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The Influence of Food Hydrocolloids on Changes in the Physical Properties of Ice Cream

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Abstract This study was carried out to investigate the effect of hydrocolloids on the changes in physical properties of a model ice cream. The model ice cream contained water, sugar, skin milk powder, corn oil, and 4 different hydrocolloid stabilizers (gelatin, pectin, hydroxyethylstarch, locust bean gum), was manufactured in a batch type freezer. The following physical characteristics of ice cream were examined: flow behavior, overrun, air cell size, ice crystal size, and melt resistance. With regard to flow behavior, all of aged mixes had a lower apparent viscosity relative to the mix before aging, and ice cream mix containing locust bean gum had the highest viscosity. Air cell size was observed to range from 20 to 38 μm, and ice cream with locust bean gum showed the largest size. There was an inverse correlation between overrun and air cell size. The ice crystal sizes of all samples ranged from 25 to 35 μm. Ice cream with added pectin contained the smallest ice crystal size, which was significantly difference from other stabilizers ($p<0.05$), and resulted in superior melt resistance with increased melting time compared to other samples.

Keywords: ice cream, stabilizer, flow behavior, ice crystal, air bubble

Introduction

Ice cream is a complex food colloid system containing fat globules, air cells and ice crystals dispersed in a freeze-concentrated dispersion/solution of proteins, salts, food hydrocolloids, and sugars (1). One of the most important goals of ice cream manufacturers is to produce a product with a small ice crystal size distribution that results in a smooth texture until consumption. In order to obtain a small ice crystal size, various technological parameters can be manipulated such as freezing rate, storage temperature, and the addition of stabilizers that function of cryo-protectants.

In this view, some stabilizers are required in ice cream production to enhance qualitative and sensorial aspects. Hydrocolloids can retard the growth of ice and lactose crystals during storage, especially when subjected to temperature fluctuation (2, 3). The functionality of a given stabilizer may be enhanced as the polymer concentration increases, but different stabilizers are not equally effective at retarding ice crystal growth at the same concentration levels and viscosity (4). Air in ice cream provides a light texture and influences the physical properties of melt down and hardness. However, it is not just the amount of air incorporation or overrun, but also the distribution of air cell size that influences these parameters (5).

Freezing rate is recognized as an important parameter for the melt resistance of ice cream. Proper control of initial freezing also results in maximum shelf life, because ice crystals remain below the threshold size for the longest time (6). In general, the crystal structure formed during the freezing process changes, affecting the coarseness of the product and reducing the number of crystals during storage and transportation of the products. These changes are due to differences in the product matrix and radii of the curvature of individual crystals (7-9). Proper control of ice crystal formation in the ice cream freezer results in a large number of small ice crystals with a smooth texture and good storage stability, and a lower drawing temperature results in smaller ice crystals (9).

In this study, four different commonly used hydrocolloid stabilizers, gelatin, pectin, locust bean gum (LBG), and hydroxyethylstarch (HES), were used in ice cream mix formulation. Gelatin, commercially derived from collagen, owes its importance to its unique rheological characteristic in creating the 'melt in the mouth' texture, type of a gelatin gel (9, 10). Starch is mainly used as a gelling system and includes maize, wheat and potato starches which can contain between 14 and 27% amylase and 73-86% amylopectin (11). Pectin is obtained from citrus fruits and consists of a linear chain of α-(1-4)-linked D-galacturonic acid units, in which varying proportions of the acid groups are present as methyl esters (12). LBG, a plant seed galactomannan, is composed of a 1-4-linked β-D-mannan backbone with 1-6-linked α-D-galactose side groups (13, 14) and has a wide variety of food and industrial applications due to its capacity to form very viscous solutions at a relatively low concentration (15, 16). Starches with differing hydrolytic rates have different nutritive values and physiological properties (17).

In this study, HES was investigated with regard to its properties in ice cream production. There have been few studies examining HES as an ice cream stabilizer. In the pharmaceutical industry, HES is a more slowly degraded starch derivation, widely used for therapy and prophylaxis of all kinds of volume (18). Levy and Andry (19) previously showed that HES leads to more stable micro-particles by interfacial cross-linking.

This study was conducted to elucidate the influence of each hydrocolloid stabilizer on the physical characteristics

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of ice cream with regard to ice crystal formation, air cell size, overrun, melt resistance, and flow behavior.

Materials and Methods

Preparation of model ice cream mixes Model ice cream mixes were prepared using the formulation described in Table 1. Four kinds of stabilizer were added to model ice cream mixes: gelatin (GR reagent, Ducksan Co., Seoul, Korea), pectin (Fluka, Buchs, Switzerland), HES (Myeongshin Co., Seoul, Korea) and LBG (Myeongshin Co.). Each mix was blended, pasteurized at 65°C for 30 min, homogenized at 9,100 rpm (X-103CD; Jeiotech, Gyeonggi, Korea) and cooled to 4°C. The mix was then aged in a screw blade agitator (TOPS; Misung Co., Seoul, Korea) at 4 for 14 hr. Aged mixes were transferred to a batch freezer (F-101; Bosung, Gyeonggi, Korea) and then first frozen to the exhausting temperature of -6.7°C. After the first freezing step, the mixes were hardened to a core temperature of -25°C in an air blast freezing unit (DF-40; Daesung, Seoul, Korea) at -40°C.

Table 1. Formulation of ice cream mix

Component	Mixing ratio (%)
Corn oil[1)]	10
Skim milk powder[2)]	12
Sugar	13
Emulsifier[3)]	0.1
Stabilizer	0.3
Water	64.6
Vanilla flavor[4)]	0.003

[1)]Jeiljedang Co., Seoul, Korea.
[2)]Meail dairy industry, Pyeongtak, Gyeonggi, Korea.
[3)]Tween 20, Daejung Co., Incheon, Gyeonggi, Korea.
[4)]Daichun Co., Yongin, Gyeonggi, Korea.

Flow behavior Flow behavior was measured with a rotational viscometer (VISCO STAR-L; J.P. Selecta S.A., Abrela, Spain) at 4°C. Spindle 1 (Ø18×65) was rotated at a shear rate of 24.43/sec during measurement.

Overrun Overrun was calculated as the weight ratio between the initially blended model mix and the aged ice cream (Eq. 1) as determined previously by Arbuckle (20). From each sample, 163 mL was used for the overrun test.

$$\text{Overrun (\%)} = \left(\frac{W_m \cdot V_i}{W_i \cdot V_m} - 1\right) \times 100 \qquad \text{(Eq. 1)}$$

W_m: weight of model mix, V_i: volume of model mix
W_i: weight of aged ice cream, V_i: volume of aged ice cream

Ice crystal and air cell size measurement Ice crystal size was measured by an organic solvent extraction method proposed by Min and Lee (21). Approximately 0.5 g of ice cream was placed on a pre-cooled slide glass (-50°C). Ice crystals were separated from ice cream using a mixture of hexane and kerosene (10:90, v/v) pre-cooled in a deep freezer before use. The ice crystal temperature was maintained constant by using a deep frozen box and cold tray connected to a cryostat (RBC-11; JEIO Tech, Seoul, Korea). The observation system (Fig. 1) consisted of cryopolarization microscope (CX40; Olympus, Tokyo, Japan) and CCD camera (VC45CSHR-12; Olympus) which were located in the cold chamber (-50°C) in order to prevent the melting of ice crystals during measurement. Digitalized images of ice crystals were transmitted to an external data logging system. Air cell size was measured at 0-5°C with an optical microscopy system (ATC 2000; Leica, St. Gallen, Switzerland) combined with a CCD camera (VC45CSHR-12; Olympus). Approximately 0.5 g of ice cream was placed between the cover and slide glass and then pressed slightly. The long axis, radius of each ice crystal, and air cell size were analyzed with the image analysis program (Image Tool 3.0; UTHSCSA, Austin,

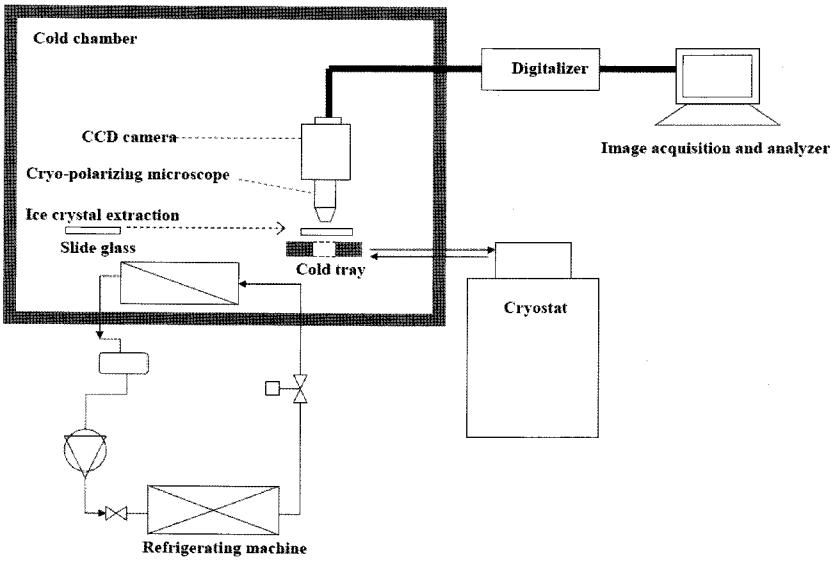

Fig. 1. Schematic diagram of the ice crystal and air cell size measuring and analysis system.

TX, USA). For statistical analysis, more than 100 ice crystals and air cells were analyzed.

Melt resistance measurement Samples were stored at -35 °C for 24 hr before each melting test. Melt resistance was conducted in an incubator (D-6450; Heraeus, Seelbach, Germany) maintaining a constant temperature of 25°C and relative humidity of 55%. Ice cream sample of 130 g was placed on a mesh grid (mesh size 10×10 mm). Melted sample passed through the grid and was collected on the sample tray of a chemical balance (GF-200; AMD, Phoenix, AZ, USA). The weight of melted sample was recorded as a function of time at 1 min intervals. Initial melting time (IMT) and total melting time (TMT) were measured. IMT was defined as the time needed to drop the first portion of melted ice cream mix, and TMT was determined as the time required to drop 80 g of melted product.

Statistical analysis The data obtained from three replications were analyzed by ANOVA using the SAS statistical program (SAS Institute, Cary, NC, USA), and differences among the means were compared using Duncan's multiple range test.

Results and Discussion

Flow behavior of model mixes The flow behavior of each non-frozen model mix is shown in Fig. 2. The aged mixes had lower viscosities than the mixes before aging. The addition of locust bean gum (LBG) resulted in the highest pre-aging viscosity, with a value of 746.70 cP followed by, in decreasing order, pectin, HES, and gelatin. The same trend was seen for the aged samples. The differences in viscosity are likely due to the molecular structure of the hydrocolloids. Min *et al.* (7) reported the highest viscosity with the use of LBG compared to hydrocolloid matrixes of Na-alginate, carboxymethyl cellulose, gelatin, κ-carrageenan, and pectin in frozen food systems. LBG is a non-gelling galactomannan composed of a 1-4-linked β-D-mannan backbone with 1-6-linked α-D-galactose side groups (13, 14) that produces high viscosity solutions due to the simple entanglement of polysaccharide

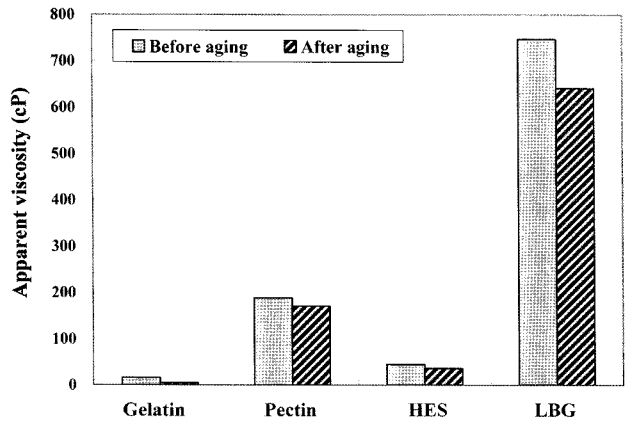

Fig. 2. Influence of added stabilizers on the apparent viscosity of ice cream mixes before and after aging.

chains (22). In contrast, the addition of gelatin resulted in the lowest viscosity, and also caused Newtonian flow behavior which is different from other ice cream mixes that showed pseudoplastic flow behavior. Similar results were reported in the work of Marcotte *et al.* (10), which showed linearity between shear rate and shear stress. Min *et al.* (7) reported a low apparent viscosity of the gelatin containing matrix, indicating the impossibility of a shear gradient.

The trend of aged mixes having a lower viscosity than samples before aging might be due to the induction of air cells and foaming in the model mix during aging. This decreased viscosity due to aging would positively affect the mixes texture and improve its appeal to consumers. It is postulated that a certain level of viscosity is essential for proper whipping and air retention (23, 24). Foam formation can provide a range of unique textures associated with many foods including cake, bread, ice cream, and confectionary products (25). Viscosity is considered an important characteristic of ice cream, since it frequently accompanies a desirable body and texture. The viscosity values of an ice cream mix are principally influenced by the fat and stabilizers it contains (23, 24).

In this view, the influence of stabilizer on viscosity should be carefully considered in the relation to the recrystallization of ice and the stability of air cells in the manufacturing of ice cream.

Overrun and air cell size The influence of each stabilizer on overrun and air cell size is shown in Fig. 3. The mean air cell size associated with each stabilizer ranged from 20 to 40 μm, in accordance with an air cell size range of 20-50 μm generally observed in ice creams (26). Figure 4 shows the circlet air cell shape in ice cream supplemented with different stabilizers and the discrete changes in size due to the different stabilizers.

There was an inverse correlation between overrun and air cell size, since increased overrun resulted in smaller air cell size. The highest overrun of 22.90% was observed in the gelatin supplemented ice cream which also had the smallest air cell size, 20.02 μm. In contrast, LBG resulted in a low overrun of 16.56% and the largest air cell size, 37.61 μm. The cumulative frequency of air cell sizes (Fig. 5) shows a typical sigmoid shape, and similar trends were observed in the research of Wildmoser *et al.* (27). The air cell diameter at 50% of the cumulative frequency, X_{a50}, was observed to be 31.3, 21.3, 20.4, and 19.9 μm in LBG, pectin, HES, and gelatin supplemented ice creams, respectively (Fig. 5). The decreasing order of X_{a50} values shows the same pattern seen with mean air cell size. Sofjan and Hartel (5) also showed that decreased air cell size results in increased overrun. Similar results were observed by other researchers (5, 20, 28). Increased overrun enhanced the destruction of larger air cells into smaller ones during freezing so that a higher percentage of smaller air cells were observed (5). There was no significant difference ($p>0.05$) of mean air cell size among ice cream mixes supplemented with gelatin, pectin, or HES. In contrast, ice cream mix with added LBG showed a significantly larger air cell size compared to other stabilizers (Fig. 3).

In general, it is recognized that smaller dispersed air

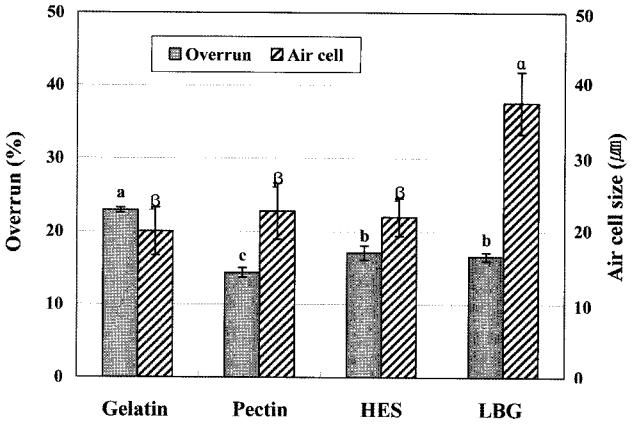

Fig. 3. Changes in overrun and air cell size with added stabilizers. [a-c,α-β]Means within each bar with different superscripts are significantly different ($p<0.05$).

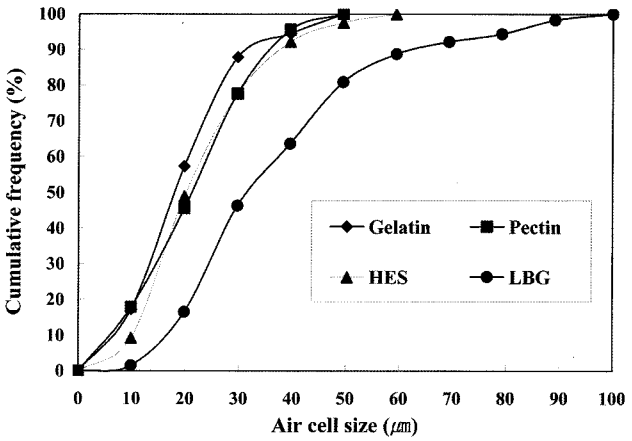

Fig. 5. Cumulative distributions of air cell size influenced by different stabilizers.

cells can lead to more stable foam, and a creamier mouthfeel of the product (29). The effect of a small air cell size distribution, as enhanced by surfactants, could minimize ice recrystallization resulting in improved sensory qualities (26, 30). In controlling both quality and quantity, it is important to maintain a uniform amount of air (24). In this view, gelatin, pectin, and HES were appropriate as stabilizers according to their small mean air cell size and cumulative frequency (Fig. 3 and 5). The largest mean size and frequency of large air cells were observed in the LBG mixes with low overrun. This might be related to the high viscosity of LBG due to its polysaccharide chains and high molecular weight compared to other stabilizers. The high viscosity of LBG appears to obstruct the incorporation and dispersion of air cells into the model mix during the aging process, resulting in a low overrun and large air cells. Therefore, the properties of each particular stabilizer are considered an important factor influencing the overrun and development of air cells in ice cream. In the work of Chang and Hartel (31), there was a strong correlation between stabilizer concentration and air cell size, resulting in decreased air cell size with increasing stabilizer concentrations up to 10 min after removing samples from a batch ice cream freeze. In our research, air cell size was measured immediately after hardening. However, if air cell size is measured in relation to storage time, somewhat contrary results could be obtained due to the delay in air cell growth with increased viscosity. Increased viscosity in the fluid decreases the diffusion rate of gas between bubbles and retards disproportion of air cell sizes maintaining a small air cell size (32).

Ice crystal size and morphology During the batch freezing of ice cream, the development of ice crystal size distribution and morphological properties was influenced by many factors. Formulation factors (fat, emulsifier, and stabilizer contents) and processing parameters (freezer temperature and scraping power) can both influence ice crystal development. The mean ice crystal size and cumulative frequency associated with each stabilizer are shown in Fig. 6 and 7, respectively. In this study, mean ice crystal size was observed to be in the range of 25 to 35 μm. The largest mean size and widest distribution of ice crystals were observed in the gelatin-supplemented ice

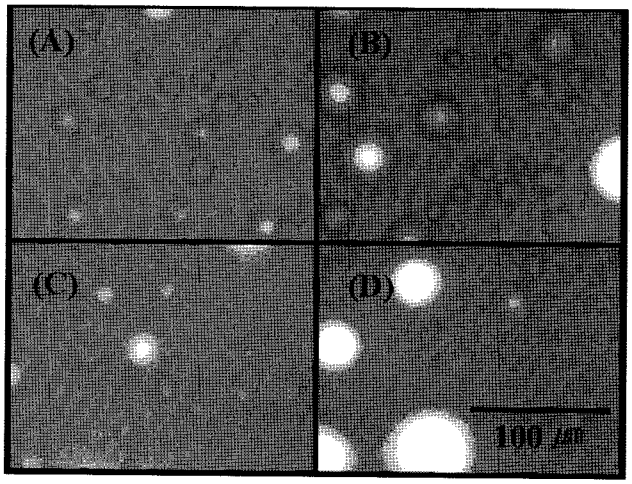

Fig. 4. Microscopic images of air cells with each added stabilizer. (A) Gelatin, (B) Pectin, (C) HES, (D) LBG

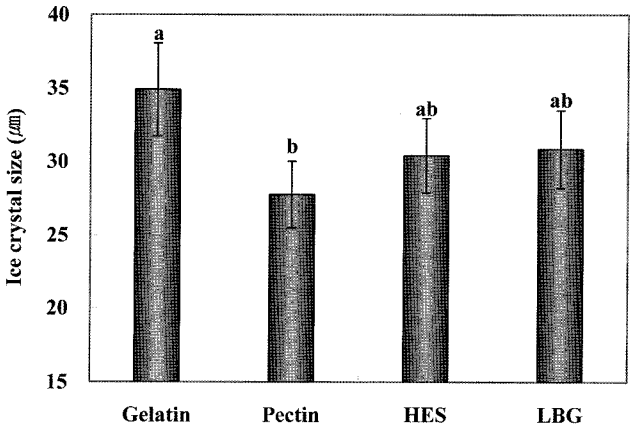

Fig. 6. Comparison of ice crystal sizes in ice cream with added stabilizers. [a,b]Means within each bar with different superscripts are significantly different ($p<0.05$).

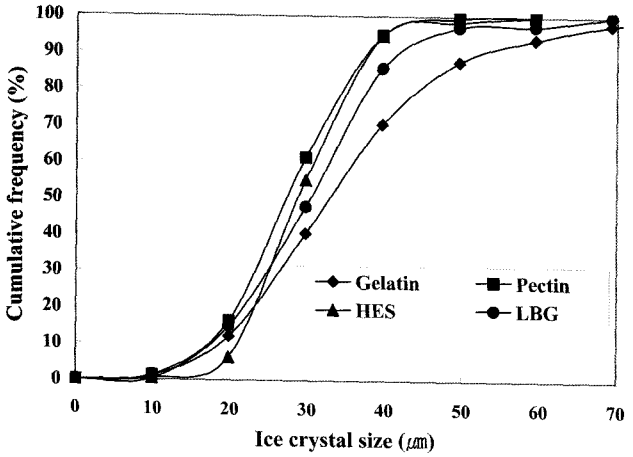

Fig. 7. Cumulative distributions of ice crystal size influenced by different stablizers.

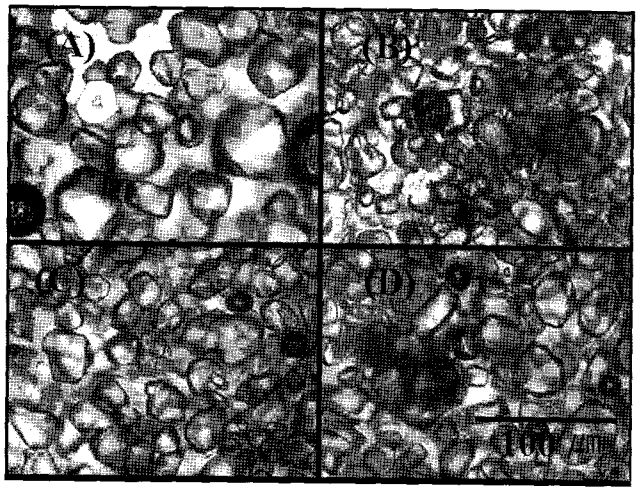

Fig. 8. Microscopic image of ice crystal size with each added stabilizer. (A) Gelatin, (B) Pectin, (C) HES, (D) LBG

cream mix, with a mean size of 34.89 μm. The addition of pectin resulted in the smallest ice crystal size, 27.76 μm (p <0.05). There was no significant difference in ice crystal size between HES and LBG supplemented ice cream. The proportion of ice crystal sizes at 50% of the cumulative frequency, the X_{i50}, was 32.7, 30, 28.5, and 27.3 μm in gelatin, LBG, HES, and pectin, respectively, showing the same decreasing order seen with mean ice crystal size (Fig. 7).

The X_{i50} value is regarded as a good representation of ice crystal distribution, representing a better measure of central tendency than does the arithmetic mean when the data is skewed (33) if a data point falls at 50% of the cumulative frequency. Flores and Goff (33) reported that the X_{i50} is typically lower than mean sizes when there is a preponderance of small crystals compared to large ones, and similar results were obtained from our studies. The ice crystal sizes obtained from this study tended to be smaller than those seen in other investigations. Berger and White (34) reported that ice crystals in ice cream varied from 10 to 160 μm, with a mean size of 48 μm. Min and Lee (21) observed a similar crystal size and distribution during the storage of ice cream. Ice crystal size can represent a quality parameter of ice cream. A crystal size of around 55 μm could result in coarseness and a sandy texture, but that depends on the crystal shape and size distribution (35).

In this study, most ice crystals exhibited a spherical shape and agglomerated form and this phenomenon was dependent on the type of added stabilizer. Figure 8 shows the microscopic morphology of ice crystals in each stabilizer supplemented ice cream mix, which were significantly different sizes among different stabilizers (p<0.05). Most ice crystals exhibited a round shape as shown in the microscopic images. Min *et al.* (7) reported that the transition of dendrite into compact round crystals resulted from the process of iso-mass recrystallization during the structuring of a low thermodynamic potential form.

Changes in ice crystal size showed an inverse correlation with viscosity, with smaller ice crystal size associated with high viscosity. These results were comparable with studies investigating the relationship between the viscosity of ice cream mixes with stabilizers and the crystallization of ice. Flores and Goff (33) found that the addition of stabilizers causes a significant increase in the viscosity of an ice cream mix, and affects ice crystallization rates. Similar phenomena were observed in other model systems (36, 37). Stabilizers affect continuous phase viscosity and other rheological properties of ice cream mixes. Min *et al.* (7) reported that delayed recrystallization of ice is influenced by various hydrocolloids. These different effects of hydrocolloids on ice crystal formation appear to originate from properties of viscosity and the migration of water molecules between two adjacent ice crystals (21). In addition, stabilizers have a significant influence on ice crystallization, which is related to freezing point depression, glass transition temperature, and viscosity (5). Different stabilizers depressed the freezing points of water to different extents depending on the number of small molecules present (5).

Melt resistance Figure 9 shows the typical melting test profiles and time versus melted weight of ice cream with added LBG. IMT and TMT were determined from time points, a to b and a to c, respectively. These results are summarized in Table 2. In this study, a longer melting time meant enhanced melt resistance and good shape retention of the ice cream at room temperature. Bolliger *et al.* (38) reported increased melt resistance and greater shape

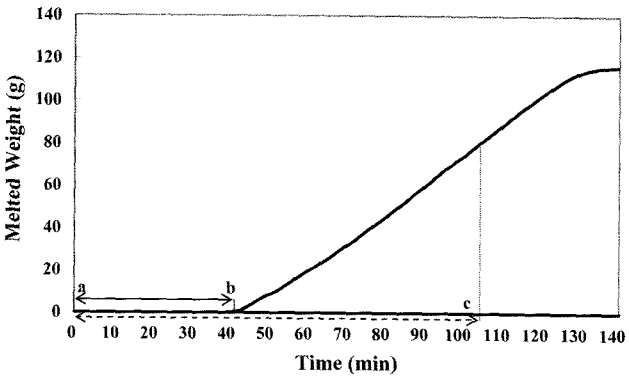

Fig. 9. Typical profile of time versus melt weight in melt resistance tests of ice cream mixed with LBG.

Table 2. Influence of freezing rate on melt resistance

Stabilizer	Melting time[1)]	
	IMT[2)] (min)	TMT[3)] (min)
Gelatin	40.0±5.29^{B}	127.3±4.28^{A}
Pectin	59.7±1.53^{A}	118.4±4.75^{AB}
HES	48.3±1.16^{B}	93.6±6.16^{C}
LBG	45.0±7.55^{B}	109.3±5.97^{B}

[1)]Means within same column with different superscripts are significantly different ($p<0.05$).
[2)]IMT: Initial melting time, [3)]TMT: Total melting time.

retention in low temperature extrusion samples compared with a conventional freezer. In our work, fine and homogeneously dispersed ice crystals in a hydrocolloid matrix might enhance the melt resistance. Among stabilizers, pectin provided the greatest degree of melt resistance, followed by HES, LBG, and gelatin in decreasing order with regard to IMT. This profile is likely related to the mean ice crystal size of each stabilizer, with enhanced melt resistance associated with smaller ice crystals. Pectin resulted in the smallest ice crystal size, and the longest melting time. This pattern was also applicable to other stabilizers.

In contrast, there was no relationship between mean ice crystal size and TMT. This might be explained by the different flow behavior of each stabilizer-supplemented ice cream. TMT was largely influenced by viscosity during the melting process. Some of the melted samples attached to the mesh grid due to high viscosity during the melting test. In addition, some fat destabilization phenomena, coalescence or flocculation may influence the melting of ice cream. Therefore, there are likely many factors, such as stabilizers, fat destabilization, and the viscosity of ice cream mixes that influence melting. Bolliger *et al.* (38) reported that the portion remaining during the meltdown test was able to hold its shape for several hours with little structural collapse after melting. This would suggest that appropriate analysis of melting includes a combination of the rate of drip and shape factors resulting from the extent of fat network formation, similar to our experimental results. Many studies have been done concerning the melt resistance of ice cream. The melting time of ice cream is mainly governed by fat aggregation through different networks resulting from the presence of fat, proteins or other stabilizers (39-42). Hyvönen *et al.* (43) measured the perceived melt rate using time-intensity methodology, and found that increased fat content slowed down the perceived melting of ice cream (44). In emulsifier-supplemented ice cream, meltdown appeared to be likely due to the effect of polysaccharides on the viscosity of the melting serum rather than a fat based effect. Further studies are needed to examine the influence of stabilizers on melt resistance with regard to the molecular properties of stabilizer, fat destabilization, and flow behavior of the ice cream mix, considering that shape retention of ice cream is one of more important factors for its appeal to consumers.

Acknowledgments

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