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Characterization of ice recrystallization in ice cream during storage using the focused beam reflectance measurement

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ABSTRACT

Ice recrystallization was investigated in two commercial and differently stabilized ice creams using an original and real time particles counting and sizing method: the focused beam reflectance measurement (FBRM). Ice cream A (with locust bean gum – LBG – as primary stabilizer) and ice cream B (with carrageenan as primary stabilizer) were stored for 154 days at four different temperatures (−5, −8, −12 and −18 °C) and three amplitudes of temperature fluctuations (±0.1 °C, ±0.75 °C, ±2.5 °C). Crystal's size distributions were assessed at various time and recrystallization kinetic data were derived by fitting the experimental results to the asymptotic Ostwald ripening model. As expected, recrystallization rates increase with mean storage temperature and amplitude of temperatures fluctuations. Carrageenan seems to be more effective than LBG in slowing down ice crystal growth during storage. Mean ice crystal size increased as a function of time$^{1/3}$ for both ice creams. The temperature dependence of recrystallization rate fitted Arrhenius well, with activation energies fairly similar for ice creams A and B.

1. Introduction

Because of its predominance in food products, any changes in the physical state of water during distribution and storage throughout the cold chain cause important changes in foods stability, structure and texture. This is especially a problem in frozen foods where these modifications resulted in quality loss at the end of the cold chain due to recrystallization phenomena. Generally, ice recrystallization leads to the decrease in the total number of crystals and to an increase in the ice crystals size. In frozen desserts such as ice cream, the ice crystals can become so large that they can be detected in the mouth, inducing an unacceptable coarse and grainy taste and texture for the consumer. Ice recrystallization is promoted by fluctuating temperatures but can also occur at constant temperature during long-term storage, especially above the glass transition temperature (Donhowe and Hartel, 1996a, 1996b).

There are several recrystallization mechanisms but the process is always a result of a minimization of surface free energy of the entire crystal phase and of an equalization of the chemical potential among all phase (Hartel, 1998; Hagiwara et al., 2006). Among the recrystallization mechanisms, isomass, accretion and migratory have been identified to be predominant in the ice cream storage (Harper and Shoemaker, 1983; Donhowe and Hartel, 1996a). Isomass recrystallization occurs in single crystals and consists of a rounding of edges or internal structure reorganization without overall mass changes, involving sharper and smoother crystals. Accretion recrystallization describes the physical coalescence between two crystals in close contact in an unfrozen solution, resulting in one larger crystal. Migratory recrystallization (also called Ostwald ripening) is characterized by the growth of large crystals at the expense of the small ones due to the difference in equilibrium temperature caused by surface energy contributions. Water molecules at the surface of small crystals are not firmly bound because of the high curvature and thus, the high surface free energy. Consequently, these water molecules tend to diffuse through the freeze concentrated matrix and to deposit onto the surface of the larger ones, causing the growth of larger crystals and the disappearance of smaller ones.

Ice recrystallization in frozen foods has been widely studied. Martino and Zaritzky (1988, 1989) as Bevilacqua and Zaritzky (1982) studied ice recrystallization in frozen beef and developed a very simple ice crystal size growth model considering the average crystal curvature. Their works pointed out a limit diameter value independent of temperature and initial crystal size. A general asymptotic model developed by Lifshitz and Slyozov (1961) and Wagner (1961) (Pronk et al., 2005), also called Ostwald ripening equation (Eq. (1)) (Donhowe and Hartel, 1996a; Hartel, 2001) is generally used to describe the recrystallization process:

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Nomenclature

c	crystal chord length (m)
$d_{3,2}$	Sauter mean diameter (m)
k	recrystallization rate (m s^{-1})
$\overline{L}$	mean crystal size (m)
$\overline{L}_0$	initial mean crystal size (m)
n	power-law exponent in Eq. (1)
N	number of experimental points
$n_{i,0}$	unweighted counts number
$n_{i,p}$	weighted counts number at the exponent p
p	exponent in Eq. (2)
R	gas constant = 8.314
t	time (s)
T	temperature (K)
ΔT	amplitude of temperature fluctuation (K)

Subscripts

exp	experimental
i	size class number
th	theoretical

$$\overline{L}(t) = \overline{L}_0 + kt^{1/n} \quad (1)$$

where $\overline{L}$ is the mean crystal size at any time t, $\overline{L}_0$ the initial mean crystal size; k the rate of recrystallization and n a parameter that depends on the mechanism, which determines the recrystallization process.

Donhowe and Hartel (1996a, 1996b) studied the effect of the storage at constant and fluctuating temperatures on ice recrystallization in ice cream. They showed that ice recrystallization was more rapid under fluctuating temperature conditions than for constant temperature. The recrystallization rate increased with the increasing temperature and with the increasing extent of temperature fluctuation, and was well fitted by both the Arrhenius and the Williams–Landel–Ferry (WLF) equations. Using the Lifshitz–Slyozov–Wagner (LSW) model in Eq. (1), they reported that the mean size of ice crystals increased with time$^{1/3}$. This result was in agreement with those of several researchers who reported a crystals size increasing depending on time to a power between 1/3 and 1/2 in either food model systems (sugar or salt solutions) or in ice cream (Sutton et al., 1996; Ablett et al., 2002; Fernández et al., 2008).

The extent of recrystallization during the storage is also influenced by the formulation of the food system. Among the ingredients of ice cream, stabilizers are of special importance for recrystallization, despite their low concentrations in ice cream mix. Several researchers have investigated the effect of stabilizers on ice recrystallization rate in ice cream (Harper and Shoemaker, 1983; Hagiwara and Hartel, 1996; Miller-Livney and Hartel, 1997; Sutton and Wilcox, 1998a, 1998b; Flores and Goff, 1999a, 1999b; Goff et al., 1999; Bolliger et al., 2000; Patmore et al., 2003; Regand and Goff, 2003; Damodaran, 2007; Aleong et al., 2008; Soukoulis et al., 2008; Kurultay et al., 2010; Bahramparvar and Mazaheri Tehrani, 2011; BahramParvar et al., 2013). It was recognized that stabilizers do not significantly affect the initial crystal size distribution in ice cream at the scraped surface heat exchanger step, as well as they do not significantly affect ice growth during hardening, but that stabilizers have an important impact on ice recrystallization by reducing the rate of growth of ice crystals during storage and handling throughout the cold chain (Harper and Shoemaker, 1983; Caldwell et al., 1992a, 1992b; Ben-Yoseph and Hartel, 1998; Sutton and Wilcox, 1998a, 1998b; Flores and Goff, 1999a, 1999b). Despite of the various studies, there is no consensus on the mechanism by which the stabilizers limit or inhibit the ice recrystallization, may be due to the differences in type (specific characteristics and functionalities) and/or in the concentration of stabilizer (Bahramparvar and Mazaheri Tehrani, 2011). In ice cream industry, stabilizers are often used in blend because of specific characteristics of each of them and synergistic effects between them. Gums such as guar gum (GG), locust bean gum (LBG) and cellulose gum are frequently used as primary stabilizers and carrageenan as a secondary stabilizer to prevent wheying off of mix (Marshall et al., 2003). The ratios and concentrations as well as the kind of hydrocolloids used in the formulation blends determine the effect on recrystallization rates and the mechanisms involved. Some authors attributed the mechanism to an increased viscosity of the unfrozen phase slowing down water molecular mobility (Harper and Shoemaker, 1983; Caldwell et al., 1992a, 1992b; Goff et al., 1993; Hagiwara and Hartel, 1996; Miller-Livney and Hartel, 1997; Herrera et al., 2007; Hagiwara et al., 2009). Regand and Goff (2003) have studied gelling and non-gelling stabilizers in ice cream model systems. They concluded that steric hindrance and water holding capability are the main mechanisms by which gelling stabilizers retard water mobility, while non-gelling stabilizers reduce water diffusion to others crystals by an increase in microviscosity of the unfrozen phase. Rather than water mobility, Sutton and Wilcox (1998a, 1998b) have postulated that stabilizers act by weakly adsorbing to the growing ice crystal and thereby impeded the incorporation of further water molecules onto the surface, but they did not elucidate the mechanism of this obstruction. Recently, Damodaran (2007) suggested hydrogen bonding between stabilizers molecules (gelatine hydrolysate) and water molecules in the prism face of ice crystals as a possible mechanism of inhibition of ice recrystallization by protein stabilizers in ice cream. Whatever the mechanism, the stabilizers seem to prevent the free water molecules to swell the already formed crystals.

Most of the studies related to the measurement of the recrystallization rate (Hagiwara et al., 2006) by determining the ice crystals size distribution and ice crystals mean size deals with microscopy and image analysis (Donhowe et al., 1991; Sutton et al., 1994; Donhowe and Hartel, 1996a, 1996b; Regand and Goff, 2003; Hagiwara et al., 2006; Damodaran, 2007; Aleong et al., 2008; Fernández et al., 2008). This two steps procedure (microscopic visualization then image analysis) has proven its efficacy but the technique is cumbersome and time consuming. Thus, it can be useful to have a more automated measurement technique able to provide results faster. This can be done with the focused beam reflectance measurement (FBRM), a counting and sizing method widely used to carry out in-line characterization of particle processes as crystallization (Kougoulos et al., 2005a, 2005b; Kempkes et al., 2008; Czapla et al., 2010; Hishamuddin et al., 2011). However, its application in food processes is not very widespread and is still innovative (Haddad Amamou et al., 2010; Arellano et al., 2012; Erabit et al., 2013; Ndoye et al., 2013). The FBRM technology is based on light backscattering using a revolving laser beam. The latter is projected inside the studied suspension through a sapphire window at the end of the FBRM probe (Fig. 1). The focused beam rotates at a high rate of speed (about 2 m/s), so that particle seems to be immobile despite the stirring