



## Staling of frozen partly and fully baked breads. Study of the combined effect of amylopectin recrystallization and water content on bread firmness

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### ARTICLE INFO

#### Article history:

Received 12 July 2010

Received in revised form

1 October 2010

Accepted 6 October 2010

#### Keywords:

Partly baked bread

Retrogradation

Staling

Freezing

### ABSTRACT

Partly baked (PB) and fully baked (FB) breads were frozen at  $-18\text{ }^{\circ}\text{C}$  for 7, 21, 63, 92, 126 and 188 d and were analysed after its thawing (FB) or thawing and final baking (PB). The starch retrogradation, the moisture content and the firmness were measured as properties closely related to the aging of bread. The temperature of glass transition of the maximally freeze-concentrated state,  $T_g'$ , was also measured and established in  $(-18 \pm 0.8)\text{ }^{\circ}\text{C}$ . This value cannot ensure molecular immobility in both types of bread during its frozen storage at  $(-18 \pm 2)\text{ }^{\circ}\text{C}$ . Consequently, the rearrangements of starch component molecules, needed for its recrystallization and for the diffusion of water during frozen storage, could take place and could justify the changes observed in the bread. PB bread showed a significant decrease in firmness with frozen storage, while the firmness of the FB bread did not change significantly, although an increase when compared with the control, not frozen bread, was detected. A regression study led to the conclusion that the combined effect of starch component crystallization and water loss could explain the firming evolution and that both variables exerted an effect of similar intensity on crumb firmness.

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### 1. Introduction

Bread staling remains responsible for huge economic losses. An approach for increasing the shelf life of bread is by applying freezing temperatures in bread distribution. Freezing a pre-baked bread is an easy way to prolong the shelf life of the bread and to keep its freshness (Barcenas et al., 2004). At present more than 60% of the bread consumed in Spain is made of frozen pre-baked bread. The bread is partially baked till the crumb is formed and before the Maillard reaction starts in the crust. Several studies have focused on determining the optimal time and temperature for partly baking (Ferreira et al., 1999; Fik and Surowka, 2002) and the quality of fresh bread after thawing and final baking (Carr et al., 2006; Ferreira et al., 1999; Fik and Surowka, 2002), concluding that bread obtained by the partly baking process has sensory and textural properties close to those of the bread obtained by conventional baking. However,

storage life of PB bread is often estimated to extend up to 6 months. In this period of time, bread characteristics perceived by consumers change when compared with fresh bread (Carr et al., 2006). Some authors have studied the effects of freezing and frozen storage on the staling of partly baked wheat bread (Barcenas et al., 2003; Barcenas and Rosell, 2006) by determining the retrogradation enthalpy of amylopectin, the crumb hardness evolution and the crumb microstructure. These authors attributed the changes observed to the damage of bread structure by the ice crystals.

The effect of freezing and frozen storage on baked goods was studied in the 1950s (Pence and Standridge, 1955). However, since then very little has been published about the effects of these processes on the staling of full baked bread. Some authors have compared the effects of freezing and frozen storage on texture and sensory quality of part and full baked breads prepared industrially (Fik and Surowka, 2002), and on breads containing hydrocolloids (Mandala et al., 2008). However, to our knowledge, the effects of frozen storage on amylopectin recrystallization of full baked bread have not been reported.

Staling involves two important changes in bread texture: the softening of the crust and the hardening of the crumb (Baik and Chinachoti, 2000). The first is a consequence of the migration of water from the crumb to the crust, driven by the higher water

Abbreviations: DSC, Differential scanning calorimetry; FB, Fully baked; FW, Frozen water; PB, Partly baked;  $T_g'$ , Temperature of glass transition of the maximally freeze-concentrated state;  $T_m'$ , Ice melting temperature of the maximally freeze-concentrated state; UFW, Unfrozen water.

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activity of the crumb in relation to the crust (Eliasson and Larsson, 1993). The hardening of the crumb is a complex phenomenon in which multiple mechanisms operate. Factors affecting crumb bread staling have been extensively investigated (Chinachoti and Vodovotz, 2001; Zobel and Kulp, 1996). All of them involve starch retrogradation. However, at present, many reports have shown that starch retrogradation is not the only factor (Baik and Chinachoti, 2000). Water plays a critical role in bread staling. When the retrogradation of amylopectin occurs, water molecules are incorporated into the crystallites and the distribution of water is shifted from gluten to starch/amylopectin, thereby changing the nature of the gluten network (Gray and BeMiller, 2003). Besides the molecular order of starch, water also plays an important role in crumb firmness due to its plasticizing effect on the crumb network (Hug-Iten et al., 2003).

Starch retrogradation is a temperature- and time-dependent phenomenon, which involves reassociation of starch component molecules into a partially crystalline, ordered structure. This, under favourable conditions, results in partial crystallization of both amylose and amylopectin. The kinetics of recrystallization of the two starch polymers differs considerably. Pure amylose in water crystallizes within hours whereas amylopectin in water requires several days for crystallization (Miles et al., 1985). Because firming of bread also develops over several days, most staling models view the changes in the amylopectin as the primary cause for crumb firming (Zobel and Kulp, 1996). The slow crystallization of amylopectin was referred to as a nucleation-limited growth process, which occurred above the glass transition temperature in a mobile, viscoelastic, fringed-micelle network (Roos, 1995). Our previous studies (Ronda and Roos, 2008) have shown that starch recrystallization during storage was enhanced by a prefreezing treatment at temperatures above  $T_g'$ . This temperature, that determines the glass transition of a maximally freeze-concentrated state, is used to characterize frozen materials (Roos, 1995). Above  $T_g'$ , molecular mobility is enhanced and consequently molecular rearrangements for nucleation can take place. Further storage at a higher temperature enhances the growth and the maturation of crystals leading to more rapid recrystallization in comparison with unfrozen systems (Ronda and Roos, 2008). This could explain deterioration of frozen bread when stored at temperatures around  $-18^\circ\text{C}$  and the more rapid aging after thawing than is found for the fresh, not frozen bread.

The aim of the present study was to investigate and compare the effects of frozen storage time at the usual commercial and domestic temperature of  $-18^\circ\text{C}$ , on FB and PB bread staling. Breads, after thawing and at the end of baking of part baked bread, were analysed and stored at  $4^\circ\text{C}$  to evaluate their aging kinetics and shelf life. The evolution of the starch retrogradation, quantified by DSC, and of the crust and crumb water were analysed considering these properties to be closely related to the aging of bread. Firmness, instrumentally measured, was used to quantify the bread staling. A regression study to correlate crumb firmness with the enthalpy of melting of recrystallized starch components and the crumb water content was carried out to establish the relative importance of these properties in the bread staling phenomenon.

## 2. Materials and methods

### 2.1. Bread processing stages

The bread used for this study, of baguette-type, was produced industrially. The formula included wheat flour, water (57% flour basis), yeast (2%), salt (1.8%), and bread improver (0.5%) with ascorbic acid, diacetyl tartaric ester of monoglyceride,  $\alpha$ -amylase and hemicellulase (Puratos, Spain). The proximate analysis of the

bread was: ash 2.0%; protein 9.8%; fat 1.5%; total dietary fibre 2.2%; and total carbohydrate 56.6%.

The bread was produced using a Sancassiano continuous kneading system (Alba, Italy) and a Mecatherm production line (Barembach, France) in a direct process. After kneading and make-up, the dough underwent a 90 min fermentation process and pre-baking was performed for 12 min at  $185^\circ\text{C}$ . After the cooling phase, the PB bread was deep-frozen to  $-18^\circ\text{C}$ , packaged in plastic bags in a cardboard box, and kept in frozen storage at  $(-18 \pm 2)^\circ\text{C}$ . PB bread samples for analysis were taken at 7, 21, 63, 92, 126 and 188 d and thawed at room temperature (45 min). After unwrapping, PB breads were baked at  $190^\circ\text{C}$  for 16 min in a Salva convection oven (Lezo, Spain) and cooled at room temperature. FB breads were prepared from PB just after its first baking stage following the same procedure described for its second baking stage. Some loafs of FB bread were measured after 3 h of baking (control bread, 0 d frozen time) and the rest were frozen exactly as PB during the same periods of time. PB and FB had a delay of 3 h before the analysis.

To study the effect of frozen storage on shelf life (aging kinetics at over-zero temperatures), breads after thawing (FB) or thawing and final baking (PB) were stored 1, 2 and 8 d under refrigeration at  $(4 \pm 2)^\circ\text{C}$ .

### 2.2. Physical measurements

A Mettler Toledo-DSC 822e (Schwerzenbach, Switzerland) equipped with a ceramic sensor FSR5 of high sensitivity, liquid nitrogen cooling system and nitrogen purge gas was used. DSC was performed on 20–25 mg of bread crumb taken from the centre of the bread loaf. The glass transition ( $T_g'$ ) and the ice melting ( $T_m'$ ) temperatures of the maximally freeze-concentrated systems were obtained as reported by Roos and Karel (1991) and Ronda and Roos (2008). The water (FW) fraction that was frozen was derived from the relationship between the enthalpy of ice melting in the maximally freeze-concentrated system and the latent heat of ice melting ( $334\text{ J/g}$ ). The unfrozen water (UFW) fraction was determined as the difference between total water weight fraction (determined in oven at  $103^\circ\text{C}$ ) and the FW fraction (Ribotta and Le Bail, 2007). Starch retrogradation was evaluated from DSC endotherms obtained during the temperature scanning from  $0^\circ\text{C}$  to  $105^\circ\text{C}$  at a heating rate of  $5^\circ\text{C}/\text{min}$ . DSC analyses were carried out for fresh bread (control) and in PB and FB breads stored 7, 21, 92 and 188 d in the freezer. The samples for the study of the crumb retrogradation kinetics at  $(4 \pm 2)^\circ\text{C}$  were stored in the DSC pans ( $40\ \mu\text{L}$ ) for 0, 1, 2 and 8 d. Each measurement was performed in duplicate. The melting enthalpy was expressed in  $\text{J/g}$  of solids.

Water content was determined in crumb, crust and the whole bread in duplicate following the standard method 44-15A (AACC, 2000).

Firmness was determined in triplicate with a TA-XT2 texture analyser (Stable Microsystems, Surrey, UK). A compression test with an aluminium 25 mm diameter cylindrical probe was used. The force to penetrate to 50% depth, at  $1\text{ mm/s}$  speed test was recorded. The analysis was carried out at  $(20 \pm 2)^\circ\text{C}$  for bread slices of 20 mm thickness taken from the centre of the loaf.

### 2.3. Statistical analyses

In order to assess significant differences among samples, a multiple comparison analysis of samples using the program Statgraphics Plus V5.1 was done (Bit stream, Cambridge, Massachusetts, USA). Fisher's least significant differences (LSD) test was used to describe means with 95% confidence. Correlation and regression studies were carried out with the software Statistica V6 (Tulsa, OK, USA).

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