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Water properties in wheat flour dough
I: Classical thermogravimetry approach

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Abstract

Thermogravimetric analysis allowed inspection of the behavior of water within a wheat flour dough. In such a system water is partitioned between coexisting phases which are none the less far from the true thermodynamic equilibrium. The vaporization rate revealed that water is released in two main steps, the first corresponding to a mere diffusion process, the second being instead related to the desorption of water more tightly bound to the gluten network. It was observed that the overall dough moisture, the extent of mixing, the dough resting time after mixing can modify water partition between phases and the way water is released during the temperature scan. Some effect was also recognized in dough samples to which original water soluble proteins had been added. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Water; Bread dough; Thermogravimetric analysis; TGA

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1. Introduction

Baking is a process where the increase of temperature accelerates the rate of any transformation that takes place within the dough, like starch gelatinization and gluten reticulation (just to mention the most important), but is accompanied by a substantial water loss (about 40% of the dough moisture in the case of bread) which reduces the extent of these modifications that are largely sustained by the available water. Both the amount and the mobility of water are indeed recognized to have a crucial role in starch gelatinization, as shown in a number of papers dealing with either starch aqueous suspensions (Biliaderis, 1983; Chinachoti, Kim-Shin, Mari & Lo, 1991; Hoseney, Zeleznak & Yost, 1986; Li, Dickinson & Chinachoti, 1996; Richardson, Baianu & Steinberg, 1986; Wootton & Bamunuarachi, 1979), or dough samples (Eliasson, 1993; Fessas & Shiraldi, 1999; Zanoni, 1991), and in the formation of the gluten network, which is rapidly hydrated during mixing and undergoes an early cross-linking even at room temperature when enough water is available (MacRitchie, 1987; Sarwin, Laskawy & Grosch, 1993; Schofield, Bottomley, Timms & Booth, 1983; Tanaka & Bushuk, 1973; Weegels, Hamer & Schofield, 1996; Weegels, Hamer & Schofield, 1997a,b). Previous studies (Bushuk & Mehrotra, 1977a,b) on water in bread dough were performed by use of DTA and focused on the determination of "boiling thermogram" and "melting thermogram" (both terms would nowadays be rejected by the International Conference of Thermal Analysis and Calorimetry), namely on the evaluation of water vaporization and fusion enthalpies. The authors of these papers however noticed substantial discrepancies between "boiling" and "melting" modes, but were not able to propose a satisfactory explanation for them, therefore leaving a number of doubts about the actual kind of interactions between water and other dough components. Finally, the experimental results reported in these papers concerned the overall water content without considering water partition between the various physical phases of the dough, which is instead a fundamental point to be investigated.

It is well known that a wheat flour dough is a heterogeneous system, since it is composed of thermodynamically incompatible polymers (Closs, Conde-Petit, Roberts, Tolstoguzov & Escheer, 1999; Grinberg & Tolstoguzov, 1997; Polyakov, Grinberg, Ya & Tolstoguzov,

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1997), which therefore form separate aqueous phases, each of which is richer in a given polymer with respect to the nominal dough composition (Larsson & Eliasson, 1996a; Tolstoguzov, 1997). Driven by a chemical potential gradient, water is exchanged between these phases during mixing and baking (Larsson & Eliasson, 1996b). In a real dough, water partition can none the less be incomplete because of the limited molecular mobility. In this respect, the increase of temperature experienced on baking should enhance the progress toward the true equilibrium condition; however, since the concomitant modifications of the polymer components of the system are accompanied by substantial changes of their properties, like affinity for water, excluded volume and molecular structure (in the case of gluten), rearrangements of the existing phases and new repartition of water are to be expected.

In any moment of the process, some water occupies inter-phase regions (Sahi, 1994) and can be supposed to move freely and generate a vapor phase with a given partial pressure; the rest of the water has to pass through phase boundaries and reach the inter-phase region to contribute to the overall water activity of the system, a_w. The latter process can be rather slow when the system texture has already turned into a gel-like condition, for example because of starch gelatinization. Once again, a further increase of the temperature could sustain the rate of this process and the corresponding water vaporization. It should be noticed that the water fraction tightly bound to the substrates is released during baking only at the temperature attained within the external regions of a bread loaf or in a biscuit.

The above picture is qualitative and cannot be of practical use to improve baking; some interest could instead be found in detailed and quantitative data about water loss in baking, such as those offered by the thermo-gravimetry analysis (TGA) performed with a modern instrument.

The TG trace of a sample undergoing dehydration is the mass-vs-T plot and describes how much and how fast water is released when the system is heated up at a constant rate, β, so as to have $T = T_o + \beta\ t$ (where t stands for time). When the mass loss is due to a single process, like the dehydration of a salt, the TG trace shows a sigmoid descending trend with a flexus at some intermediate temperature where the water loss rate is maximum. A more direct impact is offered by the trace of the time derivative, DTG (derivative of thermo gravimetry), which shows a well defined peak, the maximum of which corresponds to the flexus point in the TG trace. Some modern instruments are designed in order to give a combined output, namely TG, DTG and DSC: the latter signal allows determination of the enthalpy drop relevant to the process that produces the mass loss. In the case of water vaporization from food samples, it is very easy to verify that the corresponding enthalpy is rather close to 2.3 kJ g^{-1} (referred to the mass released), namely, the vaporization enthalpy of pure water in this temperature range.

When the system contains water in different conditions, either because of different molecular mobility or because of tertiary force links of different strength with the substrate, the DTG trace shows either several peaks or shouldered peaks which can be mathematically singled out. For each of them the corresponding enthalpy drop is accessible to ensure that the underlying process still deals with water vaporization. A better control of this can be attained by coupling the instrument with a gas chromatographer (but these instruments have difficulties to follow the processes in line) or a IR spectrophotometer.

The present paper reports the results of TGA investigations on azyme wheat flour dough just mixed or let to rest after mixing and also in presence of extra flour water soluble proteins (albumins and globulins in the sense of Osborne).

2. Materials and methods

2.1. Flour and extracts

Wheat flour was a commercial product with the following non-starch content (% flour mass, w/w.): proteins 9.85±0.47 (Kjedahl nitrogen, conversion factor = 5.7), water 14.5±0.2, lipids 1.19±0.01, ash 0.45±0.01. Moisture was determined gravimetrically by heating samples in a ventilated oven at 105°C for 20 min. Soluble proteins (albumins and globulins in the sence of Osborne) were extracted from centrifuged water suspensions of flour according to (Fessas & Schiraldi, 1998). Gluten and Starch from wheat flour were commercial products (Sigma, G5004, S5127).

The wheat flour dough was prepared with wheat flour and distilled water, without adding any salt and yeast for the sake of reducing the number of variables that could affect water partition within the dough. The recipe was modified by changing the water content and/or by adding water soluble proteins (extracted from the same lot of flour). The following type of dough were accordingly considered:

1. Dough with an overall 42% moisture.
2. Dough with an overall 50% moisture.
3. Dough with an overall 42% moisture added with 0.48% (referred to the flour dry mass) lyophilized soluble proteins.

For each type of dough, 30 g of water–flour mixture were manually mixed for 10 min (2 min in a beaker, and 8 min with manual kneading). Dough of type 1 was also prepared starting from a 500 g flour–water mixture with mechanical mixing (Hobart mixer, USA) for 10 and 30 min.

Manually mixed starch–water suspension (54.2% total moisture content) and manually mixed Gluten (64.9% total moisture content) samples were also prepared.

2.2. TGA

The TGA instrument was a SETARAM TG-DSC111 (France) with the simultaneous output of the thermal effect (heat flow-vs-*T*), TGA trace, namely, mass loss-vs-*T* (see TG in Fig. 1a), and its time derivative DTG. The typical sample mass was 30 mg. Each run was repeated at least twice. The raw data were analyzed with the software IFESTOS (Fessas & Schiraldi, in press). All the TGA traces were normalized to a 100 mg water content: accordingly the DTG traces were expressed as mg of lost water per degree K (with reference to the scanning rate used), and the related heat flow as specific heat *C*p/J K^{-1} g^{-1} (see Fig. 1b).

3. Results and discussion

The typical TGA run for the present work was carried out at 2°C min^{-1} heating rate starting from $T_o = 25°C$ and ending at 200°C. Fig. 2 reports the relevant DTG trace where two main peaks appeared which were resolved by mathematical deconvolution of the overall signal. The simultaneous DSC trace allowed to verify that both peaks were related to water vaporization, the relevant enthalpies being 2.30 ± 0.05 and 2.25 ± 0.05 kJ g^{-1} (referred to the mass lost).

The same figure shows the α -vs-*T* trend overlapped to the DTG trace, where α stands for the extent of starch gelatinization namely α = gelatinized/total strarch mass-ratio. The α data were drawn from a previous work (Fessas & Schiraldi, 1999) aiming at defining the actual progress of starch gelatinization in a baking process simulated with DSC runs performed with open and sealed pans. The α-vs-*T* trend reported in the figure is the one expected for a 2°C min^{-1} heating rate.

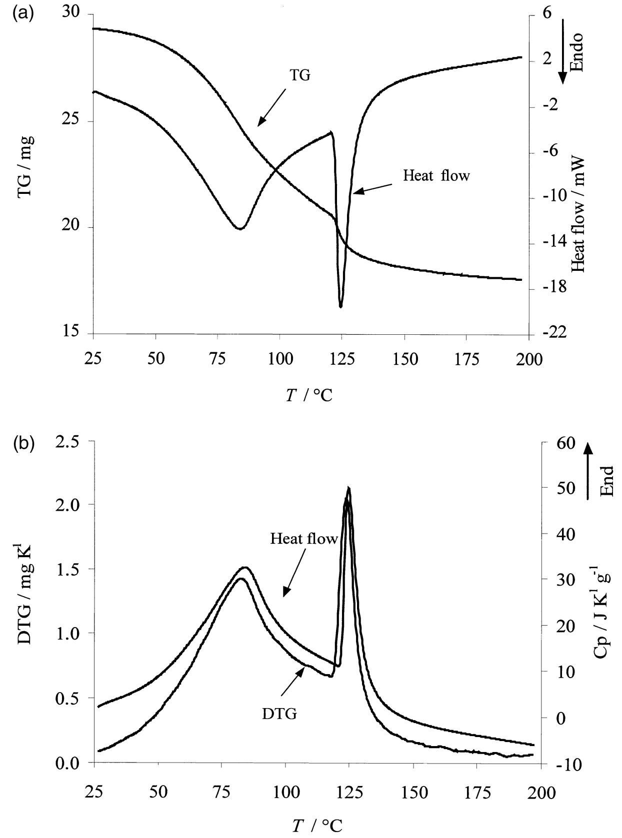

Fig. 1. (a) Raw TGA trace (TG) of a manually mixed dough with 42% moisture content (30 mg sample, 2°C/min heating rate) and related heat flow. (b) DTG and *C*p traces of the same sample (see text).

Reminding that the onset of starch gelatinization occurs at about 45°C, the mass loss recorded below this temperature was referred to as imbibing water ($a_w = 1$) that did not encounter any particular difficulty to diffuse from the core to the surface of the sample through a medium that remained practically unmodified. A substantial increase of the medium viscosity, due to starch gelatinization and gluten cross-linking, occurred above 45°C, but its effects were overcome by the enhanced molecular mobility sustained by the increase of temperature: the water loss rate thus continued to rise and reached a maximum at 85–87°C, when starch gelatinization and gluten reticulation were almost completed within the dough sample. This means that during the early 30 min time span (at this heating rate) the starting dough turned into crumb.

The presence of a maximum can be explained taking into account that the water loss makes the viscosity of the system to increase and therefore the vaporization rate to drop down; moreover, the formation of a tight gluten network, whose rate becomes large at this temperature (~80°C), can produce an overwhelming increase of the system viscosity which could no longer be counterbalanced by the increase of temperature; this process and also the lack of imbibing water would govern the descending tail of this DTG peak.

In order to understand the mechanism underlying the water loss related to this DTG peak, experiments were performed at the same heating rate with samples of different mass. It was found that the onset slope of the DTG trace was related to the sample mass (see Fig. 3), being slower for larger samples and therefore affecting also the temperature of the relevant DTG maximum. The migration paths of water molecules toward the vapor phase strictly depend on the dough structure (e.g. presence of pipes, internal vacuoles, etc.) and the dough surface (flat, wrinkle, porous, etc.). The actual transport

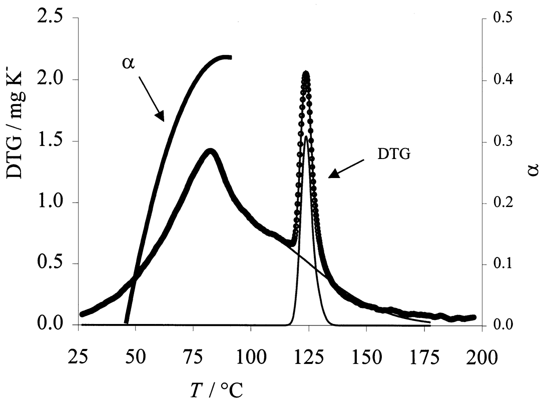

Fig. 2. Deconvolution of the DTG trace of a manually mixed dough with 42% moisture content (30 mg sample, 2°C/min heating rate) and starch gelatinization degree α of a dough undergoing heating at the same heating rate in a open DSC pan (see text).

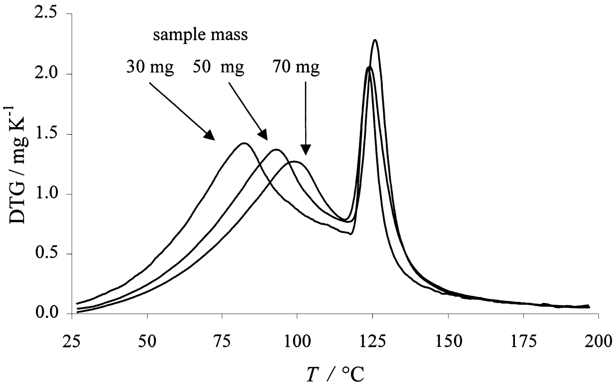

Fig. 3. DTG traces of 30, 50, 70 mg samples of manually mixed dough with 42% moisture content (2°C/min heating rate).

mechanism can be difficult to be unambiguously assessed; a phenomenal description can none the less be proposed with the aim at understanding whether the water release is mainly governed by physical (molecular mobility, medium viscosity, etc.) or chemical (specific binding, solvation equilibria, etc.) constraints. In the present case the early water release can be phenomenally modeled with a simple diffusion mechanism. As a matter of fact, in a diffusion regimen the water loss rate, dm_W/dt, should depend on the specific surface area, namely A/V (where A and V stand for surface area and volume, respectively); since the samples considered were given a ball shape, dm_W/dt had to be proportional to the inverse of sample radius, which is in turn proportional to the $m_w{}^{-1/3}$. Fig. 4 shows that this was indeed the case, since the DTG values observed at a given temperature for the three different dough sample masses showed a straight line trend versus $m_W^{-1/3}$ throughout the temperature range below the maximum.

Another aspect to be checked was the effect of the heating rate on the position of the DTG peak. With this aim two different heating rates were applied at a given mass sample: the experiment was repeated for all the sample masses considered above. A 20°C upward shift of the first DTG peak was observed in every case, whereas no substantial change appeared for the high T signal. As an example, Fig. 5 shows the results obtained for a 70 mg dough sample. Such a behavior depends on the fact that the diffusing species cannot immediately react to the driving force imposed, namely, the increase of temperature, requiring some time to relax: for this reason, when the heating rate is large, the DTG peak appears at a higher temperature.

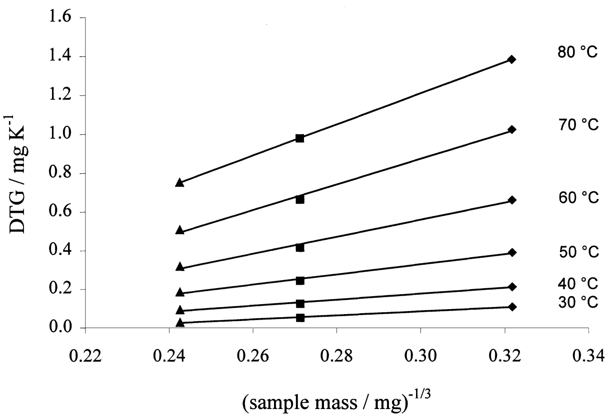

Fig. 4. Trend of DTG value-vs-(sample mass)$^{-1/3}$ at various temperatures. Data obtained from the traces reported in Fig. 3.

In all the cases so far presented, the second DTG peak remained practically unmodified, either for its shape and underlying area or for the temperature of its maximum, which occurred rather above 100°C. This peak appeared rather sharp and narrow looking like the signal produced by a well defined transition: in other words, its shape could not be explained with a diffusion mechanism, but required a more chemical interpretation. One had, therefore, to determine which of the dough component should be responsible for trapping water well above the boiling point and releasing it so abruptly. Because of their major fraction in the dough recipe, starch and gluten had to be considered first.

Samples of a starch aqueous suspension and a gluten dough were examined separately. Fig. 6 reports the relevant DTG traces: starch gave a single diffusional peak accounting for the whole water content, with an almost vertical ending tail due to the exhaustion of solvent, while the gluten dough gave a two peak trace, which seemed rather analogous to that observed for wheat flour dough samples. It was therefore conclusive that the high-T DTG peak in the traces of flour dough samples had to be assigned primarily to their gluten component.

The above findings suggested that water in the dough would mainly be in two states, namely,

1. free to diffuse through a medium, whose viscosity increases with increasing T because of the drying

and to transformations affecting starch and gluten; and
2. tightly bound to the gluten network and able to flash off only at rather higher temperatures.

Reminding the general considerations reported in the introduction of this paper, namely, that water in a freshly mixed dough is shared between different phases (mainly, starch, gluten, pentosans, etc.) and occupies the inter-phase regions, one has to realize that, when the dough is heated up, separated phases are still present in the system and water accordingly rearranges its partition over the phases, being subjected to different driving forces. Below 45°C, dough water simply increases its vapor tension and mobility at increasing *T*; at 45°C and above, water swells starch granules, solvates amylose and amylopectin molecules and sustains gluten cross-linking, playing also the role of plasticizer between polymer chains: it becomes less free to move away and therefore requires higher temperatures to flash off. Being engaged within polysaccharide gels and gluten network, water largely contributes to perfect the structure of these substrates, which attain a well defined conformation where some water is more tightly bound.

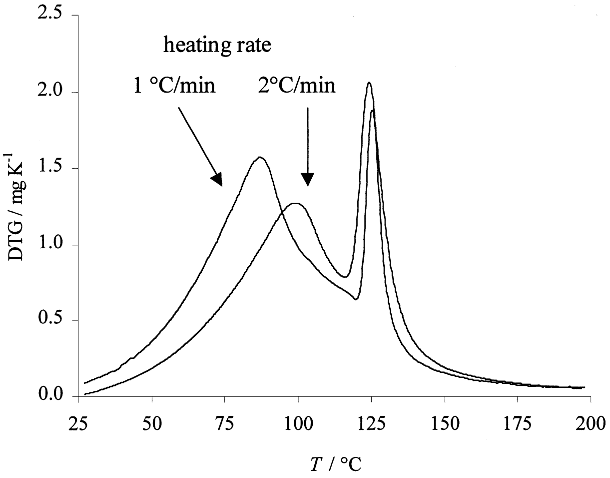

Fig. 5. DTG traces at 1 and 2°C/min heating rate of manually mixed dough (70 mg samples with 42% moisture content).

As mentioned in the introduction, water partition between the different dough phases does not correspond to a true thermodynamic equilibrium: it may instead be referred as to a starting metastable state that can depend on a number of factors, like, for example, the overall dough moisture and the extent of mixing. At the same time, one has to expect that the starting partition of water may be important in the redistribution that takes place when the dough is heated. Effects of dough moisture, mixing, and resting time after mixing are in fact well recognized in the technological practice (Endo, Okada & Nagao, 1987; Kilborn & Tipples, 1972; Sarwin et al., 1993; Tipples & Kilborn, 1975).

Experiments were, therefore, performed with dough samples of different moisture (42 and 50% w/w): Fig. 7 shows that in the DTG trace of the dough with higher water content the two peaks were closer together, since

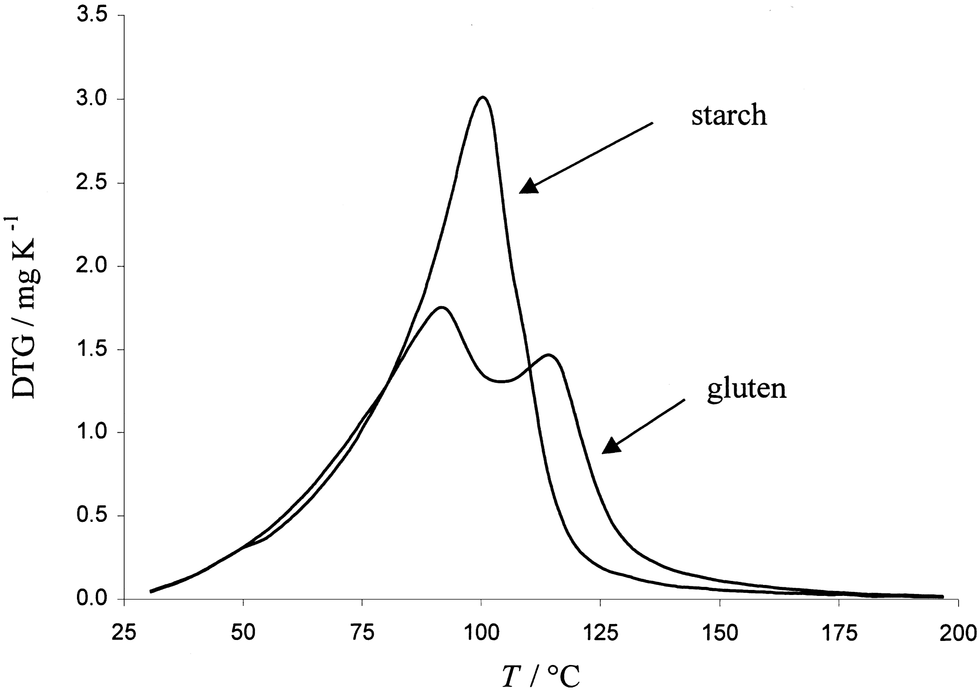

Fig. 6. DTG traces (30 mg samples, 2°C/min heating rate) of manually mixed starch–water suspension (54.2% moisture content) and manually mixed gluten (64.9% moisture content).

the high-T one occurred at a lower temperature (maximum at 108°C instead of 125°C), the position of the other being the same for either dough. The main observation however was that the solvent richer dough was almost wholly dehydrated at 125°C, i.e. when the other dough underwent the fastest release of its tightly bound water. As a simple interpretation, one could argue that, when the dough moisture is increased, water in the gluten phase would include a poorly linked fraction which is able to flash off when the gluten network is not yet too tight.

Mixing mainly affects gluten cross-linking being related to the mechanical energy trapped within the dough. As a side effect, the water fraction bound to gluten should also be modified. To check this aspect, dough samples that had experienced different mixing times (at the same rpm speed and torque) and different rest time after mixing were examined with TGA.

Fig. 8 shows the DTG traces obtained from two fresh dough samples which were mixed for 10 and 30 min, respectively. The first trace showed two well defined peaks, while the second was characterized by a much less profound inter-peak valley and a substantially smaller high-T peak.

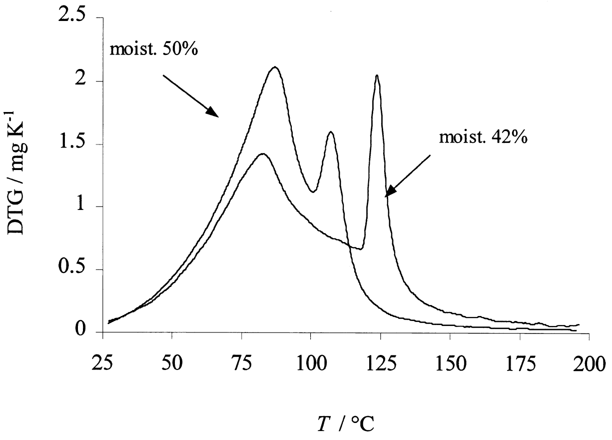

Fig. 7. DTG traces (30 mg samples, 2°C/min heating rate) of manually mixed dough with 42% and 50% moisture content.

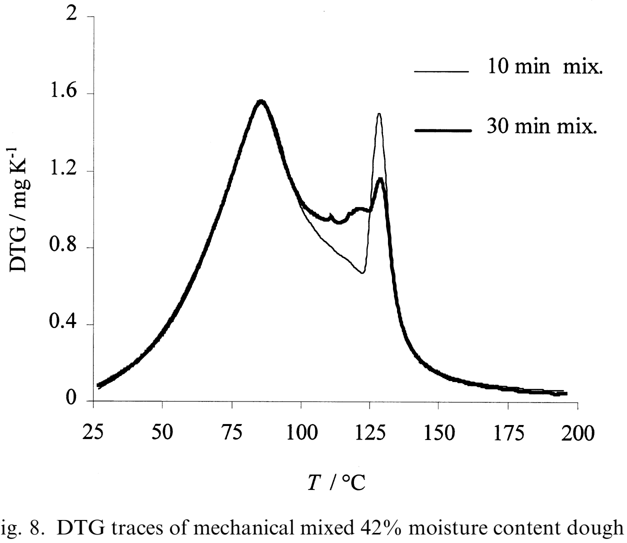

Fig. 8. DTG traces of mechanical mixed 42% moisture content dough (see text) after 10, 30 min mixing (30 mg samples, 2°C/min heating rate).

Deconvolution of these traces showed that the overall signal might be reproduced as the sum of three main contributions (see Fig. 9a and b). An intermediate signal was recognized between the two main peaks: it could correspond to a water fraction less tightly bound being still related to the gluten phase, in as much as its amount (14.1%) seemed increased (in the more mixed dough) at the expenses of that of strongly bound water (2.8%). This interpretation, however, must be referred to as a reasonable speculation that has to be confirmed with ad hoc further investigations. Looking at the corresponding proportion in the less mixed dough, 1.7 and 8.9% were found for the intermediate and the high T components.

It was also noticed that the overall amount of water corresponding to these peaks (intermediate and second peak) was larger (16.9%) in the more mixed dough than in the less mixed dough (10.6%). This would mean that the gluten phase is able to keep more water when mixing was longer.

Samples from the same dough lots were examined after 2 h rest after mixing. Fig. 10 shows that no significant differences existed between their DTG traces, where two well resolved peaks appeared with a slight shoulder along the descending branch of the first one. Rest after mixing had apparently canceled any difference induced by the different mixing time. The shoulder along the descending tail of the first peak occurred at about the same temperature as that of the intermediate component discussed above (Fig. 9a and b). In both traces the high T component was slightly (about 3 K) shifted to higher T with respect to the analogous signals observed for either dough before resting.

This experimental evidence suggested two main conclusions:

- mixing produces perturbations that affect water distribution within the dough and relax during a sufficiently long rest time;
- mixing reduces the strength of the water–gluten bonds, although this condition is not stable and tends to turn to a more tightly bound state.

This view seemed in line with the results reported by (Larsson & Eliasson, 1996a,b) who investigated the phases of a ultracentrifuged dough: these authors found that the water content of the gluten phase increased when the starting dough was overmixed and the separated gluten phase seemed stickier.

The overall picture could be accordingly adjusted. Below 45°C water can freely vaporize according to its partial pressure. Above 45°C, water vaporization is controlled by the diffusion through two main media, namely, a starch (amylose + amylopectin) gel and a gluten network which is undergoing reticulation. This accounts for the first DTG peak observed. A tightly bound water fraction, that mainly deals with gluten, is released only at higher *T* with a maximum rate occurring at 125°C and produces the second DTG peak. In the presence of excess water, this fraction is less tightly bound and can be released at lower *T*. Mixing unbalances the water partition mimicking the effects of a water enrichment, but a sufficiently long rest after mixing allows water to attain a more stable state (maximum evaporation rate at 128°C).

One could accordingly conclude that the dough undergoing baking can keep some memory of its initial phase separation and water partition. This has therefore a crucial role in bread making and should be taken into account when the dough recipe is modified by addition of other hydrophilic ingredients, like water soluble proteins, pentosans, salts, simple sugars, etc (Biliaderis, 1995; Chinachoti et al., 1991; Fessas & Schiraldi, 1998; Gudmundsson, 1991; Michniewicz, 1991, 1992)

Such an effect was verified by examining the behavior of dough samples whose composition was slightly modified by adding small amounts of water soluble proteins, like flour albumins and globulins (in the sense of Osborne). The DTG trace showed minor changes only for the high-*T* peak which moved toward slightly higher temperatures (130°C). These proteins seemed therefore to affect the water partition in the opposite way as compared to adding extra moisture, Fig. 11).

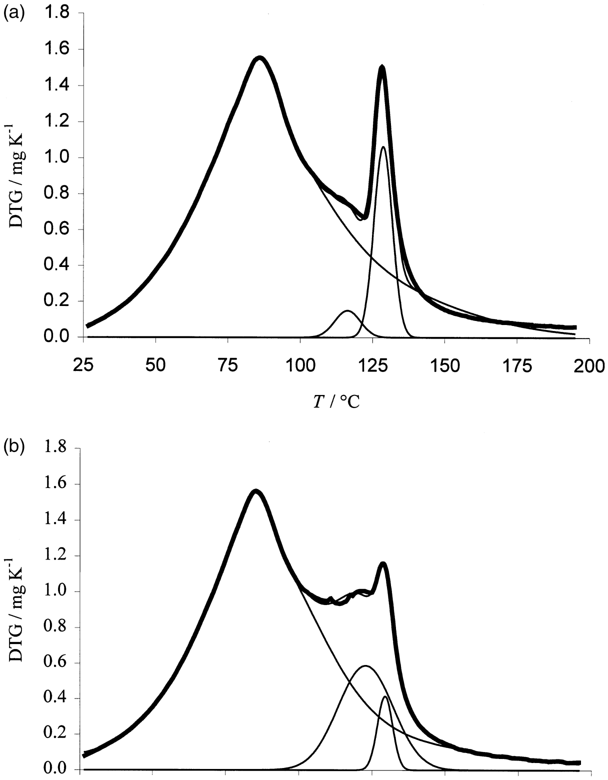

Fig. 9. Deconvolution of the DTG traces shown in Fig. 8.

The modifications induced by addition of pentosans to the standard dough recipe will be reported in a separate paper, since they imply a detailed and specific discussion which is outside the scope of the present work.

To complete the picture of the water properties in a wheat flour dough the present investigation is to be extended to the determination of water activity. This property will be also evaluated in bread at various aging times.

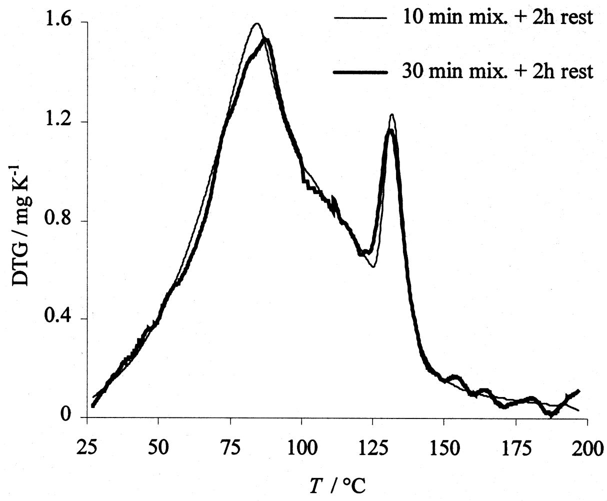

Fig. 10. DTG traces of mechanical mixed 42% moisture content dough (see text) after 10 and 30 min mixing and 2 h rest after mixing (30 mg samples, 2°C/min heating rate).

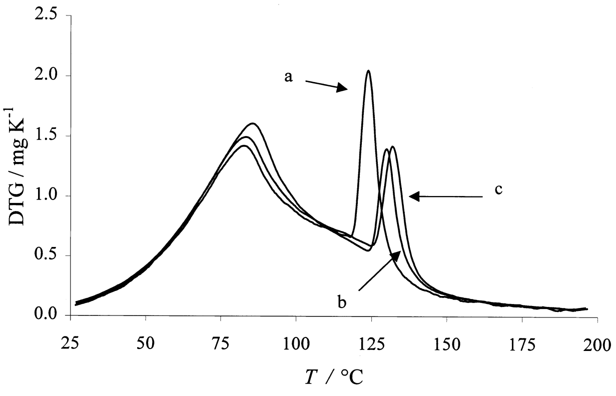

Fig. 11. DTG traces of manually mixed dough (30 mg samples with 42% moisture content, 2°C/min heating rate). Curves a, b and c are related to dough as it is, dough added with 25 mg of albumins, dough added with 25 mg of globulins, respectively (see text).

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