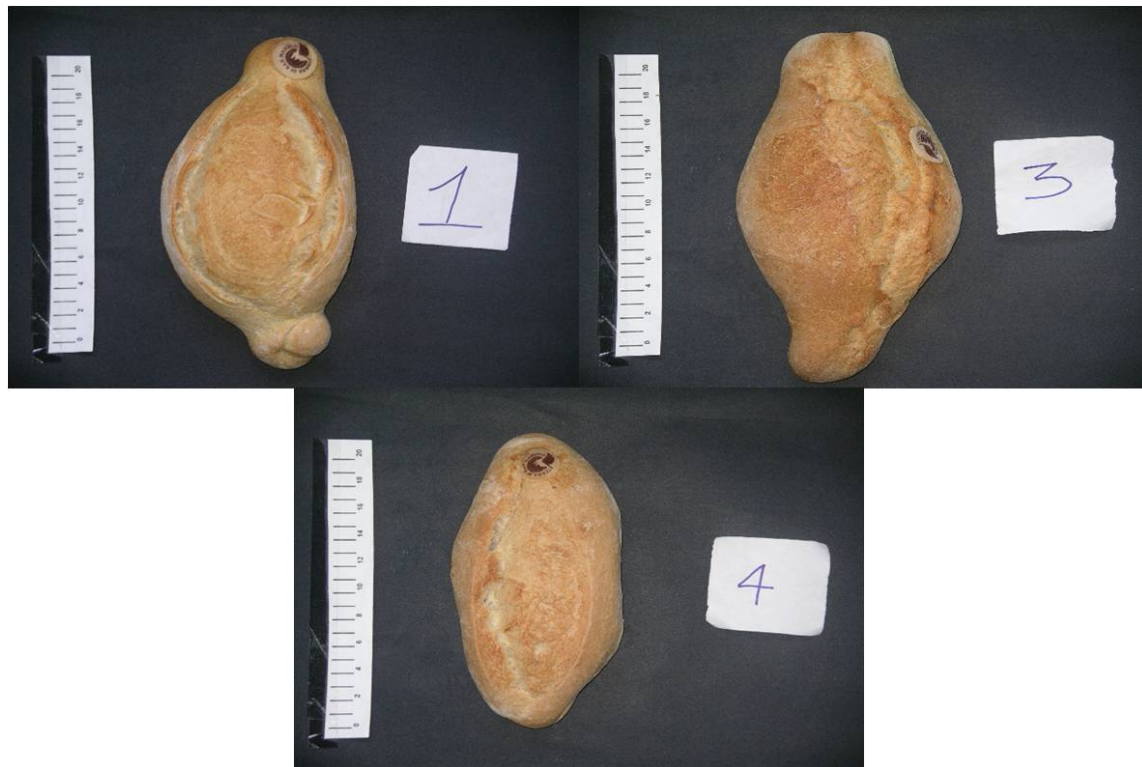


Figure 3.12 Samples of “Filone Terra di San Marino”.

## 3.2 Analyses

### ***3.2.1 Dough and bread Texture Profile Analysis (TPA)***

TPA is based on the recognition of texture as a multi-parameter attribute. The test consists of compressing a bite-size piece of food two times in a reciprocating motion that imitates the action of the jaw and extracting from the resulting force-time curve a number of textural parameters.

Texture measurements can be very valuable for the quality control and process optimization as well as for the development of new products with desirable properties and characteristics. Especially crumb firmness is a common quality characteristics for bakery products since it is strongly correlated with consumer's perception of bread freshness.

This test was applied on dough and on final products prepared by using different procedures. The TPA setting changed in function of the different samples analysed.

Doughs:

- a. Frozen;
- b. Frozen, with yeast;
- c. 100% based on Kamut<sup>®</sup> flour .

Final products (bread):

- a. Obtained by using frozen dough;
- b. Coming from different bakeries:
  - I. “Coppia Ferrarese”;
  - II. “Pane Comune Romagnolo”;
  - III. “Filone Terra di San Marino”.
- c. Obtained with different amount of ginger powder;



## TPA on dough

- a. TPA was carried out on fresh dough and on frozen samples after a thawing process of 90 min at room temperature.
- b. After the storage period of 1, 7 and 14 days at -18/-20°C 6 slices of 2 cm thickness for each samples were thawed for two hours at room temperature before carrying out the TPA test (Figure 3.13).

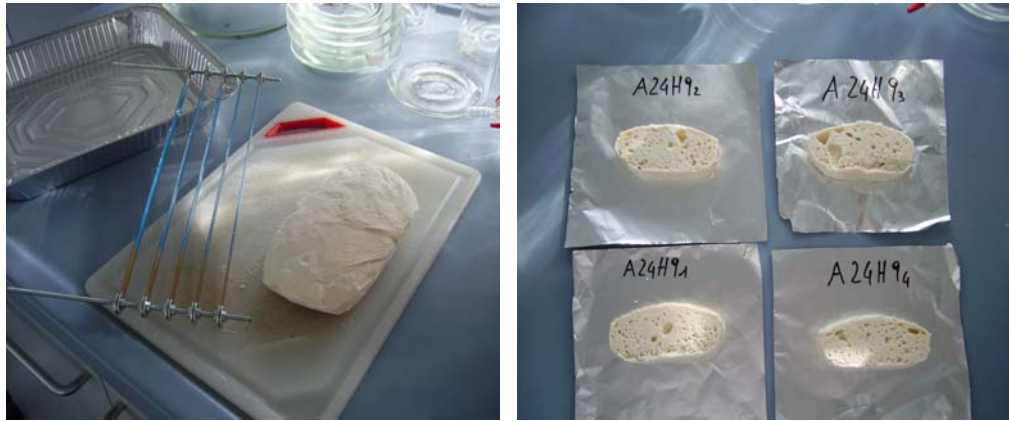


Figure 3.13 Sample for TPA test.

- c. The TPA test was carried out on sample during different times of control (see Figure 3.7).

The TPA test on dough was conducted on about 100g sample with a Texture Analyser mod.TA.HDi 500 (Stable Micro Systems, Surrey, UK) by using a 50mm diameter aluminium cylinder probe (a and c) and a 20mm diameter aluminium cylinder probe (b) and with the following setting:

Pre-test Speed 10,00 mm/s;

Test Speed 0,50 mm/s;

Post-test Speed 10,00 mm/s;

Strain 60,0%;

Time 75 s;

Load Cell 50 Kg;

Trigger Force auto-50 g

as described previously (Armero, E. and Collar, C., 1997).

TPA parameters varied widely with the strain and some of them also depend on the period which elapsed between the two compressions.

Springiness increased with an increasing period between compressions until 75s, at which time the dough had completely recovered from the first compression. An extended period between compressions would also lead to increments in dough volume due to the fermentation process.

The shape of first peak was clearly asymmetrical, with low resilience values, corresponding to a plastic flowable material. Springiness indicates the percentage recovery and resilience shows the speed of the recovery. Resilience was only dependent upon strain. At higher strains, doughs showed the same elasticity rate (springiness) but at a lower speed (resilience). Cohesiveness was only dependent upon strain; the higher the strain the lower the cohesiveness. As the strain was increased the structural elements of the dough were permanently broken. Specific TPA parameters are only meaningful when measured at certain intervals of deformation. When measuring cohesiveness, it is important that the sample is only partially deformed.

As a first point, adhesiveness increased rapidly as the strain increased.

Secondly, when strain was increased, the dough required more time to recover its shape and a negative force was measured by the probe for a longer period.

Gumminess and chewiness are secondary parameters.

Comparisons with hardness showed that gumminess and chewiness depend mostly on hardness rather than on cohesiveness or springiness.

TPA conditions selected were:

1. a period of 75s as the time required for the dough to recover after the first compression.
2. a strain of 60% of dough compression provide sample differentiation in a meaningful way for cohesiveness. Strain values lower than 60% resulted in too low adhesiveness (Armero, E. and Collar, C., 1997).

The primary textural properties were measured in the absence of dough adhesiveness by using a plastic film on the dough surface to avoid the distortion induced by the negative peak of adhesiveness. Dough adhesiveness was measured separately by running a second TPA without the plastic film and disregarding the others parameters (Angioloni, A. and Collar, C, 2008).

### TPA on bread

- a. The final products were cooled at room temperature for 2 hours before carrying out the test. The TPA test was carried out on bread slices of 2 cm thickness.
- b. The “Coppia Ferrarese” bread showed a peculiar shape with two different zones, the core and four extremity horn-shaped (“cornetti”). The core were softer than the extremity that were more crispy. TPA test was carried out on the sample core (Figure 3.14).

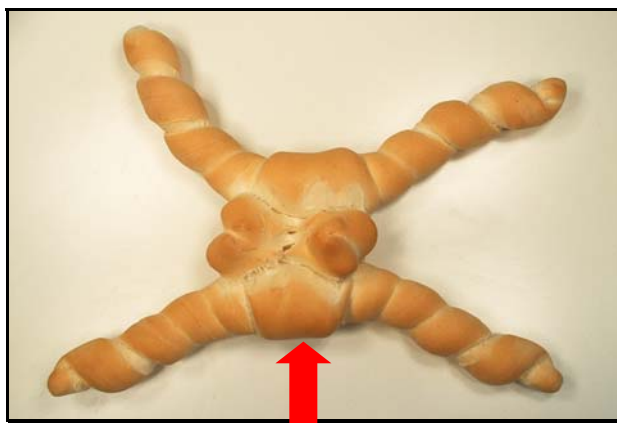


Figure 3.14 The arrow shows the zone taken for the test.

From “Pane Comune Romagnolo” and “Filone Terra di San Marino” bread slices of 2cm thickness were taken and the test was carried out on the centre of the slices (Figure 3.15).



Figure 3.15 Slice of “Pane Comune Romagnolo”.

- c. From each final product were taken 4 slices of 2 cm thickness and from the centre of the slices were taken sample of 2x2cm (Figure 3.16).

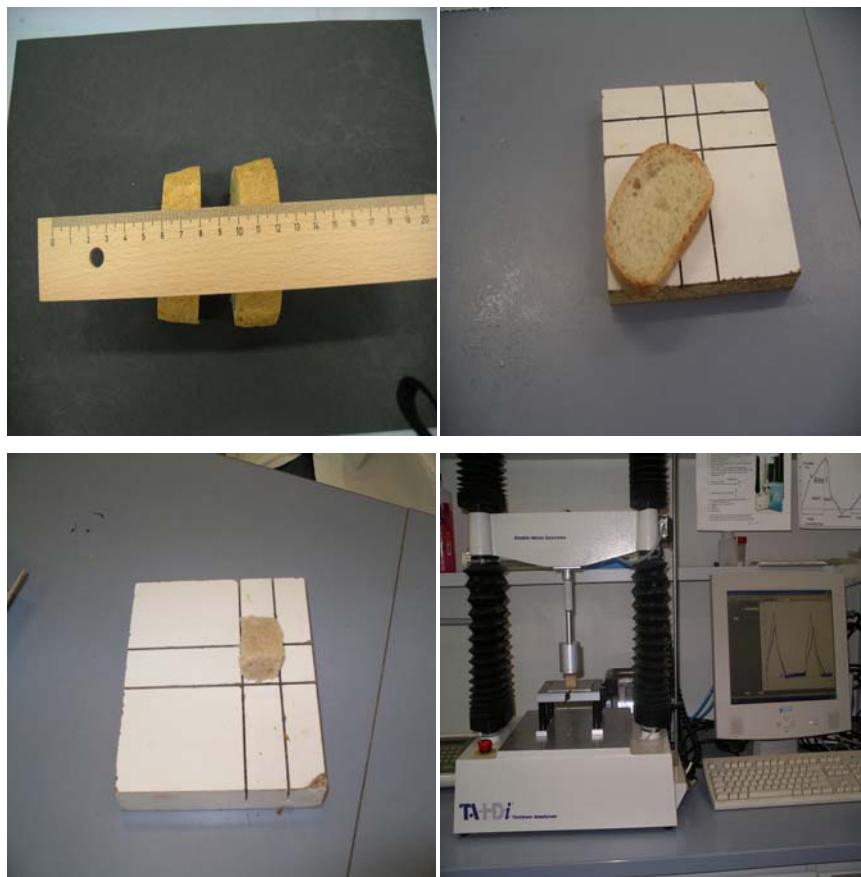


Figure 3.16 Operations to prepare the sample to analyse.

When the probe is larger than the sample (c), the forces registered are largely due to uniaxial compression. However, when the opposite is true (a and b), the forces derive largely from puncture, a combination of compression and shear.

It is interesting to note that in the first paper on TPA, Friedman (1963) reported that “all samples are made up with an area at least that of the plunger base,” so that their tests combined compression and shear forces. Bourne (1968) was the first to adopt true uniaxial compression to perform TPA with an Instron. This experimental condition was subsequently used by other authors working with the same instrument.

Generally speaking, most recent work done on TPA normally uses compression devices larger than the sample size, so that the forces registered in TPA tests are largely due to uniaxial compression forces.

For this reason in the last work (c) was chosen a sample smaller than the probe.

After placing the samples on the instrument base, they were squeezed twice with an aluminium cylinder-shaped probe with a diameter of 20mm (a and b I.(“Coppia Ferrarese”)) and 50 mm (b II. and III.(“Pane Comune Romagnolo” and “Filone Terra di San Marino”) and c).

The test was conducted on samples a and b (I, II and III) with the following setting:

Mode TPA;  
Pre-test Speed 10.0 mm/s;  
Test Speed 0.5 mm/s;  
Post-test Speed 10.0 mm/s;  
Strain 50,0%;  
Time 30 s;  
Load Cell 50 Kg;  
Trigger Force auto-10 g.

For the last experiment (c) the setting was changed according to Gàmbaro A. *et al* ( 2002):

Mode TPA;

Pre-test Speed 2.0 mm/s;

Test Speed 3.0 mm/s;

Post-test Speed 3.0 mm/s;

Strain 40,0%;

Time 5 s;

Load Cell 50 Kg;

Trigger Force auto-10 g.

### ***3.2.2 Dough uni-axial extensibility (Kieffer method)***

#### **Dough Relaxation**

After kneading, the dough (15-20g) was removed thoroughly from the equipment and rolled gently into a ball. This was placed in a small plastic pot and placed in a container or other place, in which there was as constant relative humidity as possible, to relax for about 20 min.

The dough form was taken and brush both the grooved base and the top form with paraffin oil, to avoid sample adhesion. If the dough to be tested appears to be very soft it may be necessary to place lametta strips along the length of the grooved regions, to aid removal of the samples after relaxation.

The sample was then rolled into a dough ball and then into sausage shape, both with as little manual manipulation as possible and then the sausage shaped sample was placed on the grooved base with its length perpendicular to the groove direction and finally the top form was placed over the bottom form from above.

It is very important to place the dough in this same direction on the form for comparative results.

The top form was pressed down until the two forms contact each other and excess dough was removed, which has extruded from the sides, with a spatula. This form cuts the sample into strips and allows the dough to relax, whilst preventing loss of moisture.

This form was placed in the clamp and screw down. Any excess dough which may have extruded further on clamping should be removed either with a spatula to ensure that each sample is separated. Any contact dough strips will be damaged by separation of the sample after they have undergone relaxation.

The dough clamp (with forms) was placed into a container, or other place, in which there is as constant a relative humidity and temperature as possible (optimally at around 24°C), and, following the official method, the dough has to relax for 40 min.

Different resting times were chosen for the samples containing yeast compared to the samples without yeast. It's not advantageous to leave dough with yeast to rest for 40 min (according to the official method) because the fermentation goes ahead and during this phenomenon the rheological properties of the dough change and it would be difficult to manage the sample and carry out the analysis and the results wouldn't be consistent.

### **Dough sample loading**

After the relaxation time the tension in the clamp is released and the dough form slid out slightly. The top form should be slid backwards gently over the grooved base, revealing each dough strip one at time, when required.

Before removing each strip the edges was cleared and separated from other neighbouring samples by running the spatula end down the length of each strip's side. The spatula end was then dipped in paraffin oil and carefully run under the length of a sample, pushing against the groove bottom, for accurate removal (taking care not to penetrate, stretch or deform the dough sample). The strip was placed across the grooved region of the same plate and the lametta strip removed gently.

Before testing one should ensure that the hook probe is lowered to just above the upper surface of the spring loaded clamp. Holding down the spring-loaded clamp lever, insert the plate (with sample in position) into the rig. Once the handle is released slowly the tensile test can begin.

Once trigger force of 5g has been attained, the hook will proceed to extend the dough sample centrally until its elastic limit (at the peak force) is exceeded and the dough separates (Figure 3.17).

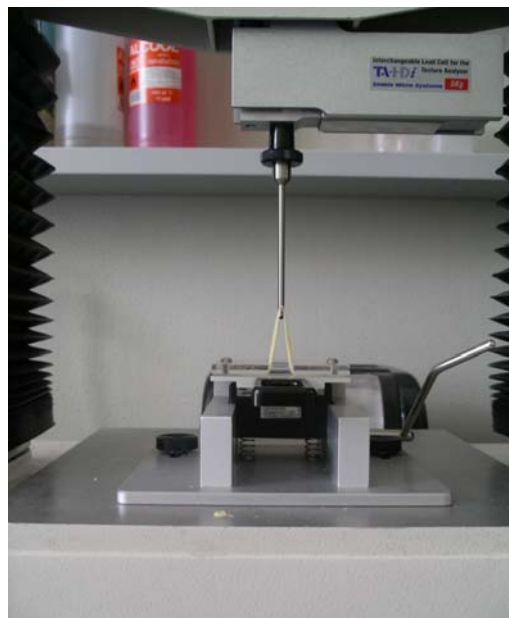


Figure 3.17 Kieffer rig.



### Dough extensibility graphs.

An example of a plot of dough extension using the Kieffer rig is reported in the Figure 3.18.

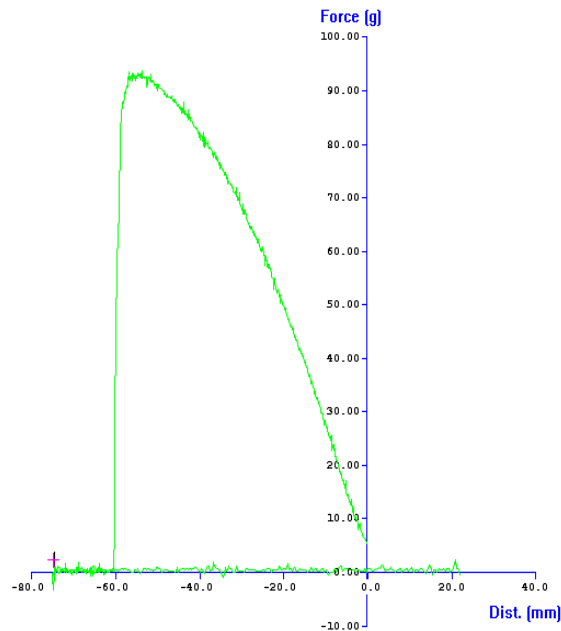


Figure 3.18 Example of force-distance curve obtained by texture analyser according to Kieffer test.

Like the extensograph, the values of major importance are:

- The peak force, the resistance to extension;
- The distance at which this peak force occurs and the distance at the extension limit, at the rupture point, which are the measurements of extensibility.
- The area under the force vs. distance curve, the dough force.

Kieffer test was performed on different dough samples:

- Frozen;
- Frozen, with yeast;
- 100% based on Kamut<sup>®</sup> flour;
- Made with different amount of ginger powder;
- Obtained by using different fermentation method.

Different rest time at different temperatures have been applied:

- a. For the samples without yeast was followed the standard method (40 min at 30°C);
- b. The samples were thawed at room temperature for 2 hours, then were prepared and were analysed immediately after the preparation to prevent an excessive fermentation in order to avoid samples difficult to manage.
- c. Immediately after mixing, in order to avoid dough dehydration, doughs were put in plastic boxes for 10 min to lose mechanical energy stored up during mixing and handling. After resting phase, dough samples were pressed into a Teflon mould and cut into dough stripes using the appropriate mould and kept for a further 10 min at 30°C under saturated atmosphere and then measured.
- d. Immediately after mixing, in order to avoid dough dehydration, doughs were put in plastic boxes for 10 min to lose mechanical energy stored up during mixing and handling. After resting phase, the doughs samples were prepared and kept for 15 min at 20°C and then measured.
- e. Immediately after mixing, in order to avoid dough dehydration, doughs were put in plastic boxes for 10 min at 25 °C to lose mechanical energy stored up during mixing and handling. After resting phase the doughs were prepared and kept 10 min at 25°C and then measured.

The Kieffer test on dough was conducted with a Texture Analyser mod. TA.HDi 500 (Stable Micro Systems, Surrey, UK) with the following setting:

Mode Measure Force in Tension

Option Return To Start

Pre-start Speed 4.00 mm/s

Test Speed 3.30 mm/s

Post-test Speed 10.00 mm/s

Distance 100,0 mm

Load Cell 5 Kg

Trigger Force Auto-5g

From the test the following parameters were obtained:

- a. The resistance to extension (g) and extensibility (mm) were determined in tension mode by recording the peak force and the distance at the extension limit (Collar, C. *et al.*, 1999; Smewing, J., 1995).

The force-deformation curves were recalculated into stress–strain data, taking into account changes in the sizes of the extended specimen. The following values were used for the dough volume calculation: 1.24 g/cm<sup>3</sup> as specific weight (for both samples) and 0.350 ± 0.01 and 0.330 ± 0.02 as weight of the pieces of dough in the apparatus gap, for A and B samples, respectively (Dunnewind B. *et al.*, 2004). The fracture properties of dough were computed from the stress–strain data.

The maximum stress or fracture stress ( $\sigma_{\max}$ ) and the Hencky strain ( $\varepsilon_H$ ) at fracture were taken as measures of the resistance to extension and extensibility, respectively.

The formulas for the calculation of fundamental rheological parameters can be derived from geometry.

The initial length of the sample  $l_0$  and the length  $l_t$  at time  $t$  are:

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

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

in which  $y_0$  is the distance which the hook has to travel from the surface of the lower plate to the point where the actual extension starts,  $y_t$  is the displacement of the hook from the point at which the actual extension starts, and 9 is half the width of the gap in the lower plate through which the hook passes (Figure 3.19A).

The relative deformation or Hencky strain and the strain rate can then be written as :

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

The measured force  $F_m$  is not the force acting on the dough  $F_d$ . Assuming that the hook passes exactly through the centre of the gap,  $F_m$  is divided equally over both stretches of dough at each side of the hook.  $\sin \alpha$  can therefore be expressed in forces as well as in lengths (Figure 3.19B):

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

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

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

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

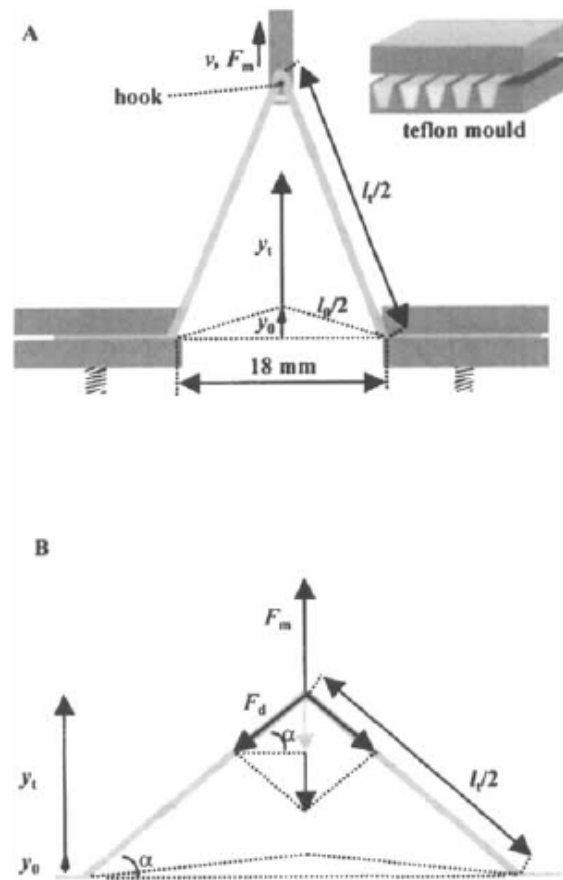


Figure 3.19A Schematic drawing of the Kieffer Extensibility Rig with its Teflon mould.

Figure 3.19B Schematic drawing of the forces acting on the dough piece (EQ.5) (modified from Dunnewind B. *et al.*, 2004).

b. Parameteres determined:

- The peak force, the resistance to extension;
- The distance at which this peak force occurs which are the measurement of extensibility.
- The area under the force vs distance curve, the dough force.

c. and d. The resistance to extension (force), extensibility (distance) and dough force (area) were determined by recording the peak force, the maximum distance limit before break and the area under the force vs. distance curve (Angioloni, A., 2005).

e. Parameters determined:

- The peak force, the resistance to extension;
- The distance at which this peak force occurs and the distance at the extension limit, at the rupture point, which are the measurements of extensibility.
- The area under the force vs distance curve, the dough force.

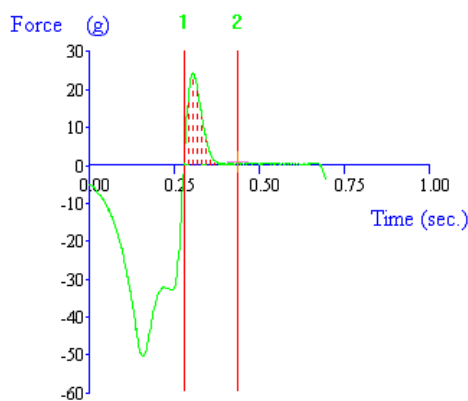
### 3.2.3 Dough stickiness

Before using the Chen and Hoseney cell, rotate the internal screw to move the piston and increase the sample chamber to its maximum capacity. Place a small quantity of prepared dough (5g) into the chamber and remove the excess dough with a spatula so that it is flush with the top of the chamber.

Screw on the extruder lid. Rotate the internal screw a little way to extrude a small amount of dough through the holes and remove this first extrusion from the lid surface using a spatula. Rotate the screw once again to extrude a 1mm high dough sample. Place the perspex cap over the exposed sample surface to minimise moisture loss, whilst allowing the prepared dough surface to rest for 30 seconds to release the stress produced by extrusion. After this time remove the cover and place the cell directly under the 25mm cylinder probe attached to the load cell. Commence the adhesive test.

The dough can then be removed from the lid surface and extruded again to repeat the test, using the above procedure.

An example of a plot using the dough stickiness method is as shown below:



The negative region of the plot when the test commences is a result of 40g of force being applied for 0.1s to compress the sample slightly.

The positive region of the plot however is of overall importance. The maximum force reading, i.e. highest positive peak, the positive area and the distance between the anchors set ('travel'), are all indicators of the stickiness or rheological properties of the dough.

The test was carried on:

- a. Doughs with different percentage of ginger powder.
- b. Doughs obtained by using different fermentation method.

The dough stickiness test on dough was conducted with a Texture Analyser TA.HDi 500 (Stable Micro Systems, Surrey, UK) applying the following setting:

Option Adhesive Test

Pre-start Speed 0.50 mm/s

Test Speed 0.50 mm/s

Post-test Speed 10.00 mm/s

Distance 4,0 mm

Force 40 g

Time 0.10 s

Load Cell 5 Kg

Trigger Type: Auto 5 g



### **3.2.4 Fundamental rheological tests**

Small deformation rheological measurements, by oscillation tests, were performed on a controlled stress–strain rheometer mod MCR 300 ( Physica/Anton Paar; Ostfildern Germany), using parallel-plate geometry (25 mm plate diameter and 2 mm plate gap). The upper serrated 25 mm plate was lowered until the thickness of sample was adjusted to 2 mm and the excess was trimmed off. The exposed surface was covered with a thin layer of mineral oil to prevent moisture loss during testing. The plates were coated with sandpaper. There is good evidence to suggest that use of sandpaper, as plate roughening, eliminates slip between the dough and the plate. In the case of many systems, it has been demonstrated that plate roughening does reduce, if not eliminate, slip between the sample and the plate (Keentok, M. *et al.*, 2002).

For dynamic oscillatory measurements to be of real value, they should be performed in the linear viscoelastic region (LVE-range: linear viscoelastic). This implies that the dynamic moduli ( $G'$  and  $G''$ ) and loss tangent ( $\tan\delta$ ) should be independent of the applied stress or strain. Also, the stress-strain relationship should be linear. In a stress controlled rheometer the region of linearity is normally determined by performing a stress/strain amplitude sweep; the sample under investigation is subjected to a range of stress values and dynamic moduli are measured. Working with deformations of small amplitude is a necessary condition to ensure the non destructive nature of the test this, in turn, allows us to express the results in terms of linear viscoelastic properties, typical of an equilibrium state system.

A strain sweep test was used in this thesis to identify the linear viscoelastic region; on the basis of this data, a target strain of 0.01%, which was within this linear region, was chosen for measurement. A frequency sweep test, ranging from 0.1 to 10 Hz, was used to study the dough rheological characteristics. The storage modulus ( $G'$ ), loss modulus ( $G''$ ) and  $\tan\delta$  ( $G''/G'$ ) were determined.  $G'$  is a measure of the solid or elastic character of the material and  $G''$  a measure of its liquid or viscous character, while  $\tan\delta$  indicates the relative contributions of the viscous ( $G''$ ) and elastic ( $G'$ ) characteristics of the material. When the material behaves more like a solid, ie when the deformation within the linear range is essentially elastic and recoverable, the elastic character,  $G'$ , exceeds the viscous character,  $G''$ , and  $\tan\delta < 1$ . On the other hand, when the material behaves more like liquid or viscous system, then the viscous character,  $G''$ , predominates and  $\tan\delta > 1$ . These parameters were evaluated at frequency of 1 Hz.

The test was carried out on:

- a. Frozen dough;
- b. Doughs with different amount of ginger powder;
- c. Doughs obtained by using different fermentation method.

Although cereal rheology is a recent addition to the field of breadmaking, it is one of the oldest branches of rheological study, the world's first rheological instruments was developed in 1836 to study the rheology of dough. However while yeast plays a crucial role in breadmaking, almost all empirical and fundamental rheological testing and studies are conducted on doughs formulated without yeast. The standard rheological testing instruments in use today by the milling and baking industries (farinographs, mixographs, alveographs, extensigraphs) test non-yeasted doughs. These instruments were originally developed to overcome the inconsistent results obtained from test baking. Furthermore, omitting yeast has the advantage of speeding up and making rheological testing simple. Yeasted doughs have also been avoided in dough rheology research, often with non-yeasted dough rheological findings being compared with baking data to provide a link between non-yeasted rheology and baking performance. The need for rapid testing and the general success of non-yeasted rheological measurements in predicting flour performance in breadmaking has seen the continued avoidance of yeast in dough rheology. However, this approach does not provide direct information on the rheological changes occurring within bread doughs during fermentation and the stages between mixing and baking of the dough.

Only a handful of studies into the rheological properties of yeasted bread dough have been made. Fundamental rheological studies of yeasted doughs have been made on wheat flour sourdough (Wehrle, K. and Arendt, E.K., 1998) and on cracker sponge and dough (Oliver, G. and Brock, C.J., 1997). All these studies of yeasted doughs have allowed the yeast to remain active during the measurements. Such an approach can be problematic in gathering accurate rheological information, particularly when conducting fundamental rheological measurements where the test are extremely sensitive and may take a long time.

Allowing fermentation to continue during rheological measurements would confound these measurements because it is impossible to ascertain what properties result from prior fermentation, the parameter of interest, or from fermentation during the measurement (Newberry, M.P. *et al.*, 2002).

To try to avoid or at least to minimize this problem was decided to apply a shorter test on the samples with yeast (b and c) compared with the test carried on the samples without yeast (a).

### ***3.2.5 Image analysis***

#### **Introduction**

Quality is a key factor for modern food industry because the high-quality of product is the basis for success in today's highly competitive market. In the food industry, the quality evaluation still heavily depends on manual inspection, which is tedious, laborious, and costly, and is easily influenced by physiological factors, inducing subjective and inconsistent evaluation results. To satisfy the increased awareness, sophistication and greater expectation of consumers, it is necessary to improve quality evaluation of food products. If quality evaluation is achieved automatically, production speed and efficiency can be improved in addition to the increased evaluation accuracy, with an accompanying reduction in production costs. As a rapid, economic, consistent and even more accurate and objective inspection tool, computer vision systems have been used increasingly in the food industry for quality evaluation purposes. The application potential of computer vision to the food industry has long been recognised. The food industry ranks among the top 10 industries using computer vision technology, which has been proven successful for the objective and non-destructive quality evaluation of several food products. Being an objective, rapid and non-contact quality evaluation tool, computer vision has been attracting much R&D attention from the food industry and rapid development has been increasingly taking place on quality inspection of a wide range of food products (Du, C-J. and Sun, D-W., 2006).

Recent advances in hardware and software have aided in this expansion by providing low cost powerful solutions, leading to more studies on the development of computer vision systems in the food industry. As a result automated visual inspection is under going substantial growth in the food industry because of its cost effectiveness, consistency, superior speed and accuracy. Traditional visual quality inspection performed by human inspectors has the potential to be replaced by computer vision systems for many tasks. There is increasing evidence that machine vision is being adopted at commercial level (Brosnana, T. and Sun, D-W., 2004).

#### **Fundamentals of computer vision**

Following its origin in the 1960s, computer vision has experienced growth with its applications expanding in diverse fields: medical diagnostic imaging; factory automation; remote sensing; forensics; autonomous vehicle and robot guidance. Computer vision is the construction of explicit and meaningful descriptions of physical objects from images. The

term which is synonymous with machine vision embodies several processes. Images are acquired with a physical image sensor and dedicated computing hardware and software are used to analyse the images with the objective of performing a predefined visual task. Machine vision is also recognised as the integrated use of devices for non-contact optical sensing and computing and decision processes to receive and interpret an image of a real scene automatically. The technology aims to duplicate the effect of human vision by electronically perceiving and understanding an image.

Basically, the system consists of two parts: hardware (illumination system, image acquisition device, computer) for image acquisition, and software for image processing and analysis (Figure 3.20).

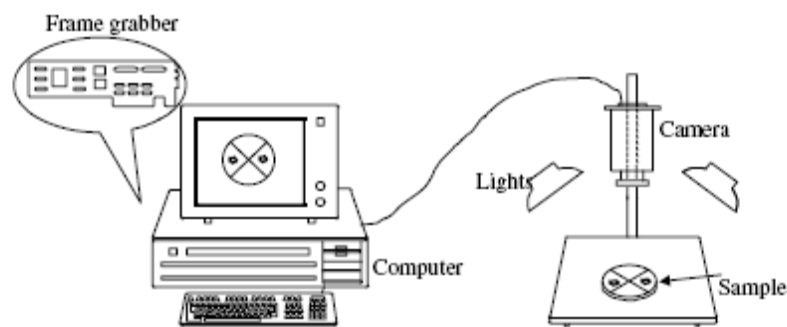


Figure 3.20 Components of a computer vision system (modified from Wang, H-H. and Sun, D-W., 2002a).

As with the human eye, vision systems are affected by the level and quality of illumination. Sarkar (1991) found that by adjustment of the lighting, the appearance of an object can be radically changed with the feature of interest clarified or blurred. Therefore the performance of the illumination system can greatly influence the quality of image and plays an important role in the overall efficiency and accuracy of the system. In agreement Gunasekaran (1996) noted that a well-designed illumination system can help to improve the success of the image analysis by enhancing image contrast. Good lighting can reduce reflection, shadow and some noise giving decreased processing time. Various aspects of illumination including location, lamp type and colour quality, need to be considered when designing an illumination system for applications in the food industry .

There are many image acquisition devices based on different sensors that can be used to generate an image, such as ultrasound, X-ray and near infrared spectroscopy. Images can also be obtained by using displacement devices and document scanners. Usually in a machine

vision system, images for analysis are gathered by using a video camera or digital camera (Brosnana, T. and Sun, D-W., 2004).

Array or area type cameras that consist of a matrix of minute photosensitive elements (photosites) from which the complete image of the object is obtained based on output proportional to the amount of incident light. Alternatively line scan cameras use a single line of photosites which are repeatedly scanned up to 2000 times per minute to provide an accurate image of the object as it moves under the sensor. Monochrome and colour cameras have been used throughout the food industry for a variety of applications (Leemans, V. *et al.*, 1998; Pearson, T.C. and Slaughter, D.C., 1996; Yang, Q., 1996).

However, flatbed scanners can be used for image acquisition. A flatbed scanner is an image gathering devices that incorporate a fixed relations between the illuminant source (lamp) and the solid state sensors of the scanning head and it was used for the image acquisition of dough for experiment on dough obtained by using different fermentation methods.

Image processing and image analysis are recognised as being the core of computer vision (Krutz, G.W. *et al.*, 2000). Image processing involves a series of image operations that enhance the quality of an image in order to remove defects such as geometric distortion, improper focus, repetitive noise, non-uniform lighting and camera motion. Image analysis is the process of distinguishing the objects (regions of interest) from the background and producing quantitative information, which is used in the subsequent control systems for decision making.

The appearance of baked products is an important quality attribute, correlating with product flavour and influencing the visual perceptions of consumers and hence potential purchases of the product. Features such as the internal and external appearance contribute to the overall impression of the products quality. Consequently such characteristics have been evaluated by computer vision (Brosnana, T. and Sun, D-W., 2004).

The image analysis was carried out on final products:

a. Different breads coming from different bakeries:

- i. “Coppia Ferrarese”;
- ii. “Pane Malatestiano”;

b. Bread realized with different amount of ginger powder;

And also on doughs obtained by using different fermentation methods.

## Image Analysis on bread

For image analysis the final products in the experimental work a, b were sliced in regular slice of 20mm thickness. The inner slice was considered for crumb grain features measurements (Figure 3.21).

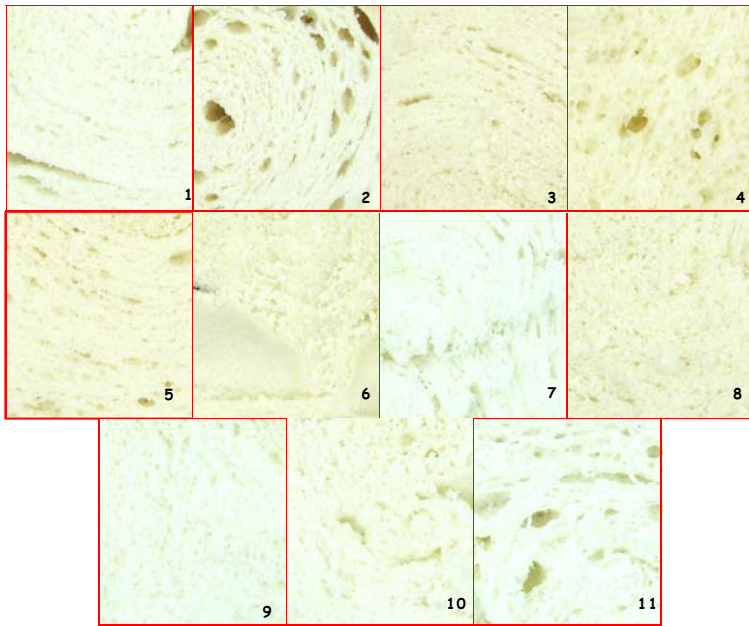


Figure 3.21 Portion of slice's "Pane Comune romagnolo".

The images were captured using an image acquisition system for color digital camera similar to that developed by Mendoza and Aguilera (2004) namely:

- samples were illuminated using four florescent lamps (60-cm length) with a colour temperature of 6500 K (Philips, Natural Day-light, 18W) and a colour rendering index (Ra) close to 95%. The four lamps were arranged as a square 35 cm above the sample and at an angle of 45° with the sample plane to give a uniform light intensity over the food sample.

- A colour digital camera (CDC) Power Shot A70 (Canon, Tokyo, Japan) was located vertically at a distance of 12.5 cm from the sample. The angle between the camera lens axis and the lighting sources was approximately 45°. Sample illuminators and CDC were inside a wood box with internal walls that are painted black to avoid the light reflection from the room.

- Images were captured with the mentioned CDC at its maximum resolution (2048x1536 pixels) and stored by connecting the CDC to the USB port of a PC. Canon Remote Capture Software (version 2.7.0) was used for acquiring the images directly in the computer.

A portion of each image was considered and digital images were processed by an advanced Image Analysis software (Image Pro-Plus<sup>®</sup> version 6.2 Media Cybernetics, USA) (Figure 3.22).

Crumb morphological analysis was performed. Crumb morphological features included: max area, mean cell area, number of cells and cell density (number of cells/cm<sup>2</sup>) (a); the number of cells, the percentage and the mean area of cells belonging to four class of varying area (b).

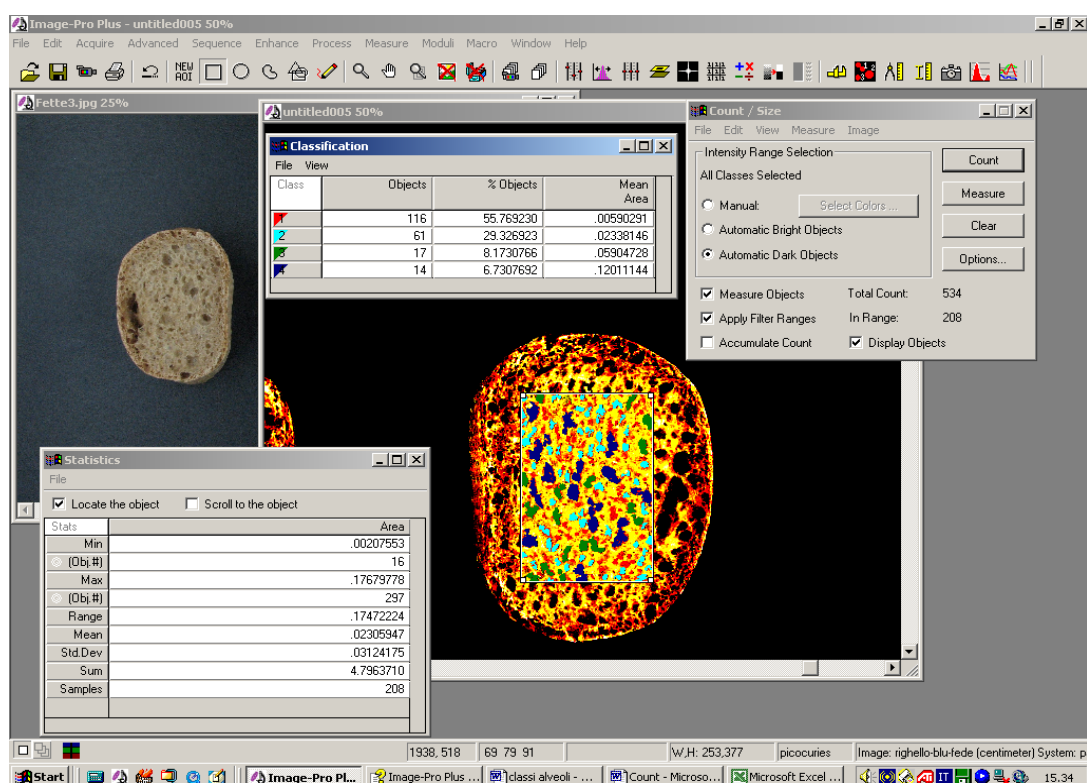


Figure 3.22 Elaboration of a slice of bread with ginger powder.

## Image Analysis on dough

A particular sample preparation procedure was set up in order to evaluate the changes in gas cell size and distribution during proofing and to avoid structural deformation (Figure 3.23). At the end of mixing, after 10 min rest, a petri dishes was put on top of the sample and after fixed proofing time (immediately after mixing, after 30 min and 60 min of fermentation) the images were acquired using a colour plane scanner mod. SnapScan40 (AGFA, Belgium, Europe). All images obtained under the same conditions (true color-24 bit, resolution of 300bit x pixel (BPP)), were taken by positioning the doughs on a scanner held on a black box, in order to exclude the surrounding light (Rocculi P. *et al.*, 2005). The images were then processed by Image Analysis software (Image Pro-Plus<sup>®</sup> version .6.2 Media Cybernetics, USA).

Dough morphological features included: total number of objects; objects classes: the objects were then divided in 4 different classes of varying area (cm<sup>2</sup>) (Class 1:0-0.001; Class 2: 0.001-0.01; Class 3: 0.01-0.1; Class 4: 0.1-1) and for each class were evaluated the number and the percentage of objects; the total area and the percentage.



Figure 3.23 Sample's preparation.



### **3.3 Statistical analysis**

Analysis of variance (ANOVA) and the test of mean comparisons according to Fisher's least significant difference (LSD) were applied, with a level of significance of 0.05.

Data were also evaluated using Pearson's correlation analysis between large and small dough rheological properties.

The statistical package STSG Statistica for Windows, version 6.0 ("Statsoft" Inc., Tulsa, OK) was used.

## 4. RESULTS AND DISCUSSION

### 4.1 Frozen dough without yeast

#### *Effects of storage time on frozen dough viscoelastic properties*

In general, dough viscoelastic characteristics can be assessed by measuring hardness, storage ( $G'$ ) and loss modulus ( $G''$ ),  $\sigma_{\max}$  and  $\varepsilon_H$  of the samples. These quality attributes are important and can be considered as key parameters in the evaluation of rheological performance of both conventional and frozen dough. Therefore, the tables which represent the fundamental (Table 4.1) as well as the empirical (Table 4.2) dough viscoelastic properties during sub-zero storage time are presented below.

Samples	Time (days)	$G'$ (kPa)	$G''$ (kPa)	$\tan\delta$
A	0	19.10 a	7.02 a	0.371 c
A	15	12.00 b	4.47 b	0.366 d
A	30	11.15 bc	4.11 c	0.369 c
A	45	10.50 c	3.84 d	0.382 a
A	60	9.87 c	3.79 d	0.385 a
B	0	12.00 a	4.81 a	0.400 b
B	15	8.18 b	3.21 b	0.391 b
B	30	7.23 c	3.01 b	0.414 b
B	45	6.33 d	3.18 b	0.420 a
B	60	6.34 d	2.46 c	0.425 a

Within column, within each sample, values with the same following letter do not differ significantly from each other ( $p \leq 0.05$ )

Table 4.1 Effect of storage time on dough fundamental rheological properties (mean values at 1 Hz of frequency) (modified from Angioloni, A. *et al.*, 2008).

Samples	Time (days)	Texture Profile Analysis			Kieffer	
		Hardness (N)	Springiness	Adhesiveness (N·s)	$\epsilon_H$	$\sigma_{\max}$ (kPa)
A	0	62a	0.95a	25.20b	2.36a	15.55a
A	15	53b	0.83b	25.25b	2.33a	12.76b
A	30	53b	0.78b	25.39b	2.25b	10.12c
A	45	51b	0.74c	25.95b	2.19c	10.08c
A	60	50b	0.73c	31.09a	2.16c	9.81c
B	0	52a	0.93a	39.71b	2.15a	4.11a
B	15	40b	0.91a	39.99b	2.11a	3.82a
B	30	39b	0.84b	40.86b	2.10a	3.20b
B	45	33c	0.75c	50.90a	2.01bc	2.94b
B	60	33c	0.75c	52.32a	1.97c	2.78b

Within column, within each sample, values with the same following letter do not differ significantly from each other ( $p \leq 0.05$ ).

Table 4.2 Effect of storage time on dough empirical rheological properties (modified from Angioloni, A. *et al.*, 2008).

Although many studies have considered the rheological properties of frozen dough, it is actually difficult to have a completely clear picture about the state-of-the-art because contradictory results have been found. Different experiments showed, a decrease in the extensibility of frozen dough (Varriano, M. *et al.*, 1980; Wolt, M.J. and D'Appolonia B.L., 1984; Havet, M. *et al.*, 2000; Giannou, V. *et al.*, 2003) but in other researches the opposite effect was also identify (Inoue, Y. and Bushuk, W., 1991). This might be due to the fact that when yeast or additives are included in dough formulations this can mask the effect of freezing on the dough viscoelastic structure. Kline and Sugihara (1968) and Hsu (1979) suggested that dough weakening can be attributed to the release of reducing substances, such as glutathione from yeast during freezing. Glutathione weakens the dough by cleaving disulphide bonds in the gluten proteins, an important factor in determining the rheology of gluten. ANOVA analyses were used in order to study the influence of storage time (at -18 °C) on dough rheology. Significant differences between the means at the 95% confidence level were found. The results about the small viscoelastic properties, reported in Table 4.1, show

that the longer the storage time the lower the storage ( $G'$ ) and loss modulus ( $G''$ ). On the other hand, different behaviour was measured for  $\tan\delta$ . Since a more substantial decrease of the elastic component was found in comparison with the viscous part, we can observe an increase of  $\tan\delta$  with the increase of storage time. These results can be interpreted as a reduction in dough elasticity with the increase of sub-zero storage period. Similar patterns were observed also for the dough large rheological properties (Table 4.2). The longer the storage time the lower the Kieffer and TPA parameter values. Adhesiveness was the only characteristic for which an opposite behaviour was measured. Moreover, all the measured viscoelastic attributes showed that the effect of freezing is particularly strong in the first 15 days of storage. This result might be explained by the reduction of gluten cross-linking caused by ice crystallization and water redistribution provoked by a modification in the water binding capacity of dough constituents. The mechanical action of ice crystals may have resulted in the disruption of the gluten matrix resulting in a network that was less continuous, more ruptured and separated from starch granules. This hypothesis is in accordance with the results obtained from other authors (Havet, M. *et al.*, 2000; Giannou, V. *et al.*, 2003), even if the studies were conducted on samples where yeast or additives have been used.

### ***Relationship between large and small dough rheological properties***

Using Pearson's correlation analysis, a range of correlation coefficients ( $r$ ) from 0.64 to 0.94 was obtained for the relationships between Texture Profile Analysis, Kieffer and rheometer parameters. Table 4.3 shows that  $\epsilon_H$ ,  $\sigma_{\max}$ ,  $G'$  and  $G''$  were positively correlated vs. hardness ( $r = 0.88$ ;  $r = 0.94$ ;  $r = 0.93$ ;  $r = 0.88$ ) and that  $G'$  and  $G''$  were positively correlated vs.  $\epsilon_H$ ,  $\sigma_{\max}$  ( $r = 0.83$ ;  $r = 0.77$ ;  $r = 0.88$ ;  $r = 0.83$ ). On the other hand, springiness,  $G'$  and  $G''$  were negatively correlated vs. storage time ( $r = 0.94$ ;  $r = 0.65$ ;  $r = 0.69$ ). The obtained results, shown in Table 4.3, are in accordance with the fact that all the parameters that are considered as indicators of a coherent and elastic dough ( $G'$ , hardness,  $\sigma_{\max}$ ) are positively correlated among themselves. On the other hand only storage modulus and springiness are negatively correlated vs. storage time. All these results indicate that large and small viscoelastic parameters have a significant role in the evaluation of frozen dough viscoelastic performance.

	Time (days)	Hardness (N)	Adhesiveness (N·s)	G' (kPa)	G'' (kPa)
Springiness	-0.94**				
Adhesiveness		-0.90**			
G' (kPa)	-0.65*	0.93**	-0.73*		
G'' (kPa)	-0.69*	0.88**	-0.64*		
tan $\delta$		-0.88**	0.96**	-0.73**	
$\epsilon_H$		0.88**	-0.90**	0.83*	0.77*
$\sigma_{\max}$ (kPa)		0.94**	-0.93**	0.88**	0.83*

(\* p<0.05; \*\* p<0.01).

Table 4.3 Relationship within empirical and fundamental dough rheological properties (modified from Angioloni, A. *et al.*, 2008).

## CONCLUSIONS

Small deformation (in fundamental units) and large deformation methods can be used in order to evaluate the influence of sub-zero storage time on non-fermented dough viscoelastic performance. The frozen storage period had a significant effect on the dough's hardness, springiness, adhesiveness,  $\sigma_{\max}$ ,  $\epsilon_H$ , storage and loss modulus. In general, the longer the sub-zero storage time the lower the positive viscoelastic attributes. As the changes in dough rheology are might be due to the formation and the mechanical action of ice crystals, the effect of freezing was particularly concentrated in the first 15 days of storage. It is clear that the use of frozen dough allows easier and more profitable baking, reducing labour and production cost while facilitating transportation. However, the quality of the final product prepared from frozen dough is often inferior to the freshly one. Nevertheless, the obtained results are useful in order to understand what really occur when dough, without any preservatives, is stored at freezing temperature for two months. Further researches and analyses should be conducted to better understand the issues mechanism involved and to improve the overall knowledge about the use of freezing in bakery technology.

## 4.2 Frozen dough and bread made with frozen dough

### *Effect of storage time on rheological properties of frozen and deep-frozen dough*

Although research on dough rheology has been performed for several years, some of the most important problems concerning frozen dough such as decreased yeast viability and gas production ability or diminished gluten network ability to retain CO<sub>2</sub> and progressive loss of dough cohesiveness and stability, have not been fully resolved yet.

It still remains difficult to thoroughly understand dough behaviour and use its textural properties in order to predict bread characteristics. Especially in frozen dough preparation, where freezing and sometimes prolonged frozen storage intervene between dough formation and bread making and several factors still have not fully investigated, this becomes even harder (Giannou, V. and Tzia C., 2007).

Two main problems arise in the case of yeasted bread dough freezing: yeast viability and the rheology of the dough which are both affected by freezing (Le Bail, A. *et al.*, 1999).

### TPA test on doughs

Table 4.4 shows the effect of the storage time on frozen and deep-frozen dough empirical rheological properties.

Characteristics	Dough type and storage time (days)					
	Frozen dough			Deep-frozen dough		
	1	7	14	1	7	14
Hardness (N)	0.87 b	0.79 b	7.99 a	0.75 a	0.70 a	0.78a
Resilience	0.09 b	0.08 b	0.15a	0.15 a	0.21 a	0.15 a
Springiness	0.96 a	0.97 a	0.97 a	0.98 a	1.00 a	1.00 a
Cohesiveness	0.44 b	0.43 b	0.50a	0.56 a	0.52 a	0.54 a
Gumminess (N)	0.39 b	0.34 b	4.01 a	0.42 a	0.37 a	0.42 a
Adhesiveness (N·s)	1.55 b	1.21 b	2.73 a	1.29 a	0.68 b	1.03 ab

Mean values for each characteristics within each dough type annotated by the same letter are not significantly different ( $p \leq 0.05$ ).

Table 4.4 Effect of storage time on frozen and deep-frozen dough empirical rheological properties (Tpa test).

We can observe the most remarkable change on frozen dough after 14 days of storage; hardness, resilience, gumminess and adhesiveness increase at the last day of control.

Instead there were not significant differences among the deep-frozen samples.

This should be attributable to the minor time necessary to the deep-frozen doughs compared to the frozen doughs to reach the temperature of -12.5°C, that allows to maintain good rheological properties (Cauvain, S.P., 1996; Peressini, D. and Sensidoni, A., 2004).

### Dough Uni-axial Extensibility (Kieffer Method)

Characteristics	Dough type and storage time (days)					
	Frozen dough			Deep-frozen dough		
	1	7	14	1	7	14
Resistance to extension (g)	33.58a	25.31b	17.68c	35.40b	46.62a	42.76a
Extensibility at max (mm)	37.38b	38.60b	51.68a	36.70a	37.95a	39.07a
Area (g·mm)	253.75a	183.19b	200.54b	255.34b	318.42a	314.55a

Mean values for each characteristics within each dough type annotated by the same letter are not significantly different ( $p \leq 0.05$ ).

Table 4.5 Effect of storage time on frozen and deep-frozen dough empirical rheological properties (Kieffer test).

Dough extension properties are very important when evaluating frozen doughs because they influence oven spring and loaf volume of the final baked product (Bhattacharyaa, M. *et al.*, 2003).

The Table above shows a difference in the behaviour of frozen dough compared to the deep-frozen dough as resulted from Kieffer test.

The frozen doughs showed a modification of resistance to extension and extensibility during storage time while the deep-frozen doughs were more stable during the same period of time.

This data was in agreement with the literature (Cauvain, S.P., 1996; Perissini, D. and Sensidoni, A., 2004; Giannou, V. *et al.*, 2007).

From different experiments a decrease in the extensibility of the dough subjected to freezing could be observed, and also the opposite effect (Havet, M. *et al.*, 2000).

Wolt and D' Appolonia (1984) found a decrease in extensibility with an increase in frozen storage time, which was attributed to overall gluten network deterioration. Inoue and Bushuk (1991) observed no significant changes in rheological properties during short-term storage (Bhattacharyaa, M. *et al.*, 2003).

In our experimentation was observed an increase in the extensibility on the frozen dough suggesting dough weakening, while extensibility of deep-frozen dough did not change significantly as the storage time increased from day 1 to day 14.

The resistance to extension of deep-frozen dough increased as the storage time increased from day 1 to day 14, possibly due to the stiffening of the dough during frozen storage.

The resulting loss of dough strength observed in frozen sample (Table 4.5) can be attributed firstly, to the release of disulphide reducing substances from dead yeast cells, and secondly, to the disruption of the gluten network by ice crystals (Giannou, V. *et al.*, 2003).

In the deep-frozen sample the loss of dough strength is less marked probably due to the higher freezing rate ( $0.40^{\circ}\text{C}/\text{min}$ ) compared to that of frozen samples ( $0.15^{\circ}\text{C}/\text{min}$ ). In fact the rapid freezing allows the formation of ice micro-crystals, which do not affect the integrity of the gluten network. This is without doubt one of the most important factors to minimize the damage caused by cold (Angioloni, A. *et al.*, 2008).

Nevertheless, excessively rapid lowering of the temperature might seriously compromise the yeast vitality. This implies that there is an optimum freezing speed which both allow intense nucleation of the crystals and reduces shock on yeast cells (Angioloni, A. *et al.*, 2008).

Even though the freezing rate is known to influence yeast viability and the rheology of the dough, the duration of the storage is also an important parameters.

However the temperature history during storage has a large influence on the structure of ice crystals which might recombine due to the temperature fluctuations (Le Bail, A. *et al.*, 1999).

It is therefore very important to use storage equipment capable of maintaining the temperature as constant as possible. For that reason, excessively long storage periods should be avoided.

In general storage time should be not more than 45-60 days (Angioloni, A. *et al.*, 2008).



***Effect of storage time on rheological properties of bread made by using frozen and deep-frozen dough***

**TPA test on bread**

Characteristics	Dough type and storage time (days)							
	Frozen dough				Deep-frozen dough			
	0 (fresh sample)	1	7	14	0 (fresh sample)	1	7	14
Hardness (N)	1.77 c	2.45bc	3.14b	23.72a	1.37 c	1.86b	2.74a	3.14a
Resilience	0.38 a	0.38 a	0.33a	0.34 a	0.42 a	0.42a	0.39 a	0.41 a
Springiness	0.97 a	0.99 a	0.98a	0.98 a	0.99 a	0.98a	0.99 a	0.98 a
Cohesiveness	0.69 a	0.66ab	0.61c	0.65bc	0.69 b	0.73a	0.71ab	0.72ab
Chewiness(N)	1.18 c	1.67bc	1.96b	15.58a	0.88 c	1.37b	1.96a	2.16a

Mean values for each characteristics within each dough type annotated by the same letter are not significantly different ( $p \leq 0.05$ ).

Table 4.6 Effect of storage time on empirical rheological properties (TPA test) of bread made by using frozen and deep-frozen dough.

As we can observe from the table above the most significant changes in bread made by using frozen dough were detected after 14 days of storage; hardness and chewiness increase appreciably at fourteenth day of storage.

The samples obtained by deep-frozen dough showed the same trend as the samples obtained by frozen dough even if the values at fourteenth day of storage were lower compared to the frozen dough and the increase was quite gradual.

## **CONCLUSIONS**

Dough rheology characteristics were evaluated using two different rheological methods: TPA test (performed on bread as well) and Kieffer test.

Obtained results confirmed that the freezing rate influences the dough rheological properties. Actually TPA results showed a different behaviour between frozen dough and deep-frozen dough. Frozen dough showed a remarkable change of rheological characteristics (increase of hardness, resilience, gumminess and adhesiveness) after 14 days of storage whilst the deep-frozen dough preserved unchanged its rheological characteristics over the storage time. This behaviour was confirmed by results of Kieffer test.

These differences were not detected between bread obtained by using frozen or deep-frozen dough.

Bread obtained by using frozen dough showed the most significant changes after 14 days of storage, in this case the bread obtained by using a deep-frozen dough showed the same trend even if the values at fourteenth days of storage were lower compared to the frozen dough and the increase was more gradual.

### **4.3 Doughs obtained by using different fermentation methods**

Fermentation is an important stage in the manufacturing of various bakery products, bread included. During fermentation, the expansion of previously incorporated air bubbles provides the characteristic of the aerated structure of bread. As a result of fermentation, the light, aerated, and spongy structure of fermented dough product is developed (Mert, B., 2008).

The rheological characteristics of fermented dough are determined by many factors. At the beginning of the mixing process, physical actions such as hydration take place, the gluten network is formed when proteins and starch granules absorb water.

Enzyme activity of amylases, proteases and hemicellulases causes the breakdown of several flour components. Microbial growth and metabolic activity begin after a lag phase, depending on the activity of starter cultures. Changes in pH caused by the production of lactic acid also alter the rheological behaviour of dough.

Even small chemical and physical changes in the gluten network can lead to significant changes in rheological characteristics. The increasing amount of CO<sub>2</sub> in the dough leads to the formation of bubbles. In bread dough, the gas-holding capacity is one of the most important factors affecting the volume of the final product. Dough additives, such as emulsifiers and fat, are used to improve gas-holding capacity. In sourdough, the formation, of gas bubbles leads to an increased volume and decreased density (Wehrle, K. and Arendt, E.K., 1998).

The impact of such changes in dough rheology must be considered in order to choose an appropriate proof time and to obtain good quality bread. The dough must contain a large volume of gas and also gas retention in reserve for oven rise. The proofing is really important in the bread making process in providing a link between the bubble structure created in the mixer and in the final baked loaf (Angioloni, A. *et al.*, 2006).

Although the rheology of the fermenting dough has a direct influence on the final acceptability of baked products, it is not generally included in the experimental studies. The common practice is to relate the rheological properties of non-yeasted dough with the baking performance of the yeasted products. One of the main reasons for this common practice is the lack of a suitable instrument that can conduct fast rheological measurements during fermentation. The fermenting dough is a complex and continuously evolving dynamic system requiring quick measurement techniques (Mert, B., 2008).

## *Effect of fermentation time and of different type of fermentation on rheological properties of doughs*

### **Kieffer dough extensibility test**

Characteristics	T 0			T60		
	<i>Straight dough</i>	<i>Sponge</i>	<i>Poolish</i>	<i>Straight dough</i>	<i>Sponge</i>	<i>Poolish</i>
Resistance to extension (g)	18.26cB	29.37aA	25.49 bB	24.38bA	31.78aA	29.34 aA
Extensibility at max (mm)	22.87aA	22.3 aA	18.99 bA	15.37 aB	15.2 aB	13.99 bB
Extensibility at break (mm)	40.57aA	38.36aA	35.19 bA	27.14 abB	28.96 aB	25.28 bB
Area (g·mm)	170.56cA	247.47aA	197.61bA	136.78 bB	183.58aB	149.61bB

Mean values for each characteristic within each time annotated by the same lower case letter are not significantly different (P<0.05).

Mean values for each characteristics within each dough type annotated by the same upper case letter are not significantly different (P<0.05).

Table 4.7 Effect of fermentation time and type of fermentation on empirical rheological properties (Kieffer test).

After mixing (T0) there were significant differences among the doughs about resistance to extension, extensibility and area parameters.

The dough realized by using straight dough method showed the lowest value of resistance to the extension and of area and the highest value of extensibility at break.

Dough obtained by using a sponge method showed the highest value of resistance to extension and area.

Dough realized with poolish method showed the lowest value of resistance to the extension and extensibility (at max and at break).

After 60 min of fermentation some of these parameters showed the same trend as after mixing. In fact the dough obtained by using straight dough method had the lowest value of resistance to the extension and the dough realized with poolish method showed the lowest value of extensibility at max.

The dough realized by using sponge method showed the highest value of resistance to extension and area both after mixing and after 60 min of fermentation.

The low pH of the sponge and its unique rheological characters are carried through to the dough where they have effect of producing a more extensible gluten network after second mixing, infact the dough realized by using sponge method showed, after 60 min of fermentation, the highest value of extensibility at break (Cauvain, S.P., 2001).

The dough development continues during fermentation. An hypothesis supported by Halton is that the resistance of fermenting doughs in stretching tests increased during fermentation, whereas that of non-fermenting doughs decreased with time (Bloksma, A.H., 1990).

Our results are in agreement with this finding; in fact the doughs analysed immediately after mixing showed a lower value of resistance to extension compared to the doughs after the fermentation process. Thus, fermentation appeared to increase the resistance to extension of the doughs.

Besides the fermentation allows the dough to become less extensible after 60 min, considering that the samples showed a significant decrease of extensibility at max and at rupture point. Moreover the doughs became less strong, as confirmed by a significant decrease of area.

The effects of sponge-dough fermentation on rheological properties of dough were examined by a number of investigators (Landis, Q. and Freilich, J., 1934; Freilich, J. and Frey, C.N., 1939; Ikezoe, K. and Tipples, K.H., 1968; Barber, B. *et al.*, 1980). Preston and Kilborn (1982), Kilborn and Preston (1982), and Casutt *et al.* (1984) used the extensigram to measure dough rheological properties during fermentation. They showed that fermentation decreases extensigram length, maximum height and area.

Similarly, Pizzinatto and Hosney (1980) reported that fermentation of cracker sponges changes the extensibility. Most of these reports are concerned with the effects of fermentation on rheological or physical properties, but only a few studied the mechanism of change in rheological properties (Shiiba, K. *et al.*, 1990).

Our results of extensibility and area, obtained by using Kieffer test, are in agreement with these previous works.

## Dough stickiness test

Characteristics	Dough type and fermentation time (min)					
	T0			T60		
	<i>Straight dough</i>	<i>Sponge</i>	<i>Poolish</i>	<i>Straight dough</i>	<i>Sponge</i>	<i>Poolish</i>
Stickiness (g)	61.84 a A	54.02 b A	50.99 b B	58.94 a A	53.84 bA	55.09 bA
Area (g·mm)	7.01 a A	5.91 b A	5.04 c B	7.09 a A	5.99 b A	6.3 bA

Mean values for each characteristic within each time annotated by the same lower case letter are not significantly different ( $P < 0.05$ ).

Mean values for each characteristics within each dough type annotated by the same upper case letter are not significantly different ( $P < 0.05$ ).

Table 4.8 Effect of fermentation time and type of fermentation on empirical rheological properties (Dough stickiness test).

The dough realized by using straight dough method showed the highest stickiness both after mixing and after 60 min of fermentation.

Instead no significant differences were detected between doughs realized by using sponge and poolish method.

Fermentation did not affect in a significant way the stickiness of the dough, unless the dough made by using a poolish method, in which there was an increase of stickiness after 60 min of fermentation.

## Fundamental rheological properties

Characteristics	Dough type and fermentation time (min)					
	T0			T60		
	<i>Straight</i> <i>dough</i>	<i>Sponge</i>	<i>Poolish</i>	<i>Straight</i> <i>dough</i>	<i>Sponge</i>	<i>Poolish</i>
$\delta$ (°)	23.05aA	21.64aA	21.64aA	23.67abA	22.8bA	24.37aA
G' (Pa)	8903bA	11818aA	8574bB	8267cA	10966aB	9864bA
G'' (Pa)	3797bA	4685aA	3414bB	3622bA	4608aB	4469aA
tan $\delta$	0.43aA	0.4aB	0.4aA	0.44abA	0.42bA	0.45aA
G* (Pa)	12700bA	16503aA	119989bB	11888cA	15574aB	11989bA
$\eta^*$ (Pa·s)	1540bA	2023aA	1473bB	1437cA	1893aB	1472bA

Mean values for each characteristic within each time annotated by the same lower case letter are not significantly different (P<0.05).

Mean values for each characteristics within each dough type annotated by the same upper case letter are not significantly different (P<0.05).

Table 4.9 Effect of fermentation time and type of fermentation on fundamental rheological properties (mean values at 1 Hz of frequency).

Immediately after mixing, the dough obtained with sponge showed significant differences for almost all the parameters unless  $\delta$  (°) and tan $\delta$  compared to the doughs obtained by using straight and poolish method.

The lower storage modulus found in dough realized by using poolish at T0 showed that the addition of sourdough reduce the dough elastic component. Similar results were found from Angioloni (2006).

After 60 min of fermentation doughs realized by using poolish method showed higher phase angle ( $\delta$ ) than the samples proofed with straight and sponge method, in agreement with a previous work (Angioloni, A. *et al.*, 2006).

All doughs showed the same behaviour for storage modulus (G') and loss modulus (G'') over the range of frequencies measured (from 0.1 to 10 Hz). In fact all doughs showed an elastic modulus (G') higher than the viscous modulus (G''), indicating that the doughs had a solid, elastic-like behaviour.

The phase angle ranges from  $0^\circ$  (ideally elastic material, Hookean solid) to  $90^\circ$  (ideally viscous material, Newtonian liquid). For all viscoelastic materials, the phase angle is between  $0^\circ$  and  $90^\circ$ ; the lower the values, the more elastic the material.

During the fermentation process there was an increase of phase angle and a decrease of storage modulus ( $G'$ ) of straight-dough and sponge-dough, indicating a decrease of elasticity of the dough that became less stiff and less elastic (Lee, S. *et al.*, 2004) as a result of a fermentation and therefore it will be more easy to laminate.

In fact the decrease of pH over fermentation time (data not shown) induces an increase in  $\tan\delta$  accompanied with a decrease in  $G'$  (Song, Y. and Zheng, Q., 2007).

The storage modulus values started to decrease after a lag period required for  $\text{CO}_2$  to diffuse through the dough matrix and saturate it. The decrease is due to the accumulation of  $\text{CO}_2$  bubbles (leavening) and change in the structure of the dough matrix (Mert, B., 2008).

A general decrease of extensibility evaluated by Kieffer test was instead registered in all the samples.

This finding is probably the result of the lower efficiency of empirical methods in appreciating microscopic structural changes (Angioloni, A. and Collar, C., 2009).

Similar to the elastic modulus, the loss modulus of dough samples also decreased during fermentation. This decrease of the loss or viscous modulus could be also attributed to the increased volume of the gas phase upon fermentation (Mert, B., 2008).

After 60 min of fermentation, the complex modulus ( $G^*$ ) decreased, indicating decreased resistance to deformation. This result is in agreement with the decrease of the area value, index of dough's force, obtained from the Kieffer test.

Instead doughs realized by using a poolish method showed a different trend. There was an increase of the elastic, loss and complex modulus over the fermentation time.

The increase of  $G'$  indicate that the sample stiffness increased after 60 min of fermentation probably it would be necessary more time for this method of fermentation realized by using a sourdough to reach similar rheological characteristics of straight dough and sponge dough

The use of sourdough in the poolish method increased the phase angle values even if not significant difference were found between T0 and T60; in agreement with Angioloni (2006).



## Image Analysis

If breadmaking can be characterized as a series of aeration stages, then the mechanisms by which gas cells in the dough create the cellular structure of the bread crumb need to be studied. Understanding how air bubbles nucleate in the bread dough during mixing is a fundamental first step because it was shown conclusively over 60 years ago that these air bubbles are the only nuclei available for subsequent gas cell growth (Baker, J.C. and Mize, M.D., 1941). A further reason for studying bubbles in dough is that dough exhibits extremely complex rheological properties and bubble numbers and sizes will affect dough rheology (Bellido, G.G. *et al.*, 2006).

Measuring bubble growth during proving is a difficult challenge that has not yet been fully achieved. Whitworth and Alava (1999) measured the bubble size distribution ex-mixer and after intermediate proof, and also produced computed tomographic images of proving doughs, but resolution limits precluded the measurement of the bubble size distribution and its change with time (Dobraszczyk B.J. *et al.*, 2001).

It is unclear what happens to the gas cells during fermentation and baking. It is generally believed that the loss of gas is due to the rupture of the walls of the gas cell.

Some authors suggested that the integrity of the gas cells is maintained by the existence of a liquid film of surface-active materials at the gas-liquid interface, and that the gas cells are stabilized by the liquid film. In their view, this liquid film plays an important role in gas retention. The gas cells remain discrete during the first stage, until discontinuities develop in the starch-protein matrix, and the degree to which such discontinuities occur is largely dependent on the gluten proteins. The rheological properties of the bulk phase determine the extensibility at this stage. With increasing fermentation time, the surface area of the liquid film will increase. The stability of the liquid film determines the behaviour of the dough at this stage. Surface-active materials probably stabilize the film so that it can expand across a larger area without rupturing (Gan, Z. *et al.*, 1995).

Fermentation may also cause changes in cell-wall components, through the activities of endogenous or added enzymes (Autio, K. and Laurikainen, T., 1997).

Characteristics			Dough type and fermentation time (min)								
			T 0			T30			T60		
			<i>Straight dough</i>	<i>Sponge</i>	<i>Poolish</i>	<i>Straight dough</i>	<i>Sponge</i>	<i>Poolish</i>	<i>Straight dough</i>	<i>Sponge</i>	<i>Poolish</i>
TOTAL OBJECTS			949aB	671bC	1129aB	1444aA	1033bB	1494aA	1390abA	1223bA	1466aA
CLASSE 1	Objects	N°	283abB	242bC	310aB	436aA	335bB	389aA	399aA	394.78aA	385.38aA
		%	29.65bA	36.00aA	27.93bA	30.06bA	32.61aB	26.04cA	28.79bA	32.31aB	26.27cA
	Area	TOT	0.23abB	0.20bC	0.26aB	0.36aA	0.28bB	0.32aA	0.33aA	0.33aA	0.32aA
		%	4.98bA	7.89aA	4.77bA	5.85bA	7.19aA	4.54cA	5.02bA	7.09aA	4.57bA
CLASSE 2	Objects	N°	576aB	389bC	702aB	888aA	631bB	954aA	860abA	748bA	930aA
		%	60.84aA	57.68bB	62.10aB	61.39bA	60.84bA	63.86aA	61.58bA	61.03bA	63.33aAB
	Area	TOT	1.67aB	1.01bC	2.04aB	2.50bA	1.65cB	2.80aA	2.46aA	2.00bA	2.78aA
		%	35.59aA	39.54aA	36.35aA	40.23aA	42.34aA	39.36aA	37.23aA	42.96aA	39.06aA
CLASSE 3	Objects	N°	86aB	39bC	113aA	116bA	65cB	148aA	127aA	78bA	148aA
		%	9.08aA	6.03bA	9.60aA	8.20bA	6.35cA	9.91aA	9.24aA	6.43bA	10.17aA
	Area	TOT	2.17abB	1.00bC	2.92aA	2.79bA	1.64cB	3.68aA	3.15aA	1.95bA	3.75aA
		%	46.08abA	38.96bA	47.17aA	43.74bA	42.15bA	50.42aA	47.25abA	41.68bA	50.38aA
CLASSE 4	Objects	N°	4.00aA	1.88bA	4.00aA	4.56aA	2.00aA	2.78aA	4.78aA	2.89aA	3.38aA
		%	0.43aA	0.29aA	0.37aA	0.35aA	0.20aA	0.19aB	0.38aA	0.23aA	0.24aAB
	Area	TOT	0.63abA	0.34bA	0.64aA	0.69aA	0.34aA	0.40aA	0.70aA	0.40aA	0.46aA
		%	13.35aA	13.61aA	11.71aA	10.18aA	8.31aA	5.68aB	10.50aA	8.28aA	5.99aB

Mean values for each characteristic within each time annotated by the same lower case letter are not significantly different ( $P < 0.05$ ).

Mean values for each characteristics within each dough type annotated by the same upper case letter are not significantly different ( $P < 0.05$ ).

Table 4.10 Dough morphological features.

By using image analysis the structure of the different doughs realized by using different fermentation method and the changes over the fermentation time was observed.

The sample realized by using sponge method showed the smaller amount of objects of the four different classes over the fermentation time. Even the area occupied by the objects was smaller compared to the doughs realized by using the others fermentation method (straight method and the poolish method).

The smaller area occupied by the objects means that the dough showed a more compact structure, this was in agreement with the lower value of  $\delta(^{\circ})$  obtained from rheological measurements.

During proof and baking the growth of gas bubbles determines the expansion of the dough and therefore the ultimate volume and texture of the baked product (He, H. and Hosney, R.C., 1991). The limit of expansion of these bubbles is related directly to their stability, due to coalescence and the eventual loss of gas when the bubbles fail. The rheological properties of the bubble walls will therefore be important in maintaining stability against premature failure during baking, and also in relation to gas cell stabilization and gas retention during proof, and thus to the final structure and volume of baked product (Dobraszczyk, B.J. and Morgenstern, M.P., 2003).

While an increase of total objects in the different doughs was detected after 30 min of fermentation, a further increase at T60 was measured only for the dough obtained with the sponge dough method.

Over the fermentation time the different classes showed a different trend.

For the dough realized by using straight-dough method the number of the objects belonging to class 1 increased after 30 min of fermentation and after 60 min decreased, but not in a significant way, while the number of the objects belonging to the class 3 and 4 increased over the fermentation time. This is probably due to the effect of coalescence that involves the fusion of gas bubbles through the rupture of the thin film between them.

The same trend was detected also for the dough realized by using poolish method.

Instead the dough realized by using a sponge method showed a different trend: the number of the objects of the four different classes increased over the fermentation time.

## **CONCLUSIONS**

The rheological properties of fermenting dough were investigated using empirical and fundamental analysis and image analysis was used to integrate this information throughout the evaluation of the structure of the different doughs over the fermentation time.

Dough viscoelastic properties changed over fermentation time; in general the elastic behaviour of dough obtained with straight and sponge method decreased during fermentation. Actually doughs showed an increase of resistance to extension and a decrease of extensibility, obtained from Kieffer test, after 60min of fermentation. Different types of fermentation had a clear effect on final rheological characteristics. The results of fundamental rheological test showed that the incorporation of sourdough (poolish method) provoked changes that were different from those seen in the others type of fermentation. Actually dough realized by using poolish method showed the lower storage modulus ( $G'$ ) after mixing ( $T_0$ ) and the higher phase angle ( $\delta$ ) after 60 min of fermentation. Such changes may be attributed to a number of intrinsically related factors, including variations in the rate or amount of acid produced (Angioloni, A. *et al.*, 2006).

There is evidence from the literature that the image analysis for quantitative assessment of bread crumb attributes is effective, but unfortunately there is a lack of information about application of image analysis to evaluate the dough structure. In this experimentation the image analysis was applied to evaluate dough structure and the changes over the fermentation time. In particular some relationship between the values obtained by using image analysis (area of objects) and fundamental rheological measurements ( $\delta$ ) were found.

## **4.4 Doughs made by Kamut® flour**

### ***Effect of different ingredients on rheological properties of dough***

#### **Kieffer dough extensibility test**

The resistance to extension and extensibility were determined in uniaxial extension mode using the Kieffer Extensibility Rig in order to evaluate the effect of the addition of oil and emulsifier on the rheological properties of the doughs.

The three doughs showed significant differences on resistance to extension and extensibility parameters immediately after mixing (Table 4.11). Control dough (CTR T0) showed higher value of resistance to extension respect to the doughs supplemented by emulsifier and olive oil (EMU T0 and OIL T0 respectively). Thus, addition of oil or emulsifier appeared to decrease the strength of the doughs. Similar observations on wheat flour dough samples were made by Indrani and Venkateswara (2007).

The resistance to extension was modified by lecithin addition which decreased this parameter values in comparison to control dough. This result confirmed the weakening effect described for lecithins (Gómez, M. *et al.*, 2004; Stampfli, L. *et al.*, 1996).

Moreover incorporation of these ingredients resulted in a more extensible dough as indicated by the higher values of extensibility at max (EMU T0 = 40.09 mm and OIL T0 = 42.24 mm) when compared to the control value (CTR T0 = 37.55 mm).

#### **Texture profile analysis**

According to TPA results reported in Table 4.11 there is a clear effect of emulsifier on dough rheological properties.

Dough cohesiveness of the doughs decreased with oil (0.78) and emulsifier (0.69) addition when compared to the control value (0.80). However the decrease was greater for the dough containing emulsifier. Similar observations were made by Indrani (2007) on wheat flour dough samples with the addition of oil.

## ***Effect of fermentation process and cold storage on rheological properties***

### **Fermentation process**

Fermentation process induced a significant fall of resistance to extension only in the sample prepared with oil (Table 4.11). All the samples showed a decrease of extensibility indicating that the samples after 4 hours of leavening became less extensible.

The sample with oil showed the highest value of extensibility. In fact, although the differences among the samples were not significant, this dough was the most extensible not only immediately after mixing (OIL T0 = 42.24 mm) but also after the fermentation process (OIL L4 = 22.92mm).

Furthermore the sample CTR showed the highest value of resistance to extension (force) (CTR T0 = 32.65g and CTR L4 = 38.78g ).

TPA results showed certain textural changes that took place over the fermentation time in the dough differently formulated (Table 4.11). In particular the hardness of the samples prepared with oil and emulsifier fell with fermentation time. The decrease in hardness values was greater for the dough containing emulsifier than for the others samples; after 4 hours the sample prepared with oil was the less hard (OIL L4 = 41.86 N).

These results are in agreement with those found for the parameter resistance to extension obtained by the Kieffer test, in which sample prepared with oil was the less resistant to the extension.

The resilience accounted for an increase of its value over the fermentation time. Furthermore in the dough with oil resilience showed the lowest value both after mixing (OIL T0) and after 4 hours of fermentation (OIL L4).

Adhesiveness values decreased significantly for all the samples over the leavening time indicating easier machinability. Finally there were no differences among the samples after 4 hours of leavening.

Characteristic	Dough type and leavening time (hours)					
	CTR		EMU		OIL	
	T0	L4	T0	L4	T0	L4
<u>Kieffer test</u>						
RESISTANCE TO EXTENSION (g)	32.65bA	38.78aA	28.57bB	34.69aB	28.57aB	26.53bC
EXTENSIBILITY AT BREAK (mm)	37.55aB	21.47bA	40.09aAB	22.08bA	42.24aA	22.92bA
AREA (g·mm)	958.16aA	562.24bA	928.57aA	527.55bA	989.79aA	417.35bB
<u>Texture Profile Analysis</u>						
HARDNESS (N)	51aB	51aA	79aA	46bAB	54aB	42bB
COHESIVENESS (adimensional)	0.80aA	0.68bB	0.69aB	0.72aAB	0.78aA	0.75aA
RESILIENCE (adimensional)	0.05bA	0.07aAB	0.05bA	0.07aA	0.04bB	0.07aB
GUMMINESS (N)	41aB	34bA	54aA	33bA	42aB	31bA
ADHESIVENESS (N·s)	194aB	163bA	234aA	174bA	208aB	165bA

CTR=control dough; EMU=dough with emulsifier; OIL=dough with extra virgin olive oil.

Mean values for each characteristics within each dough type annotated by the same lower case letter are not significantly different (P<0.05).

Mean values for each characteristics within each time annotated by the same upper case letter are not significantly different (P<0.05).

Table 4.11 Effect of different ingredients and of fermentation time on empirical rheological properties of dough.

## ***Cold storage***

In order to study the changes in dough extensibility induced by the cold storage, the Kamut® doughs were prepared adopting the same recipe described above and successively they were subjected to refrigeration at 4°C for 24 and 48 hours. Results, showed in Table 4.12, evidenced significant differences for all the parameters for fresh sample (CTR T0, EMU T0, OIL T0) compared to the samples analysed after 24 and 48 hours of storage. Between 24 and 48 hours of storage there were not significant differences with the exception of resistance to extension for the sample with emulsifier. The resistance to extension increase, after 24 hours of cold storage, is probably due to the development of gluten network. Sample with emulsifier was less resistant to extension (after 48 hours) than the other doughs, while the extensibility decreased for all the stored samples (S24 and S48) compared to the samples analysed immediately after mixing (T0).

After 48 hours of storage, dough containing oil (OIL S48) and emulsifier (EMU S48) were more extensible and less resistant to extension than control dough (CTR S48). These results proved the importance of oil or emulsifier addition in order to obtain a dough with better rheological properties after 48 hours of storage at 4°C.

Moreover we can observe that the positive effect of emulsifiers on dough rheological properties is maximum after 48 hours of storage at 4°C. In fact after 48 hours samples with emulsifier and with oil showed lower values of hardness compared with the control dough.

Although immediately after mixing the hardness and gumminess parameters of dough with emulsifier (EMU T0) accounted for the highest values (Table 4.12), after 48 hours of cold storage they fell significantly, showing that this dough was softer than control. This result might be explained by the delayed action of emulsifier.



Characteristics	Dough type and storage time (hours)								
	CTR			EMU			OIL		
	T0	S24	S48	T0	S24	S48	T0	S24	S48
Kieffer test									
RESISTANCE TO EXTENSION (g)	32.65bA	55.10aA	54.08aA	28.57cB	56.12aA	43.88bB	28.57bB	48.98aA	45.92aB
EXTENSIBILITY AT BREAK (mm)	37.55aB	20.51bB	20.16bB	40.09aAB	21.64bAB	22.05bA	42.24aA	22.86bA	23.14bA
AREA (g·mm)	958.16aA	760.20bA	722.45bA	928.57aA	807.14bA	643.88cB	989.80aA	772.45bA	723.47bA
Texture Profile Analysis									
HARDNESS (N)	51cB	63aA	57bA	79aA	52bB	47bB	54aB	48bB	51abB
COHESIVENESS (adimensional)	0.80aA	0.68bB	0.70bB	0.69bB	0.73abAB	0.74aAB	0.78aA	0.74aA	0.76aA
RESILIENCE(adimensional)	0.05bA	0.07aA	0.08aA	0.05bA	0.07aA	0.07aAB	0.04cB	0.06bB	0.07aB
GUMMINESS (N)	41aB	42aA	40aA	54aA	38bB	35bB	42aB	35bB	38abAB
ADHESIVENESS (N·s)	194aB	186aA	178aA	234aA	185bA	170bA	208aB	192abA	180bA

CTR= control dough; EMU= dough with emulsifier; OIL=dough with extra virgin olive oil.

Mean values for each characteristics within each dough type annotated by the same lower case letter are not significantly different (P<0.05).

Mean values for each characteristics within each time annotated by the same upper case letter are not significantly different (P<0.05).

Table 4.12 Effect of cold storage on empirical rheological properties of dough.

## Microstructure

The objective of the microstructure analysis, performed by SEM, was to elucidate the eventual relationships between rheological properties and dough microstructure.

The microstructure of control dough (without additives) appeared compacted and homogeneous (Figure 4.1a) with starch granules closely embedded by gluten matrix. The microstructure of dough added by the emulsifier appeared quite different (Figure 4.1b). In fact, after mixing, the starch granules appeared “submerged” in a well compacted matrix and, differently from Ribotta (2004), with any void amount among granules and gluten network.

Furthermore the addition of olive oil (3%) gave rise to big changes in dough microstructure (Figure 4.1 c). In particular, starch granules looked embedded in a lax gluten matrix which in some zones formed flat strands similar to gluten sheets. In general this microstructure is scarcely uniform.

After the first 24 hours of cold storage, gluten matrix of control dough appeared open and its strands were thin but still quite uniform (Figure 4.1d). On the other hand gluten matrix of dough with emulsifier lost its initial packed structure and appeared quite open (Figure 4.1e). The addition of the olive oil (OIL) induced on cold stored dough a more uniform and porous microstructure respect to the dough immediately after mixing (Figure 4.1f). In general the differences among the three doughs observed after mixing step were not so evident after 24 hours of refrigeration.

After 48 hours of cold storage the strands forming gluten network of control dough lost its uniformity (data not shown). On the other hand the dough EMU and OIL appeared characterised by flat and short strands maintaining a quite structured matrix

The changes of dough microstructure induced by the fermentation process can be seen in Figure 4.1 (micrographs g. h. and j).

In particular, after 4 hours of fermentation, the microstructure of control dough (Figure 4.1g) appeared more expanded and the gluten matrix more extended due to the gas production. On the other hand, the addition of emulsifier or olive oil (Figure 4.1h and 4.1j respectively) caused especially in dough added by oil an increase of void fraction (more clear in dough OIL) due to the CO<sub>2</sub> production during leavening process.

Different relationships between textural properties and dough microstructure may be evidenced. In particular dough microstructure after mixing seemed related to the values obtained from TPA test. In fact the sample with emulsifier showed a well compacted matrix probably responsible of the higher value of hardness (78.98 N). Furthermore the decrease in

hardness values after the fermentation step evidenced in dough containing emulsifier or oil, may be associated to the increase of void fraction observed by SEM analysis. Finally in cold stored samples added by emulsifier, the falling of hardness after 24 hours at 4°C, can be consistent to the opening of microstructure.

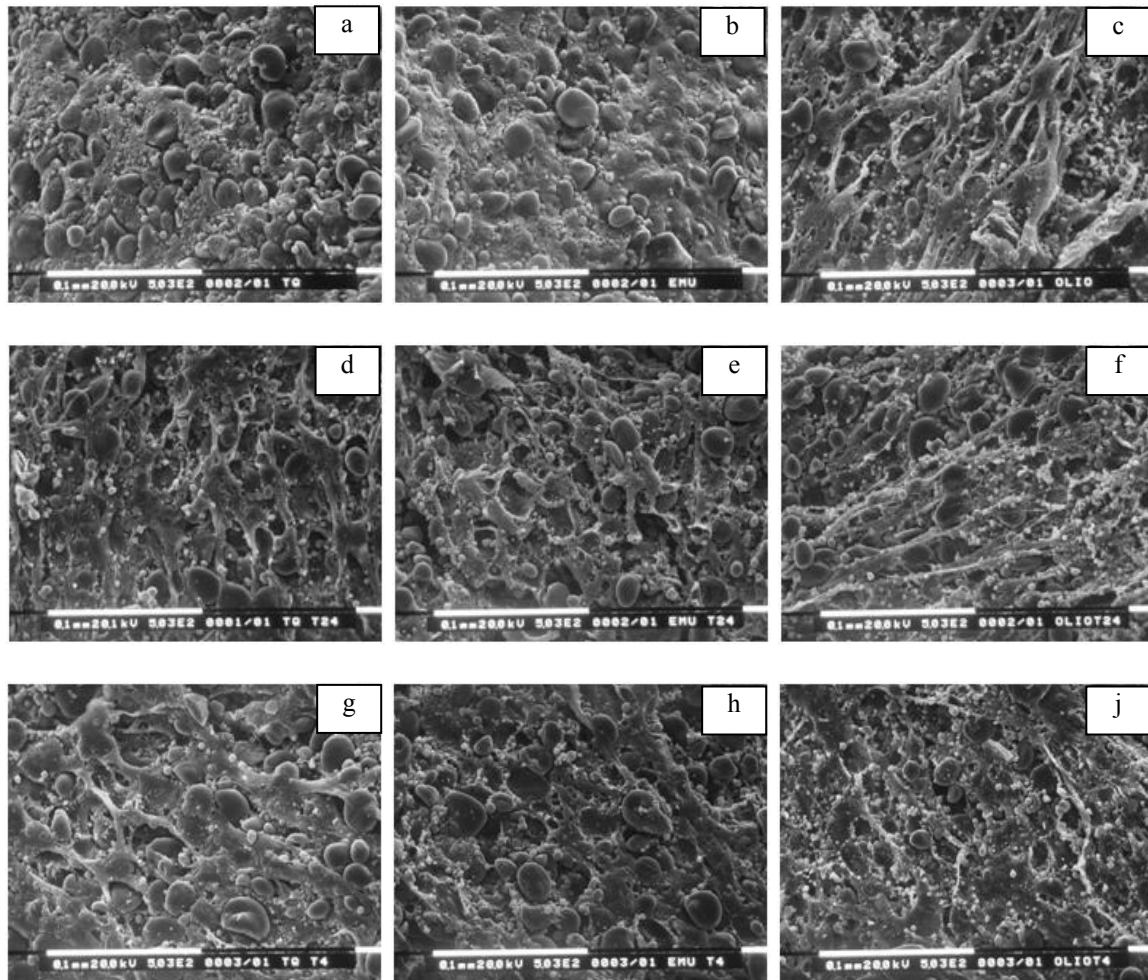


Figure 4.1: SEM micrographs of control dough, dough added by emulsifier and by olive oil: immediately after mixing (a, b and c for the three doughs respectively); after a 24 hour cold storage at 4°C (g, h and i for the three doughs respectively); after a 4 hour fermentation process (d, e and f for the three doughs respectively).

## ***CONCLUSIONS***

The present study demonstrated that some ingredients are helpful to improve rheological characteristics of Kamut<sup>®</sup> dough also when subjected to low temperatures. In particular the addition of some ingredients may improve the efficiency of dough mixing step (and consequently the texture of baked product).

The rheological properties of dough were positively influenced by the presence of emulsifiers. Over the fermentation time the addition of emulsifiers allowed to obtain a more extensible and less resistant dough. The decrease in hardness values (TPA test) was greater for the dough containing emulsifier than for the others samples; after 4 hours the sample prepared with oil showed the lowest hardness value (OIL L4 = 41.86 N).

After 48 hours at 4°C dough containing oil and emulsifier were more extensible and less resistant to extension than control dough. These results showed the importance of emulsifiers addition in order to obtain dough with good rheological properties.

This benefit could be particularly useful to small bakeries that usually adopt cold storage to reduce costs of personnel and, at the same time, they need, after refrigeration, a dough sufficiently “machinable”.

## 4.5 Dough and bread with ginger powder

In general, literature reported variable detrimental effects on dough handling and bread quality when fibres or different ingredients are added to wheat flour in sufficient high amount to ensure human physiological benefits. This because of the significant dilution of the main endogenous biopolymers – gluten and starch – that govern dough unique viscoelasticity. As a direct consequence, either rheological measurements or pasting profile are required in order to properly assess dough viscoelastic and viscometric behaviour. Hardness, cohesiveness, adhesiveness, storage and loss moduli are widely used for dough macroscopic and molecular viscoelastic assessment. All mentioned analyses measure those properties that are principally related to the gluten network (Angioloni, A. and Collar, C., 2008).

### *Results of rheological measurements on dough*

#### **Fundamental rheological properties**

Doughs	$\delta$ (°)	G' (Pa)	G'' (Pa)	$\tan \delta$	$\eta^*$ (Pa·s)	G* (Pa)
Control	28.39a	9267c	5004b	0.54a	1677c	14271c
With 3% of ginger powder	27.78ab	10289bc	5402b	0.53ab	1849bc	15691bc
With 4.5% of ginger powder	27.36ab	10728b	5547b	0.52ab	1922b	16274b
With 6% of ginger powder	26.98b	12878a	6526a	0.51b	2298a	19403a

Within column values with the same following letter do not differ significantly from each other ( $p \leq 0.05$ ).

Table 4.13 Influence of ginger powder on dough fundamental rheological properties (frequency sweep) (mean value at 1Hz of frequency).

The results of the frequency sweep test are shown in Table 4.13.

All doughs showed that the elastic modulus (G') was higher than the viscous modulus (G''), indicating that the doughs had a solid, elastic-like behaviour.

Addition of ginger powder decreased the phase angle and increased the elastic modulus, indicating increased degree of elasticity of the dough that become more elastic and therefore it

will be more difficult to laminate. Dough realized with 6% of ginger powder showed significant differences in terms of  $G'$  and  $G''$ , compared to the others doughs.

Addition of ginger powder increased the complex modulus ( $G^*$ ), indicating increased resistance to deformation and significant differences were found between control dough and sample with 6% of ginger powder.

Sample with ginger powder showed an higher value of complex viscosity ( $\eta^*$ ) compared to the control dough.

Similar results were found by Daramola and Osanyinlusi (2006) that studied ginger and starch interactions by using Rapid Visco Analyser (RVA). High peak viscosities of ginger modified cassava starches in comparisons to the native starch suggest cross linkage of starch molecules by active components of the ginger root. Cross-linked starch is more resistant to the loss of viscosity at low pH values than other kinds of starch.

The nature of modification effected by active components of ginger is cross-linking. This could be due to the bi-functional entities containing an activated double bond as well as hydroxyl/methoxy phenyl residues present in active ginger components such as gingerol, dehydroginger-dione and shogaol (Daramola, B. and Osanyinlusi, S.A., 2006).

All the parameters obtained by using a rheometer and an RVA directly or indirectly assess dough stiffness/thickness characteristics. Storage modulus and phase angle are mainly focused on gluten related properties while peak viscosity is instead linked to starch features (Angioloni, A. and Collar, C., 2008).

Viscoelastic properties are closely linked to gluten performance while pasting/gelling profiles are strongly associated to starch (Dobraszczyk, B.J. and Morgenstern, M.P., 2003). The observation can be explained on the basis that both fundamental microscopic measurements and mimetic macroscopic tests take into account not only the performance of the two individual biopolymers involved in dough structure, but also the functionality derived from interactions between them (Angioloni, A. and Collar, C., 2008).

### ***Empirical rheological properties***

#### **Kieffer dough extensibility test**

Dough	Resistance to extension (g)	Extensibility at break (mm)	Area (g·mm)
Control	12.35c	44.35a	132.42b
With 3% of ginger powder	17.88b	34.95c	146.76b
With 4.5% of ginger powder	13.45c	37.97bc	113.53c
With 6% of ginger powder	21.86a	39.27b	192.72a

Within column values with the same following letter do not differ significantly from each other ( $p \leq 0.05$ ).

Table 4.14 Influence of ginger powder on dough empirical rheological properties (Kieffer test).

Addition of ginger powder strengthened the gluten network as shown by the decrease of the extensibility suggesting that cross-linking was taking place in the dough. Ginger's addition, at the microstructural level, gives rise to a network with a higher density of cross-links. As a result, a more highly cross-linked network becomes shorter range and therefore sample with 6% of ginger powder shows lower values of extensibility compared to the control dough.

Moreover the sample obtained with the higher level of ginger powder showed the higher value of resistance to extension and area indicating a sample more resistant to the extension. These results are in agreement with the increase of  $G^*$ .

Fibre replacement or new ingredients, such as ginger powder, of flour encompasses a gluten diluting effect, a disruption of the starch–gluten matrix that force gas cells to expand in a particular dimension and an increased concentration of insoluble and soluble dietary fibres leading to a significant changes in dough functional properties as observed by uniaxial extensional measurements (Angioloni, A. and Collar, C., 2008).

## Dough stickiness test

Dough	Force (g)	Area (g·mm)
Control	93.54a	13.27a
With 3% of ginger powder	101.36a	10.08b
With 4.5% of ginger powder	78.47b	9.97b
With 6% of ginger powder	74.16b	6.79c

Within column values with the same following letter do not differ significantly from each other ( $p \leq 0.05$ ).

Table 4.15 Influence of ginger powder on dough empirical rheological measurements (dough stickiness).

The addition of ginger powder allows to obtain a dough less sticky. Actually the force value decreased with the increase of the amount of ginger powder. Consequently the area, index of work of adhesion, decreased.

This is very important in order to obtain a dough easier to handle.

Anything that gives a weaker dough (less elastic with lower  $G'$  and lower  $\delta$ ) will be measured as a sticky dough (higher area), suggesting that tack is primarily controlled by a rheological mechanism (Hoseney, R.C. and Smewing, J.O., 1999).



## ***Results of measurements on bread***

### **Image Analysis**

Bread	Total objects	Class 1 % objects	Class 2 % objects	Class 3 % objects	Class 4 % objects
Control	182.04c	62.69a	27.96a	6.65a	2.70ab
With 3% of ginger powder	199.14b	62.49a	26.41ab	8.91a	2.18b
With 4.5% of ginger powder	199.87b	61.85a	25.50bc	9.13a	3.53a
With 6% of ginger powder	217.61a	64.26a	26.21ac	7.38a	2.15b

Within column values with the same following letter do not differ significantly from each other ( $p \leq 0.05$ ).

Table 4.16 Crumb morphological features of different breads with different amount of ginger powder.

As we can see from the Table 4.16 the majority of pores were small and belonging to the class 1, index of crumb structure fine and uniform.

The increase of ginger powder does not influence the percentage of objects belonging to the different classes.

### **Texture Profile Analysis (TPA)**

	Control	3% of ginger powder	4.5% of ginger powder	6% of ginger powder
Hardness (N)	1.39b	1.51b	1.46b	1.89a
Cohesiveness	0.86a	0.87a	0.84b	0.83b
Resilience	0.53b	0.56a	0.53b	0.52b
Springiness	0.996ac	0.998a	0.996bc	0.997ab
Gumminess (N)	1.20b	1.31b	1.23b	1.56a
Chewiness (N)	1.20b	1.30b	1.22b	1.55a

Within row values with the same following letter do not differ significantly from each other ( $p \leq 0.05$ ).

Table 4.17 Influence of ginger powder on bread empirical rheological properties (TPA test).

The TPA results showed certain textural changes that took place in the dough differently formulate (Table 4.17). In particular the hardness, gumminess and chewiness of the samples prepared with higher amount of ginger powder increased, indicating that the sample became

more hard. This is in agreement with the lower value of  $\delta$  and the higher value of  $G'$  obtained from rheological measurements.

Significant differences were found just on sample with 6% of ginger bread; the same trend was observed on fundamental rheological measurements on dough.

Therefore we can suppose that the highest amount of ginger powder (6%) changes the textural characteristics of the samples.

## ***CONCLUSIONS***

Small deformation oscillatory measurements and large deformation mechanical tests provided useful information on the rheological properties of different samples analysed.

The dough with the highest amount of ginger powder (6%) showed the highest value of elastic modulus ( $G'$ ) (fundamental rheological measurements) and it was the most resistant to the extension showing the highest value of force (g) (empirical rheological measurements: Kieffer test).

This sample, after baking, showed the highest values of hardness and gumminess.

Therefore is important to choose an advantageous amount of ginger powder to employ in formulation in order to obtain an healthy baked goods with good rheological properties as well. In our experimentation this purpose was reached by the sample with the 4.5% of ginger powder.

## 4.6 Final products coming from different bakeries

### *Analysis on fresh sample of “Coppia Ferrarese” and “Pane Comune Romagnolo”*

#### Moisture content and specific volume

Table 4.18 and Table 4.19 show the moisture content and the specific volume of the fresh sample of “Coppia Ferrarese” and “Pane Comune Romagnolo”.

Sample	Moisture content (%)			Specific volume (m <sup>3</sup> /g)
	Crust	Crumb	Extremity Horn-shaped ("cornetti")	
A	11.96±0.68	24.09±0.58	7.26±0.57	3.58±0.40
B	10.3±0.58	20.81±0.51	7.98±0.12	4.42±0.19
C	11.93±0.87	21.51±0.23	8.95±0.78	4.01±0.17
D	13.07±0.43	25.9±0.7	6.02±0.76	2.8±0.18
E	11.28±0.8	20.16±0.41	9.87±0.74	4.96±0.73
F	11.6±0.64	23.63±0.71	7.2±0.75	3.44±0.49
G	12.64±0.68	25.32±0.37	9.87±0.81	3.57±0.40
H	11.41±0.63	23.32±0.68	8.66±0.71	3.27±0.98
I	11.36±0.87	19.4±0.3	8.48±0.43	4.79±0.96

Values represent mean of three replicates ± standard deviation.

Table 4.18 Moisture content and specific volume of fresh “Coppia Ferrarese”.

Sample	Moisture content (%)		Specific volume (m <sup>3</sup> /g)
	Crust	Crumb	
1	14.3ab	35.6bc	4.23 b
2	12.4c	32.4c	3.86 c
3	12.2c	34.8bc	4.23 b
4	11.2c	34.6bc	5.04 a
5	13.1b	34.5bc	4.08 b
6	13.5b	39.2a	4.29 b
7	15.0b	37.3b	2.62 d
8	13.2c	36.3b	3.83 c
9	13.4bc	37.2b	3.92 c
10	13.6b	32.5c	4.25 b
11	12.7bc	34.4bc	4.92 a

Within column values with the same following letter do not differ significantly from each other ( $p \leq 0.05$ ).

Table 4.19 Moisture content and specific volume of fresh “Pane Comune Romagnolo”.

## Texture Profile Analysis (TPA)

Sample	Hardness (N)	Cohesiveness	Resilience	Springiness	Gumminess (N)	Chewiness (N)
A	6.37±0.05	0.50±0.01	0.15±0.01	0.87±0.01	3.14±0.03	2.74±0.02
B	4.61±0.03	0.45±0.02	0.14±0.04	0.90±0.03	2.06±0.04	1.86±0.04
C	6.86±0.04	0.48±0.05	0.14±0.03	0.82±0.06	3.33±0.05	2.74±0.08
D	5.59±0.04	0.44±0.03	0.11±0.07	0.80±0.06	2.16±0.11	1.67±0.09
E	5.59±0.02	0.46±0.07	0.11±0.05	0.83±0.05	2.25±0.08	1.86±0.07
F	5.49±0.03	0.48±0.05	0.15±0.03	0.84±0.04	2.35±0.08	1.96±0.07
G	5.89±0.05	0.54±0.08	0.17±0.09	0.89±0.05	3.14±0.04	2.84±0.03
H	5.68±0.02	0.43±0.02	0.18±0.07	0.94±0.06	2.45±0.09	2.25±0.08
I	5.68±0.02	0.52±0.07	0.16±0.03	0.86±0.03	2.94±0.09	2.55±0.08

Values represent mean of three replicates ± standard deviation.

Table 4.20 Empirical rheological properties obtained from TPA test on “Coppia Ferrarese” bread.

Sample B showed the lowest value of hardness (4.61 N) and a highest value of springiness (0.90), while sample C showed the higher value of hardness (6.86 N).

Sample	Hardness (N)	Resilience	Cohesiveness	Springiness	Gumminess (N)	Chewiness (N)
1	6.82b	0.35c	0.74b	0.93a	4.62c	4.31b
2	21.98a	0.63a	0.68c	0.84c	16.10a	13.70a
3	6.80b	0.53b	0.67c	0.80c	4.34c	3.47c
4	3.33d	0.36c	0.71b	0.88b	1.93e	1.69d
5	4.92c	0.28d	0.61d	0.94a	3.30d	3.09c
6	5.11c	0.43c	0.87a	0.94a	4.92b	4.52b
7	5.97b	0.33d	0.68c	0.91b	3.60d	3.30c
8	6.02b	0.36c	0.68c	0.93a	4.55c	4.24b
9	5.62bc	0.36c	0.67c	0.94a	3.51d	3.30c
10	4.38c	0.23e	0.68c	0.89b	3.60d	3.22c
11	3.36d	0.28d	0.62d	0.94a	2.05e	1.92d

Within column values with the same following letter do not differ significantly from each other ( $p \leq 0.05$ ).

Table 4.21 Empirical rheological properties obtained by TPA test on “Pane Comune Romagnolo”.

Specific volume of bread samples is a characteristic quality parameter as it indicates dough inflating ability and oven spring (Giannou, V. and Tzia, C, 2007).

In general the greater the product volume the lower will be the hardness value (i.e. it will be softer).

Sample 4 had the highest value of specific volume (Table 4.19) and as a consequence the lowest value of hardness (Table 4.21).

All the samples analysed showed similar value of hardness, gumminess and chewiness except sample 2 that had the highest values of hardness, gumminess and chewiness and one of the lowest value of the springiness.

These values are in agreement with the lowest moisture content of the sample (Table 4.19), in fact the increase in firmness is related to the decrease in moisture. Moisture content has been shown to be inversely proportional to the rate of firming. Bread firmness is caused mainly by the formation of cross-links between partially solubilised starch and gluten proteins. In bread, water acts as a plasticizer. When moisture decreases, it accelerates the formation of cross-links between starch and protein and, thus, the bread firms faster. Therefore, crumb moisture and firmness are closely related (He, H. and Hoskeney, R.C. 1990)

However, over one century ago, Boussingault (1852) showed that bread firming occurred without a loss of water.

The samples were realized in craft bakery and as a consequence were handmade by using different ingredients in formulation and following different bread making processes. For this reason, although all the samples analysed belong to the same typology of bread, it is not an unusual outcome that a sample, sample 2 in this experimentation, showed textural characteristics clearly different compared to the other samples.

## Image Analysis

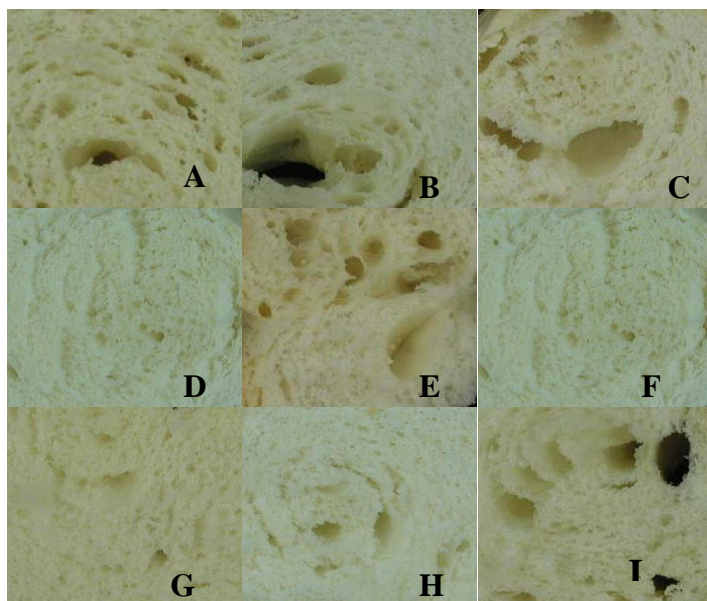


Figure 4.2 Portion of “Coppia Ferraese”’s slices.

Samples	Max Area (mm <sup>2</sup> )	Mean Area (mm <sup>2</sup> )	Number of cells	Number of cells/cm <sup>2</sup>
A	7.63 ±2.31	0.35±0.07	202.3±16.05	51
B	14.37±3.86	0.57±0.03	208.5±11.15	52
C	22.33±3.36	0.57±0.10	173.8±17.13	43
D	5.53±1.12	0.39±0.03	292±14.44	73
E	12.59±2.87	0.83±0.04	117.8±11.24	29
F	5.99±1.66	0.27±0.04	251.2±9.43	63
G	3.59±1.40	0.24±0.08	258.5±13.60	65
H	5.12±1.40	0.27±0.04	209.2±9.31	52
I	9.26±2.71	0.61±0.04	148±14.77	37

Values represent mean of three replicates ± standard deviation.

Table 4.22 Crumb morphological features of different sample of “Coppia Ferrarese”.

Among the samples, significant differences were found about crumb morphological structure. Samples D, F and G showed the highest number of cells, the highest cell density (number of cells/cm<sup>2</sup>) and the lowest mean area (mm<sup>2</sup>) indicating a more fine and homogeneous crumb structure.



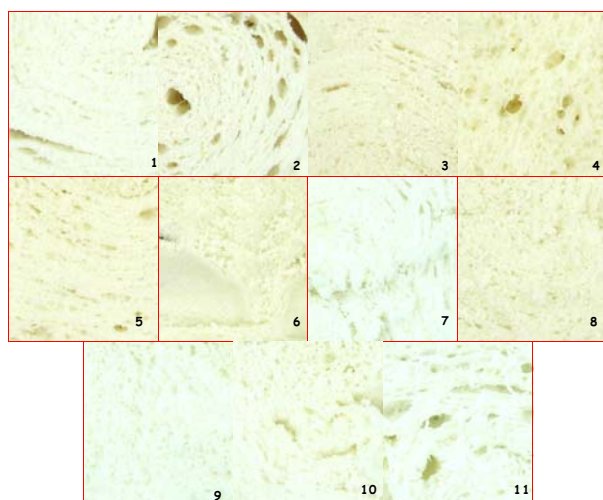


Figure 4.3 Portion of “Pane Comune Romagnolo”’s slices.

Samples	Max Area (mm <sup>2</sup> )	Mean Area (mm <sup>2</sup> )	Cells number	Number of cells /cm <sup>2</sup>
1	24.71a	0.51b	133.7c	45c
2	26.40a	1.02a	151.3bc	50b
3	4.29c	0.27c	200.2a	67ab
4	10.94b	0.44bc	179.5b	60b
5	3.80c	0.29c	163.5b	55b
6	29.86a	0.58b	136.2c	45c
7	14.27b	0.56b	147.0bc	49bc
8	7.30bc	0.38c	242.7a	81a
9	5.97bc	0.33c	238.8a	80a
10	12.29b	0.66b	135.0c	46c
11	12.82b	0.80ab	164.0b	55b

Within column values with the same following letter do not differ significantly from each other ( $p \leq 0.05$ ).

Table 4.23 Crumb morphological features of different sample of “Pane Comune Romagnolo”.

In general significant differences were found among the samples. Samples 3, 8 and 9 showed a significant higher value of number of cells and number of cells/cm<sup>2</sup> compared to the other samples, indicating a fine crumb structure.

*Analysis on sample of “Coppia Ferrarese” and “Pane Comune Romagnolo”  
after 1 day of storage*

**Moisture content**

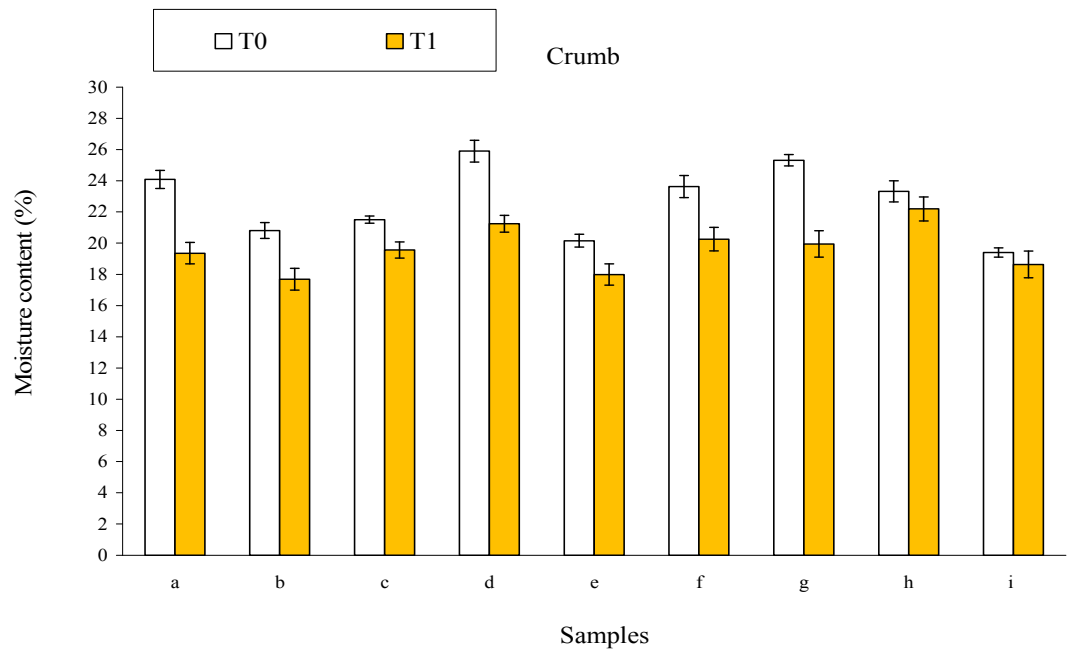


Figure 4.4 Crumb moisture content variation of “Coppia Ferrarese” samples during storage.

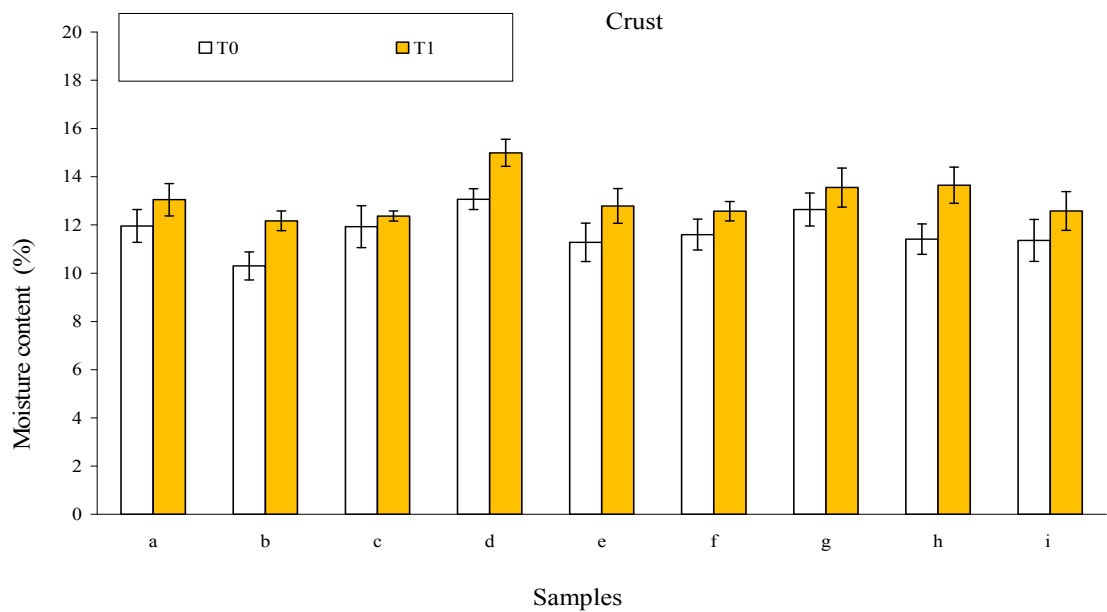


Figure 4.5 Crust moisture content variation of “Coppia Ferrarese” samples during storage.

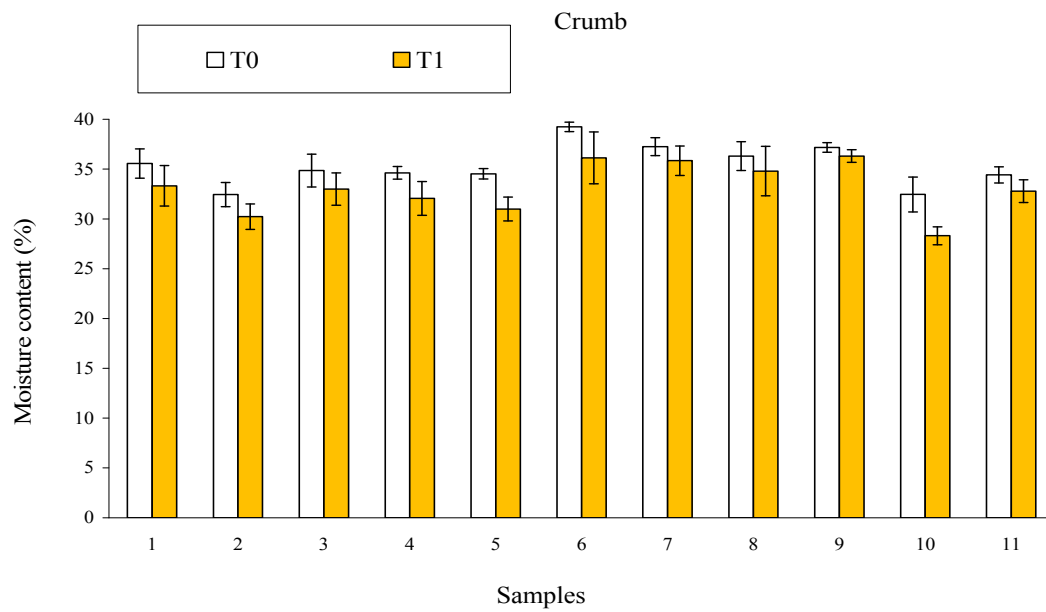


Figure 4.6 Crumb moisture content variation of “Pane Comune Romagnolo” samples during storage.

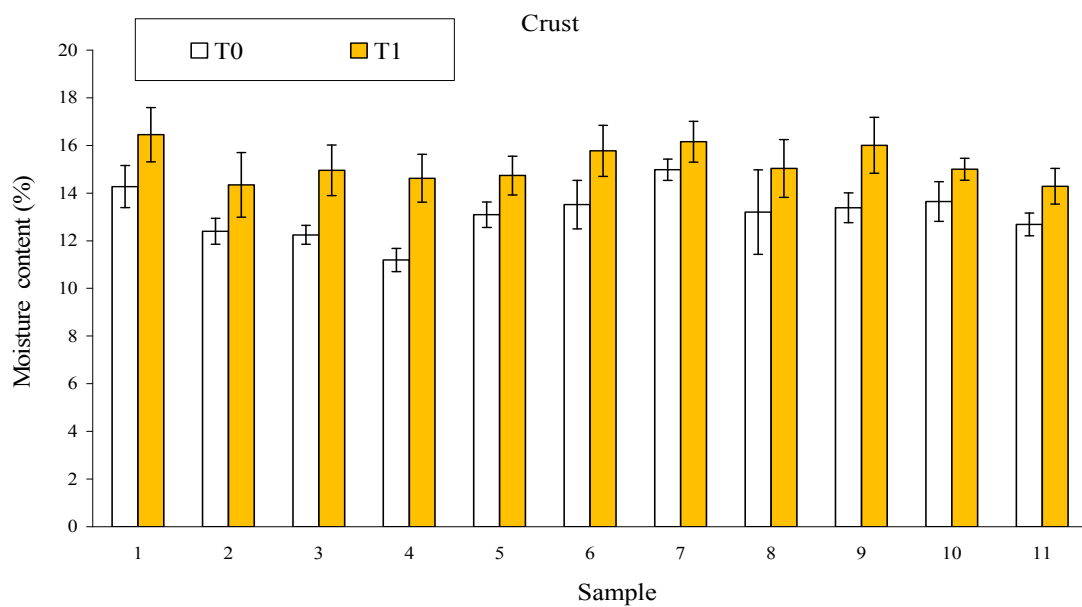


Figure 4.7 Crust moisture content variation of “Pane Comune Romagnolo” samples during storage.

Bread staling falls into 2 categories: crust staling and crumb staling. Crust staling is generally caused by moisture transfer from the crumb to the crust, resulting in a soft, leathery texture and is generally less objectionable than is crumb staling. Crumb staling is more complex, more important, and less understood. The firmness of bread varies with position within a loaf, with maximum firmness occurring in the central portion of the crumb (Gray, J.A. and Bemiller, J.N. 2003).

Many investigations have examined the phenomenon of crumb-firming, and many theories have been proposed and discussed in previous reviews (Herz, K.O., 1965; Willhoft, E.M.A., 1973; Zobel, H.F., 1973; Maga, J.A., 1975; Knightly, W.H., 1977; Kulp, K. and Ponte, J.G.Jr., 1981; Zobel, H.F. and Kulp, K., 1996).

Water is involved in the following changes in the bread system: drying out, moisture equilibration between crumb and crust, and moisture redistribution between and among bread components. Drying out of the bread, as demonstrated by Boussingault (1852), does not explain staling, but may accelerate reactions leading to staling. Thus, moisture relationships within the crumb are important considerations when studying bread staling.

As baked bread begins to cool, a moisture gradient forms in the loaf. Differences in vapor pressures between the crust and the internal region of the loaf result in moisture migration from the crumb to the crust. Over time, the moisture content in the center of the loaf decreases, while that in the external region increases (Gray, J.A. and Bemiller, J.N. 2003).

In the Figures 4.4, 4.5, 4.6, 4.7 is possible to see this phenomenon.

Axford (1968) studied the effect of loaf volume on the rate of staling and found that lower specific volume increased the staling rate and higher specif volume lowered it (Maleki, M. *et al*, 1980).

### **Texture Profile Analysis (TPA)**

The TPA test was employed to evaluate the influence of the staling process on the bread's crumb structure.

The analyses was carried out on fresh sample (T0) and on the sample after 1 day of storage at room temperature (T1). Since this type of bread is for daily consumption, the changes in textural characteristics were followed during the first 24 hours of storage.

The hardness of the sample analysed increase over the storage time, due to the decrease on the crumb moisture content (Figure 4.4 and Figure 4.6).

Firmness is linked with the force required to compress the food between the molars. Crumb firmness is common quality characteristics for bakery products since it is strongly correlated with consumers’ perception of bread freshness (Giannou, V. and Tzia, C., 2007).

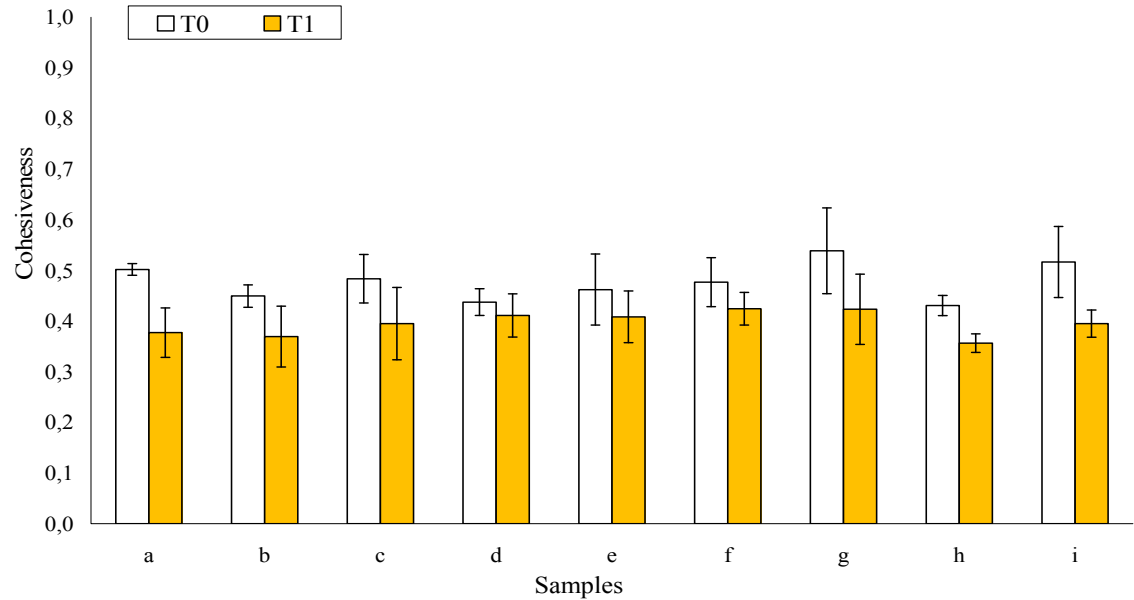


Figure 4.8 Cohesiveness variation of “Coppia Ferrarese” samples during storage.

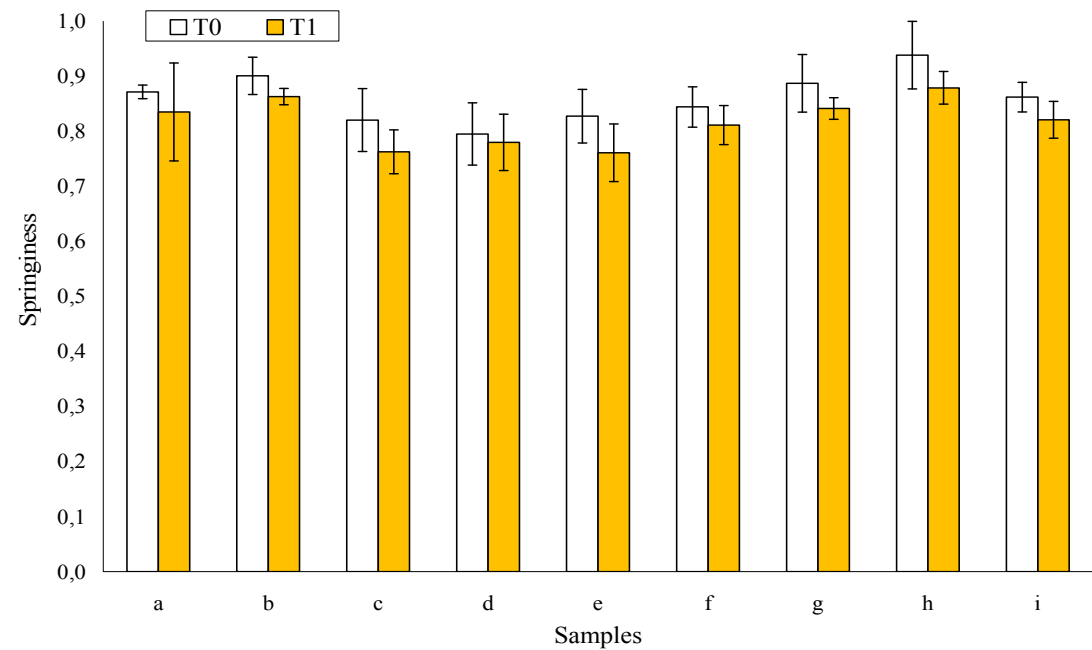


Figure 4.9 Springiness variation of “Coppia Ferrarese” samples during storage.

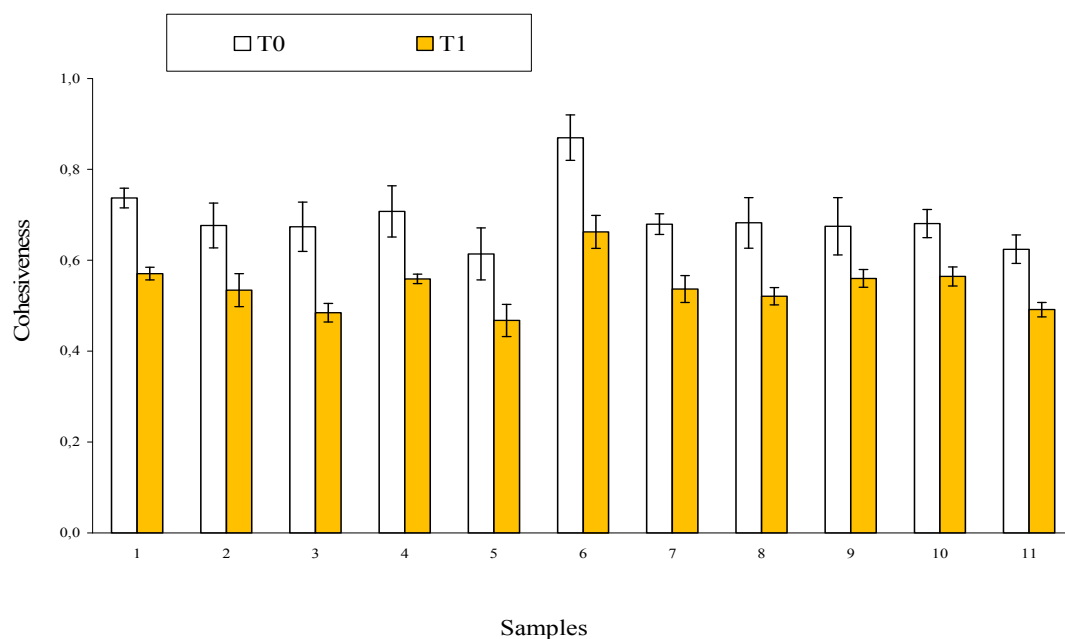


Figure 4.10 Cohesiveness variation of “Pane Comune Romagnolo” samples during storage.

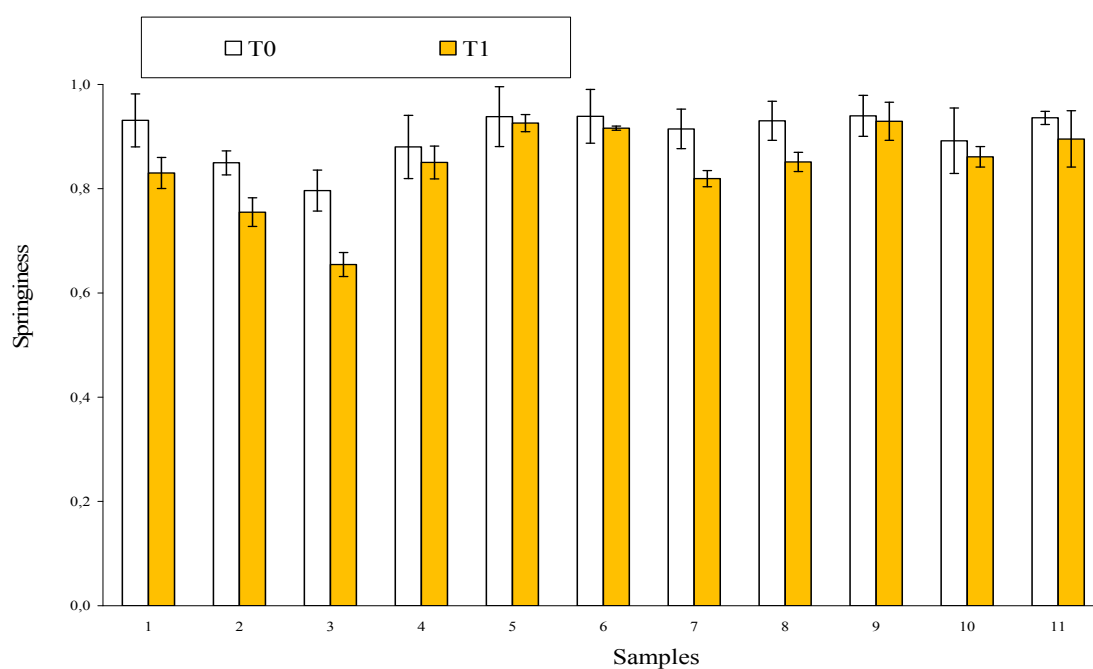


Figure 4.11 Springiness variation of “Pane Comune Romagnolo” samples during storage.

Parameters such as cohesiveness and springiness, indicator of quality in fresh bread, decrease during the storage.

As we can see from the figure above in our experiment the values are not always significantly different and this is probably due to the short period of storage, only 1 day. Similar results were obtained from Bollain (2005).

## ***FILONE TERRA DI SAN MARINO***

### **Moisture content and specific volume**

Time (days)	0	1	2	3	4
Sample 1	37.95 b	36.00 a	35.34 b	32.59 a	30.16 a
Sample 2	39.44 a	38.18 a	36.45 ab	34.31 a	28.99 ab
Sample 3	40.37 a	37.38 a	37.66 a	31.66 a	26.12 b

Within column values with the same following letter do not differ significantly from each other ( $p \leq 0.05$ ).

Table 4.24 Effect of storage time on crumb moisture content of different samples

Time (days)	0	1	2	3	4
Sample 1	14.90 a	15.98 a	15.94 a	15.20 a	15.01 a
Sample 2	15.90 a	16.72 a	16.69 a	15.58 a	14.69 a
Sample 3	15.91 a	16.65 a	16.52 a	15.38 a	14.08 a

Within column values with the same following letter do not differ significantly from each other ( $p \leq 0.05$ ).

Table 4.25. Effect of storage time on crust moisture content of different samples

Time (days)	0	1	2	3	4
Sample 1	4.31 b	4.19 b	4.18 a	4.30 a	3.86 a
Sample 2	4.49 ab	4.47b	4.45 a	4.47 a	4.46 a
Sample 3	5.28 a	5.20a	4.94a	4.91 a	4.45 a

Within column values with the same following letter do not differ significantly from each other ( $p \leq 0.05$ ).

Table 4.26 Effect of storage time on specific volume of different samples

## Texture Profile Analysis

Table 4.27 shows the changes on the textural characteristics within each bread types between 0 and 4 days of storage.

In fact the TPA, performed about 4 hours after baking, revealed a softer crumb, lower chewiness and an higher resilience values in fresh bread compared with the stored bread.

For all bread types, crumb hardness, gumminess and chewiness significantly increased over the storage time whilst the crumb resilience (the ability of the crumb to recover from compression), for sample 1 and 2, significantly decreased , in agreement with other research (Crowley P. *et al.*, 2002).

Bakery products have a very short shelf-life and their quality is dependent on the period of time between baking and consumption. During storage, a decrease in bread freshness parallel to an increase in crumb hardness produces a loss of consumer acceptance known as staling (Arendt, E.K. *et al.*, 2007).

All textural changes found are considered to have a negative impact on the quality of this type of bread, e.g. the crumb becomes harder (increased firmness), requires more energy to disintegrate during chewing (increased gumminess) and requires a longer time for mastication (increased chewiness).

These changes, in particular the increase in the hardness values, may be attributed to the crumb moisture content decrease (Table 4.24) bound to starch retrogradation phenomenon.

Finally, cohesiveness, indicator of quality in fresh bread, decreased over the storage time.



Parameters	Samples and time of storage (days)														
	Sample 1					Sample 2					Sample 3				
	0	1	2	3	4	0	1	2	3	4	0	1	2	3	4
Hardness (N)	16.1c	33.0b	42.5b	62.5a	72.7a	12.8c	19.9c	39.4b	46.7b	66.3a	13.3c	25.8bc	32.7b	76.5a	78.0a
Resilience	0.24a	0.14b	0.11c	0.11c	0.09c	0.27a	0.17ab	0.11b	0.23ab	0.09b	0.23a	0.16a	0.15a	0.11a	0.22a
Cohesiveness	0.53a	0.38b	0.31cd	0.33bc	0.29d	0.51a	0.40b	0.33c	0.32c	0.29c	0.50a	0.44b	0.36c	0.32d	0.32cd
Gumminess(N)	8.43c	12.45b	12.94b	20.00a	20.97a	6.57c	7.84c	12.74b	15.0b	18.82a	6.47c	10.98bc	11.96b	24.79°	25.3a
Chewiness (N)	8.43c	12.45b	12.94b	20.00a	20.97a	6.86c	8.04c	12.84b	15.39ab	18.82a	6.67c	10.98c	12.05b	24.01a	25.09a

Mean values for each parameter within each sample annotated by the same letter are not significantly different (P<0.05)

Table 4.27 Changes in texture within each bread type between 0 and 4 days storage.

The Table 4.28 shows the differences in texture among bread types within each storage time. It is commonly known that the addition of sourdough had a significant impact on the textural characteristics of the bread. Application of sourdough in bread production has several documented effects including leavening, acidification, improvement of aroma, anti-staling, delayed firmness and staling and microbiology stability (Crowley, P. *et al.*, 2002).

Bread made with sourdough keeps the crumb softer over a longer time than a sample made without sourdough; unfortunately this was not supported with our results.

In fact samples 1 and 3, realized with sourdough, showed at the last day of control an higher value of hardness, even if the differences are not significant, compared to the sample 2 realized with sponge-dough process.

This result may be attributed to a not optimally management of the sourdough ( storage at not optimal temperature, etc.).

Parameters	Storage time (days) and bread type														
	0			1			2			3			4		
	1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
Hardness (N)	16.4a	13.1b	13.6ab	33.7a	20.3b	26.3b	43.4a	40.2ab	33.4b	63.8ab	47.7b	78.1a	74.2a	67.7a	79.6a
Resilience	0.24a	0.27a	0.23a	0.14a	0.17a	0.16a	0.11b	0.11b	0.15a	0.11a	0.23a	0.11a	0.09a	0.09a	0.22a
Cohesiveness	0.53a	0.51a	0.50a	0.38b	0.40b	0.44a	0.31b	0.33b	0.36a	0.33a	0.32a	0.32a	0.29b	0.29b	0.32a
Gumminess (N)	8.6a	6.7b	6.6b	12.7a	8.0b	11.2a	13.3a	13.1a	12.2a	20.4ab	15.3b	24.6a	21.4a	19.2a	25.3a
Chewiness (N)	8.6a	7.0ab	6.8b	12.07	8.2a	11.2b	13.2ab	13.1a	12.3a	20.4ab	15.8b	24.5a	21.4ab	19.2b	25.6a

Mean values for each characteristics within each time annotated by the same letter are not significantly different ( $P < 0.05$ ).

Table 4.28 Differences in texture among bread types within each storage time.

## **CONCLUSIONS**

Moisture content, specific volume, texture and crumb grain characteristics are the major quality attributes of bread products. Instrumental methods for measuring textural properties are widely used as tools for the objective measurement of texture properties. Digital image analysis is a relatively new, non-destructive technique that can be used for the evaluation of crumb grain structure given integrative information to rheological measurements.

The TPA test was useful to characterize and differentiate different bread samples.

Bread rapidly loses freshness, it is subjected to mould spoilage and its limited shelf-life has an important economical impact on the baking industry and consumers. Changes in flavour and texture taking place during storage are common called staling. This phenomenon which makes the product dry and hard is frequently attributed to starch retrogradation, which is considered as the main factor responsible for the observed increase in crumb firmness during storage.

Numerous compressibility methods have been developed to quantify the firming of bread, which has been shown to correlate with bread staling as measured by consumer acceptability. Hence, compressibility measurements are most commonly used to determine the degree of bread staleness (Gray, J.A. and Bemiller, J.N., 2003).

Hardness, for example, which has been determined successfully by using a texture analyser by using a TPA test, is often used as a measure of bread staling.

The different sample analyzed, “Coppia Ferrarese”, “Pane Comune Romagnolo” and “Filone Terra di San Marino”, showed a decrease of crumb moisture and increase in hardness over the storage time.

Parameters such as cohesiveness and springiness, evaluated by TPA, indicator of quality in fresh bread, decreased during the storage.

In our test the cohesiveness decreased over the storage time for all the samples analyzed.

By using empirical rheological test we found several differences among the samples, due to the different ingredients used in formulation and the different process used to prepare the sample, but since these products are handmade, the differences could be account as a surplus value.

## CONCLUSIONS

Empirical tests are easy to perform and often used in practical factory situations, providing data that are useful in evaluating performance during processing and for quality control. These measurements are not strictly “rheological” tests since: the sample geometry is variable and not well defined; the stress and strain states are uncontrolled, complex and non-uniform and it is not possible to define any rheological parameters such as stress, strain, strain rate, modulus or viscosity. Instead with fundamental rheological approach the properties that are measured are reproducible and can be compared between different samples, test sizes and shapes, and test methods, while the empirical measurements are purely descriptive and dependent on the type of instrument, size and geometry of the test sample and the specific conditions under which the test was performed. In order to study the structural aspects of food products, image analysis techniques are largely used for the integration of the information coming from empirical and fundamental rheological measurements.

During the Ph.D. activity, these techniques have been applied to dough and breads differently formulated and processed. The main results obtained of the different aspects investigated are following reported:

Small deformation (in fundamental units) and large deformation methods were used in order to evaluate the influence of sub-zero storage time on non fermented and on fermented dough viscoelastic performances. The frozen storage period had a significant effect on dough hardness, springiness, adhesiveness (evaluated by TPA); extensibility and resistance to extension (obtained by Kieffer test) and on storage and loss modulus. In terms of fundamental and empirical rheological properties, the longer the sub-zero storage time the lower the positive viscoelastic attributes.

Different types of fermentation (straight dough, sponge and poolish method) had a clear effect on the final rheological characteristics of dough. The fermenting dough is a complex and continuously evolving dynamic system requiring quick measurement techniques and for this reason, even if yeast plays a crucial role in breadmaking, almost all empirical and fundamental rheological testing and studies are conducted on doughs formulated without yeast. In this part of the research fundamental measurements were applied on yeasted dough; to try to avoid or at least to minimize the influence of the fermentation process, a shorter test was applied on the samples with yeast compared to the test carried out on samples without

yeast. Fundamental rheological measurements evidenced that the incorporation of sourdough (poolish method) provoked remarkable dough changes. Actually dough obtained with poolish method showed the lowest storage modulus ( $G'$ ) after mixing and the highest phase angle ( $\delta$ ) after 60 min of fermentation, compared with doughs obtained with straight dough and sponge methods.

The experimentation on dough made by Kamut<sup>®</sup> flour demonstrated that some ingredients are helpful to improve rheological characteristics of the dough also when subjected to low temperatures. In particular the addition of some ingredients may improve the efficiency of dough mixing step (and consequently the texture of baked product). The rheological properties of dough were positively influenced by the presence of emulsifiers; after 48 hours at 4°C dough containing oil and emulsifier were more extensible and less resistant to extension than control dough. Interesting relationships were found between textural properties obtained by using TPA test and dough microstructure evaluated by using SEM.

Detrimental effects on dough handling and bread quality when fibres or different ingredients are added to wheat flour in sufficient high amount to ensure human physiological benefits, were found in the literature. This because of the significant dilution of the main endogenous biopolymers – gluten and starch – that govern dough unique viscoelasticity. Rheological measurements were applied in order to properly assess dough viscoelastic behaviour as a function of different ginger powder concentrations. The rheological measurements on dough showed that the samples realized with the highest amount of ginger powder (6%) was the most elastic (highest value of  $G'$ ), and the most resistant to the extension (highest value of force from Kieffer test). This sample, after baking, showed the highest values of hardness and gumminess evaluated by using TPA test; test conditions were chosen in order to measure mainly uniaxial compression forces.

The study of commercial handmade products (realized in small bakeries) evidenced that empirical rheological tests are really useful to study structural modifications bound to the different ingredients used in formulation and processing conditions.

More in general the knowledge of formulation, processing and storage conditions together with the evaluation of structural and rheological characteristics is fundamental for the study of complex matrices like bakery products, where numerous variable can influence their final quality (e.g. raw material, bread-making procedure, time and temperature of the fermentation and baking).

## REFERENCES

- Angioloni, A. and Collar, C., 2008. "Functional response of diluted dough matrixes in high-fibre systems: A viscosimetric and rheological approach". *Food Research International* 41, 803-812.
- Angioloni, A. and Collar, C., 2009. "Small and large deformation viscoelastic behaviour of selected fibre blends with gelling properties" *Food Hydrocolloids* 23, 742-748.
- Angioloni, A., 2005. "Proprietà reologiche degli impasti ottenuti con *Triticum dicoccum* Schübler e *Triticum Spelta* L. Valutazione con il microestensografo Kieffer. *Tecnica Molitoria* 6, 605-610.
- Angioloni, A., Balestra, F., Pinnavaia, G.G. and Dalla Rosa, M., 2008. "Small and large deformation tests for the evaluation of frozen dough viscoelastic behaviour". *Journal of Food Engineering*, 87, 527-531.
- Angioloni, A., Romani, S., Pinnavaia, G.G. and Dalla Rosa, M., 2006. "Characteristics of bread making doughs: influence of sourdough fermentation on the fundamental rheological properties". *European Food Research and Technology*, 222, 54-57.
- Arendt, E.K., Liam A.M. Ryan and Dal Bello, F., 2007. "Impact of sourdough on the texture of bread. *food microbiology*". *Food Microbiology*, 24, 165-174).
- Armero E. and Collar C., 1997. "Texture properties of formulated wheat doughs. Relationship with dough and bread technological quality". *Z Lebensm unters Forsch A* 204, 136-145
- Autio, K. and Laurikainen, T., 1997. "Relationships between flour/dough microstructure and dough handling and baking properties- Review". *Trends in food research & technology*, 8, 181-185.

- Autio, K. and Sinda, E., 1992. "Frozen doughs: rheological changes and yeast viability". *Cereal Chemistry* 69 (4): 409–413.
- Axford D.W.E., Colwell, K.H., Cornford, S.J. and Elton, G.A.H., 1968. "Effect of loaf specific volume on the rate and extent of staling in bread" *Journal of the science of food and agriculture*, 19, pp 95-101.
- Baker, J.C. and Mize, M.D., 1941."The origin of the gas cell in bread dough". *Cereal Chemistry*, 18, January., 19–34.
- Baker, J.C. and Mize, M.D., 1941."The origin of the gas cell in bread dough". *Cereal Chemistry*, 18, January., 19–34.
- Barber, B., Benedito de Barber, C. and Planells, V., 1980. " The sponge for breadmaking. Its influence on the rheological and fermentative characteristics of wheat flour doughs". *Rev. Agroquim. Technol. Aliment.*, 20, 403.
- Barnes, H.A., Hutton, J.F. and Walters, K., 1989. "An Introduction to Rheology", Elsevier.
- Bellido, G.G., Scanlon M.G., Page, J.H., Benedikt Hallgrimsson, 2006. "The bubble size distribution in wheat flour dough" *Food Research International* 39, 1058-1066.
- Bhattacharyaa, M., Langstaffa Tami, M. and Berzonskyb, W.A., 2003. "Effect of frozen storage and freeze–thaw cycles on the rheological and baking properties of frozen doughs". *Food Research International*, 36, 365-372.
- Bloksma, A.H. and Bushuk, W., 1988. "Rheology and chemistry of dough". In *Wheat: Chemistry and Technology*, 3rd Ed., Volume 11, (Y. Pomeranz, ed.) pp. 131-217, American Association of Cereal Chemists, St. Paul, Minnesota.
- Bloksma, A.H., 1962. "Slow creep of wheat flour doughs". *Rheologica Acta* 2,217-230.
- Bloksma, A.H., 1990. "Rheology of the bread making process". *Cereal Foods World* 35(2), 228-236, 959-960.



- Bollain, C., Angioloni, A. and Concepción, C., 2005. "Bread staling assessment of enzyme-supplemented pan breads by dynamic and static deformation measurements". *European Food Research Technology*, 220, 83-89.
- Bourne, M.C., 1968. "Texture Profile of ripening pears". *Journal of Food Science*. 33, 223-226
- Bourne, M.C., 1978. "Texture Profile Analysis". *Food Technology*. 32 (7), 62-66, 72.
- Bourne, M.C., Moyer, J.C. and Hand, D.B., 1966. "Measurement of food texture by a Universal Testing Machine". *Food Technology* 20(4), 522-526.
- Boussingault J.B., 1852. "De la transformation du pain tender en pain rassis" *Ann Chem. Phys.* 3 Ser. 36, p.490
- Breene, W.M., 1975. "Application of texture profile analysis to instrumental food texture evaluation". *Journal Texture Studies* 6, 53-82.
- Brosnana, T. and Sun, D-W., 2004."Improving quality inspection of food products by computer vision – a review. *Journal of Food Engineering* 61, 3-16.
- Calzada, J.F. and Peleg, M., 1978. "Mechanical interpretation of compressive stress-strain relationships of solid foods". *Journal Food Science* 43, 1087-1092
- Carter, R.E., 1990. "Rheology of Food, Pharmaceutical and Biological Materials with General Rheology", Elsevier Applied Science, London.
- Casutt, V., Preston, K.R. and Kilborn, R.H., 1984. "Effects of fermentation time, inherent flour strength and salt level on extensigraph properties of full-formula remix-to-peak processed doughs. *Cereal Chemistry*, 61, 454.
- Cauvain, S.P. and Young, L.S., 2006. "The Chorleywood Bread Process" Eds. Cauvain, S.P. and Young, L.S, CRC Press Boca Raton Boston New York Washington DC. pp. 17-23; pp. 24-39.

- Cauvain, S.P., 1996. "The freezing and Retarding of Fermented Dough". CCFRA Review No. 3; March
- Cauvain, S.P., 2001. "Breadmaking". In (Eds. Gavin Owens), *Cereals processing technology* CRC press Boca Raton Boston New York Washington, DC.
- Cauvain, S.P., 2003. "Breadmaking-an overview". In (Eds. S.P. Cauvain), *Bread making-Improving quality*. CRC press Boca Raton Boston New York Washington, DC.
- Cauvain, S.P., 2004. "Improving the texture of bread". In (Eds. David Kilcast) *Texture in food- Volume2: Solid Foods* CRC press Boca Raton Boston New York Washington, DC.
- Chamberlain, N., Collins, T.H. and Elton, G.A.H., 1961. "The Chorleywood Bread Process". BBIRA Report 59, CCFRA, Chipping Campden, Gloucestershire.
- Chen, W.Z. and Hosney, R.C., 1995a. "Development of an objective method for dough stickiness". *Lebensm.-Wiss. U.-Technol.* 28, 467-473.
- Chen, W.Z. and Hosney, R.C., 1995b. "Wheat flour compound that produces sticky dough: isolation and identification". *Journal Food Science.* 60, 434-437.
- Claassens, S.W., 1958. "The adhesion-cohesion, static friction and macrostructure of certain butters. 1.A method of measuring the adhesion-cohesion of butter". *S. Afr. J. Agric. Sci.* I, 457-463.
- Claassens, S.W., 1959a. "The adhesion-cohesion, static friction and macrostructure of certain butters. 11. Factors affecting the values recorded with thehesion balance". *S. Afr. J. Agric. Sci.* 2. 89-1 18.
- Claassens, S.W., 1959b. "The adhesion-cohesion, static friction and macro structure of certain butters. 111. An apparatus for measuring the apparent coefficient of static friction of butter". *S. Aff. J. Agric. Sci.* 2, 247-254.

- Claassens, S.W., 1959c. "The adhesion-cohesion, static friction and macrostructure of certain butters. IV. Factors affecting static friction measurements of butter". S. Afr. J. Agric. Sci. 2, 387-408.
- Collar, C., Andreu, P., Martinex, J.C. and Armero, E., 1999. "Optimization of hydrocolloid addition to improve wheat bread dough functionality: a response surface methodology study. Food Hydrocolloids 13 (6), 467-475.
- Collins, T.H., Overton, M.J., Redman, B.I., Roberts, J.A. and Smith, M.J., 1968. "A summary of breadmaking processes". FMBRA Report 13, CCFRA, Chipping Campden, Gloucestershire.
- Crowley, P., Schober, T.J., Clarke, C.I. and Arendt, E.K., 2002. "The effect of storage time on textural and crumb grain characteristics of sourdough wheat bread". European Food Research Technology, 214, 489-496.
- Daramola, B. and Osanyinlusi, S.A., 2006. "Investigation on modification of cassava starch using active components of ginger roots (*Zingiber officinale* Roscoe). Africa Journal of Biotechnology, Vol. 5 (10), 917-920.
- Deshpande, S.S. and Bhattacharya, K.R., 1982. "The texture of cooked rice". Journal Texture Studies 13, 31-42.
- Dhaliwal, A.S., Mares, D.J. and Marshall, D.R. 1990. "Measurement of dough surface associated with the 1B/1R chromosome translocation in bread wheats". Journal of Cereal Science. 12, 165-175.
- Dhaliwal, A.S., Mares, D.J., Marshall, D.R. and Skerritt, J.H., 1988. "Protein composition and pentosan content in relation to dough stickiness of 1B/1R translocation wheats". Cereal Chemistry. 65, 143-149.
- Dobraszczyk, B.J. and Morgenstern, M.P., 2003. "Rheology and breadmaking process". Journal of Cereal Science 38, 229-245.

- Dobraszczyk, B.J. and Vincent, J.F.V., 1999. "Measurement of mechanical properties of food materials in relation to texture: the materials approach". In Food Texture: measurement and perception (A.J. Rosenthal, ed.), Aspen Publishers, MD, USA.
- Dobraszczyk, B.J., 1997. "The rheological basis of dough stickiness". Journal Texture Studies 28, 139-162.
- Dobraszczyk, B.J., 2003. "Measuring the rheological properties of dough". In (Eds. S.P. Cauvain), Bread making-Improving quality. CRC press Boca Raton Boston New York Washington, DC.
- Dobraszczyk, B.J., Campbell, G.M. and Gan, Z., 2001. "Bread: a unique food". In: Cereal and cereal products. Chemistry and technology, (edited by A.V. Dendy & B. Dobraszczyk)
- Drake, B., 1966. "Advances in the determination of texture and consistency of foodstuffs". pp. 277-287, Proc. 2nd. Int. Congress Food Sci. Technol., Warsaw, Poland.
- Du, C-J. and Sun, D-W., 2006. "Learning techniques used in computer vision for food quality evaluation: a review. Journal of Food Engineering, 72, 39-55.
- Dunnewind, B., Sliwinski, E.L., Grolle, K. and Van Vliet, T., 2004. "The kieffer dough and gluten extensibility rig- an experimental evaluation". Journal Texture Studies 34, 537-560.
- Faridi, H. and Faubion, J.M., 1986. "Fundamentals of Dough Rheology", American Association of Cereal Chemists, St. Paul, MN, USA.
- Faridi, H. and Faubion, J.M., 1990. "Dough Rheology and Baked Product Texture", AVI Van Nostrand Reinhold, New York.
- Ferry, J.D., 1980. "Viscoelastic Properties of Polymers", John Wiley and Sons.
- Fiszman, S.M. and Damasio, M.H., 2000. "Instrumental measurement of adhesiveness in solid and semi-solid foods. a survey". Journal of Texture Studies 31, 69-91.
- Freilich, J. and Frey, C.N., 1939. " Dough oxidation and mixing studies. II Effects of remixing after fermentation. Cereal Chemistry, 16, 495.

- Friedman, H.H., Whitney, J.E. and Szczesniak, A.S., 1963. "The Texturometer: a new instrument for objective texture measurement". *Journal of Food Science* 28, 390-396.
- Gàlbaro, A., Varela, V. and Giménez, A., 2002. "Textural quality of white pan bread by sensory and instrumental measurements". *Journal of Texture studies* 33, 401-413.
- Gan, Z., Ellis, P.R. and Schofield, J.D., 1995. "Gas cell stabilisation and gas retention in wheat bread dough". *Journal of Cereal Science* 21, 215-230.
- Gélinas, P., Deaudelin, I. and Grenier, M., 1995. "Frozen dough: effects of dough shape, water content, and sheetingmolding conditions". *Cereal Foods World* 40 (3):124–126, 1995.
- Giannou, V. and Tzia, C., 2007. "Frozen dough bread: quality and textural behaviour during prolonged storage – prediction of final product characteristics". *Journal of Food Engineering* 79 (3), 929–934.
- Giannou, V., Kessoglou and V., Tzia, C., 2003. "Quality and safety characteristics of bread made from frozen dough". *Trends in Food Science and Technology* 14, 99–108.
- Giannou, V., Tzia and C., Le Bail, A., 2006. "Quality and safety of frozen Bakery Products". In (Eds. Da-Wen Sun) *Handbook of frozen food processing and packaging*. CRC Taylor&Francis Group Boca Raton London New York.
- Gómez, M., Del Real, S., Rosell, C.M., Ronda, F., Carlos, A.B. and Caballero, P.A., 2004. "Functionality of different emulsifiers on the performance of breadmaking and wheat bread quality". *European Food Research and Technology* 219, 145-150.
- Gore, P.J., 1991. "An update on sticky doughs". In *Cereals International Proceedings*, Sept. 9-13, (D.J. Martin and C.W. Wrigley, eds.) pp. 228-230, Brisbane, Australia.
- Grausgruber, H., Schoggl, G. and Ruckenbauer, P., 2002. "Investigations on the validity of the micro-extensigraph method to measure rheological properties of wheat doughs". *European Food Research Technology*, 214,79-82

- Gray, J.A. and Bemiller, J.N., 2003. "Bread staling.molecular basis" and control". *Comprehensive reviews in food science and food safety*, Vol.2, 1-21.
- Gunasekaran, S., 1996. "Computer vision technology for food quality assurance". *Trends in Food Science and Technology*, 7(8), 245–256.
- Hammond JR., F.R., 1989. Tack. In *Handbook of Pressure Sensitive Adhesives Technology*, (D. Satas, ed.) Van Nostrand Reinhold, New York.
- Haridas-Rao, P.H., Leelavathi, K. and Shurpalekar, S.R. 1989. "Effect of damaged starch on the chapati-making quality of whole wheat flour,, *Cereal Chemistry*. 66, 329-333.
- Havet, M. and LeBail, A. "Frozen bread dough: impact of the freezing rate and the storage duration on gassing power". In: *Proceedings of the Workshop on Process Engineering of Cereals*. France: Montpellier, October 8, 1999.
- Havet, M., Mankai, M. and Le Bail, A., 2000. "Influence of the freezing condition on the baking performances of French frozen dough". *Journal of Food Engineering* 45, 139–145.
- He, H. and Hoseney, R.C. 1990. " Changes in bread firmness and moisture during long term storage" *Cereal Chemistry* 67 (6), 603-605.
- He, H. and Hoseney, R.C., 1991. "Gas retention of different cereal flours". *Cereal Chemistry*, 68, 334-336.
- Heddleson, S.S., Hamann, D.D. and Lineback, D.R., 1993. "The Dahlquist criterion: applicability of a rheological criterion to the loss of pressure-sensitive tack in flour-water dough". *Cereal Chemistry*. 70, 744-748.
- Heddleson, S.S., Hamann, D.D., Lineback, D.R. and Slade, L., 1994. "Pressure-sensitive adhesive properties of wheat flour dough and the influence of temperature, separation rate and moisture content". *Cereal Chemistry*. 71, 564-570.
- Henry, W.F., Katz, M.H., Pilgrim, F.J. and May, A.T., 1971. "Texture of semi-solid foods: sensory and physical correlates". *Journal Food Science* 36, 155-161.

- Herz, K.O., 1965. “Staling of bread: A review“. Food Technology 19, 1828.
- Holland, D., 1994. Letter to the editor: which is more critical-the stress or the strain. Journal Rheol. 38, 1941-1943.
- Hoseney, R.C. and Chen, W.Z., 1994. “Adhesion testing apparatus and method for flowable materials such as wheat dough”. U.S. Patent 5,280.
- Hoseney, R.C. and Smewing, JO., 1999. “Instrumental measurement of stickiness of doughs and other foods”. Journal of Texture Studies 30, 123-136.
- Hoseney, R.C., 1994. “Principles of Cereal Science and Technology”. Second Edition. Published by the American Association of Cereal Chemists, Inc. St. Paul, Minnesota, USA.
- Hoseney, R.C., Rogers, P.F., Krishnarau, L. and Chen, W.Z., 1990. “Factors affecting the stickiness of wheat flour doughs”. pp. 338-343, Fifth International Symposium on PreHarvest Sprouting in Cereals. Westview Press. Boulder.
- Hsi-Mei, L. and Tze-Ching, L., 2006. “Bakery products”. In (Eds.Y. H. Hui), Handbook of food science, technology, and engineering. CRC press Taylor and Francis Group, Boca Raton London New York.
- Hsu, K., Hoseney, R. and Seib, S., 1979. “Frozen dough. I. Factor affecting the stability of yeasted doughs”. Cereal Chemistry 54, 419–424.
- Huang, W.N. and Hoseney, R.C., 1998. “Isolation and identification of a wheat flour compound causing sticky dough”. Cereal Chemistry. 76,276-28 1.
- Hwang, P. and Bushuk, W., 1973. “Some changes in the endosperm proteins during sprouting of wheat”. Cereal Chemistry. 50, 147-160.
- Ibrahim, Y. and D’appolonia, B.L., 1979. “Sprouting in hard red spring wheat”. Baker’s Digest 53(5), 17-19.
- Ikezoe, K. and Tipples, K.H., 1968. “Farinograph studies on the effect of various oxidizing agents in the sponge-and-dough system. Cereal Science Today, 13, 327.

- Indrani, D. and Venkateswara Rao, G., 2007. “Rheological characteristics of wheat flour dough as influenced by ingredients of *parotta*”. *Journal of Food Engineering* 79, 100-105.
- Inoue, Y. and Bushuk, W., 1991. “Studies on frozen doughs. I. Effects of frozen storage and freeze-thaw cycles on baking and rheological properties”. *Cereal Chemistry* 68 (6), 627–631
- Jowitt, R., 1974. “The terminology of food texture”. *Journal Texture Studies* 5, 351-358.
- Juliano, B.O., Perez, C.M., Barber, S., Blakeney, A.B., Iwasaki, T., Shibiuya, N., Keneaster, K.K., Ching, S.O., Laignelet, B., Launay, B., Del Mundo, A.M., Tokoyama, J., Tatsumi, K. and Webb, B.D., 1981. “International cooperative comparison of instrument methods for cooked rice texture”. *Journal Texture Studies* 12, 17-38.
- Keentok, M., Newberry, M.P., Gras, P., Bekes, F. and Tanner, R.I., 2002. “The rheology of bread dough made from four commercial flours”. *Rheologica Acta* 41, 173-179.
- Kieffer, R., Garnreiter, F. and Belitz, H-D., 1981b. “Beurteilung von Teigeigenschaften durch Zugversuche im Mikromapstab. 2 „. *Lebensm. Unters. Forsch.* 172, 193-194.
- Kieffer, R., Kim, J.-J. and Belitz, H.-D., 1981a. Zugversuche mit Weizenkleber im Mikromapstab. *Z. Lebensm. Unters. Forsch.* 172, 190-192
- Kilborn, R.H. and Preston, K.R., 1982. “A modified extensigraph procedure for measuring the stretching properties of fermented doughs”. *Cereal Chemistry*, 59, 381.
- Kilcast, D. and Roberts, C., 1998. “Perception and measurement of stickiness in sugar-rich foods”. *Journal Texture Studies* 29, 81-100.
- Kline, L. and Sugihara, T., 1968. “Factors affecting the stability of the frozen bread dough. I. Prepared by straight dough method”. *Beakers Digest* 42, 44–50.
- Knightly, W.H., 1977. “The staling of bread: A review”. *Bakers Digest* 51(5), 52.
- Konstance, R.P., 1993. “Axial compression properties of calcium caseinate gels”. *Journal Dairy Science* 76, 3317-3326.



- Krutz, G. W., Gibson, H. G., Cassens, D. L. and Zhang, M., 2000. "Colour vision in forest and wood engineering". *Landwards*, 55, 2–9.
- Kulp, K and Ponte, J.G. Jr. 1981. "Staling of white pan bread: fundamental causes". *CRC Critical Rev Food Sci Nutr* 15:1.
- Kulp, K., Lorenz, K. and Brümmer, J., 1995. "Frozen and Refrigerated Doughs and Batters". Minnesota: American Association of Cereal Chemists, pp. 1–3, 93, 148.
- Landis, Q. and Freilich, J., 1934. "Fermentation response and fermentation tolerance". *Cereal Chemistry*, 11, 396.
- Le Bail, A., Grinand, C., Le Cleach, S., Martinez, S. and Quilin, E., 1999. "Influence of storage conditions on frozen French bread dough". *Journal of Food Engineering* 39, 289-291.
- Le Bail, A., Havet, M. and Pasco, M., 1998. "Influence of the freezing rate and of storage duration on the gassing power of frozen bread dough". In: *Proceedings of the Symposium of the International Institute of Refrigeration*. Nantes, France: International Institute of Refrigeration.
- LeDuff, L., 1985. "Pain Francais précuit congelé et son procédé de fabrication". French Patent. Patent number FR 2 589 044-A1.
- Lee, S., Pyrak-Nolte, L.J. and Campanella, O., 2004. "Determination of ultrasonic-based rheological properties of dough during fermentation". *Journal of Texture Studies* 35, 33-51.
- Leemans, V., Magein, H. and Destain, M.F., 1998. "Defects segmentation on Golden Delicious apples by using colour machine vision". *Computers and Electronics in Agriculture*, 20, 117–130.
- Maga, J.A. 1975. "Bread staling". *CRC Critical Reviews Food Technology* 5,443.
- Maleki, M., Hosene, R.C. and Mattern, P.J., 1980. "Effects of loaf volume, moisture content and protein quality on the softness and staling rate of bread". *Cereal Chemistry*, 57(2), pp 138-140.

- Masi, P., Cavella, S. and Piazza, L., 2001. "An interpretation of the rheological behavior of wheat flour dough based on fundamental tests". In Bread staling (Eds. Chinachoti P., and Vodovotz Y.) CRC press Boca Raton Boston New York Washington, DC.
- Mendoza, F. and Aguilera, J.M., 2004. "Application of image analysis for classification of ripening bananas". *Journal Food Science* 69, 471–477.
- Mert, B., 2008. "A new instrumental setup for determination of small amplitude viscoelastic properties of dough during fermentation". *European Food Research Technology*, 227, 151-157.
- Mohsenin, N.N. and Mittal, J.P., 1977. "Use of rheological terms and correlation of compatible measurements in food texture research". *Journal Texture Studies* 8, 395-408
- Mossman, A.P., Fellers, D.A. and Suzuki, H., 1983. "Rice stickiness. I. Determination of rice stickiness with an Instron Tester". *Cereal Chemistry*. 60, 286-292.
- Muller, H.G., 1975. " Rheology and the conventional bread and biscuit making process", *Cereal Chemistry*, 52, 89r-105r.
- Newberry, M.P., Phan-Thien, N., Larroque, O.R., Tanner, R.I. and Larsen, N.G., 2002. "Dynamic and elongation rheology of yeasted bread doughs". *Cereal Chemistry* 79 (6), 874-879.
- Noguchi, G., Shinya, M., Tanaka, K. and Yoneyama, T., 1976. "Correlation of dough stickiness with texturometer reading and with various quality parameters". *Cereal Chemistry*. 53, 72-77.
- Okabe, M., 1979. "Texture measurement of cooked rice and its relationship to the eating quality". *Journal Texture Studies* 10, 131-152.
- Oliver, G. and Brock, C.J., 1997. "A rheological study of mechanical dough development and long fermentation processes for cream-cracker dough production. *Journal Science Food Agriculture*, 74, 294-300.

- Olkku, J. and Rha, C.K., 1975. "Textural parameters of candy licorice". *Journal Food Science*, 40, 1050-1054.
- Padua, M.R. de, and Padua-Maroun, H. 1984. "Rheological behaviour of Venezuelan arepa dough precooked corn flour". *Cereal Chemistry*. 61, 37-41.
- Pearson, T.C. and Slaughter, D. C., 1996. "Machine vision detection of early split pistachio nuts". *Transactions of the ASAE*, 39(3), 1203–1207.
- Peleg, M., 1976. "Texture Profile Analysis parameters obtained by an Instron Universal testing machine". *Journal Food Science*, 41, 721-722.
- Peleg, M., 1983. "The semantics of rheology and texture". *Food Technology*. 37 (11), 54-61.
- Peleg, M., 1987. "The basics of food rheology". In *Food Texture, Instrumental and Sensory Measurement* (H.R. Moskowitz, ed.) pp. 3-33, Marcel Dekker, New York.
- Peressini, D. and Sensidoni, A., 2004. *Frozen Dough Conference Proceedings*. Parma, Italy.
- Pizzinatto, A. and Hosney, R.C., 1980. "Rheological changes in cracker sponges during fermentation. *Cereal Chemistry*, 57, 185.
- Pons M., and Fiszman, S.M., 1996. "Instrumental texture profile analysis with particular reference to gelled systems." *Review Paper Journal of Texture Studies* (27) 597-624.
- Preston, K.R. and Kilborn, R.H., 1982. "Sponge-and-dough bread: Effects of fermentation, bromated and sponge salt upon the baking and physical dough properties of a Canadian red spring wheat. *Journal Food Science*, 47, 1143.
- Proctor, B.E., Davison, S. and Brody, A.L., 1956a. A recording strainage denture tenderometer for foods. 11. Studies on the masticatory force and motion, and the force-penetration relationship. *Food Technol.* 10(7), 327-331,
- Proctor, B.E., Davison, S. and Brody, A.L., 1956b. A recording strainage denture tenderometer for foods. III. Correlation with subjective tests and the Canco Tenderometer. *Food Technol.* 10(8), 344-346.

- Ram, B.P. and Nigam, S.N. 1983. "Texturometer as a tool for studying varietal differences in wheat flour doughs and gluten proteins". *Journal of Texture Studies* 14, 245-249.
- Ramírez-Wong, B., Sweat, V.E., Torres, P.I. and Rooney, L.W. 1993. "Development of two instrumental methods for corn masa texture evaluation". *Cereal Chemistry*, 70, 286-290.
- Ramírez-Wong, B., Sweat, V.E., Torres, P.I. and Rooney, L.W., 1994. "Cooking time, grinding, and moisture content effect on fresh corn masa texture". *Cereal Chemistry*. 71, 337-343.
- Ranhotra, G.S., Loewe, R.J. and Lehmann, T.A., 1977. "Breadmaking quality and nutritive value of sprouted wheat". *Journal Food Science*. 42, 1373-1375.
- Rao, M.A. and Steffe, J.F., 1992. "Viscoelastic Properties of Foods", Elsevier Applied Science, New York.
- Ribotta, P.D., Leon, A.E. and Anon, M.C., 2001. "Effect of freezing and frozen storage of doughs on bread quality". *Journal of Agricultural and Food Chemistry* 49 (2):913–918.
- Ribotta, P.D., Pérez, G.T., León, A.E. and Añón, M.C., 2004. "Effect of emulsifier and guar gum on micro structural, rheological and baking performance of frozen bread dough. *Food Hydrocolloids* 18, 305-313.
- Rocculi, P., Romani, S. and Dalla Rosa, M., 2005. "Effect of MAP with argon and nitrous oxide on quality maintenance of minimally processed kiwifruit". *Postharvest Biology and Technology* 35 319-328..
- Ross., K.A., Pyrak-Nolte, L.J. and Campanella, O.H., 2004. "The use of ultrasound and shear oscillatory test to characterize the effect of mixing time on the rheological properties of dough". *Food Research International* 37, 567-577.
- Sai-Manohar, R.S. and Haridas-Rao, P.H. 1997. "Effect of mixing period and additives on the rheological characteristics of dough and quality of biscuits" *Journal Cereal Science*, 25, 197-206.

- Sarkar, N.R., 1991. "Machine vision for quality control in the food industry". In I. Fung, & E. Matthews (Eds.), *Instrumental methods for quality assurance in foods* (pp. 167–187). New York: Marcel Decker.
- Scott Blair, G.W., 1936. Ph.D. Thesis: Viscous and elastic properties of plastic bodies. (original not seen) Fac. Sci., Univ. London.
- Selemulyo, V.O. and Zhou, W., 2007. "Frozen bread dough: Effects of freezing storage and dough improvers". *Journal of Cereal Science* 45 (1): 1-17.
- Shama, F. and Sherman, P., 1973. "Evaluation of some textural properties of foods with the Instron Universal Testing Machine". *Journal Texture Studies* 4, 344-353.
- Sherman, P., 1969. "A texture profile of foodstuffs based upon well-defined rheological properties". *Journal of Food Science*. 34, 458-462.
- Sherman, P., 1970. "Industrial Rheology: with particular reference to foods, pharmaceuticals, and cosmetics", Academic Press, London.
- Shiiba, K., Negishi, Y., Okada, K. and Nagao, S., 1990. "Chemical changes during sponge-dough fermentation". *Cereal Chemistry*, 67 (4), 350-355.
- Shuey, W.C., 1975. "Practical instruments for rheological measurements on wheat products". *Cereal Chemistry*, 52, 42r-81r.
- Sievert, D., Hoskeney, R.C., Delcour, J.A., 2007. "Bread and other baked Products". Standard Article. Wiley- VCH Verlag GmbH & Co. KGaA, Weinheim.
- Sila-Bhattacharya, S. and Narasimha, H.V., 1997. "Puncture and stress relaxation behavior of blackgram (*Phaseolus mungo*) flour-based papad dough". *Journal of Food Process Engineering* 20, 301-316.
- Smewing, J., 1995. "The Measurement of Dough and Gluten Extensibility Using the SMS/Kieffer Rig and the TA.XT2 Texture Analyser". Stable Micro Systems Ltd., Surrey, UK.

- Song, Y. and Zheng, Q., 2007. “Dynamic rheological properties of wheat flour dough and properties”. Trends in food science and technology 18, 132-138.
- Stampfli, L., Nersten, B. and Molteberg, E.L., 1996. “Effects of emulsifiers on farinograph and extensograph measurements“. Food Chemistry, 57 (4), 523-530.
- Steffe, J.F., 1996. “Rheological methods in food process engineering”. Second Edition. Freeman Press, USA.
- Szczesniak, A.S. and Hall, B.J., 1975. “Application of the General Foods Texturometer to specific food products”. Journal Texture Studies 6, 117-138.
- Szczesniak, A.S. and Smith, B.J., 1969. “Observations on strawberry texture: a three-pronged approach”. Journal Texture Studies I , 65-89.
- Szczesniak, A.S., 1963. “Classification of textural characteristics”. Journal Food. Science 28, 385-389.
- Szczesniak, A.S., 1971. “Effect of mode ~Frehydration on textural parameters of precooked freeze-dried sliced beef”. Journal Texture Studies 2, 18-30.
- Szczesniak, A.S., 1975a. General Foods Texture Profile revisited - Ten years perspective. Journal Texture Studies 6, 5-17.
- Szczesniak, A.S., Brandt, M.A. and Friedman, H.H., 1963. “Development of standard rating scales for mechanical parameters of texture and correlation between the objective and the sensory methods of texture evaluation”. Journal Food Science 28, 397-403.
- Tang, Q., Mccarthy, O.J. and Munro, P.A., 1995. “Effects of pH on whey protein concentrate gel properties: comparisons between small deformation (dynamic) and large deformation (failure) testing”. Journal Texture Studies 26.
- Van Vliet, T., 1999. “Rheological classification of foods and instrumental techniques for their study”, in: Food Texture: measurement and perception, (A.J. Rosenthal, ed.), Aspen Publishers, MN, USA.

- Varriano-Marston, E.K., Hsu, K.H. and Mahdi, J., 1980. "Rheological and structural changes in frozen dough". *The Bakers Digest* 54 (1): 32–41,
- Walstra, P. and Peleg, M., 1991. "Rheological and fracture properties of cheese. General considerations". *Bulletin of the IDS* 268, 3-4.
- Wang, H-H. and Sun, D-W., 2002a. "Correlation between cheese meltability determined with a computer vision method and with Arnott and Schreiber". *Journal of Food Science*, 67 (2), 745-749.
- Wang, S.M., Watts, B.M., Lukow, O.M., Schlichting, L. and Bushuk, W. 1996. "Dough profiling: an instrumental method for dough stickiness measurement". *Cereal Chemistry* . 73, 445-451.
- Wang, S.M., Watts, B.M., Lukow, O.M., Schlichting. L. and Bushuk, W., 1996. "Dough profiling: an instrumental method for dough stickiness measurement". *Cereal Chemistry*. 73, 445-451.
- Wehrle, K. and Arendt, E.K., 1998. "Rheological changes in wheat sourdough during controlled and spontaneous fermentation". *Cereal Chemistry*, 75, 882-886.
- Whitworth, M.B. and Alava, J.M., 1999. "The imaging and measurement of bubbles in bread doughs". In G.M. Campbell, C. Webb, S.S. Pandiella, & K. Niranjan (Eds.), *Bubbles in food* (pp. 221-232). St. Paul, MN: American Association of Cereal Chemists.
- Whorlow, R.W., 1992. "Rheological Techniques", 2nd edn, Ellis Horwood, Chichester, UK.
- Willhoft, E.M.A., 1973. "Mechanism and theory of staling of bread and baked goods, and associated changes in textural properties". *Journal of Texture Studies* 4, 292.
- Wolt, M.J. and D'Appolonia, B.L., 1984. "Factors involved in the stability of frozen dough. I. The influence of yeast-reducing compounds on frozen dough stability". *Cereal Chemistry* 61, 209–212.

- Yang, Q., 1996. "Apple stem and calyx identification with machine vision". *Journal of Agricultural Engineering Research*, 63, 229–236.
- Zeller, F.J., Gunzel, G., Fischbeck, G., Gerstenkorn, P. and Weipert, D., 1982. "Veränderungen der Backeigenschaften des Weizens dmh die WeizenRoggen-Chromosomentranslokation 1B/1R". *Getreide Mehl Brot* 36, 141-143.
- Zobel, H.F. 1973. "A review of bread staling". *Bakers Digest* 47(5), 53.
- Zobel, H.F. and Kulp, K., 1996. "The staling mechanism". In: *Baked Goods Freshness*. Hebeda RE, Zobel H, editors. Marcel Dekker, New York. p 1-64.