Sponge cake microstructure, starch retrogradation and quality changes during frozen storage

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Summary The effect of frozen storage (6 months) on the microstructure (microscopy, flatbed scanning and image analysis), quality (texture, moisture and specific volume) and starch retrogradation of the cake crumb were evaluated. After 2 months of storage, texture (firmness, cohesiveness and resilience) was significantly ($P < 0.05$) affected and starch retrogradation was observed, while by the fourth month, the crystallinity increased and crumb fractures were noticeable. Additionally, the shrinkage of starch granules was observed as the starch circularity (Sc) values significantly decreased ($P < 0.05$) by the sixth month of storage. Although structural changes were not detected by image analysis, it was demonstrated that cake microstructure damage is related to physical changes because the Sc was significantly correlated ($P < 0.05$) with moisture and specific volume and therefore with the cake quality and texture. Moreover, sugar re-crystallisation occurred during frozen storage, and it was significantly correlated ($P < 0.05$) with the deterioration in cake quality.

Keywords Cake, freezing, frozen storage, starch retrogradation, starch shrinkage, structure, texture.

Introduction

Sponge cake is one of the most consumed cakes in America due to its porous structure that allows the production of wet cakes of good quality such as 'Génoise' or the 'Biscuit de Savoie' in Europe (Levy, 1981) or the 'Three milks cake (Tres leches)' in Argentina and Mexico. This type of product is usually produced in two stages. In the first stage, the product is baked and frozen, usually in a factory; second, it is delivered to the retail store where it is kept frozen until the point of sale when it is thawed and decorated. This process results in a good alternative because the product handling is easier. However, during frozen storage, the quality of the product is reduced, which results in economic losses to the industry.

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According to Gélinas *et al*. (1999), the shelf life of cakes depends on their formulation, packaging material and storage conditions. The process of freezing and frozen storage can reduce the staling rate. However, a detrimental effect on textural (firmness) and quality properties (specific volume and moisture content) during sub-zero temperature storage has been reported (Owen & Duyne, 1950; Karaoğlu *et al*., 2008; Gómez *et al*., 2011; Jia *et al*., 2014). Additionally, although not related to cakes, the growth of ice crystals and their re-crystallisation affect the structure of the bread crumb (Berglund *et al*., 1990, 1991; Polaki *et al*., 2010). The increase in firmness is associated with the redistribution of water in the crumb (Guy *et al*., 1983), starch interactions (Gómez *et al*., 2007) and storage temperature (Cauvain, 1998). In addition, during frozen storage, the starch and protein networks that determine the crumb structure of the cake undergo important changes such as that were observed in certain model systems (Bean & Yamazaki, 1978). The starch re-arrangement in the bread itself has been studied qualitatively and quantitatively (Hug-Iten *et al.*, 2001; Primo-Martín *et al.*, 2007) by evaluating the crystallinity by X-ray diffraction or the retrogradation enthalpy by differential scanning calorimetry (DSC). This type of analysis (Ji *et al.*, 2010; Sozer *et al.*, 2011) could be applied to frozen cake samples by following the changes in the starch during storage and relating them to textural and structural properties evaluated at both the macro- and micro-level, as well as by applying microscopy techniques and image analysis. Hence, the aim of this work was to provide a more in-depth knowledge about the relationships among the quality changes in sponge cake (textural and physical properties) and variations in crumb structure evaluated by microscopy at different scales when the cake is stored under freezing conditions.

Materials and methods

Materials

Sponge cake was prepared using the formulations based on Gómez *et al.* (2011) and Guillard *et al.* (2003) with some adjustments: 28% wheat flour (Harinera de México, S.A. de C.V., Cuauhtémoc, DF, México), 30.2% fresh egg (San Juan, Proteína Animal S.A. de C.V., San Juan de los Lagos, Jalisco, México), 18.9% refined sugar (Great Value; Walmart, Bentonville, AR, USA), 12.9% water (Bonafont, Liquimex S.A. de C.V., Toluca, Estado de México, México), 5.8% corn starch (Maizena, Unilever de México, S. de R.L. de C.V. Tultitlán, Estado de México, México), 1.7% skim milk powder (Svelty, Nestlé S.A. de C.V, Miguel Hidalgo, DF, México), 1% baking powder (Royal, Kraft Foods De México, S. De R.L. De C.V, Álvaro Obregón, DF, México), 1% emulsifier (Grindsted®SSL; Danisco, Copenhagen K, Denmark) and 0.5% salt (La Fina, Sales del Istmo, S.A. de C.V, Coatzacoalcos, Veracruz, México).

Methods

Sponge cake preparation

The sponge cake was prepared according to the methodology reported by Díaz-Ramírez *et al.* (2013). The product (10.6 ± 0.1-cm diameter, 5.0 ± 0.1-cm height) was covered with a plastic film (125 gauge and 31.7 µm in thickness, Cryovac®D-955 Multipurpose Shrink Film; Sealed Air, Duncan, SC, USA). It was frozen (horizontal plate freezer, −28 ± 3 °C) for 240 min (–18 °C at the centre), and then it was stored for 6 months in a chest freezer (−23 °C, Kenmore, Sears Holdings Corporation, Hoffman Estates, IL, USA). The thawing process was carried out at room temperature (20 °C) during 240 min, until the temperature of the centre of the sample rose to 20 ± 2 °C. At the end of this process, the plastic film was removed. During those operations, a thermocouple was inserted into the centre of the cake. Temperatures were measured every 60 s using a K-type thermocouple (0.81-mm diameter, T/C wire) connected to a digital scanning thermometer (Digi-Sense, Mod. 69202-30, Eutech Instruments; Cole-Parmer Instrument Co, Vernon Hills, IL, USA). Data were recorded in a personal computer using the Scanlink software v 1.2.1 (Barnant Co., Barrington, IL, USA).

Sponge cake evaluation

The analyses were carried out in samples without freezing (Control), frozen and thawed without storing (0) and after being stored for 2, 4 and 6 months and then thawed (2, 4, 6).

Sponge cake quality

The whole sponge cakes were weighed on a precision balance (Ohaus Adventurer Pro AV4101; Ohaus Corp, Pine Brook, NJ, USA, 4100 g capacity and precision to 0.1 g; the cake weight varied from 200.3 ± 0.6 to 197.4 ± 0.8 g during frozen storage); the volume was determined by the seed displacement method, and the specific volume was calculated by dividing the product volume by its weight (Lee *et al.*, 1982); the volume measurement accuracy for this determination was ±10 cm^3. After the crust was removed, the sponge crumb was milled for 30 s in a kitchen blender (Braun Aromatic KSM2; Procter & Gamble, Cincinnati, OH, USA). A 4-g sample was analysed in a moisture analysis balance (MB45 Moisture Analyzer; Ohaus Corp) following the manufacturer's procedure (Pastry method: step drying program at a drying temperature of 170/130 °C).

Texture analysis

The texture was measured using a texture analyser (TA-XT plus; Stable Micro Systems, Godalming, Surrey, UK), employing a 25-mm-diameter aluminium cylinder probe and applying a double compression test to penetrate to 50% depth, at a compression speed of 5 mm s^{-1}. Samples (40 × 40 × 40 mm) were prepared by removing the sponge dome, as well as the side crusts, following the methodology as recommended by Gómez *et al.* (2011) and Karaoğlu *et al.* (2008). Due to the sample size, special care was taken to assure that the probe was centred, in this way avoiding any possible effect at the edges of the sample. The firmness, cohesiveness, gumminess and resilience were obtained from the texture profile analysis, where the peak of the first compression was defined as the firmness; the ratio of the force area during the second compression and the first compression is defined as the cohesiveness. The resilience is the ratio between the area of decompression and the area of compression during the first compression cycle. The gumminess is defined as the result of multiplying firmness by cohesiveness. Three independent sponge cakes were analysed.

X-ray diffraction analysis

X-ray diffraction analyses were performed on milled (30 s/Braun Aromatic KSM2, Procter & Gamble, Cincinnati, OH, USA) and freeze-dried (6–8 h, −50 °C, 75034 Bench Top Freeze Dryer; Labconco, Kansas City, MO, USA) cake crumb samples, with a moisture content of 12 ± 2%. The X-ray diffractometer (Miniflex 600; Rigaku Corporation, Sendagaya, Shibuka-Ku, Tokyo, Japan) was used at 40 kV and 15 mA with a CuKα radiation source (λ = 0.154 nm), scanning 2θ in the range from 2° to 60°. The step size was 0.01° with a measurement rate of 4° min^{-1}. Three independent sponge cakes were analysed.

Crystallinity analysis

The X-ray diffraction patterns were analysed according to Ribotta *et al.* (2004) using the Peak-fit V.4.12 software (SeaSolve Software Inc, Framingham, MA, USA) and calculating the total mass crystallinity, $\mathrm{Xcr}_{(\mathrm{T})}$, (eqn 1), where I_{T} is the area of the crystalline phase (all the peaks) and I_{A} is the area of the amorphous phase;

$$\mathrm{Xcr}_{(\mathrm{T})} = \frac{I_{\mathrm{T}}}{I_{\mathrm{T}} + I_{\mathrm{A}}} \qquad (1)$$

Differential scanning calorimetry

Milled (30 s/Braun Aromatic KSM2, Procter & Gamble, Cincinnati, OH, USA) and freeze-dried (75034 Bench Top Freeze Dryer; Labconco, 6–8 h per −50 °C) crumb samples were weighed, and distilled water was added at a 3:1 (v/w) water-to-sample ratio in stainless steel pans (TA Instruments Inc., New Castle, DE, USA). Thermograms were obtained using a differential scanning calorimeter (Diamond DSC; Perkin Elmer, Inc., Waltham, MA, USA). Indium was used to calibrate the system. An empty stainless steel pan was used as a reference during the measurements. The samples were heated from 30 to 200 °C, at a heating rate of 10 °C min^{-1}. The enthalpy of amylopectin retrogradation was estimated by integrating the area of the endotherm observed between 50 and 60 °C. It was expressed in J g^{-1} dry sample (db) calculated using the Pyris Thermal Analysis V7.0 Software (Perkin Elmer Inc., Waltham, MA, USA). Additionally, the onset temperature (Tonset), peak temperature and the end temperature (Tend) were reported. Three independent sponge cakes were analysed.

Crumb structure evaluation by image analysis and microscopy

The crumb images were taken from the central position of the cake (diameter slices of 10.6 cm), and these were analysed according to Díaz-Ramírez *et al.* (2013). The cell area, cell density, maximum length and shape factor were calculated, removing all objects smaller than or equal to 0.02 mm^2 and larger than or equal to 3.19 mm^2. Image analysis evaluations were carried out applying the ImageJ freeware (National Institutes Health, Bethesda, MD, USA). Microscopic evaluation was conducted on 10 × 10 × 10 mm^3 pieces taken from the central position of the cake with a razor blade, and for scanning electron microscopy (SEM) analysis only, these samples were immediately freeze-dried (24 h, −50 °C, 75034 Bench Top Freeze Dryer; Labconco, Kansas City, MO, USA). These freeze-dried samples were viewed at 30× and 10 KVA in a scanning electron microscope (JSM 5800LV; JEOL Inc., Peabody, MA, USA) as stated by Tlapale-Valdivia *et al.* (2010). Confocal scanning laser microscopy analysis was carried out as stated by Díaz-Ramírez *et al.* (2013) where two fluorescent probes, namely 0.5% fluorescein 5-isothiocyanate (FITC; Sigma-Aldrich Corp, St Louis, MO, USA) and 0.15% Rhodamine B (Sigma-Aldrich Corp) solutions, both in water, were used to stain the samples. The staining was performed in two stages. First, the FITC was added to stain the proteins, and second, the Rhodamine B was added to dye the starch after the protein reacted with the FITC. After staining, the samples were studied using a confocal scanning laser microscope (LSM 710; Carl Zeiss, Oberkochen, Baden-Württemberg, Germany) with 20× lenses.

The starch circularity (Sc) values were obtained from confocal micrographs. The images were pre-processed using Adobe Photoshop elements V. 12 software (Adobe Systems Inc., San Jose, CA, USA) converting the colour images to black and white images, and from them, only the starch granules were selected and filled with black colour, with the rest of the area being filled with white colour. These images were saved as tiff images and then were changed to greyscale (eight bit) using ImageJ software (National Institutes Health, Bethesda, MD, USA). Segmentation was carried out using the same software and applying the Otsu algorithm. The area, circularity and roundness of the starch granules were determined by removing the objects smaller than 100 μm^2 and <1000 μm^2, taking into consideration that most of analysed starch granules, (frequency histogram) were in this size range. For all microscopic techniques, three independent cake sections were analysed, and from each section, three microscopic fields were observed. A total of nine images and approximately twenty objects (starch granules) per image were evaluated for each storage time.

Data analysis

All tests were carried out in at least triplicate, and the average and standard deviation values are reported. The data were statistically treated by ANOVA and the Tukey test (SigmaPlot V.11.0 (Systat Software Inc, San Jose, CA, USA). $P < 0.05$ values were considered significantly different. Correlations were made using SigmaPlot software V.11.0 (Systat Software Inc).

Results and discussion

Cake quality

The freezing process (0) did not have an effect on the evaluated quality parameters, including texture (Table 1). Frozen storage had an effect on the quality parameters in comparison with the quality of the control sample, as the moisture (40.3 ± 0.1–39 ± 0.2) and specific volume (2.51 ± 0.03–2.34 ± 0.02 $cm^3\ g^{-1}$) values decreased (Table 1) during 6 months of frozen storage. These changes were noticeable after 4 months of storage, yielding at the end of storage a smaller and dryer cake meaning a lower quality cake. The firmness (Table 1), which is the most studied textural parameter associated with human perception of freshness (Carr & Tadini, 2003), significantly increased ($P < 0.05$), as its value after 6 months of frozen storage was approximately twice its initial value, varying from 30.4 ± 1.9 to 66.6 ± 4.0 N. This negative change was observed after 2 months of storage and produced an undesirably harder product at the end of this period. The increase in the firmness of the cake crumb was an expression of the staling process in which the starch biopolymers and moisture content were modified, as demonstrated by the correlation coefficient between firmness and moisture (Table 3) and because at the same time, a significant increase in the starch retrogradation enthalpy was noticeable (Table 2).

The cohesiveness and resilience values (Table 1) also significantly ($P < 0.05$) decreased after the second month of frozen storage, with their values changing by the end of storage from 0.68 ± 0.01 to 0.57 ± 0.01 and from 0.29 ± 0.03 to 0.18 ± 0.01 respectively. Cohesiveness measures the internal resistance of cake structure, and as this value decreases, a loss of intramolecular attraction among the ingredients and a trend to fragility with ageing is presented. Moreover, the resilience expresses how well a product 'fights to regain its original position' instantaneously after stress. According to our results, the moisture loss contributed to the generation of a more brittle crumb (Table 3), preventing a fast return to its initial condition due to the dehydration process. The lack of cohesion negatively affects the functionality of the sponge cake because its structure cannot hold up a larger quantity of liquid during the soaking, and thus, more suspended solids are produced, reducing its acceptability by consumers. In addition, the reduction in resilience also affects the appearance of the cakes because a lower volume of the cake is readily observed.

Finally, gumminess has been defined as the energy required to disintegrate a semi-solid food to a consistency that is ready for swallowing (Karaoğlu & Kotancilar, 2009). The effect of frozen storage (Table 1) on this parameter shows a tendency to increase, varying from 21.2 ± 2.7 to 36.3 ± 4.0, where high values are associated with dense and rubbery characteristics (Karaoğlu *et al.*, 2008), which are undesirable quality features of these cakes. Because this parameter is the product of firmness and cohesiveness, the migration of water towards the crust and the starch retrogradation must affect these results.

X-ray diffraction and differential scanning calorimetry analysis

X-ray diffraction patterns (Fig. 1) showed that the frozen stored samples had noticeable changes in their crystallinity as the intensities increased. A significant rise in the total crystallinity Xcr(T) (Table 2) after 4 months of frozen storage was observed, with this value Xcr(T) varying from 0.556 ± 0.050 to

0.771 ± 0.064. Moreover, the re-crystallisation process affects the cake texture, as seen in the correlation coefficient values with cohesiveness and resilience (Table 3). This means that a crystalline structure produced a more fragile cake that cannot regain its original size after stress. The more noticeable peaks in the frozen patterns were 11.9°, 13.2°, 15.5°, 18.8°, 19.6°, 22.0°, 24.7°, 29.4° and 38.3° for the 2Θ angle (Fig. 1). According to the database of the X-ray diffractometer and to the pattern of the raw materials, these peaks corresponded to sugar crystals, except for the peak at 29.4°, which had not been reported in the literature for bread products. This peak corresponded to the baking powder, specifically to calcium carbonate. Calcium carbonate is conventionally added to bread-making products (Sadd *et al*., 2008) as a 'dough conditioner' to regulate pH or to prevent the reaction between the acid salt and the soda in baking powders on cakes (Conn, 1965). In addition, the cakes contain a higher per cent of sugar in their formulation, which is normally added in its crystalline form and is solubilised during mixing, improving the air incorporation and producing more stable foams in the batters (Paton *et al*., 1981). The sugar is also considered a tenderiser as it delays the starch gelatinisation (Spies & Hoseney, 1982) and allows the formation of the desired cake structure (Bean & Yamazaki, 1978). The presence of sugar crystals measured by X-ray diffraction has been reported for a biscuit crust (Chevallier *et al*., 2000), while such a fact has not previously been observed in cake crumb. However, it is known that during freezing, the re-crystallisation of sugars can occur due to the formation of a supersaturated solution in the unfrozen phase (Blond & Le Meste, 2004); in this sense, we assumed that crystallinity rise could be attributed to physical changes in the sugar crystals such as re-crystallisation and reorientation, along with starch retrogradation, but more studies are required to confirm this finding. Moreover, the presence of sugar crystals could negatively affect the taste of the cake and therefore the consumer acceptability.

Based on DSC studies, the enthalpy change due to starch amylopectin retrogradation during frozen storage showed a significant increase at 2 months of storage (Table 2), which coincides with the time that the texture changed (Table 1). However, there was not a significant correlation between them, indicating that starch modification by frozen storage was not the only factor related to cake quality deterioration. The T onset significantly decreased (Table 2) after freezing and during frozen storage, while the peak temperature dropped at 2 months of this process. Similar tendencies were observed in frozen stored dough (Ribotta *et al*., 2003), part-baked bread (Bárcenas & Rosell, 2006) and in freeze-thawed waxy rice cake (Jongsutjarittam & Charoenrein, 2013). The interaction among the starch polymers could be influenced by the structure (Bárcenas & Rosell, 2006), moisture, lipid content and other ingredients (Biliaderis, 1990). A significant correlation between these parameters and total crystallinity (Table 3) indicated that re-crystallisation processes could induce changes in the structure and arrangement of amylopectin.

Cake-crumb structure

The freezing process (0) and frozen storage did not have a significant effect ($P < 0.05$) on all the cake crumb structural parameters (Table 1) compared with the values obtained from the control cake (Control). In this regard, it has been reported (Polaki *et al*., 2010) that the frozen storage of part-baked bread promotes a change in the shape of the crumb pores, making them rounder. This difference could indicate that some ingredients present in sponge cake formulation, such as the egg yolk, could be acting as a cryo-protective material.

From the SEM micrographs (Fig. 2a–e), the presence of fractures was observed (arrows) as the frozen storage time increased, probably as a result of the growth of ice crystals during storage. Baier-Schenk *et al*. (2005) reported that the gas pore interfaces in dough are preferential sites for ice nucleation, favouring the growth of ice crystals in these regions with a redistribution of water in the dough, probably weakening the solid matrix and producing the fractures. These fractures were apparently observable after the fourth month of frozen storage and during the next months. Although the fractures were present at the microscopic level, this fact did not affect the appearance of the cake crumb at a macroscopic level according to our results (Table 1); however, this could also be associated with the reduction in cohesiveness.

The confocal images (Fig. 3a–e) show the deterioration of the cake solid matrix during frozen storage. The cake–crumb structure is a complex system formed by a discontinuous phase of gelatinised starch granules (red) surrounded by a continuous phase of protein structures (green). It seems that the shape of the gelatinised granules, probably starch granule ghosts, changed with storage (Fig. 3) and acquired a more amorphous shape, maybe as a result of dehydration and shrinkage caused by the low-temperature storage process (6 months). The circularity values obtained (Sc) showed that the shape of the starch granules was modified by the thermal process. At the end of the storage period, they were significantly less circular (from 0.747 ± 0.041 to 0.684 ± 0.021). The protein structures seem to undergo a redistribution process (Fig. 3d and e) as thicker protein strands were observed; this fact has also been noticed in wheat dough and in part-baked bread (Naito *et al*., 2004; Bárcenas & Rosell, 2006). It might have been originated by the aggregation of protein structures caused by ice-crystal formation and by the interruption of the organised protein hydrogen bonding system that stabilises its structure (Meziani *et al*., 2011). Other studies (Autio & Sinda, 1992) have reported the loss of cross-linked polymers caused by freezing and longer thawing periods. This weakens the protein structures and separates the starch granules. Therefore, we suppose that these changes could contribute to the fragility of the cake crumb and could produce the fractures found in the crumb, thus modifying the sponge cake quality. This aggregation of protein was opposite from the findings of Baier-Schenk *et al.* (2005), who observed that the structure of thawed gluten is not altered after freeze/thaw cycles and that freezing-induced structural changes in gluten are reversible upon thawing. Apparently, this difference is a consequence of the formation of large ice crystals during frozen storage, which is independent of the freezing rate (Chen *et al.*, 2012).

Conclusions

This work showed that frozen storage affected the microscopic components (starch and protein structures) and macroscopic quality characteristics (quality and texture), with observations of these changes after 2 months of frozen storage. Microstructure damage, as measured by Sc, was significantly correlated ($P < 0.05$) with quality parameters. It seems that frozen storage favoured the re-crystallisation of sugar and that this was a factor contributing to the modification of the sponge cake texture and probably also to the re-arrangement of the starch. In this regard, more studies are needed that lead to the development of new cryo-protective materials or processes that could help to minimise the negative effect of frozen storage on baked cakes due to re-crystallisation, the goal being to increase the product shelf life.

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