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# Monitoring the crystallization of starch and lipid components of the cake crumb during staling

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#### 1. Introduction

The quality of industrial-scale bakery products depends on controlling staling (Dargue, 1975). Staling has been studied for more than a century; Bechtel, Meisner, & Bradley (1953) defined it as the term that indicated decreasing consumer acceptance of bakery products caused by changes in crumb other than those resulting from the action of spoilage organisms. It was postulated that starch produced bread staling. Several works then showed that bread staling was closely associated with starch retrogradation (Inagaki & Seib, 1992; Kim & D'Appolonia, 1977a, 1977b; Leon, Duran, & Benito De Barber, 1997; Schoch & French, 1947). The basic ingredients in cake dough are flour, fat, egg, milk, sugar and salt. Although the functionality of each one of these ingredients is known, it is difficult to assess their specific contributions in complex systems, such as cakes. Information regarding the mechanism of staling is mostly based on moisture migration (Lahtinen, Levda, Jouppila, & Salovoara, 1998; Sych, Castaigne, & Lacroix, 1987; Willhoft, 1973)

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#### ABSTRACT

Cake staling is a complex problem which has still not been fully understood. Starch polymers retrogradation, which is linked to biopolymers recrystallisation, is the most important factor affecting cake firmness in addition to water migration and fat crystallization. In this study, the effect of storage temperatures of  $4^{\circ}$ C and 20 °C on starch retrogradation and fat recrystallization was investigated. Starch retrogradation can be tracked through changes in crystalline structure via X-rays diffraction as well as through melting of crystals via calorimetry. These techniques have been coupled to study the different phenomena occurring during staling. The results revealed that the storage of cakes at 20 °C for 25 days showed more starch polymer retrogradation and more intense fat recrystallization in the  $\beta$  form than at 4 °C. Consequently, the staling was delayed when a low storage temperature like 4 °C was used, which is recommended to retain high quality cakes during storage.

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while the effects of combined ingredients on the staling rate of cakes remain poorly documented.

The staling rate of cakes is slower than in bread systems because of higher ratios of sugar and fats in the formulation (Gelinas, Roy, & Guillet, 1999). Cakes are usually expected to keep their initial quality attributes from 1 to 4 weeks or more, depending on the formulation, water activity and storage temperature. It is known that cake staling can be accelerated by changing the storage parameters, such as time and temperature. For example, it is faster during storage at 20 °C compared to 4 °C (Hodge, 1977; Pence & Standridge, 1958; Sych et al., 1987). However, few works have provided results on the evolution of the ingredients, such as starch and fat, during storage at different temperatures.

Crystallization during the staling process should be monitored in order to determine the relationship between cake texture and the retrograded starch process or lipid crystallization, throughout the storage period. The botanic origin determines the structure of starch, it is reported in the literature that native cereal starches present an A-type X-ray diffraction pattern (Katz & Derksen, 1933). During cake making, starch is subjected to heating and cooling. When it is heated in the presence of water, the linear amylose diffuses out of the swollen granules (gelatinization) leading to a loss of the granule structure (Donavan, 1979; Zobel, 1988). Recrystallization occurs upon cooling and storage, causing the appearance







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 Table 1

 Details of ingredients in the cake formula.

Ingredients	Percentage %
Flour (F)	29.5
Sugar (S)	25
Fatty substances (Fs)	20
Liquid eggs (Le)	25
Baking powder (Bp)	0.5

of other structures, such as V-type and B-type, which correspond to the formation of amylose–endogenous lipid complexes (Biliaderis, 1992; Biliaderis, Page, Slade, & Sirett, 1985; Kugimiya & Donovan, 1981; Kugimiya, Donovan, & Wong, 1980) and amylose or amylopectin retrogradation (Kalichevski & Ring, 1987), respectively. This phenomenon produces the crystallization of the amorphously melted starch.

The functional properties of fat are strongly related to its composition and the type of crystals formed at the temperature of the application. The main constituents of fats and oils are triglycerides and their different polymorphic forms have been detailed by many authors (Hagemann, 1988; Ollivon & Perron, 1992). The three major polymorphic forms are  $\alpha$ ,  $\beta'$ , and  $\beta$  in increasing order of stability. These can be identified by X-ray diffraction (Keller et al., 1988; Keller, Lavigne, Loisel, Ollivon, & Bourgaux, 1996).

In this study, our objective was to observe the structural reorganization, during storage, of some specific constituents of cake like the fatty substances and starch. Two storage temperatures were tested to determine their respective effects on the occurrence of staling.

#### 2. Materials and methods

#### 2.1. Materials

Wheat flour (14.8% water content, 9.9% protein, 1.1% fat, 71.5% starch and 0.4% ash; all on a wet basis) was supplied by Giraudineau (France), whole liquid eggs (0.8% minerals, 12.1% protein, 10.2% fat and 0.8% carbohydrate) were purchased from Ovoteam (France). Fat consisted of rapeseed oil (70%) and anhydrous milk fat (30%) supplied by Corman (Belgium). Sugar was purchased from Saint Louis, France. Baking powder (sodium bicarbonate) was supplied by Brenntag.

#### 2.2. Baking procedure

The cakes studied were made according to the recipe described below and the ingredients used are summarized in Table 1.

First, sugar, whole liquid eggs and fat were placed in the stainless steel bowl of a Kitchen Aid mixer (KSM90, Kitchenaid, St. Joseph, MI) and mixed (speed 6) for 2 min. Next, wheat flour and baking powder were added and the whole was remixed (speed 8) for 3 min. The final temperature of the dough was between  $18 \circ C$  and  $20 \circ C$ . Then, the dough was placed in the nine cavities of a silicone pan. The pan was placed in the middle of a ventilated oven and the dough was baked for  $15 \min$  at  $180 \circ C$  (Hesso, Loisel, Chevallier, & Le-bail, 2014). After baking, the cake was cooled down to room temperature. For all the experiments, cake from the same position in the pan was used.

#### 2.3. Sampling and storage

The samples were taken from cakes as indicated in Fig. 1 and stored at two temperatures,  $4 \degree C$  and  $20 \degree C$ . The water content was adjusted by desorption at  $a_w = 0.75$  over saturated NaCl solution

Position



Fig. 1. Positions of the various samples taken from the cake.

throughout the storage. All samples were prepared in duplicate and each measurement was performed three times.

#### 2.4. X-ray diffraction (WAX-SAX)

Fifty milligrams of sample, equilibrated at  $a_w = 0.75$ , was sealed in a copper ring between two adhesive tape sheets to prevent any change in water content. The sample was examined by wide-angle (WAX) and small-angle (SAX) X-ray diffraction. Measurements were performed using a D8 Discover spectrometer from Bruker-AXS. Cu K $\alpha_1$  radiation (Cu K $\alpha_1 = 1.5405$  Å), produced in a sealed tube at 40 kV and 40 mA, was selected and parallelized using a double Gobël mirror parallel optics system and collimated to produce a 500-µm beam diameter with sample alignment by microscopic video and laser. Data were monitored by a GADDS 2D detector for 10 min and normalized.

#### 2.5. Heating cell XRD

The sample was placed in a special cell, between two mica windows, which was set on a heating stage HFS91 (Linkam, UK). The detector was positioned at a focusing distance of 8.6 cm from the sample surface in a direct beam position. The heating kinetics applied to the sample were  $1 \,^{\circ}$ C/min from  $10 \,^{\circ}$ C to  $60 \,^{\circ}$ C. Every  $5 \,^{\circ}$ C, a plateau of 5 min was introduced to enable the acquisition of the diffraction spectrum.

#### 2.6. DSC analysis

DSC thermograms were recorded using an automated heat flux differential scanning calorimeter (TA Instruments, Q100). Stainless steel high-pressure cells (TA Instruments, ref: 900825.902) were used. The system was calibrated with indium and a pan containing 12  $\mu$ l of water was taken as the reference taking in consideration the water content of the sample. Eight milligrams of sample was weighed into pans and 12  $\mu$ l of water was added. Pans were sealed. Two successive scans were run in triplicate at 3 °C/min from 1 °C to 160 °C for the first scan and from 1 °C to 160 °C at 3 °C/min for the second, separated by a cooling stage at 3 °C/min.

#### 3. Results and discussion

#### 3.1. Effect of the sampling and storage temperature on staling

The X-ray diffraction diagrams (data not shown) of the different samples taken from the crumb of cakes at different positions (Fig. 1)

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