Ice recrystallisation and melting in ice cream with different proteins levels and subjected to thermal fluctuation

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A B S T R A C T

Ice cream of high quality has smooth consistency due to ice crystal sizes between 20 and 50 μm, which depends on ice cream formulation and thermal stress during storage. Ice crystals of 3 “traditional” (milk proteins and stabilisers) and 2 vegan (inulin and potato proteins) ice cream formulations were studied using a cold stage microscope under frozen conditions. Samples were observed before and after a temperature fluctuation (cycles of –18 °C and –6 °C for 14 days), and the data obtained compared with the results of a melting test. Results showed that milk proteins and stabilisers conferred structural stability to ice cream, both in terms of small crystal size (20–50 μm) and behaviour during melting, suitable for storage. Ice creams without one of the two constituents or in presence of potato proteins or inulin, showed a crystal size increase; in this case ice creams were for immediate consumption.

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1. Introduction

Ice cream is a product consumed in frozen state, where freezing and aeration processes are very important unit operations for the desired structure development (Bahramparvar & Tehrani, 2011). The formulation of the original mixture is given by lipids, proteins and functional elements such as carbohydrates, stabilisers and emulsifiers. According to Goff and Hartel (2013), the right combination of these ingredients and their interaction confer the best texture with its complex microstructure to the finished product.

In particular, the sensorial quality of an ice cream is affected, not only by its formulation, but also by the size and morphology of ice crystals that should impart a smooth texture to the finished product (Caillet, Cogne, Andrieu, Laurent, & Rivoire, 2003). An ice cream is considered of good quality in terms of texture when it has a large number of very small ice crystals that should have an average size of 40 μm, or mostly between 20 and 75 μm (Russell et al., 1999).

However, during production, storage and commercialisation, important changes may occur to ice cream structure (Caillet et al., 2003). In fact, it is known that ice cream has chemical/physical characteristics that make it thermodynamically unstable and recrystallisation phenomenon that occur during storage at low temperatures causes a gradual increase of the average size of ice crystals and the decay of its quality (Regand & Goff, 2003). This phenomenon depends on storage conditions and ice cream formulation (Donhove & Hartel, 1996a,b).

To counteract the recrystallisation phenomena, during production and storage hydrocolloids with stabilising functionality (polysaccharides and proteins) are added to the product. Hydrocolloids have a cryoprotective behaviour: hindrance of the recrystallisation phenomena, enhancement of the viscosity of ice cream mixtures, improvement of texture and mouthfeel, and shape retention of ice cream (Marshall, Goff, & Hartel, 2003). The presence of these ingredients, in fact, improves texture by affecting ice crystallisation during production and increasing resistance to thermal shock during storage (Miller-Livney & Hartel, 1997). The presence of agar, k-carrageenan and locust bean gum, often added to ice cream formulations as stabilisers, form network structures within the product that inhibit the ice recrystallisation (Miller-Livney & Hartel, 1997). The protective effect of hydrocolloids is also due to the increase of viscosity that controls crystal growth consequent on their water binding and gel forming properties (Goff, Ferdinando, & Schorsch, 1999; Miller-Livney & Hartel, 1997; Soukoulis, Chandrinos, & Tzia, 2008). Miller-Livney and Hartel (1997) suggest that macro-viscosity changes due to the presence of hydrocolloids are associated with micro-viscosity, so the diffusion of the water to the ice crystals is delayed over time.

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Taking into account that texture, in general, and ice crystals size, in particular, are crucial elements of ice cream quality, different methods of analysis have been developed to control these characteristics. Sensory analysis, texture analysis, and many microscopy methods are often used in industrial laboratories for quality control.

Microscopy techniques, used to analyse ice cream texture, have the advantage of being reasonably fast and providing much information on ice crystal characteristics. However, some of these microscopy techniques are destructive; the sample is melted and the image does not represent all the frozen system texture morphology. Electron microscope analysis provides a lot of information on ice cream texture, but the instrumentation is very expensive and sample preparation takes a rather long time. A valid alternative is offered by the cold stage microscope, similar to that discussed by Caillet et al. (2003), which allows ice-cream crystals to be analysed under frozen conditions. In fact, direct observation at low temperatures allows the sample to be kept under conditions similar to reality and minimises the artefacts that can be generated in all other microscopy methods. In the operation conditions of the cold stage microscope it is possible to study distribution and ice crystal size of ice cream and the evolution of the ice crystals during storage.

In considering the importance of ice crystals in conferring structural and texture characteristics to ice cream, in this research the dimensions of the crystals in five ice cream formulations, produced directly on an artisanal level, was studied. Three formulations were prepared with proteins of animal origin, while the other two were prepared with plant (Solanum tuberosum) proteins and inulin.

2. Material and methods

2.1. Ice cream recipes

Five ice cream recipes were formulated and divided into two groups: three recipes containing animal proteins (milk proteins), called “traditional ice cream” and two recipes, called “vegan ice cream”, containing plant proteins and polysaccharides (potato proteins and inulin, respectively) (Table 1).

Traditional ice cream mixes all contained 10% coconut oil, 12% sucrose (Daila Zuccheri, Osio Sotto, Italy), 3.2% dehydrated glucose syrup (Glucidex DE38, Roquette, France), 11% low-fat milk powder (Milch Gmbh, Germany), except for sample 3, in which functional milk protein concentrate (11%; Havero 6115, Hoogwegt, Arnhem, The Netherlands) was added (these proteins represent the milk protein fraction that does not pass through the ultrafiltration membrane during processing; the resulting protein concentrate is dried to a well dissolving powder, varying in protein content up to 85%, as reported in the information sheet). Cremodan SE30 (0.65%; Danisco, Paris, France) was added as stabiliser; this is a mixture of mono and diglycerides of E471 fatty acids, carob seed E410, sodium alginate E401, guar gum E412 and carrageenan E407.

Vegan ice cream mixes all contained the same ingredients as above, with the exception of 11% inulin (Orafti GR, Beneo Gmbh, Mannheim, Germany) and 11% protein from S. tuberosum® 300, Avebe, Groneking, The Netherlands) instead of milk proteins (Table 1).

Water was added to the percentage needed to reach 100% (w/v) for each recipe.

2.2. Production process

For each formulation, 4 batches were prepared and for each batch 20 L of ice cream mix were processed. The raw materials, in powder form, were weighed and mixed together; coconut oil, solid at room temperature, was added after melting using a microwave. The ingredients were mixed with water until they became apparently homogeneous. All the ice creams were manufactured using a Bravo Trittico (model 305, Executive EVO Pastry and Gelato Ice Cream Machine, The Big Ice Box Co., Glaslough, Ireland) with the same operating conditions. Each ice cream mix was pasteurised at 85 °C for 5 s and homogenised at pressures of 17.2 MPa and 3.5 MPa in the first and second stages, respectively. The mix was then left to cool and ripen at 4 °C for 24 h. Then, the ice cream was remixed to emulsify the two phases (aqueous and lipid) that had separated over 24 h at 4 °C. The ice cream then was frozen at −5 ± 0.5 °C; sample was subjected to a 20 min of continuous whipping. Then the ice cream samples of about 100 g were extruded into plastic cups, hardened at −30 °C for 24 h and afterward stored at −20 °C.

2.3. Ice crystal size by cold stage microscope

A light microscope (Olympus BH-2, Tokyo, Japan) and MicrOil®'s Thermal Stage MTS120 apparatus (Amsterdam, The Netherlands) were used to study ice crystals and observe their morphology at the thermal condition of the product. The microscope slide was kept in the cold-stage, an isolated chamber with a heat exchanger, that can be set in high heating/freezing rates or be constant. The microscope cold stage has a temperature range of −20 °C to +120 °C. The MTS120 stages supplied with high precision MTDC600 programmable temperature controller, with resolution and accuracy of 0.1 °C.

In particular, a sample of ice cream was removed from the freezer (−19 ± 1 °C). All the procedures, performed for the study of ice crystals, were conducted in a cold chamber at −20 °C and all materials and solvents were kept at the same temperature to avoid thermal shocks. Ice cream sample was obtained 2 cm from the surface, by removing the upper part of the frozen product. In this inner part of the ice cream, 10 mg of product was collected with a metal scoop and spread on the slide surface of the cold stage microscope.

Sample was covered with a cover slip, and a drop of butanol, at −20 °C, was added to avoid condensation. A camera was used to capture images at about 100× magnification (with some modification, as a function of ice crystal size). For each sample a variable number of photos were taken (28–35 pictures) and for each picture about 40 ice crystals were measured. ImageJ image analysis software was used to count ice crystals, and measure their size by considering the maximum distance between parallel tangents to the projection area of the particle; this parameter was chosen because of the irregular shape of the ice crystals.

2.4. Overrun determinations

Overrun (OR) was determined according to the method described by Marshall et al. (2003). A known volume of ice cream mix and frozen ice cream were weighed and overrun calculated according to the formula:

\[
\text{Overrun (%) } = \frac{\text{weight of a volume of the ice cream mix} - \text{weight of the same volume of ice cream}}{\text{weight of the same volume of ice cream}} \times 100
\]
2.6. Thermal stress

The quantity of drained ice cream (expressed in grams, on the ordinate) and consideration time (expressed in minutes, on the abscissa) was measured, to calculate the percentage of melted ice cream after 5 min time intervals for 60 min. The weight of the drained sample was recorded at a wire screen mesh and allowed to melt at 22°C for 6 h then 6°C for 6 h, in a thermostat (PID System, Thermovetro, Grandola ed Uniti, Italy). All samples were kept at −19 ± 1°C until tested. The time required for the dripping of the first drop of melted ice cream was recorded. The weight of the material passed through the screen was measured to calculate the percentage of melted ice cream after 60 min at room temperature. Finally, a graph was drawn taking into consideration time (expressed in minutes, on the abscissa) and quantity of drained ice cream (expressed in grams, on the ordinate).

2.5. Melting test

To study the melting ice cream behaviour, first dripping time and melting rate were considered, as proposed by Soukoulis et al. (2008), with some modifications. The ice cream sample, 100 ± 2 g, (slightly varied in terms of dimension from one ice cream to another) was put on a wire screen mesh and allowed to melt at 22 ± 0.5°C, in a thermostat (PID System, Thermovetro, Grandola ed Uniti, Italy). All samples were kept at −19 ± 1°C until tested. The time required for the dripping of the first drop of melted ice cream was recorded. The weight of the material passed through the screen was recorded at 5 min time intervals for 60 min. The weight of the drained sample was measured, to calculate the percentage of melted ice cream after 60 min at room temperature. Finally, a graph was drawn taking into consideration time (expressed in minutes, on the abscissa) and quantity of drained ice cream (expressed in grams, on the ordinate).

2.6. Thermal stress

Ice cream samples were placed at −18°C, and a data logger (RC-5, Elitech, London, UK) was set to check the data of the thermal stress, as reported in the graph of Fig. 1.

The cold room was connected to an adjustable thermostat (ITC 310T B 230 V, Inkbird, Shenzen, China) for both temperature and time. Then a temperature cycle of −18°C for 6 h then −6°C for 6 h was set and the cycle repeated for 14 days.

2.7. Statistical analysis

All statistical analysis was carried out using Origin 2018 Graphing & Analysis, Statgraphics Centurion XVII software and Excel. Each recipe was produced 4 times, so, for each ice cream recipe, there were 4 batches (4 batches × 5 recipes). For each batch, 3 replicates were considered, in this way for each recipe 12 data sets were statistically processed by descriptive and inferential tests (Tukey test, p < 0.05) and for melting test a non-linear regression model was performed.

3. Result and discussion

3.1. Ice cream composition and overrun

As shown in Table 1, the different ice cream composition affects the product expansion values in terms of OR; samples 2 and 3 show a higher OR (84% and 83%, respectively), followed by sample 1 with 62% OR and finally samples 4 and 5 with a low OR value (24% and 26% respectively). In the first two samples the presence of powdered milk and in sample 3 the functional dairy proteins concentrate promote foam formation; in fact, milk proteins and functional dairy proteins concentrate show important functional characteristics in forming foam due to their amphiphilic character (Singh, 2009), so increasing the volume and expansion of the ice cream. The higher expansion of samples 2 and 3 might be due to the presence of milk proteins (milk powder and functional milk proteins concentrate) and Cremodan SE30, a mixture rich in polysaccharides, such as guar gum (Goff et al., 1999), which is responsible for the further increase in OR in fact creates an excellent dispersion of air in the mixture and a stable OR. The lack of milk proteins and their effect on OR values is particularly evident in samples 4 and 5, in which neither the presence of S. tuberosum proteins, with their rheological characteristics, in particular as foaming agents (Lomolino, Vincenzi, Gazzola, Crapisi, & Curioni, 2015), and polysaccharides present in the stabiliser, could not reach the OR values observed in samples 1, 2 or 3 (Table 1). It could therefore be stated that the OR is favoured by milk proteins, especially in presence of the polysaccharides (guar gum, carrageenan, etc.) of the stabiliser CremodanSE30, added to the formulations.

3.2. Ice crystal analysis

To study ice cream characteristics and its changes induced by temperature, the 5 samples were subjected to thermal fluctuation for 14 days. Samples, subjected to this treatment, should behave differently depending on their composition and formulation. Ice crystals, size and shape, and melting dynamics represent crucial aspects in studying the consequences of thermal stress undergone by ice cream.

Fig. 2 shows the pictures of the 5 samples before (samples 1s and 5s) thermal stress treatment. As shown in Fig. 2, ice crystals of the five samples apparently differ in size both when they are prepared with different ingredients and before and after thermal stress. It is well known that ice cream icy phase, represented by ice crystals, is influenced by recrystallisation phenomena that depend on formulation characteristics, freezing process and storage conditions (Donhowe & Hartel, 1996a,b). Large crystals are perceived as rough particles on the palate and negatively affecting sensorial texture of ice cream (Goff et al., 2013).

Table 1 Ingredients used in the “traditional” and “vegan” recipes of ice cream and resulting overrun. 

<table>
<thead>
<tr>
<th>Ingredients %</th>
<th>Traditional recipes</th>
<th>Vegan recipes</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Recipe 1</td>
<td>Recipe 2</td>
</tr>
<tr>
<td>Sugar</td>
<td>12</td>
<td>12</td>
</tr>
<tr>
<td>Glucose syrup</td>
<td>3.2</td>
<td>3.2</td>
</tr>
<tr>
<td>Coconut oil</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Powder skimmed milk</td>
<td>11</td>
<td>11</td>
</tr>
<tr>
<td>Cremodan SE stabiliser</td>
<td>0.65</td>
<td>0.65</td>
</tr>
<tr>
<td>Functional milk protein</td>
<td>11</td>
<td></td>
</tr>
<tr>
<td>Potato protein</td>
<td>11</td>
<td></td>
</tr>
<tr>
<td>H2O</td>
<td>63.8</td>
<td>63.15</td>
</tr>
<tr>
<td>Overrun</td>
<td>62.0 ± 2.0a</td>
<td>86.0 ± 2.0a</td>
</tr>
</tbody>
</table>

a The quantities of ingredients are expressed in %. Overrun values were based on three different samples and data are presented as mean ± SD; values followed by the same letter are not significantly different (p < 0.05), as measured by the Tukey's multiple range test.
Fig. 2. Cold stage microscope ice cream pictures; comparison of the samples before (samples 1–5) and after (samples 1s–5s) thermal stress.
Fig. 3 shows the results of descriptive statistical analysis by box plot; as shown in Fig. 3A, samples differ from each other in terms of mean, median and rank width. In particular, sample 3 presents a mean value (27.33 µm), median (26 µm) and rank (41 µm) lower than the other samples, statistically showing the small size and the uniformity of the ice crystals. On the other hand, samples 1 and 5 show higher mean and median values (43.5 and 43, respectively, for sample 1; 42.9 and 42, respectively, for sample 5). In all cases, with exception of sample 3, ranks are rather large, and show a non-uniformity in crystal size. The presence of stabiliser (Cremodan SE30) and milk proteins, in sample 3, allowed the development of uniform and small crystals, in contrast to sample 1 in which the absence of stabiliser favoured the heterogeneous development of the icy phase. This feature is also observed in samples 4 and 5 in which the absence of milk proteins favours the heterogeneous growth of ice crystals (Fig. 3A). After thermal stress, the ice crystals of the five samples change their size and shape (Fig. 3B). In particular, sample 1s underwent great changes in terms of the average crystal size, which increased about 8 times; the median value is much lower (123 µm) than the mean, while the rank is particularly large (136 µm). These results highlight that sample 1, without stabiliser with its polysaccharide component designed to stabilise and improve the characteristics of ice cream could be damaged if subjected to thermal stress, losing the regularity of the crystals and dramatically increasing in size. In contrast, sample 3s maintained its structure in terms of size and uniformity of ice crystals: the mean value increased only 1.7 times compared with the same original sample, the median value is very close to the mean and the rank remained low (74 µm). Samples 2s and sample 5s are very similar to each other in terms of mean, median and rank values. Even if their formulation is different, ice crystals change in terms of average size (the average is about twice as high as the original samples) and shape.

Samples containing milk proteins, both in form of powdered milk (sample 2) and concentrated milk protein (sample 3) and sample 4, in which potato proteins are present, in combination with

![Box plot analyses on the five ice cream formulations before (A) and after (B) thermal stress. Data refer to the ice crystals size; calculated values are from the raw data. The box represents 25%–75% and contains the median line and the mean (□); the range within 1.5IQR is given by the bar; outliers are denoted by •.](image-url)
the polysaccharide-rich stabiliser (Cremodan SE30), show crystals size smaller than samples 1 and 5 (Fig. 3A). Some studies (Regand & Goff, 2003) showed that stabilisers, in general, have cryoprotective characteristics, very important during ice cream storage period or when product is subjected to thermal fluctuations.

In this study, the thermal stress undergone by the five samples may have generated this physical process, characterised by large crystals growth at the expense of the smaller ones, due to difference in equilibrium temperature, caused by the contribution of surface energy (Donhhowe & Hartel, 1996b). Donhhowe and Hartel (1996b) observed that recrystallisation phenomenon was favoured by the increase in temperature fluctuation, as in the case of the five formulations in this study. The extent of recrystallisation during storage or temperature fluctuation is affected by the formulation (Regand & Goff, 2003). It was observed that stabiliser characteristics and composition impact recrystallisation phenomenon, with a reduction of crystal growth. Regand and Goff (2003) attributed the stabiliser mechanism to the increase in viscosity of the unfrozen phase, which decreases the mobility of water molecules. Polysaccharides such as locust bean gum and carrageenan, present in the Cremodan SE30 used in samples 2–5, form gel-like structures delaying water mobility through steric hindrance due to the ability to bind it during ice cream storage or thermal fluctuations, as in this case. In particular, the carrageenan present in Cremodan SE30 was more effective in delaying recrystallisation and cryoprotective activity in presence of caseins with which it interacts forming a strong gel-network (Bbrahimparvar & Tehran, 2011). In particular, the protein/polysaccharide interaction that occurs in presence of carageenan promotes an increase in the viscosity of liquid phase (Camacho, Martinez- Navarrete, & Chiralt, 1998). Thus, the reduced growth of ice crystals, observed in presence of the stabiliser CremodanSE30 with its polysaccharides and especially carrageenan, is due to the increase in viscosity and to the strong network-type or gel-like structure (Regand & Goff, 2006) that reduces the mobility of water around ice crystals. In contrast, as reported by Gaukel, Leiter, and Spieß (2014), k-carrageenan shows a significant decrease of recrystallisation phenomenon when it is present at low concentration, perhaps because of the interaction of the polysaccharide with the ice crystal surface.

However, in all five samples subjected to thermal stress, recrystallisation phenomenon and ice crystal size increasing occurs to a different extent depending on the sample formulation. In fact, as suggested by Regand and Goff (2003), the ability to bind water by the different stabilisers and interactions that they establish with other components in the formation of gel-like structure, is reduced during temperature fluctuation, with consequent increase of water mobility around crystals and destabilisation of the ice cream structure. As observed in the five samples, the phenomenon of recrystallisation is particularly present in the sample without stabiliser Cremodan SE30 (sample 1), and the mean crystal size has very high values; in samples 2, 4 and 5 the presence of stabiliser Cremodan SE30 seems to control ice crystal dimension, both with or without proteins with which the polysaccharides interact. In sample 3, the possible interaction between carrageenan of the stabiliser and caseins in the concentrated milk proteins ensures a high cryoprotective effect and reduced crystal size growth (Soukoulis et al., 2008).

3.3. Melting and dripping

Since the physical structure of ice cream and its formulation affect melting process, the melting dynamics of the five ice cream samples were studied by observing the macroscopic shape modification and by quantifying the melted fluid released over time. The five samples, before and after thermal stress, were placed on a grating at 22 °C for 60 min. As shown in Fig. 4, sample 1, without stabiliser (Cremodan SE30), after 60 min at 22 °C is completely destructured and the remainder represents the 20% of the initial weight. Even sample 1s is collapsed, but probably due to the structure that was generated inside, after thermal stress, retained of 55% of its weight. In contrast, samples 2, 2s, 3, and 3s, after 60 min at 22 °C, maintain almost the original parallelelepiped shape and their weight is 84–89% initial weight in almost all cases. Once again polysaccharides, present in carob flour, sodium alginate, guar gum and carrageenan that constitute the stabiliser mixer (Cremodan SE30), in combination/interaction with milk proteins (milk powder, sample 2 and milk protein concentrate, sample 3) allowed the formation of an internal network that favoured maintenance of the structure and shape of the ice cream, a large part of its initial weight and reduced melting phenomenon. In sample 4, potato protein did not guarantee maintenance of the initial sample shape, their interaction with the Cremodan SE30 polysaccharides is not strong enough to preserve the internal network structure and the shape is modified, maintaining 75% (sample 4) and 78% (sample 4s) of initial weight.

In sample 5, however, even in presence of the stabiliser (Cremodan SE30), the absence of proteins does not allow structure and shape maintenance; the ice cream was almost collapsed and represented about 60% of the initial weight, in both sample 5 and sample 5s. In sample 5, inulin does not completely guarantee the maintenance of the initial shape and does not inhibit melting. As reported by some authors (Akin, Akin, & Kirmaci, 2007) inulin is able to bind water, making it immobilised and unable to move freely among other molecules of ice cream mixture, delaying melting; in this study inulin acts as a stabiliser in a rather limited way. The melting study was carried out by evaluating the time of “latency” before first drop formation of each sample as an indicator of structure stability. Fig. 4 shows once again the unstable structure of sample 1, in fact dripping starts after about 10 min at 22 °C. Samples 4 and 5 seem to have a better network structure than sample 1 due to the presence of the stabiliser Cremodan SE30 and, to a lesser extent, inulin; dripping of the samples begins after about 20 min. Samples 2 and 3 are much more stable due to their more structured internal matrix; dripping began after about 45 min. During the melting process, water, from melted ice, diffuses in the unfrozen fluid viscous phase and this diluted solution, which is formed, flows downwards, through the structural elements (fat globules, air cells, remaining ice crystals, polysaccharides and the net-work within the product; Marshall et al., 2003; Soukoulis et al., 2008). These elements constitute a resistance factor to fluid flow during ice cream melting. Therefore, when ice cream presents a well-structured system, due to formulation components that increase viscosity or form an internal network matrix, dripping will be delayed and melted fluid will be reduced (Fig. 4), as in case of samples 2, 2s, 3 and 3s.

Another element that affects dripping is ice crystal size; the larger they are, as in the case of samples 1 and 1s, the more the phenomenon will be favoured. This may be due to liquid flow path of melted ice cream (Muse & Hartel, 2004). When small crystals are present, as in sample 3, the path of liquid phase during melting is more tortuous, fluid must pass through more obstacles, represented by small ice crystals. Therefore melting, measured as dripping, is reduced in the presence of many small crystals. In the case of the five samples, the formulation, components interactions and ice crystals dimensions could contribute in a different extent to their melting. It is interesting to observe the five samples after thermal stress (samples 1s–5s) subjected to melting and evaluation of the beginning of dripping. As shown in Fig. 4, they maintained...
Fig. 4. Dripping and melting test: comparison of the five ice cream formulations before and after thermal stress. Graphs show the time elapsed between the start of the melting test and the first drop of melted material. All values were based on three different samples and data are presented as means ± SD; different letters indicate significant differences (p < 0.05) as measured by the Tukey’s multiple range test. Percentage figures are the residual weight of ice creams after 60 min of melting test.
the same behaviour of the unstressed samples, with the exception of sample 1s that starts on average 15 min later than sample 1.

Melting dynamics of samples was studied, weighing the amount of thawed product from the beginning of dripping of each sample, every 5 min for 60 min. Fig. 5(A and B) show the melting curves of the five samples before and after thermal stress.

As shown in Fig. 5, melting trend does not occur in a linear manner, but follows a squared polynomial model (Table 2) even though the low value of B2 coefficient shows that the curve bends slightly. The regression coefficient values are very high (R² is over 99%) indicating that the trend and data fit the proposed function. The trends of the five samples do not seem to vary much before and after thermal stress, with the exception of sample 1 that before thermal stress presents a curve with a higher trend and a value of B2 higher than the other samples. After thermal stress, melting trend of the sample 1s becomes much lower and similar to that of sample 5s. The curves of samples 2 and 3, and 2s and 3s, are rather low and remain unchanged regardless of thermal stress that they have undergone, while the curves of samples 4 and 4s are positioned at intermediate level, and sample 4s is not particularly affected by thermal stress. The observed trends, all attributable to the same model and typical for each sample, confirm what was previously observed and discussed on the effect of the different compounds present in the 5 formulations and the chemical physical interaction that could occur in the observed systems.

4. Conclusion

The 5 ice cream formulations, analysed by cold stage microscope before and after thermal stress, used to simulate the prolonged stored conditions, show different characteristics in terms of both ice crystal size and dripping and melting behaviour. In particular, milk proteins, in the presence of the hydrocolloids of the stabiliser, allowed the optimisation of some characteristics, such as the higher overrun, the small size of ice crystals and melting dynamics. Potato proteins, in the presence of stabiliser, give a certain initial quality to ice cream, in terms of ice crystal size even if ice cream does not resist to thermal stress. Finally, ice creams in which only the protein component of milk or only stabiliser is present tend to deconstruct during prolonged storage. Even if other analytical tests are useful to study ice cream overall quality, the use of cold stage microscope together with melting test could predict the stability of ice cream obtained by different formulations.

Acknowledgements

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References


Table 2

Parameter values of the polynomial model that fits melting data of the 5 ice cream formulations. 

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Intercept (g)</td>
<td>Sample 1</td>
</tr>
<tr>
<td>0.291</td>
<td>0.07</td>
</tr>
<tr>
<td>B1 (min)</td>
<td>0.61</td>
</tr>
<tr>
<td>B2 (min²)</td>
<td>0.01</td>
</tr>
<tr>
<td>R²</td>
<td>0.995</td>
</tr>
</tbody>
</table>

Stressed Sample 1s Sample 2s Sample 3s Sample 4s Sample 5s

| Intercept (g) | 2.5 | 0.21 | 0.09 | 0.44 | 0.04 |
| B1 (min) | 0.44 | 0.09 | 0.009 | 0.44 | 0.53 |
| B2 (min²) | 0.004 | 0.002 | 0.001 | 0.0008 | 0.0004 |
| R² | 0.998 | 0.998 | 0.997 | 0.995 | 0.991 |

* The polynomial model was Y = intercept + (B1 × 1) + (B2 × 2). The quantities of melted ice cream, expressed in grams, were collected and weighed every 5 min for 60 min; tests were carried out in triplicate (SD < 5%).


