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Effect of inulin on melting and textural properties of low-fat and sugar-reduced ice cream: optimization via a response surface methodology

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Abstract

Ice cream is a dairy product with relatively higher fat and sugar content. In this work the simultaneously reduction of both butyric fat and sugar content in ice cream formulation via a response surface methodology was investigated. A rotatable central composite design (15 runs plus 5 central point replications) with different butyric fat, sugar and inulin content was employed to study the effect on overrun, viscosity, melting and textural properties of ice cream. Higher apparent viscosity resulted in a more stable system with higher overrun, where inulin controlled available water. The improvement in melting properties reflected the stable state of the air bubbles-emulsified fat-ice crystals matrix, where the putative effect of inulin to retain water compensating solids and fat reduction, retarded ice crystals melting. In instrumental texture, inulin retained free water when butyric fat and sugar were reduced, resulting in smaller ice crystals reflecting a softer texture. At the experimental conditions proposed, inulin (3%) as functional ingredient (soluble fiber and prebiotic) can be employed to reduce 30% butyric fat content and 12% sugar content, in the formulation of low-fat reduced-sugar ice cream.

Introduction

Ice cream is a complex food system with a disperse phase consist in three main structural components (air bubbles, ice crystals and emulsified fat globules) immersed in a continuous liquid phase (unfrozen water with dissolved sugar, proteins and hydrocolloids). Fat and sugar are the compounds that provide the caloric content in ice cream. Reduce or replace these important ingredients from ice cream formulations mainly affect the texture and sensorial perception (Clarke, 2004). Fat had many functions on physicochemical and sensorial properties in ice cream, for example: it is responsible of emulsion formation, increase the viscosity of serum phase, creates a film on the surface of the air cells that promote the stability in the ice cream, decrease the melting time, reduce the growth and size of ice crystals, provide texture, palatability, creaminess, releases flavor molecules and enhances mechanical properties (Goff, 1997; Adapa *et al*., 2000; Goff, 2002). Sugars in ice cream had two principal functions: provide sweetness and they control the amount and size of ice crystals, affecting ice cream softness. Sugar has the ability to decrease the freezing point of serum phase and therefore reduce the amount of ice caused for crystallization and recrystallization. The high molecular weight of sugars increase the viscosity of serum phase, promoting an increase of trap air, slow melting and low hardness of ice cream (Hagiwara and Hartel, 1996; Goff and Flores, 1999).

Obesity and overweight in many countries of the world have risen sharply over the past two decades, involving altered eating habits or the increasingly sedentary lifestyles, and the sharpest change in diet structure has involved added sugars and fat. Even low income families have the highest rates of overweight, due to high palatability and low energy cost of added sugars and fats. Strategies for obesity prevention increasingly focus on fiscal and policy measures to limit the consumption of fats and sweets (Drewnowski, 2003). The technological challenge is then to replace both fat and sugar without detrimental effect on food properties. For example, replacing fat and sugar employing inulin affected textural properties and overall acceptability in cakes (Rodriguez-Garcia *et al*., 2014). Inulin has been employed to replace fat in ice cream, decreasing melting rate, besides to increase adhesiveness and hardness (Akbari *et al*., 2016). In same manner, other gums had been employed in low fat ice cream. Javidi *et al*. (2016) reported that basil seed gum and guar

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Table 1. Experimental points design (uncoded, coded) and experimental results for the low-fat sugar-reduced ice cream formulation

Run	% Butyric fat (X_1)	% Inulin (X_2)	% Sugar (X_3)	Apparent viscosity (Cps) (Y_1)	Overrun (%)(Y_2)	Melting rate (g/min) (Y_3)	First drop (min) (Y_4)	Hardness (N) (Y_5)	Compression force (N) (Y_6)
1	4.0 (-0.5)	2.5 (0.25)	10.5 (-0.5)	1920	2.10	0.08	35	5.90	14.1
2	4.0 (-0.5)	3.5 (0.75)	13.5 (0.5)	3920	3.10	0.03	46	14.20	37.0
3	8.0 (0.5)	2.5 (0.25)	13.5 (0.5)	7040	5.00	0.10	50	6.00	17.3
4	8.0 (0.5)	3.5 (0.75)	10.5 (-0.5)	8320	5.00	0.12	30	7.00	16.8
5	6.0 (0.0)	2.0 (0.0)	9.0 (0.0)	4320	3.40	0.11	40	9.90	26.3
6	6.0 (0.0)	2.5 (0.0)	12.0 (0.0)	5120	3.20	0.12	45	10.10	11.7
7	4.0 (-0.5)	2.5 (0.25)	13.5 (0.5)	3680	3.20	0.09	38	5.70	9.4
8	4.0 (-0.5)	3.5 (0.75)	10.5 (-0.5)	6880	5.00	0.06	42	7.90	23.7
9	8.0 (0.5)	2.5 (0.25)	10.5 (-0.5)	6400	4.20	0.08	55	8.30	11.1
10	8.0 (0.5)	3.5 (0.75)	13.5 (0.5)	13930	10.00	0.04	65	4.40	7.4
11	6.0 (0.0)	2.0 (0.0)	15.0 (1.0)	7200	3.10	0.07	30	7.80	15.0
12	6.0 (0.0)	4.0 (0.0)	12.0 (-1.0)	5600	4.00	0.08	40	5.60	7.4
13	10.0 (1.0)	2.0 (0.0)	12.0 (0.0)	5130	2.80	0.06	40	9.70	9.6
14	2.0 (-1.0)	2.0 (0.0)	12.0 (0.0)	8640	2.80	0.11	60	4.80	4.4
15	6.0 (0.0)	2.0 (1.0)	12.0 (0.0)	6080	2.40	0.08	40	13.30	11.4
16	6.0 (0.0)	2.0 (0.0)	12.0 (0.0)	4720	4.20	0.08	50	9.30	13.1
17	6.0 (0.0)	2.0 (0.0)	12.0 (0.0)	5440	2.80	0.12	45	9.00	14.4
18	6.0 (0.0)	2.0 (0.0)	12.0 (0.0)	5520	2.60	0.12	45	14.00	14.0
19	6.0 (0.0)	2.0 (0.0)	12.0 (0.0)	5600	3.80	0.13	45	9.00	12.2
20	6.0 (0.0)	2.0 (0.00)	12.0 (0.0)	5320	3.35	0.15	46	10.35	13.4

gum favored the perception of creaminess, depressed coldness and coarseness perception. Basil seed gum reduced meltdown rate and can be employed as fat replacer/stabilizer in low fat ice cream. Also chia seed mucilage was employed as ice cream stabilizer, improving overrun, texture and melting rate (Campos *et al*., 2016). Nonetheless, the prebiotic capacity of inulin (Cruz *et al*., 2009; Criscio *et al*., 2010), besides their techno-functional properties as fat replacer in dairy products (Meyer *et al*., 2011; Tiwari *et al*., 2014), made inulin the better option to formulate low fat and sugar reduced ice cream, with added prebiotic. In low-calorie functional ice cream inulin increased overrun and hardness, besides to improve the viability of B. lactic, where melting rate and sensory scores of low fat and/or low-sugar formulation (Hashemi *et al*., 2014). In same manner, Fragoso *et al*. (2016) reported that thermotolerant lactic acid bacteria improved sensory, melting and textural properties of low-fat ice cream formulated with inulin.

The aim of this study was to reduce simultaneously butyric fat and sugar content, employing inulin to compensate fat and sugar, via response surface methodology, and the effect of formulation on apparent viscosity, overrun, textural and melting properties.

Material and Methods

Ice cream formulation

Fat-reduced ice cream was elaborated according the formulation described by Pintor and Totosaus (2012). Solid ingredients like sugar (15% w/v), non-fat dry milk and whey protein concentrate (8.0 and 4.0% w/v, respectively, DILAC S.A de C.V.), emulsifier (sorbitan and glyceryl monostearates, 0.25% w/v, ARCY S.A. de C.V. Ecatepec, México) were hydrated in water (aprox. 58% v/v) at 60°C to disperse anhydrous butyric fat (10% w/v, ARCY S.A. de C.V., Ecatepec, México) and vegetable fat (4% w/v, La Mixteca, Ecatepec, México). Agave inulin (Vaserco, S.R.L. de C.V., Guadalajara, México) was employed to replace both fat and sugar. The homogenized ice cream base was pasteurized at 70°C for 30 min, cooled down to 4°C in ice bath, and stored at 2-4°C during 24 h. The ice cream base was frozen in a 2 quarters frozen ice cream CIM 50RSA machine (Cuisinart, East Windsor) for 20 min until obtain a uniform frozen paste. Samples were placed in plastic containers (123 mL) and kept frozen at -25°C.

Experimental design, data analysis and optimization

The optimization of the formulation of fat-reduced ice cream to enhance its physicochemical and textural parameters was carried out employing a response surface methodology. Butyric fat and sugar were systematically replaced with agave inulin. A rotatable central composite design was proposed for optimization of ice cream formulation at five levels with 20 runs, including five replicates of central point (Table 1) (Montgomery and Runger, 2010). The experimental results were analyzed in SAS software v. 8.0 ADX interface (SAS Institute, Cary), fitting second order model to establish relationship between independent variables (butyric fat X_1, sugar X_2 and inulin X_3) with response variables Y, as follows:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{j=1}^{k} \beta_{ii} X_i^2 + \sum\sum_{i<j} \beta_{ij} X_i X_j + \in \qquad (1)$$

Where Y is the response variable that corresponds to physicochemical and textural measures, β_0, β_i, and β_{ij} are the estimate regression coefficients, and $\in$ is the experimental error. Response contour plots were generated in the same software holding one variable constant (butyric fat, central point, 6%).

Optimization of ice cream was performed selecting the desirability of the model for each one of the responses measured in the Desirability function of the Prediction profiler in same SAS ADX interface, where according to SAS support the overall desirability can be defined as the geometric mean of the desirability for each response. Multiple responses in the central composite design were maximized or minimized, according to desirable characteristics in ice cream, i.e., higher viscosity and overrun, slow melting, and a softer texture (Roland *et al*., 1999; Aime *et al*., 2001).

Apparent viscosity and overrun

The apparent viscosity were determined to ice cream base in a Brookfield RVT viscometer (Brookfield Laboratories, Middleboro), adapting the methodology reported by Akesowan (2008). Samples were cooled to 10°C and analyzed with a spindle #07 at 50 rpm after 30 s, reporting ice cream base viscosity in centipoises.

Ice cream yield, overrun, was determined as described by Marshall *et al*. (2003), according to:

$$\%\ \text{Overrun} = \frac{(\text{Ice cream weight} - \text{Ice cream base weight})}{\text{Ice cream base weight}} \times 100 \qquad (2)$$

Melting properties

Melting properties were determined according to the report by Soukoulis *et al*. (2008), with some modifications. First drop time and the melting rate were determinate by removing standardized ice cream samples from the containers and putting them on a stainless steel mesh (size 14, 1.41 mm pore size) at room temperature (25±2°C); the time (min) elapsed to obtain the first drop of melting ice cream

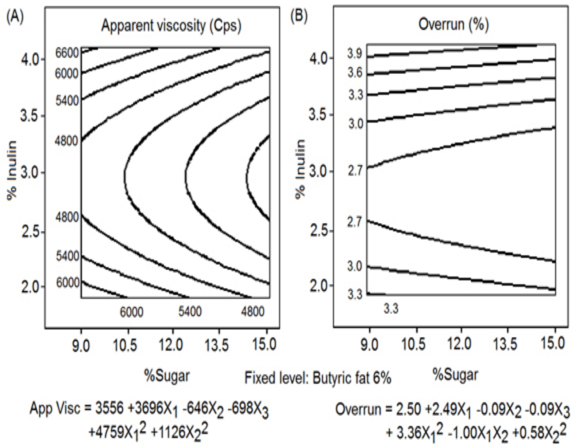

Figure 1. Contour plot and adjusted regression equation for (a) viscosity and (b) overrun (X_1: Butyric fat, X_2: Inulin, X_3: sugar).

was registered. The weight of the material that passed through the stainless steel mesh was recorded at 5 min time intervals during 1 h to obtain the melting rate (weight change per minute, according to the slope of the dripped portion as function of the time, in g/min).

Textural properties

The hardness of the ice cream was determined according to the method described by Soukoulis *et al*. (2008). Samples in plastic containers (123 mL) were temper at room temperature for 20 min and penetrated 8 mm from surface with a 10 mm diameter acrylic probe at a constant speed of one mm/s with a Brookfield LFRA 4500 texture analyzer (Brookfield Laboratories, Middleboro), reporting hardness as the peak force during penetration.

For the compression test, the methodology reported by Clark (2004) was adapted. Ice cream base was frozen in PVC cylindrical molds (15.70 cm2 diameter and 2.0 cm height) to form solid ice cream cylinders. Molds were removed and samples were compressed between two 10 cm diameter acrylic plates 40% of the original height in same the Brookfield texture analyzer at a constant rate of 1 mm/s. From force-deformation curves, compression force (maximum load peak) was calculated.

Results and Discussion

Apparent viscosity and overrun

According to the apparent viscosity results, the adjusted second-order model was highly significant (P=0.0001) and had a high correlation coefficient (R^2=0.8435). It was observed that the butyric fat linear term (X_1) and the butyric fat quadratic term

(X_1^2) had a highly significant effect ($P<0.01$) on this parameter. Inulin (X_2) and its quadratic term (X_2^2) presented a significantly ($P<0.05$) effect. No significantly ($P>0.05$) of sugar (X_3) was observed on ice cream base viscosity. In the regression equation, the positive sign of the linear and quadratic terms for butyric fat showed that apparent viscosity values increased mainly due to butyric fat effect, and the quadratic term for inulin (positive sign as well) (Figure 1a). In the contour plot it can be appreciated, at a fixed butyric fat level (6.0%), how at higher inulin concentrations and lower sugar content ice cream base the apparent viscosity increased (Figure 1a).

Fat that is dispersed and distributed through the continuous phase plays an important role in the increase of viscosity in ice-cream. When fat is reduced in the ice cream base, two phenomena are observed: a decrease in base viscosity due to a lower volume of fat aggregates in the continuous phase; and the decrease in formation of a film of fat globules on the surface of the air bubbles, reducing stability during melting (Chung and Grün, 2003). At the experimental conditions employed, when butyric fat was reduced together with sugar, inulin compensate the ice cream viscosity base, due to its capacity to form micro-crystals that interact with each other, creating small aggregates that trap water and increase the viscosity of the base (Akalin and Erişir, 2008; Akalin *et al*., 2008; Karaca *et al*., 2009). In this view, butyric fat reduction was compensated by inulin at reduced sugar contents.

Based on the results for overrun, the adjusted second-order analysis had a highly significant effect ($P=0.0001$) on this parameter, with a high correlation coefficient ($R^2=0.9520$). According to the ANOVA, the butyric fat linear term (X_1), butyric fat quadratic term (X_1^2), butyric fat×inulin interaction (X_1X_2), and the inulin quadratic term (X_2^2) had a highly significant effect ($P<0.01$) on this parameter. Inulin (X_2) presented a significantly ($P<0.05$) effect on overrun. No significantly ($P>0.05$) effect of sugar (X_3) was observed. In the regression equation it was found that butyric fat, with a positive sign, both linear and quadratic terms, increased the overrun values of ice-cream. Inulin quadratic term presented as well a positive influence on ice cream overrun (Figure 1b). The contour plot for overrun (Figure 1b), higher inulin concentrations increased overrun, at a fixed butyric fat level (6.0%), at lower sugar concentration.

High overrun were related to higher viscosities that promote more efficient air incorporation and the formation of smaller air cells (Chang and Hartel, 2002; Akin *et al*., 2007). In low-fat ice-creams where inulin

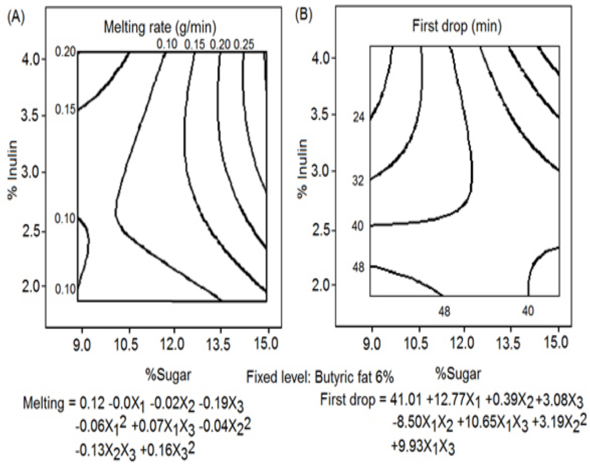

Figure 2. Contour plot and adjusted regression equation for (a) melting rate and (b) first drop (X_1: Butyric fat, X_2: Inulin, X_3: sugar).

was added, an increase in overrun values reported was related to the high viscosities caused by inulin (Akalin *et al*., 2008). At the experimental conditions, butyric fat reduction was then compensated with inulin, probably enhancing the air bubbles stability during and after freezing process, even at reduced solids (sugar) content.

Melting properties

The adjusted second-order model had a highly significant ($P=0.0017$) effect and a high correlation ($R^2=0.8426$) for the melting rate. Based on the ANOVA results, inulin (X_2), sugar (X_3), butyric fat quadratic term (X_1^2) and sugar quadratic term (X_3^2) had a highly significantly ($P<0.01$) effect on this parameter. Butyric fat (X_1), inulin quadratic (X_2^2), butyric fat×sugar (X_1X_3) interaction, and inulin×sugar (X_2X_3) interaction presented a significantly ($P<0.05$) effect. In regression equation, most of the parameters presented negative sign meaning that the increase in there ingredients concentration decreased melting rate (Figure 2a). In the contour plot (Figure 2a), at a butyric fat fixed level (6.0%), how higher sugar concentrations increased melting rate, decreasing when sugar content decreased with the incorporation of inulin as solids compensator.

Melting properties are influenced by the three structural components that make up the dispersion phase of ice-cream: ice, air and fat. The amount of ice crystals formed depends on the freezable water and therefore on the amount of solids in the ice-cream. Since both butyric fat and sugar were reduced, according to the experimental design proposed, the inulin added to compensate solutes enhancing melting rate. This was also related to apparent viscosity and overrun values, since air bubbles act as an insulating medium that prevents rapid heat transfer from the medium to the ice crystals (Marshall *et al*., 2003; Sofjan and Hartel, 2004). The more air is incorporated (higher overrun), the slower the melting will be (Chang and Hartel, 2002; Caillet *et al*., 2003). Butyric fat had a marked effect on melting, and its interactions with sugar and inulin were related to the complex interactions during free water freezing. Fat and sugar were reduced and inulin incorporation reduced the melting rate of ice cream.

For the first drop fall during melting test, the adjusted second-order model had a highly significant effect (P=0.0001) and a good correlation (R^2= 0.8986). In ANOVA, butyric fat (X_1), butyric fat×inulin (X_1X_2) interaction and inulin×sugar (X_2X_3) interaction presented a highly significantly (P<0.01) effect on the time to first drop fall. Inulin (X_2), sugar (X3), inulin quadratic term (X_2^2), and the three interactions (X_1X_2, X_1X_3, and X_2X_3) present a significantly (P<0.05) effect on this parameter. According to the proposed regression equation, butyric fat had the stronger positive effect, where higher butyric fat content extend first drop time. Only butyric fat×inulin interaction had negative sign, i.e., their interaction decreased first drop times (Figure 2b). In contour plot, at a butyric fat fixed level (6.0%), it can be observed that at lower sugar concentrations at low inulin concentration, the first drop time increased (Figure 2b).

Reducing fat increased the melting rate of ice-cream (Roland *et al*., 1999; Akalin *et al*., 2008), but in reduced-fat ice-cream, the inulin acts as a stabilizer, due to its capacity to retain and immobilize large amounts of water, causing less crystallization and longer melting time (Caillet *et al*., 2003; Muse and Hartel, 2004; Meyer *et al*., 2011). Again, the stabilizing property of the inulin compensates the fat and solids reduction. Butyric fat presented as well the most marked effect on time for first drop fall, and the interaction of this ingredient with inulin and sugar on melting represents the interaction between all the soluble compounds during emulsion formation and ice crystals formation and stabilization. Butyric fat and sugar were counterbalance by inulin to extend the time for first drop fall.

Textural properties

The adjusted model for the hardness of the formulated ice-cream had a significantly (P=0.0108) effect and a high correlation (R^2=0.6763). Based on the ANOVA, only the butyric fat (X_1) linear term presented a highly significantly (P<0.01) effect on the model. Inulin (X_2), sugar (X_3), both quadratic terms for butyric fat (X_1^2) and sugar (X_3^2), and butyric

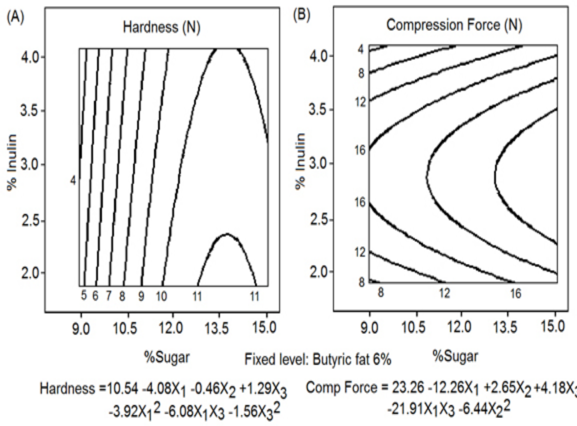

Figure 3. Contour plot and adjusted regression equation for (a) hardness and (b) compression force (X_1: Butyric fat, X_2: Inulin, X_3: sugar).

fat×sugar (X_1X_3) interaction presented a significantly (P<0.01) effect. In the regression equation, butyric fat and sugar concentration had an opposite effect on ice cream hardness. Since sugar linear term was positive, and its quadratic interaction negative, a reprimand effect of sugar on ice cream hardness was observed (Figure 3a). In contour plot, this effect can be observed since at lower sugar concentrations ice cream hardness remained practically constant at the studied inulin concentrations range, at a fixed butyric fat level (6.0%) (Figure 3a).

The model for compression force had a significantly (P=0.0204) effect and a high correlation (R^2=0.6977). The ANOVA showed that linear term for butyric fat (X_1) and butyric fat×sugar (X_1X_3) interaction presented a highly significant (P<0.01) effect on compression force, whereas both inulin (X_2) and sugar (X_3) linear terms and inulin quadratic term (X_2^2) had a significant effect (P>0.05) on this parameter. Regression equation presented negative sign for butyric fat, butyric fat×sugar interaction and inulin quadratic term (X_2^2) (Figure 3b). This means that butyric fat (and its interaction with sugar) affected the force necessary to compress the samples, but the most marked effect was provoked by inulin. Contour plot shown that an increase in inulin concentration and the decreased in sugar concentration, at a fixed butyric fat level (6.0%), reduced the force necessary to compress the samples (Figure 3b).

The two texture tests serve to determine the uniformity of the structure formed in the ice-cream. Penetration measures the hardness (force required to break the structure of the ice crystals, air bubbles and emulsified fat globules) of the ice-cream and compression test simulate the deformation that occurs during mastication between the palate and the tongue (Clarke, 2004). On reducing the amount

Table 2. Factors setting in ice cream formulation optimization (overall desirability =50.19%)

Factor	Response	Estimate value
Butyric fat= 7.0%	Apparent viscosity	4635.65
Inulin= 3.0%	Overrun	3.45
Sugar= 13.2%	Melting rate	0.0758
	First drop	48.51
	Hardness	8.37
	Compression force	19.71

of fat and solids a harder texture would be expected. When fat content is decreased and compensated with water, then ice crystals were larger, as the ice-phase volume was higher because of the increased water content. In same manner, as the amount of solutes decreases, the ice-phase volume increases, resulting in larger ice crystals, and a harder texture (Hartel, 1996; Clarke, 2004). The use of inulin, a branched polysaccharide of high molecular weight and higher capacity of interaction during ice crystallization, as compared to sugar, decrease ice-phase volume (Goff *et al*., 1993). Incorporation of inulin reduced water molecules mobility from the bulk aqueous phase to the ice crystal surface, resulting in a softer ice cream texture (Soukoulis *et al*., 2009). Texture is the result of components interaction during ice cream freezing, this is, ice crystals formation by the emulsified fat globules and air bubbles matrix. Since inulin compensate on one hand the butyric fat reduction (added water was retained by branched inulin), and on the other hand, sugar reduction (inulin as hygroscopic material that at lower concentration reduce freezable water), the resulting texture was softer (less hard and easy to compress) at lower sugar concentration with relatively low inulin content (around 3%).

Optimization of inulin, sugar and butyric fat levels to enhance textural and melting properties, looking for a softer ice cream texture with longer melting times, is shown in Table 2. Employing inulin (3%) as fat and sugar replacer, butyric fat content can be reduced from 10 to 7% (this is, 30%), and sugar can be reduced as well from 15 to 13.2% (this is, 12%).

Conclusion

Fat and sugar represents two main ingredients in ice cream formulation to define their physical characteristics. Higher apparent viscosity resulted in a more stable system with higher overrun, where inulin controlled available water. The improvement in melting properties reflected the stable state of the air bubbles-emulsified fat-ice crystals matrix, where the putative effect of inulin to retain water compensating solids and fat reduction, retarded ice crystals melting. In instrumental texture, inulin retained free water when butyric fat and sugar were reduced, resulting in smaller ice crystals reflecting a softer texture. At the experimental conditions proposed, inulin as functional ingredient (soluble fiber and prebiotic) can be employed to reduce 30% butyric fat content and 12% sugar content, in the formulation of low-fat reduced-sugar ice cream.

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