Chapter 5

Rheological characterization of crunchy biscuit

5.1 Introduction

It is well-know that during the sequences of actions that constitute food consumption, we perceive a whole range of different characteristics relating to the appearance, flavour and texture of the food. Numerous tools are available for investigating the sensory properties of foods, and the information required must be carefully defined if appropriate tools are to be selected. The systematic development of new products will inevitably depend on the use of different tools at different stages of the development cycle.

Similarly, textural characteristics such as viscosity can influence the perception of flavour, and some flavour characteristics. Texture and food structure are inextricably linked; the micro- and macro-structural composition of foods will determine the sensory perception, and any change in structure carries the risk of changing perceived texture and violating consumer expectations. The industry therefore needs to take great care to ensure the textural characteristics modifying the product, for example producing low-fat variants, because it is possible to introduce structural changes that can generate substantial textural modifications.

Texture is a quality attribute that is closely related to the structural and mechanical properties of a food. It is, therefore, essential to understand the mechanical properties of foods to relate this with the final texture and appearance.

The study of mechanical behaviour, i.e. deformation and flow, of foods under applied forces falls within the scope of food rheology. A number of textbooks have been written about rheology with various degrees of mathematical requirements [Ferry, (1980); Mohsenin, (1989); Rao and Steffe, (1992); Sherman, (1970)].

The theory of rheology describes the basic mechanical behaviour of many food products with reasonable accuracy.

There has long been a conviction that the rheological properties of dough are in some way related to baking behaviour, and specifically the rupture of the cell walls during baking is considered to be the limit of dough expansion during the proof and oven rise phases related to biaxial tensile stress and large strains at rupture [Dobraszczyk, B.J. and Roberts C. A. (1994)]. Moreover, they found that the fracture

strain depends on the flour type, deformation rate and temperature. In agreement with what has been previously stated, Van Vliet T. et al. (1992) proposed a new rheological criterion for the extensibility of dough films between gas cells that is based on strain hardening of the dough in biaxial extension exceeding a specific lower limit and according with this Dobraszczyk, 2004 found that the bubble walls with good strain hardening properties remain stable for longer during baking, allowing the bubbles to resist coalescence and retain gas for much longer. Janssen et al. (1996) studied dough rheological properties in biaxial extension for several flours and discussed the results in relation to bread making performance. Their study suggested that to obtain a high loaf volume and a fine crumb structure, wheat flour dough has to exhibit biaxial strain hardening and an extensibility exceeding a minimum level and that the resistance to deformation may vary within a certain range.

Several studies have been carried out to evaluate the viscoelastic properties of wheat flour dough, with the aim to study the influence of wheat quality, dough ingredients, and processing conditions on the fundamental rheological properties. Fundamental rheological testing at small deformations are non-destructive, and the methods used for dough characterisation can be transient such as creep recovery or stress relaxation, or dynamic such as oscillatory measurements. For this motive several studies have performed dynamic oscillatory methods to characterise the fundamental viscoelatic behaviour of dough [Pedersen L., (2003)].

Bulk rheological properties become of primary importance for the growth of gas cells [Van Vliet, 1999] and then, to simulate the proofing, it is very important to relate the growing mechanical behaviour with the matrix. In fact, Mitchell JR. et al. 1999 related the bubble growth (dR/dt) with the viscosity of the medium to predict the expansion and Huang H., 1999 considered the biaxial extensional viscosity of dough as the predominant factor in the gas growth equation during proofing.

Then, in order to obtain information on the mechanical properties of dough in this chapter, the rheological behaviour of biscuit dough is analysed. Small deformation and biaxial extension tests were performed, because the bulk properties of dough are important in determining the final biscuit quality, moreover, the viscoelastic properties, which allow the formation of spongy typical structure, are correlated with the mixing time, water content, temperature, etc.

Since the proofing performances are very sensitive to bulk rheological properties, it is important to find a link between the structure and the process condition, and then it is essential to have the constitutive relation that can describe the mechanical behaviour of dough during baking. To achieve the purpose a fundamental rheological characterization for biscuits dough, mixed with two

different flours was performed. In this way it is possible to complete the set of equations used in Chapter 1 with a constitutive equation. Then, to obtain information on the mechanical properties of dough under conditions similar to baking, biaxial extension tests were performed. From this last tests the "rupture work", necessary to break the film around the bubbles, was found and the equations written for the coalescence criterion were completed.

5.2 Dough – Biscuits Rheology

Rheology is now well-established as the science of the deformation and flow of matter: it is the study of the manner in which materials respond to applied stress or strain.

The behaviour of a material is a relationship between forces and deformations (or changes of shape) and a model gives a mathematical formulation of the relationship, rheological properties being expressed by the structure of a model and values of constants included in a model — characteristic for a material. Rheological models are related to a point, which is a physical object including a sufficient number of molecules in order to neglect the molecular structure of matter and to treat it as continuum [Malkin, 1994]. Rheological behaviour of a material depends on time and space scales of observation (experiment).

When force is applied to a solid material and the resulting stress versus strain curve is a straight line through the origin, the material is obeying Hooke's law. The relationship may be stated for shear stress and shear strain as:

$$\tau = G \cdot \gamma \tag{5.1}$$

where G is the shear modulus. Hookean materials do not flow and are linearly elastic. Stress remains constant until the strain is removed and the material returns to its original shape. According to its constitutive relation, the solid does not relax the stress generated by the time constant deformation with time. On the contrary a Newtonian liquid material has a constitutive equation as follows:

$$\tau = \eta \cdot \dot{\gamma} \tag{5.2}$$

Where η [Pa*s] is the system viscosity. Then, for this type of material an instantaneous variation of the stress, expressible as δ of the Dirac function, will be the material answer. The liquid then instantaneously relaxes the stress without tensional residual stresses. So far we have considered elastic behaviour and viscous behaviour in terms of the laws of Hooke and Newton. These are

linear laws, which assume direct proportionality between stress and strain, or strain rate, whatever the stress.

Moreover, it is well-known that a lot of materials have an intermediate behaviour between an ideal elastic solid and a Newtonian fluid, depending on the way of relaxing induced stress in the material with time:

- 1. *Visco-elastic systems*: the stress is completely relaxed over a long time, as with the liquid system;
- 2. *Elastic- viscous systems*: the material exhibits, over a long time, a residual stress not relaxed as with the solid systems (Figure 5.1).

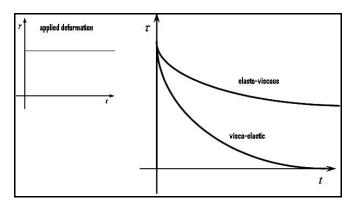


Figure 5.1 – Visco- elastic and Elasto-viscous material behaviour

Dynamic oscillatory methods were used to characterise the fundamental viscoelastic behaviour of dough, mainly in relation to protein content, gluten strength, and glutenin/gliadin ratio [Edwards and Dexter, 1999; Khatkar et al., 1995; Uthayakumaran et al., 1999] to have a behaviour of final dough and then to link the structure with the baking performance. Storage modulus (G') and loss modulus (G'') are strongly affected by water content [Navickis et al., 1982]. In fact, the water addition causes a lowering of viscosity with a small reduction of relaxation time, whereas the final biscuits will be with a smaller thickness. If the water continent is too small, during baking, a pronounced "crust effect" will become visible because of rapid dehydration [Maache-Rezzoug et al., 1998]. Moreover, a low water content makes the dough become hard causing sheeting problems.

In the literature it is possible find the effect of the other ingredients, which are generally put inside the biscuit dough to have the right characteristics, like sugar, salt, yeast, etc. As a matter of fact the addition of sugar softens the dough, causing lower viscosity and an increase of cohesiveness because of the reaction among the added sugar and the water available [Burè, 1980]. The fat addition contributes to the dough plasticity as a lubricant, while the salt improves the organoleptic qualities, increasing the gas retention, then the final volume, the consistency and elasticity of the dough.

5.3 Relaxation modulus determination

In the light of the affirmations in the previous paragraphs, the biscuit dough was characterized rheologically. The paste is considered to behave as a viscoelastic material according to the constitutive equation of the weak gel model [Gabriele D et al, 2001] owing to time dependency. Thus the following function factorization was applied:

$$G(t,T,\dot{T},X_W) = s(T,\dot{T},X_W) \cdot t^{-\alpha(T,X_W)} \cong s(T,\dot{T}) \cdot t^{-\alpha(T,\dot{T})}$$
(5.3)

The dependence on the water fraction was neglected up to coalescence because only a rather small water loss was observed. The dependence on \dot{T} was inserted as a result of the measured experimental evidence. In fact, in the microsystem a quasi-static transformation was considered that implies $\dot{T} = 0$, but to be closer to the current conditions the dependency was inserted into the properties.

The rheological behaviour, and therefore the applicability of a weak-gel equation, was obtained by the fundamental rheological tests. Samples were prepared by mixing following the standard recipe and then fundamental measures were performed at different temperatures. With the purpose of carrying out the oscillatory tests in the linear viscoelastic region, stress sweep tests were performed, necessary to determine the linear region and time sweep tests to have the delay time, necessary to relax the induced efforts to the dough sample during the loading and to allow the reaching of uniform temperature on the material. With the parameters determined from the stresses and from the time sweep tests at different T, measures of Frequency sweep tests were performed, in which G' and G" was determined as the frequency function. A typical behaviour of G' and G'' for flour 1 is shown in figure 5.2.

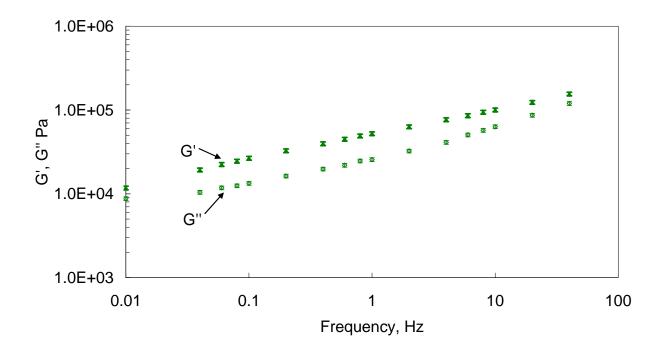


Figure 5. 2 - Frequency sweep test at T=30 $^\circ C$ for flour 1

From the figure 5.2 it is possible point out that G' and G", in the window of analyzed frequencies, are linear, according to the weak-gel model. Then the obtained experimental data were analyzed with the following equation:

$$G^{*}(\omega) = \sqrt{\left(G^{'2} + G^{''2}\right)} = A \cdot \omega^{\frac{1}{z}} \quad \text{with} \quad \frac{0\langle \frac{1}{z} \langle 1 \\ and \quad \omega_{1} \langle \omega \langle \omega_{2} \rangle$$
(5.4)

in which z represents the coordination number and A the network strength. The values of A and z, obtained by the application of the equation (5.4), are put in table 5.1 and from the trend it is possible understand the behaviour of dough with the temperature and the type of structure.

| Т | A flour 2 | | z flour 2 | z flour 1 |
|-----|---------------------|---------------------|-----------|-----------|
| С° | Pa*s ^{1/z} | Pa*s ^{1/z} | - | - |
| 30 | 61719 | 59100 | 3,44 | 3,27 |
| 45 | 17427 | 19338 | 2,97 | 3,03 |
| 60 | 15589 | 19385 | 3,49 | 3,59 |
| 75 | 27140 | 28772 | 5,07 | 5,06 |
| 90 | 87759 | 84043 | 6,66 | 6,35 |
| 100 | 204036 | 197313 | 7,98 | 8,11 |
| 110 | 182831 | 204239 | 8,24 | 8,64 |

Table 5.1 - A and z parameters with the temperature

Then, following the Winter theory for the gels, concerning the transition sol-gel, on the basis of which the relaxation modulus of a critical gel (which is in correspondence to the transition sol-gel) follows a power-law, for the relaxation modulus an equation like the following is had:

$$G(t) = S \cdot t^{-\alpha} \qquad 0 < \alpha < 1 \qquad \lambda_0 < t < \infty$$
(5.5)

where , λ_0 is the shortest relaxation time, S the "gel strength", a function of chain segment mobility and obtainable from the subsequent equation:

$$S(t) = \frac{A}{\Gamma(1-n)}.$$
(5.6)

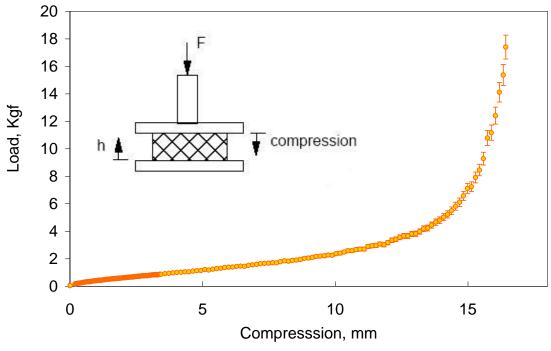
In which n is the inverse of the coordination number; and finally, A represents the exponent of relaxation obtainable from the n-trend at different temperatures.

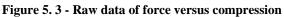
5.4 Squeezing flow parameters

With the purpose then of having the necessary parameters for the squeezing flow, K and n obtainable from the viscosity curve, compression tests were performed in a non-lubricated way with SMS at 30°C [Steffe J. F., 1996].

According to the Scott equation, the values of K and n at high shear rate were obtained, while the values at low shear come from creep tests.

A typical configuration for compression tests is represented in figure 5.3, while the data obtained for the viscosity are in figure 5.3 and the value obtained for K and n are put in table 5.2.





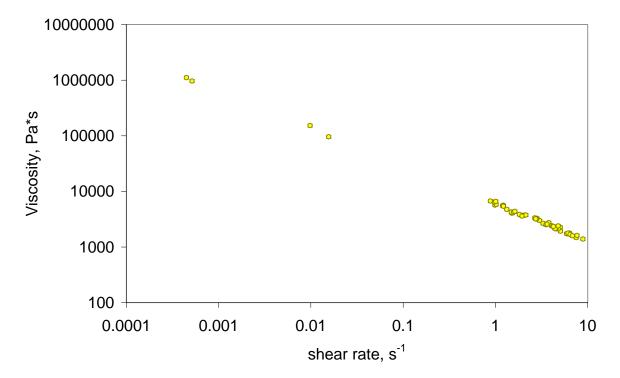


Figure 5. 4 – Viscosity behaviour for dough mixed with flour 1.

| | K | n |
|---------|---------|--------|
| | [Pa*s^n | [-] |
| Flour1 | 6094 | 0,3257 |
| Flour 2 | 5528 | 0,304 |

Table 5. 2 – K and n value for flour 1 and 2.

The shear flow measurements obtained with different techniques were fitted by a power law rule that has to be shifted at different temperatures to give the flow curve used in the squeezing model:

$$\tau(T,\dot{\gamma}) = m(T)\dot{\gamma}^{n(T)}$$
(5.7)

The power law parameters at any condition, were expressed in terms of the dynamic viscosity, therefore a shifting technique was developed to transfer the oscillatory data to shear flow. Thus given the data at the reference temperature $T_{REF}=30^{\circ}C$ a further shift was applied by overlapping dynamic and shear viscosity.

The functionality on *T* and deformation rate $\dot{\gamma}$ may be factorised. A shifting technique was applied to obtain a flow curve from the different measurements, instead of making all the measurements, which are often not available or much less reproducible at any T and $\dot{\gamma}$. This technique is based on the equivalence of the rheological behaviour by applying a superposition effect principle. Regarding the above eq.(5.7), apart from the usual time-temperature superposition, it was proposed to use dynamic measurement instead of the usual shear rate ones for the rate of deformation dependence. This has the net advantage of a better reproducibility especially for dough where it is rather difficult to obtain good shear flow data. Thus shear flow measurements carried out at a given temperature with a creep test by means of a stress controlled rheometer or as lubricated squeeze flow by means of an SMS compression instrument may give the basis for shifting. A power law can be used for fitting this sort of data:

$$\tau = k\dot{\gamma}^n = \left(k\dot{\gamma}^{n-1}\right)\dot{\gamma} \tag{5.8}$$

On the other hand, a frequency sweep oscillatory test carried out in asymptotic conditions, gives the values of the two dynamic moduli G' and G'' as a function of the applied frequency ω . When the dissipative modulus is set in terms of dynamic viscosity, even in this case a power law is found:

$$\frac{G''}{\omega} = \eta' = k' \,\omega^{n'} \tag{5.9}$$

In principle the main difference between Eqs.5.8 and 5.9 is due to the fact that the latter refers to equilibrium conditions. Nevertheless, a shifting technique may be proposed in the hypothesis that the same value of the viscosity should be found when $\omega = a_{\dot{\gamma}}\dot{\gamma}$:

$$k' \omega^{n'} = k' \left(a_{\dot{\gamma}} \dot{\gamma} \right)^{n'} = k \dot{\gamma}^{n-1}$$
(5.10)

Thus by simply overlapping the two curves, a relation between the frequency and the deformation rate is found over the entire range of interest. The result is very important and may be justified for structured viscoelastic material, admitting that a steady shear condition can be considered as a sort of equilibrium, but at a lower level of structuration. In other words, by modelling the viscoelastic behaviour as a three-dimensional network it looks like a less connected network. So the ultimate result is a cut of the relaxation times which in turn corresponds to their shift if a quasi static condition is assumed. The adopted technique is very helpful, because rather often only the dynamic viscosity is available and therefore a shift technique is used to expand the shear flow curve. It should be noted that if the two slopes are the same, a sole horizontal shift is needed:

$$n' = n - 1 \tag{5.11}$$

Then the following link between oscillatory and shear parameters at the same temperature is found:

$$k = \frac{k' (a_{\dot{\gamma}} \dot{\gamma})^n}{\dot{\gamma}^{n-l}} = k' (a_{\dot{\gamma}})^{n'}$$
(5.12)

Obviously if the value of the shear flow viscosity is known at the considered temperature T, the proposed shift becomes I. Finally, the shift value is:

$$a_{\dot{\gamma}} = \left(\frac{k}{k'}\right)^{l'/n'} \tag{5.13}$$

To take into account the temperature dependence, a shift with temperature can be considered, concerning both k and n'. Even in this case the dynamic measurements are the most available, and it is rather common to refer to an oscillatory time cure at $1H_z$ (figure 5.5). on an experimental basis. Since the viscosity was the parameter of interest only the value of G'' is considered. By applying the time temperature superposition to the dissipative modulus referred to any given temperature T_{REF} , the following expression is found:

$$G''(T, \omega a_T) = G''(T_{REF}, \omega)$$
(5.14)

The physical meaning is again a cut of the relaxation times with the consequence of their shift to a_T . It should be noted that in this case the theory is more solid than above, because it refers completely to equilibrium conditions.

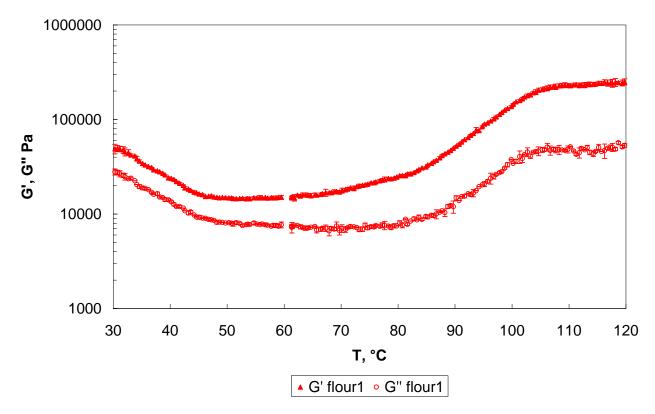


Figure 5. 5 - Oscillatory time cure at *1Hz* for flour 1

The use of Eq.5.20 suggests the validity of the weak gel model. Thus the substitution in the above Eq.5.14 leads to:

$$k'(T) \cdot (\omega a_T)^{n'(T)+I} = k'(T_{REF}) \cdot (\omega)^{n'(T_{REF})+I}$$
(5.15)

by rearranging this equation it is found:

$$k'(T) = \frac{k'(T_{REF}) \cdot (\omega)^{n'(T_{REF})+1}}{(\omega a_T)^{n'(T)+1}}$$
(5.16)

under this hypothesis, by considering a time cure test at $\omega = 1Hz$ is obtained

$$k'(T) = \frac{k'(T_{REF})}{a_T} \left(\frac{1}{a_T}\right)^{n'(T)}$$
(5.17)

Since the slopes *n*' are different it is not enough to determine the sole horizontal shift. Therefore to complete the computation once given the reference value $k'(T_{REF})$, the values of the shifting factor a_T

and n'(T) are needed. According to the weak gel theory, the link between the loss tangent δ and the power law index of frequency is at any temperature *T*:

$$\frac{\delta(T)}{n'(T)+1} = const.$$
(5.18)

which can be used to solve Eq.5.17.

 a_T computation

$$a_{T} = \left(\frac{G''(T, 1Hz)}{G''(T_{REF}, 1Hz)}\right)^{\frac{1}{n'(T)}} = \left(\frac{10^{f''(T)}}{10^{f''(T_{REF})}}\right)^{\frac{1}{n'(T)}}$$
(5.19)

where f''(T) is a proper fitting function.

<u>*n'(T)* computation</u>

$$\frac{n'(T)+1}{n'(T_{REF})+1} = \frac{\delta(T)}{\delta(T_{REF})}$$
(5.20)

$$n'(T) = \frac{\delta(T)}{\delta(T_{REF})} \cdot \left(1 + n'(T_{REF})\right)$$
(5.21)

Finally, if a time cure at 1Hz is known and so it is the weak gel parameter at a reference temperature T_{REF} , the value of k'(T) is found from Eq.5.16 and, knowing $a_{\dot{\gamma}}$, the value of k and n are found from Eqs.5.11-12.

5.5 Collapse and Rupture Work parameters

The model needs recovery and memory parameters for the collapse model after biscuit collapse. To find these parameters it is possible perform *creep-recovery* tests. The creep test applies a constant stress on the material and in this way it is possible to measure the deformation; usually the creep result is analyzed in term of compliance J(t) [Pa⁻¹], defined as the ratio of deformation and stress applied:

$$J(t) = \frac{\gamma}{\tau_0} \tag{5.23}$$

where γ is the deformation in the time and τ_0 is the applied stress. Then it is possible to complete the test with the Recovery phase, during which the stress is removed from the sample and the material recovery is analyzed in terms of compliance reduction in time. A typical creep-recovery test on biscuit dough is reported in figure 5.6. The curve shape gives important information about the stress relaxation and generally the data in the recovery zone can be fitted with the Voigt model:

$$\varepsilon'(t) = \varepsilon'_{collapse} \left(J_0 + \sum_{i=1}^3 J_i \cdot \exp\left(\frac{t_c - t}{\lambda_i}\right) \right)$$
(5.24)

where \mathcal{E}' is the deformation. The values of the parameters, obtained through a recovery measurement and elaborated thanks to a three-Voigt element model, are reported in the appendix. Then, the characteristic reference time t*, from which the system starts to remember, is a measure of the transition in dough behaviour from viscoelasticity to "plasticity", and it can be determined by a creep test, then from the first part of the curve shown in figure 5.6. Creep experimental data were fitted by using a 1-element Kelvin-Voigt model:

$$J = J_0 + J_1 \left(1 - e^{-\frac{t}{t^*}} \right) + \frac{t}{\eta}$$
(5.25)

Thus from this equation the value of t* is obtained. Below t* a viscoelastic behaviour is shown, while above t* a plastic behaviour is exhibited. The data for the memory are obtained through the following equation where ε is the void fraction:

$$t^* = t_c - \left(A \cdot t_c + B\right) \cdot \left(1 + a \cdot \varepsilon^3\right)^{1/2}$$
(5.25)

and the parameters used are in the thesis appendix.

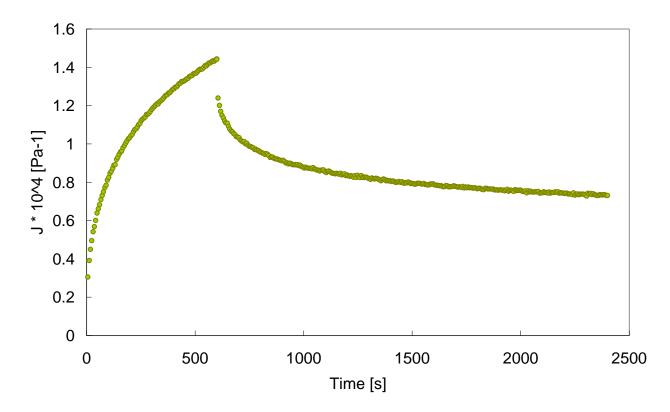


Figure 5. 6 – Creep – Relaxation on biscuit dough mixed with flour 1

As it concerns the tests of bi-axial extension, with the purpose of obtaining the *rupture work*, the experiments were performed setting one sheet of dough, with opportune dimensions, among a grate and a circular crown, in such a way as to be bound by the same plate. The sample was therefore covered with appropriate silicone oil to avoid water losses during the test. The apparatus is provided with pressure transducer and flow-meter. The test is performed insufflating compressed air to obtain a bubble of opportune dimension. The bubble height is measured up to the point of break-up with a laser, set on the top of the instrument. The elaborated results obtained are shown in the following:

| Rupture Work | | Dev. St |
|--------------|-------|---------|
| | J/m^3 | J/m^3 |
| Flour 2 | 63604 | 11448 |
| Flour 1 | 64568 | 6258 |

Table 5.3 – Rupture work values.

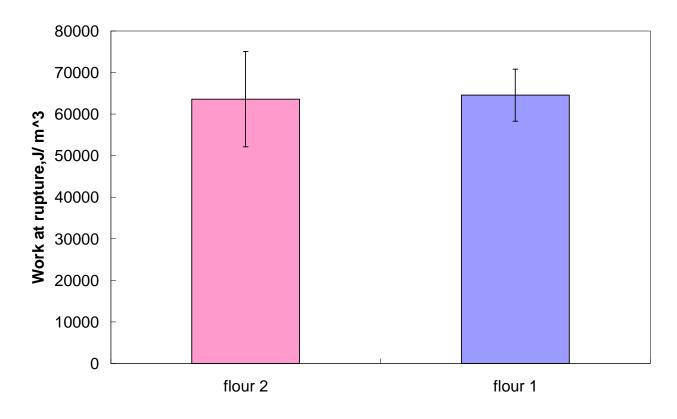


Figure 5.7 – Rupture work for flour 1 and 2

5.6 Conclusions

The possibility of checking the process, both in terms of raw material and in terms of operative variables, is an essential requisite for the food industry and this is possible only by keeping the rheological properties of the dough controlled.

It is well-known that rheological properties influence the physical properties of dough, and because the rheological properties have a great influence on the formation and dimensions of gas bubbles during the process, the possibility of knowing the rheological behaviour of dough with optimal characteristics for the process, can help to produce a standard reference. Moreover, the fundamental rheological characterization allows a constitutive equation to be obtained, which describes the behaviour of the viscoelastic material in question. Therefore, an important conclusion from the present chapter was obtained, because in this part of the work the parameters were found from the rheological analysis of standard optimal flours used in the factory and in addition, since these parameters can be used for baking model simulation to compare the experimental with the simulated baking height profiles.

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