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Effect of freezing on microstructure and degree of syneresis in differently formulated fruit fillings

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ABSTRACT

This study describes the syneresis and its effect on microstructure in fruit fillings within a wide range of the total soluble solids content and with or without hydrocolloids upon freezing. Linear models showed the relevance of the addition of pectin and gellan gum to fillings to prevent syneresis, increasing the water-holding capacity especially after freezing. Microstructural experiments by means of NMR spin–spin relaxometry combined with fluorescence microscopy allowed to observe that the continuous hydrocolloid gel, containing the dispersed solution of native fruit parts with the addition of inulin and sugars, changed its structure/distribution according to the amount of each ingredient and due to the freezing process. Relaxometry results confirmed that hydrocolloids strength was correlated ($R^2 > 0.92$) with water-holding capacity, due to a relationship between the signal given by the water chemically exchanging with biopolymers, and the changes in the degree of syneresis.

1. Introduction

Bakery products combined with various fruit fillings are currently among the fastest growing segments of the food industry, benefiting from high consumer demand for palatable fresh-fruity flavor and ready on-the-go snacks and meals. Bakery fruit fillings belong to a group of fruit preparations made from fruit-based raw material, sugar, water and stabilizing agents and are used as a filling component in bakery products such as pastries, cakes, doughnuts, etc (Wei, Wang, & Wu, 2001).

The fruit fillings designed for bakery products are expected to be stable not only at the high temperatures typical of baking processes, but also under different storage conditions, such as freezing. Thermal-instable fillings tend to degrade inside the pastry both upon baking and during frozen storage, giving rise to syneresis leading to the release of large amounts of water. The water strongly released may stain the dough surface (Agudelo, Varela, Sanz, & Fiszman, 2014), thus worsening the outer appearance of the finished product, making it inhomogeneous and partially sticky (Cropotova & Popel, 2013).

The water-holding capacity as well as melting behavior of fruit fillings depend on amount and type of added stabilizing ingredients. Many hydrocolloids increase thermal stability by acting on the melting and freezing points of fruit fillings, leading to the conservation of their initial structure at both high and low temperatures. Unfortunately, many studies investigating the quality parameters of fruit fillings have been mainly focused on color retention (Pratt, Sistrunk, & Morris, 1986; Sistrunk, Morris, & Gascoigne, 1982), bakery stability and rheological properties (Hill, Mitchell, & Sherman, 1995; Wei et al., 2001; Young, Kappel, & Bladt, 2003). The degree of syneresis of fruit fillings in terms of water-holding capacity of added hydrocolloids has seldom been mentioned (Agudelo et al., 2014). However, earlier studies revealed that some hydrocolloids possess low degree of syneresis, thus, they could be used in bakery fillings to deliver thermal stability to the final application (Young et al., 2003). A special interest is paid to gellan gum and pectin, because these hydrocolloids give an excellent thermal stability in various food applications, including fruit fillings (Cropotova, Popel, & Parshakova, 2013; Endreß, Kratz, & Kratz, 1992; May, 2000).

Historically, pectin was among the earliest hydrocolloid used to increase the thermal stability of jam and fillings (Endreß et al., 1992; May, 2000). Unfortunately, under mechanical stress pectin gel network may be damaged (Frey, 2005), leading to the release

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of colloidal water which can in turn boil during baking. This problem could be solved by integrating pectin with other stabilizers having thickening properties. In this respect, various hydrocolloid blends are of special interest for the food industry to provide a versatile range of novel functional characteristics. Gellan gum is tailored as a solution to syneresis problems because it can form a transparent gel in the presence of multivalent cations, and it is highly resistant to heat and acid (Sanderson, 1990; Sworn, 2000). The addition of inulin may also improve the quality properties of fruit fillings. From a technological point of view, the use of inulin is suitable for preventing syneresis in fruit fillings, because this ingredient possess good water-holding capacity, gelling and thickening properties (Dysseler & Hoffen, 1995) and may act as bulking agent (Tungland & Meyer, 2002). The ability of inulin to bind free water and form gels, investigated in a large number of studies (Evageliou, Tseliou, Mandala, & Komaitis, 2010; Zimeri & Kokini, 2002), presents a great interest for manufacturing thermal-stable fruit fillings.

From a structural point of view, gellan gum, inulin and pectin represent polysaccharides having linear conformations of various monosaccharides, which mainly determines their physicochemical properties (Dysseler & Hoffen, 1995; May, 2000; Sanderson, 1990). Thus, gellan gum is a tetrasaccharide, which consists of two residues of D-glucose and one of each residues of L-rhamnose and D-glucuronic acid (Sanderson, 1990). Pectin is a heteropolysaccharide made up of linear chains of α-(1 → 4)-linked D-galacturonic acid (May, 2000), while inulin represents a linear fructan consisting of β-(2 → 1) fructosyl-fructose linkages and α-D-glucose moieties (Dysseler & Hoffen, 1995).

In order to improve existing fruit fillings and develop new ones with thermal-stable properties, the water interaction with hydrocolloids at the molecular level is fundamental to be understood. Water–hydrocolloids interactions can be conveniently monitored by following the distribution of water across the biopolymers network. This can be done using light and fluorescence microscopy, which is a widely applied tool in food research investigating the structure of biopolymer blends (Blonk, van Eendenburg, Koning, Weisenborn, & Winkel, 1995; Nordmark & Ziegler, 2000). In addition, through proton transverse relaxation time weighted signals, as obtained by time domain nuclear magnetic resonance (TD-NMR), quantitative information about the water–hydrocolloids interactions can be achieved. This is possible because protons in different compartments of a sample are often characterized by different transverse relaxation times, modulated by the proton chemical exchange between water and biomolecules. Kim, Yoo, Cornillon, and Lim (2004) were able to separately observe water interacting with starch and pentosans from that interacting with gluten. Zhang, Matsukawa, and Watanabe (2004) estimated in water–carrageenan gels the T_2 of labile biopolymers protons by studying at different temperatures their chemical exchange with water. Panarese et al. (2012) studying kiwifruit tissue along ripening, by means of TD-NMR, were able to estimate the water located in the vacuoles, in the cytoplasm plus extracellular spaces and in the cell walls.

The goal of this study was to investigate the effect of freezing on water distribution in thermal-stable fruit fillings based on pectin–gellan gels with addition of inulin by means of fluorescence microscopy along with proton NMR relaxometry. The dynamic mobility of water molecules in fruit fillings with different formulations would be very useful data for manufacturers to develop or improve the quality of thermal-stable filling compositions, taking into account stability of hydrocolloid gel matrix and syneresis degree under different storage/processing conditions. Since the interactions between sugars, hydrocolloids and water highly affect the degree of syneresis, a better understanding of those phenomena could reduce the problem of water loss during storage.

2. Materials and methods

2.1. Raw material and ingredients

The ingredients used in the study for fruit fillings preparation were long-chain inulin Orafti HP (moisture content 5.0%, inulin content 100% d.m., DP ⩾ 23, Beneo, Belgium), low-methoxyl pectin GRINDSTED SF 580 (moisture content 12%, 38% degree of etherification, pH of 1% dissolution 3.8 to 4.2, Danisco, Danemark), low-acyl gellan gum KELCOGEL F (moisture content 10%, 2–3 × 10^5 Daltons m.w., acyl level 41%, gelling temperature 30–50 °C, CP Kelco, U.S.), citric acid (“EcoChimie” LTD, Chisinau, Republic of Moldova), homogenized apple puree (14 °Brix, “Orhei-Vit”, Republic of Moldova) and white sugar (moisture content 5.0%, sucrose content 99.85% d.m., JV “Südzucker Moldova”, Republic of Moldova).

2.2. Chemicals

The fluorescent dye Rhodamine B was purchased from Sigma–Aldrich (Steinheim, Germany).

2.3. Preparation of fruit fillings

Filling samples were prepared in laboratory conditions under atmospheric pressure according to the experimental plan, shown in Table 1, from apple puree, sugar, inulin, pectin, gellan gum and citric acid. According to Table 1, the following five experimental factors were investigated on two levels (2^5 factorial design) with added central point: amount of added inulin (4%, 6% and 8%), pectin (0.5%, 0.8% and 1.1%) and gellan gum (0%, 0.3% and 0.6%), fruit part (45%, 67.5% and 90% apple puree) and the total soluble solids content (30, 50 and 70 °Brix). Thus, the filling formulations were made up of 30, 50 and 70 °Brix as follows. First, apple puree was blended with different amount of sugar, stirred and boiled for five minutes. The amount of added sugar varied from 16.07% (w/w) to 55.05% (w/w) depending on the total soluble solids content of each apple filling established by experimental design plan displayed in Table 1. Inulin, pectin and gellan gum were dissolved in hot distilled water under continuous mixing and then simultaneously added in previously prepared apple filling mix. After homogenization under intense blending the solution of citric acid (50% w/w) was added in amount of 0.6%. The filling composition was heat evaporated to reach the required °Brix according to the planned experiment. The total soluble solids content was measured by an ABBE benchtop refractometer (precision ± 0.1 °Brix). The prepared fillings were put immediately into glass jars, sterilized and stored at room temperature (20 ± 2 °C) for 6 months. In order to verify the physical stability in terms of syneresis, products have been subjected to one extra month of storage at freezing conditions. This necessity is explained by the fact that fruit fillings stability at frozen state is quite relevant, because it represents their ability to retain the initial composition and integrity during storage (Agudelo et al., 2014). Afterwards, the analyses were carried out in three replicates before and after one month of frozen storage at −18 ± 1 °C.

Before analysis frozen samples were thawed at room temperature (20 ± 2 °C) for one hour and then accurately stirred. The twenty-seven combinations of five design factors according to the selected design of experiments type H_{A5} (Table 1) were run in triplicate. The values of the experimental factors were chosen in a base of preliminary experimental results. All experiments adjusted by the design planned in coded and encoded form of process variables, were conducted randomly.