

## Effect of Frozen Storage Temperature on the Quality of Premium Ice Cream

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### Abstract

The market sales of premium ice cream have paralleled the growth in consumer desire for rich flavor and taste. Storage temperature is a major consideration in preserving the quality attributes of premium ice cream products for both the manufacturer and retailers during prolonged storage. We investigated the effect of storage temperature (-18°C, -30°C, -50°C, and -70°C) and storage times, up to 52 wk, on the quality attributes of premium ice cream. Quality attributes tested included ice crystal size, air cell size, melting resistance, and color. Ice crystal size increased from 40.3 μm to 100.1 μm after 52 wk of storage at -18°C. When ice cream samples were stored at -50°C or -70°C, ice crystal size slightly increased from 40.3 μm to 57-58 μm. Initial air cell size increased from 37.1 μm to 87.7 μm after storage at -18°C for 52 wk. However, for storage temperatures of -50°C and -70°C, air cell size increased only slightly from 37.1 μm to 46-47 μm. Low storage temperature (-50°C and -70°C) resulted in better melt resistance and minimized color changes in comparison to high temperature storage (-18°C and -30°C). In our study, quality changes in premium ice cream were gradually minimized according to decrease in storage temperature up to -50°C. No significant beneficial effect of -70°C storage was found in quality attributes. In the scope of our experiment, we recommend a storage temperature of -50°C to preserve the quality attributes of premium ice cream.

**Keywords:** ice cream, storage temperature, ice cream qualities, crystal size, air cell size

*Received May 6, 2015; Revised August 28, 2015; Accepted October 2, 2015*

### Introduction

In recent years, market sales of premium ice cream have paralleled the growth in consumer desire for rich flavor and taste. Ice cream is an aerated suspension of crystallized fat and water in a highly concentrated sugar solution; it contains hydrocolloids, casein micelles, and proteins (Eisner, 2005). The grade of ice cream depends on the amount of milk fat content. Generally, ice creams containing more than 12% are recognized as premium level (Choi and Shin, 2014). Fat content in premium ice cream generally ranges from 11% to 15% and the ice cream is 60% to 90% overrun (Goff, 2008). Overrun refers to the amount of air pushed into the ice cream.

Premium ice cream tends to be more overrun, which produces a denser, heavier, and creamier structure, and induces richer mouth feel than regular ice cream. The quality attributes of ice cream depend on various factors, including ice crystal size, overrun, initial freezing temperature, and storage temperature. Among these, storage temperature is of particular interest to ice cream manufacturers and retail stores, because the physical characteristics of ice cream associated with mouth feel are greatly influenced by storage temperature (Buyck *et al.*, 2011; Choi and Shin, 2014; Park *et al.*, 2006). In general, the industry standard for ice cream storage is -28.9°C (Buyck *et al.*, 2011). The International Dairy Foods Association has stated that any ice cream product stored at higher than -28.9°C has incurred heat-shock damage and should not be sold via retail (Buyck *et al.*, 2011; International Dairy Foods Association, 1997). The final quality of the ice cream product, particularly the smooth texture and the cooling sensation perceived by the consumers, is largely

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influenced by the size and morphology of the ice crystals and air bubbles (Caillet *et al.*, 2003). Consumers desire small ice crystals ( $< 55 \mu\text{m}$ ), as they perceive the product to be less icy and sandy in texture (Buyck *et al.*, 2011). Maintaining a uniform distribution of small ice crystals is critical for obtaining a smooth texture and mouthfeel during consumption (Flores and Goff, 1999). Ice creams with small ice crystals experience minimal recrystallization during frozen storage (Choi and Shin, 2014; Trgo *et al.*, 1999). Recrystallization is a result of storage temperature fluctuations that cause some ice crystals to melt and recrystallize freely into larger crystals (Buyck *et al.*, 2011). Appropriate air cell structure in ice cream promotes a light texture and influences melt down (Park *et al.*, 2006). Air cell diameters ranging from 30 to 150  $\mu\text{m}$ , with a mean diameter of 40  $\mu\text{m}$ , is considered as the criteria for good quality ice cream (Caillet *et al.*, 2003; Hagiwara and Hartel, 1996). Air cells tend to coalesce into larger cells at storage temperatures higher than  $-25^\circ\text{C}$ , resulting in coarsening of the foam structure (Sofjan and Hartel, 2004). In general, storage at temperatures below  $-30^\circ\text{C}$  is considered deep frozen storage and lower temperature storage could minimize the quality degradation of ice cream products. However, excessively low temperature storage is not desirable in the energy consumption and storage costs for manufacturers and retailers. This study was carried out to investigate the effect of storage temperature ( $-18^\circ\text{C}$ ,  $-30^\circ\text{C}$ ,  $-50^\circ\text{C}$ , and  $-70^\circ\text{C}$ ) and storage time (up to 52 wk) on the quality attributes (ice crystal size, air cell size, melt resistance, color) of premium ice cream.

## Materials and Methods

### Sample preparation

In this study, premium vanilla ice cream manufactured from NTR company was selected. Commercial market products were sampled (130 mL) and tested. The samples were directly purchased from the manufacturer to avoid variations among retail stores. The major ingredients of tested ice creams were cream concentrate, skim milk, sugar, egg yolk, and vanilla flavor, with fat content near 12%. All quality attributes were immediately analyzed after purchasing the ice cream (0 wk storage, control).

### Frozen storage

Four different frozen storage temperatures were selected including  $-18^\circ\text{C}$ ,  $-30^\circ\text{C}$ ,  $-50^\circ\text{C}$ , and  $-70^\circ\text{C}$ . In general, storage at temperatures below  $-30^\circ\text{C}$  is considered deep frozen storage. Each quality attribute was analyzed at 4, 8, 12, 16, 28, 40, and 50 wk. Three ice cream products were prepared for each storage temperature and storage time. Control was day 0 sample after direct purchase from retail store.

### Quality attributes

#### Ice crystal size measurement

Ice crystal size was measured using a low temperature stereo microscope (CX40; Olympus, Japan) and organic solvent extraction methods (Fig. 1) described by Min and Lee (1997). The ice crystal measurement system consisted

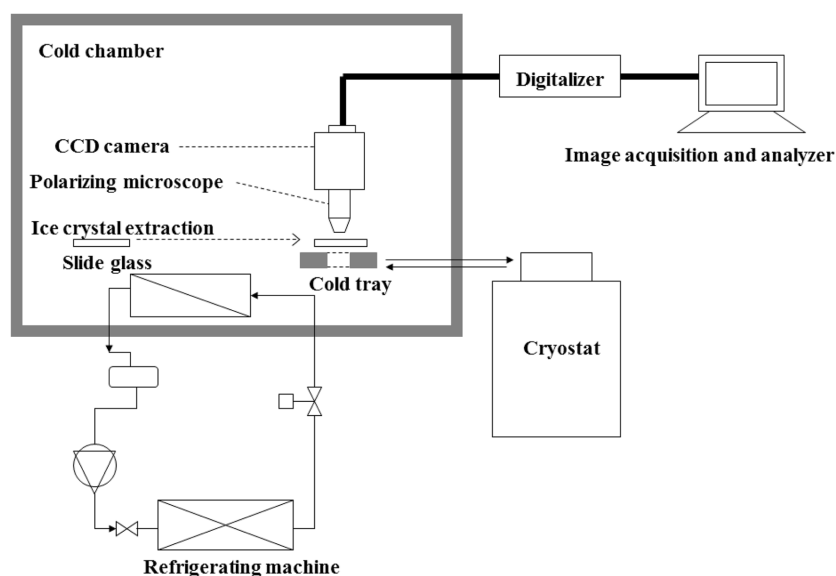


Fig. 1. Schematic diagram of the ice crystal and air cell size measurement unit in the cold chamber.

of a cryopolarization microscope (CX40, Olympus, Japan) and CCD camera (VC45CSHR-12, Olympus, Japan) housed in a cold chamber at  $-50^{\circ}\text{C}$ . This set-up prevented ice crystal melting during observation. Approximately 0.5 g of ice cream was placed on cold-set slide glass at  $-50^{\circ}\text{C}$ . Ice crystals were removed from the ice cream matrix using an organic solvent mixture (hexane:kerosene, 0.1:0.9, v:v). The organic solvent mixture was precooled to  $-50^{\circ}\text{C}$  to prevent ice crystal melting before analysis. Separated ice crystals were then placed on a cold tray coupled to a cryostat at  $-30^{\circ}\text{C}$  (RBC-11, JEIO TECH, Korea). Ice crystal size was analyzed with an image analysis program (Image Tool 3.0; UTHSCSA, USA). More than 100 ice crystal images at each experimental condition were acquired for statistical analysis.

#### Air cell size measurement

Air cell size was measured at  $0\text{--}5^{\circ}\text{C}$  using an optical microscope (ATC 2000, Leica, Switzerland) coupled with a CCD camera (VC45CSHR-12, Olympus, Japan). Approximately 0.5 g of ice cream was loaded on the slide glass and then slightly depressed. Air cell size was measured as the longest axis using an image analysis program (Image Tool 3.0, UTHSCSA, USA). More than 100 ice crystal images at each experimental condition were acquired for statistical analysis.

#### Melting resistance

Samples were moved from storage at  $-18^{\circ}\text{C}$ ,  $-30^{\circ}\text{C}$ ,  $-50^{\circ}\text{C}$ , or  $-70^{\circ}\text{C}$  to a  $-30^{\circ}\text{C}$  freezer 24 h before melting analysis for uniform control of the initial temperature. Ice cream samples (130 g) were placed on a mesh grid ( $10 \times 10$  mm) and placed in an incubator at  $25^{\circ}\text{C}$ , with a relative humidity of 55%. Molten ice cream dripped into sample trays poised on a balance for weight determination. The weight of molted ice cream was recorded every 1 min. Melting curves were analyzed to determine the melting resistance.

#### Color measurement

Ice cream color was measured using a portable colorimeter (CR 400, Minolta Co., Japan). The color meter was calibrated with a white standard plate ( $L^* = 97.83$ ,  $a^* = 0.43$ ,  $b^* = 1.98$ ). CIELAB  $L^*$  indicates the lightness. CIELAB  $a^*$  and  $b^*$  indicates for the color-opponent dimensions, based on nonlinearly compressed (e.g., CIE XYZ color space) coordinates. The lightness,  $L^*$ , represents the darkest black at  $L^* = 0$ , and the brightest white at  $L^* = 100$ . The color channels,  $a^*$  and  $b^*$ , will represent true

neutral gray values at  $a^* = 0$  and  $b^* = 0$ . The red/green opponent colors are represented along the  $a^*$  axis, with green at negative  $a^*$  values and red at positive  $a^*$  values. The yellow/blue opponent colors are represented along the  $b^*$  axis, with blue at negative  $b^*$  values and yellow at positive  $b^*$  values.

#### Statistical analysis

The data was analyzed using SAS 9.1.3 (SAS Inst. Inc., USA). Fisher's least-significant difference (LSD) procedure was used for multiple comparisons among treatments at the 95% confidence interval ( $p < 0.05$ ). Multiple comparisons were conducted among different storage temperatures at the same storage period, since this study focused on storage temperature effects. To evaluate the ice crystal size changes ( $Y$ ) as a function of storage period, ice crystal size as a function of storage time ( $x$ ) was empirically fitted using logarithmic regression as follows:

$$Y = \beta_0 + \beta_1 \times \ln(x) \quad (1)$$

## Results and Discussion

### Ice crystal size

Ice crystal sizes for ice cream samples were measured as a function of storage temperature ( $-18^{\circ}\text{C}$ ,  $-30^{\circ}\text{C}$ ,  $-50^{\circ}\text{C}$ , and,  $-70^{\circ}\text{C}$ ) and time (0–52 wk). Ice crystal size gradually increased with prolonged storage time (Fig. 2). Low temperatures ( $-70^{\circ}\text{C}$  and  $-50^{\circ}\text{C}$ ) minimized the growth of ice crystals in comparison to high temperatures ( $-30^{\circ}\text{C}$  and  $-18^{\circ}\text{C}$ ). For control ice cream, initial ice crystal size was  $40.3 \pm 3.4 \mu\text{m}$ , and after 52 wk of storage at  $-18^{\circ}\text{C}$ , ice crystal size increased to  $100.1 \pm 11.9 \mu\text{m}$ . For ice cream stored for 52 wk at  $-70^{\circ}\text{C}$ , ice crystal size increased only

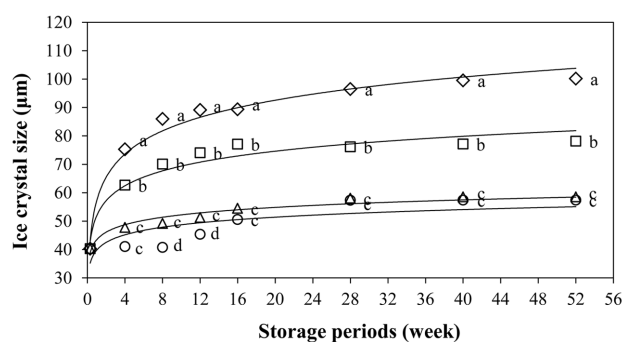
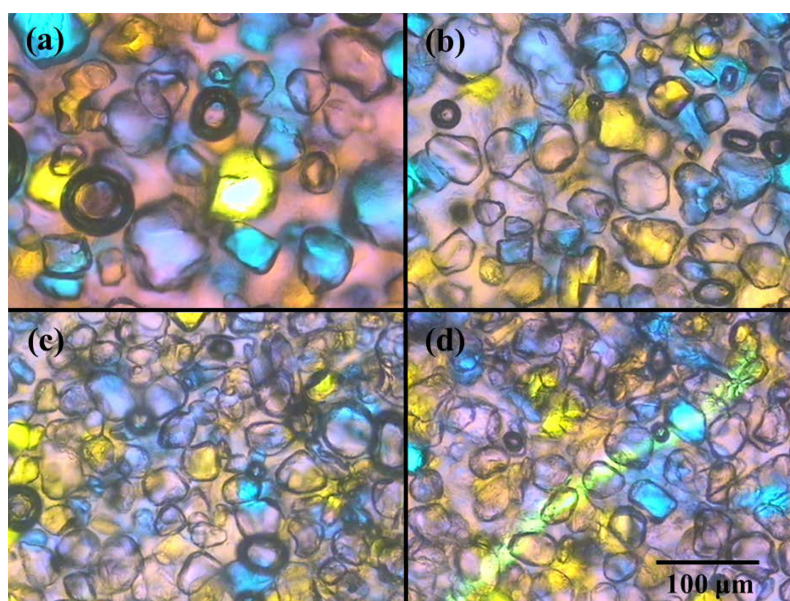


Fig. 2. Changes in ice crystal size at different storage temperatures ( $\diamond$ :  $-18^{\circ}\text{C}$ ,  $\square$ :  $-30^{\circ}\text{C}$ ,  $\triangle$ :  $-50^{\circ}\text{C}$ ,  $\circ$ :  $-70^{\circ}\text{C}$ ) and periods (0–52 wk). <sup>a-d</sup> Values with different letters within the same storage period are significantly different ( $p < 0.05$ ).



**Fig. 3.** Microscopic images of ice crystals at different storage temperatures: (a)  $-18^{\circ}\text{C}$ , (b)  $-30^{\circ}\text{C}$ , (c)  $-50^{\circ}\text{C}$ , and (d)  $-70^{\circ}\text{C}$  after 52 wk of storage.

slightly from  $40.3 \pm 3.4 \mu\text{m}$  to  $57.4 \pm 3.4 \mu\text{m}$ . There was no significant difference in ice crystal size for storage temperatures of  $-50^{\circ}\text{C}$  and  $-70^{\circ}\text{C}$  after 12 wk of storage ( $p > 0.05$ ). Our results are consistent with a previous report that indicated that low storage temperature ( $-45.6^{\circ}\text{C}$ ) produced smaller mean ice crystal size than the  $-23.2^{\circ}\text{C}$  or  $-26.1^{\circ}\text{C}$  storage conditions (Buyck *et al.*, 2011; Cook and Hartel, 2010). Low temperature stereo microscopic images of ice crystals were acquired for ice cream stored at  $-18^{\circ}\text{C}$ ,  $30^{\circ}\text{C}$ ,  $-50^{\circ}\text{C}$ , and  $-70^{\circ}\text{C}$  for 52 wk. The largest ice crystal size was observed at  $-18^{\circ}\text{C}$ , followed by that at  $-30^{\circ}\text{C}$ ,  $-50^{\circ}\text{C}$ , and  $-70^{\circ}\text{C}$  (Fig. 3). Smaller ice crystals ( $< 55\text{--}60 \mu\text{m}$ ) in ice cream are more desirable to consumers, as the product is perceived to be less icy and sandy in texture (Buyck *et al.*, 2011). The growth of ice crystals during frozen storage originates in the ice recrystallization stage. Ice recrystallization, also called ripening or coarsening, is the final step of crystallization and produces changes in crystal shape and size as a function of heat- and mass-transfer (Cook and Hartel, 2010). In our study, ice cream stored for up to 52 wk at deep frozen storage temperatures of  $-70^{\circ}\text{C}$  to  $-50^{\circ}\text{C}$  experienced minimal ice recrystallization. Low frozen storage temperatures suppress the molecular mobility and vapor pressure of ice crystals, inhibiting ice recrystallization. The fit of an empirical model to ice crystal size versus storage time are summarized in Table 1. The slope of the fit suggests that ice crystallization and size increase rapidly with increas-

**Table 1.** Estimated coefficients for logarithmic regression of the relationship between ice crystal size ( $Y$ ) and storage period ( $x$ )

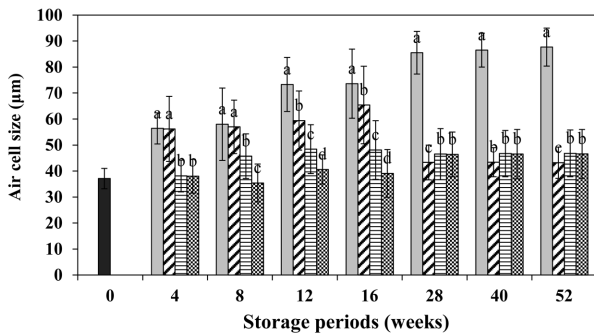
	Slope ( $\beta_1$ )	Intercept ( $\beta_0$ )	$R^2$
$-18^{\circ}\text{C}$	11.76	57.32	0.98
$-30^{\circ}\text{C}$	7.56	51.98	0.95
$-50^{\circ}\text{C}$	3.79	43.50	0.95
$-70^{\circ}\text{C}$	3.85	39.85	0.66

ing temperature for high temperatures ( $-18^{\circ}\text{C}$  to  $-30^{\circ}\text{C}$ ). However, there were no significant difference in ice recrystallization between  $-50$  and  $-70^{\circ}\text{C}$  storage, meaning that, in terms of ice crystal formation, there is no need to store ice cream below  $-50^{\circ}\text{C}$ .

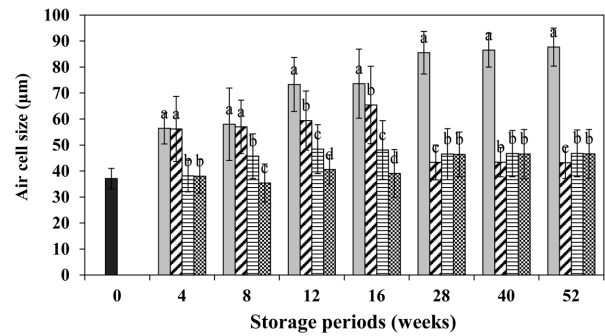
#### Air cell size

Air cell size was measured as a function of storage temperature ( $-18^{\circ}\text{C}$ ,  $-30^{\circ}\text{C}$ ,  $-50^{\circ}\text{C}$ , and  $-70^{\circ}\text{C}$ ) and time (0–52 wk). Initial air cell size in the ice cream matrix was  $37.1 \pm 3.9 \mu\text{m}$ , and then gradually increased to  $87.7 \pm 7.3 \mu\text{m}$  when stored at  $-18^{\circ}\text{C}$  for 52 wk (Fig. 4). Air cell size was  $65.4 \pm 14.9 \mu\text{m}$  when stored at 16 weeks at  $-30^{\circ}\text{C}$ , and then decreased with further increase in storage time. For the deep frozen storage temperatures ( $-50^{\circ}\text{C}$  and  $-70^{\circ}\text{C}$ ), no drastic increase in air cell size was observed; air cell size increased from  $37.1 \mu\text{m}$  to  $46\text{--}47 \mu\text{m}$  after storage at  $-50^{\circ}\text{C}$  or  $-70^{\circ}\text{C}$ .

Creaminess is related to the distribution of air cells in



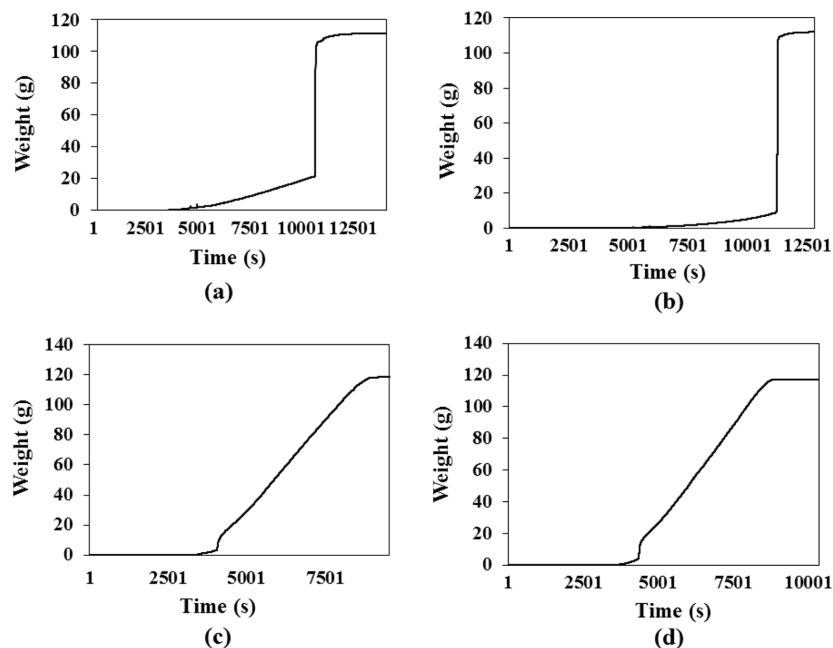
**Fig. 4.** Changes in air cell size with different storage temperatures (□: -18°C, ▨: -30°C, ■: -50°C, ▩: -70°C) and time periods (0-52 wk). <sup>a-d</sup>Values with different letters within the same storage period are significantly different ( $p < 0.05$ ).



**Fig. 5.** Changes in melting time for different storage temperatures (□: -18°C, ▨: -30°C, ■: -50°C, ▩: -70°C) and periods (0-52 wk). <sup>a-d</sup>Values with different letters within the same storage period are significantly different ( $p < 0.05$ ).

the ice cream matrix and its overrun. In general, a homogenous distribution of small air cells in the ice cream matrix is desirable since small and dispersed air cells produce a stable foam and creamier mouthfeel (Eisner *et al.*, 2005). Coalescence of air cells leads to the loss of two small bubbles and the formation of a single large bubble, which causes an increase in air cell size (Chang and Hartel, 2002). Smaller air cells with a narrow size distribution improves the rheological properties of ice cream and has a positive influence on creaminess and mouthfeel as the melting ice cream is being consumed (Eisner *et al.*, 2005; Hanselmann and Windhab, 1998). Air cells are in

close proximity and coalescence can be an important mechanism that leads to coarsening of the air cells (Chang and Hartel, 2002). We found that changes in air cell size and ice crystal size produced similar trends with respect to storage temperature and time. Similar associations of small ice crystal size with narrow air cell distributions have been previously reported (Flores and Goff, 1999; Park *et al.*, 2006; Sofjan and Hartel, 2004). We found that ice cream storage at low temperatures (-50°C and -70°C) minimized changes in air cell size, probably as a result of suppressed vapor pressure and decreased mass- and heat-transfer.



**Fig. 6.** Melting curves of ice cream as a function of time: (a) -18°C, (b) -30°C, (c) -50°C, and (d) -70°C after 52 wk of storage.

### Melt resistance

We characterized ice cream melting resistance by measuring the amount of time required for melting. Overall, the time required for melting increased with an increase in storage time for  $-18^{\circ}\text{C}$  and  $-30^{\circ}\text{C}$ ; however, no clear trends were observed for storage temperatures of  $-50^{\circ}\text{C}$  and  $-70^{\circ}\text{C}$  (Fig. 5). Ice cream melting was evaluated by measuring the weight of molten ice cream as a function of time. The ice cream sample stored at  $-18^{\circ}\text{C}$  gradually melted up to about 20 g at 3 h, and then melted drastically, with a nearly vertical melting curve. A similar trend was observed for the sample stored at  $-30^{\circ}\text{C}$ , but not for the samples stored at  $-50^{\circ}\text{C}$  or  $-70^{\circ}\text{C}$ , which melted continuously (Fig. 6). The melting resistance of ice cream is affected by many factors, including the amount of air incorporated, the nature of the ice crystals, and the network of fat globules formed during freezing (Bolliger *et al.* 2000; Muse and Hartel, 2004). A uniform distribution of small ice crystals and air cells results in gradual melting, a phenomenon that can be attributed to a reduced rate of heat transfer, since the trapped air functions as thermal resistance layers (Muse and Hartel, 2004; Sakurai *et al.*, 1996). We found that a homogenous distribution of small ice crystals and air cells is associated with melting resistance.

### Color

The color of frozen and molten ice cream was assessed using colorimetry. Since  $a^*$  and  $b^*$  values were not significantly influenced by storage temperature and time, only the  $L^*$  value was reported in Fig. 7. The  $L^*$  value, which indicates whiteness, is one of the most important parameters for vanilla ice cream since consumers prefer lighter ice cream colors with enhanced whiteness. The

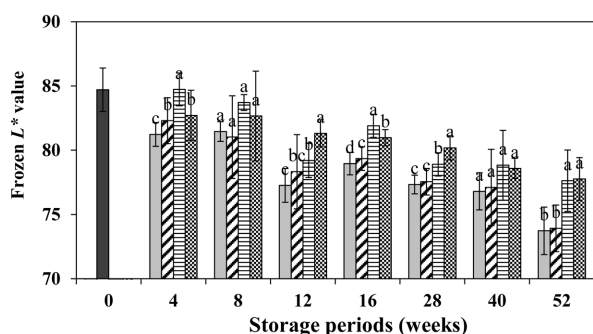


Fig. 7. Changes in  $L^*$  values for different storage temperatures (□:  $-18^{\circ}\text{C}$ , ▤:  $-30^{\circ}\text{C}$ , ▥:  $-50^{\circ}\text{C}$ , ▦:  $-70^{\circ}\text{C}$ ). <sup>a-d</sup>Values with different letters within the same storage period are significantly different ( $p < 0.05$ ).

control ice cream produced an  $L^*$  value of  $85.2 \pm 2.2$ , which decreased with prolonged storage times at  $-18^{\circ}\text{C}$  or  $-30^{\circ}\text{C}$ , but not for temperatures of  $-50^{\circ}\text{C}$  or  $-70^{\circ}\text{C}$ , for which  $L^*$  did not change significantly ( $p < 0.05$ ). The whiteness of dairy products is influenced by several factors that include light scattering by fat and protein particles (Metzger *et al.*, 2000; Rudan *et al.*, 1999). In our study, low temperature storage at  $-50^{\circ}\text{C}$  or  $-70^{\circ}\text{C}$  delayed changes in  $L^*$  values of ice cream with minimization of milk fat and protein denaturation.

### Acknowledgements

This work was carried out with the support of the “Cooperative Research Program for Agriculture Science & Technology Development (Project No. PJ009440)” Rural Development Administration, Republic of Korea.

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