Fortification with iron chelate and substitution of sucrose by sucralose in light uvaia sherbet (*Eugenia pyriformis* Cambess): physical, chemical and sensory characteristics

Tales Márcio de Oliveira Giarola ·
Cristina Guimarães Pereira · Jaime Vilela de Resende

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Abstract In this work, iron fortified light uvaia sherbet, with low sucrose content, was developed and its physical, chemical and sensory characteristics were evaluated. The central composite rotational design (CCRD), applicable to the response surface methodology, was used to analyze the formulations. In the formulations, in addition of iron fortification (9 to 15 mg/100 g), the sucrose was substituted by micronized sucralose in a proportion of 66–94 %. The responses were analyzed with respect to changes in pH, total solids, ash, carbohydrates, proteins, calories, overrun, nucleation and thawing temperatures, rheological parameters and sensory attributes. Protein contents and acidity were similar in all formulations. There was a reduction of over 25 % in the caloric value. The rheological results showed pseudoplastic behavior and significant viscosity differences among the tested sherbets. In the overrun and thawing behavior results the sucrose concentration had a significant influence as the formulations with substitution by 28 g of sucralose/kg of sucrose showed greater air incorporation. In the flavor attribute there was not significance in relation to the iron fortification. Sherbets prepared with substitution of sucrose by sucralose and fortified with iron showed good acceptability, more stability and more resistant to thawing.

Keywords Iron fortification · Frozen dessert · Thermal properties · Microstructure · Sensory analysis

Introduction

Sherbet is defined as an edible frozen product or as a food product obtained from an emulsion of fat and protein, with or without the addition of other ingredients or substances, or from a mixture of water, sugar and other ingredients or substances that have undergone freezing (Brasil 1999). Sherbets are basically products elaborated with milk and/or dairy products and/or other food raw materials which contain only a small proportion of fats and proteins, which may be wholly or partially of non-milk origin and can be added to other food ingredients. Sherbets have wide acceptance in human nutrition, especially among children and young people where there is a high incidence of anemia. Similar to ice cream, sherbet is processed with a high amount of fruit pulp, therefore presenting a more acidic flavor. Moreover, it has lower overrun, i.e., less air incorporation (25 to 50 %), higher sugar content (sucrose), lower thawing point, and coarser texture than that of ice cream (Marshall and Arbuckle 1996).

Sucralose has a flavor profile most similar to that of sucrose, compared to all other sweeteners, but without the unwanted bitter/metallic aftertaste. Its carbon-chlorine bonds are stable and not hydrolyzed during digestion, being rapidly excreted in the feces. In their evaluation during 15 years, approximately 140 studies were conducted in animals and humans, and it was concluded that sucralose does not present any teratogenic effects, toxicity or carcinogenicity (Bortolozo and Quadros 2007). The sweetness perception is rapid and lasts for slightly longer period than that of sucrose without revealing a bitter or metallic aftertaste. It has high water solubility and thermal stability in acidic or aqueous medium as well as during storage. It is compatible with other food ingredients, including flavorings, spices and preservatives (Cândido and Campos 1996).

T. M. d. O. Giarola · C. G. Pereira (✉) · J. V. de Resende
Department of Food Science, Laboratory of Food Refrigeration,
Federal University of Lavras, Campus Universitário, P.O. Box 3037,
37200-000 Lavras, MG, Brazil
e-mail: crisgp13@yahoo.com.br

Iron is an attractive component for fortification, since its natural level in milk is relatively insignificant. However, the potential of iron to act as a catalyst in fat oxidation is quite high, making it necessary to ensure that it is added under conditions where this functionality is eliminated (Martínez-Navarrete et al. 2002; Umbelino and Rossi 2006). Copper, cobalt, manganese and vitamin C are needed for assimilation of iron (Martínez-Navarrete et al. 2002). Vitamin C is a potent activator of non-heme iron (foods of animal origin) absorption. A medium with ascorbic acid forms a soluble chelate with iron 3+. Absorption of iron contained in a diet can be multiplied by 5 times when it contains Vitamin C. This vitamin does not intervene only in the absorption of non-heme iron. It favors the incorporation of iron in ferritin and encourages the use of iron reserves, mainly of the spleen. The condition commonly associated with iron deficiency is called iron deficiency anemia (Lynch 1997; Cook and Reed 2001). The addition of iron to a food product is considered the best way to combat iron deficiency over the long-term since it does not require individual cooperation and the initial cost and maintenance is lower when compared to a pharmacological supplementation program (Baltussen 2004).

Brazil is rated a major producer of tropical fruits; however, the perishability of fruits restricts their trade. Therefore, processing technologies are necessary to increase shelf-life and reduce post-harvest losses (Karwowski et al. 2013). The uvaia is native and abundant in the Brazilian Atlantic Forest, has enormous potential for acceptability and success as a result of its high ascorbic acid content, being extremely palatable and with little or no use in the processing industry. The uvaia is absent from the market for reasons almost techniques: the fruit mash, rust and dries out easily. This practically prevents the uvaia marketing. The fruit is consumed *in natura*, in the form of juices, jellies and sweet paste. It presents around 90.7 % moisture; vitamin C content of 33 to 39.52 mg/100 g, 1.53 % acidity (TA), 1.16 g of this being citric acid/100 g, total soluble solids (TSS) of 7.5 °Brix and a TSS/TA ratio of 4.90 (Donadio 1997). This study aimed to develop a light uvaia sherbet fortified with different iron levels and evaluate the effect of adding iron chelate and substitution of sucrose by sucralose on its physical, chemical and sensory characteristics.

Materials and methods

This study was approved by the local ethics committee of Federal University of Lavras, approval CAA 12370113.2.0000.5148.

Formulations and production of sherbets

The production and maturation of the sherbets were conducted in batch (Arbuckle 1977). The ingredients were dispersed,

under stirring (3500 rpm), in water preheated to 50 °C using an industrial blender. The mixture was then pasteurized in a pan with rotating spindle at 72 °C/30 min and then homogenized in a two stage process (Manton-Gaulin DJ4, MantonGaulin Manufacturing Company, Everett, USA) at 17.5 and 2.5 MPa for the first and second stages, respectively. The temperature of the mixture was adjusted to 4 °C and kept at this temperature for 24 h to mature. All formulations were subjected to the same freezing conditions in a discontinuous horizontal batch freezer (Refrigás, Bauru, Brazil), with a 5 min beating time. All samples were packaged, placed in a refrigerated chamber at −25 °C for 48 h to complete the hardening stage and stored in a horizontal freezer at −30 °C.

The basic formulation was composed of 2 % milk fat (cream with 60 % fat, Verde Campo, Lavras, Brasil), 4 % total non-fat milk solids or NFMS (skim milk powder, 95 % solids, Cosulati, Pelotas, Brazil), 0.84 % sucrose (Companhia União, São Paulo, Brazil), 10 % dried corn syrup (38 DE, Corn Products, São Paulo, Brasil), 1 % stabilizer and emulsifier mix (Starmix Premium®, Kerry Brasil, Campinas, Brasil), 25 % uvaia pulp and 28.84 % water. After the addition of sucrose and iron, the mass was balance to 4000 g of the ready sherbet light mixture fortified with different iron concentrations by adding water to a total of 42.84 % for the sample containing the maximum sucrose by sucralose substitution.

Table 1 shows the formulations that were established to obtain the best concentration in iron fortification within the Recommended Daily Intake (RDI) range for iron adopted in Brazil that is from 8 to 16 mg/100 g with 10 % bioavailability (Brasil 2005). Table 1 also shows the substitution of sucrose by sucralose in order to obtain a substitution level in the range of 60 to 100 %, which corresponds to the development of products classified as “light”.

Experimental design

The experiment was conducted in a central composite rotational design (CCRD) applicable to the response surface methodology. The effect of the sucrose substitution by sucralose and the addition of iron were evaluated. The results of all of the analyzes were evaluated by the response surface method using Statistica 8.0 software, with the polynomial used to adjust the model defined by Eq. (1).

$$y = \beta_0 + \beta_1 X_1 + \beta_{11} X_1^2 + \beta_2 X_2 + \beta_{22} X_2^2 + \beta_{12} X_1 X_2 + \varepsilon \quad (1)$$

where β_0 , β_1 , β_{11} , β_2 , β_{22} , β_{12} , are the regression coefficients; X_1 is the iron concentration; X_2 is the proportion of substitution of sucrose by sucralose; and ε is the experimental error. The criteria used for the adaptation of the model were

Table 1 Formulations of iron fortification and substitution of sucrose by sucralose per 4000 g of sherbet

Formulation	CCRD		Iron	Sucralose	Sucrose
	Fe	Sucralose			
1	−1	−1	10 mg–7.60 g	0.6533 g	10 %–56.0 g
2	+1	−1	14 mg–10.64 g	0.6533 g	10 %–56.0 g
3	−1	+1	10 mg–7.60 g	0.8399 g	30 %–168.0 g
4	+1	+1	14 mg–10.64 g	0.8399 g	30 %–168.0 g
5	−1.41	0	9 mg–6.84 g	0.7466 g	20 %–112.0 g
6	+1.41	0	15 mg–11.40 g	0.7466 g	20 %–112.0 g
7	0	−1.41	12 mg–9.12 g	0.6159 g	6 %–33.6 g
8	0	+1.41	12 mg–9.12 g	0.8773 g	34 %–178.2 g
9	0	0	12 mg–9.12 g	0.7466 g	20 %–112.0 g
10	0	0	12 mg–9.12 g	0.7466 g	20 %–112.0 g
11	0	0	12 mg–9.12 g	0.7466 g	20 %–112.0 g
12	–	–	–	–	100 %–560.0 g

the determination coefficient values ($R^2 > 75\%$) and variance analyses.

Determination of overrun

The time that the mixture remained in the producer was 5 min for all the formulations. The overrun was determined according to the method described by Whelan et al. (2008). Equal volumes (50 ml) of the base mixture before whipping and of sherbet after whipping collected at the output of the producer were weighed and the overrun was calculated according to Eq. (2):

$$\text{Overrun (\%)} = \frac{\text{weight of sherbet mix} - \text{weight of sherbet}}{\text{weight of sherbet}} \quad (2)$$

Chemical composition of sherbets

The protein content was obtained by the determination of total nitrogen by distillation *MicroKjedahl* instrument (Aoac 2005), using the factor 6.25 for the protein concentration calculation. The total solids (TS) of the sherbets were quantified by the gravimetric method as a result of moisture loss in an oven at 105 °C until constant weight, according to the technique described by Association of Official Analytical Chemists - AOAC (2005). The amount of fat was determined using the Rose-Gottlieb method. The ash fraction was obtained gravimetrically evaluating the weight loss of the material subjected to heating at 550 °C in oven (Aoac 2005) and the total quantities of carbohydrates, by subtracting the total solids from the sum of total fat, ash and protein content (Aoac 2005). The calorific value was calculated according to the technique described by Anvisa (2005).

pH and rheological behavior of the samples

The pH of each sample was determined with a Micronal pH meter (Micronal, São Paulo, SP, Brazil). Rheological measurements of the samples were made immediately after mixing the iron, sucrose and sucralose and also after freezing, storage and thawing. The analyzes were performed using a Brookfield DVIII Ultra concentric cylinder rotational viscometer (Brookfield Engineering Laboratories, Stoughton, USA) with a 13R/RP small sample adapter (19.05 mm diameter and 64.77 mm depth, Brookfield Engineering Laboratories, Stoughton, USA) and an SC4-34 coaxial shear sensor (9.39 mm diameter and 24.23 mm in length; Brookfield Engineering Laboratories, Stoughton, USA). A thermostat bath (New Ethics, Vargem Grande Paulista, Brazil) was coupled to the viscometer to control the temperature of the sample at 5 °C. Analyses were made after the maturation period and after storage and freezing. The samples were subjected to an increasing deformation rate ramp, varying linearly from 1.59 to 4.88 (s^{-1}) for 18 min, 20 readings being taken. The rheological parameters were obtained by the Power Law model (Eq. (3)), using the Rheocalc software (version V.3.1, Brookfield Engineering Laboratories, Stoughton, USA).

$$\sigma = k\dot{\gamma}^n \quad (3)$$

Where σ is the shear stress (Pa) k is the consistency index ($Pa.s^n$), $\dot{\gamma}$ is the deformation rate (s^{-1}), and n is the flow index.

Freezing and thawing temperatures

The freezing and thawing temperatures were determined using differential scanning calorimeter (DSC-60A, Shimadzu, Tokyo, Japan) connected to a computer for data acquisition.

The system temperature control was conducted with liquid nitrogen. The instrument was calibrated for temperature and heat flow with indium ($T=156.6\pm6^\circ\text{C}$, $\Delta H=-30.25\text{ J/g}$) and zinc ($T=28.5\pm1.5^\circ\text{C}$, $\Delta H=104.71\text{ J/g}$). Approximately 3 mg of the samples were transferred to stainless steel pans that were then hermetically sealed. An empty pan was used as reference. The temperature protocol used was as follows: Samples were equilibrated at 20°C and frozen to -30°C , stabilized and heated from -30 to 25°C at a rate of 3°C min^{-1} .

Thawing behavior

To determine the melting behavior of the samples, a day prior to testing, the samples were transferred to a -15°C freezer and left overnight. The sherbet samples (51 g each) were put on a wire screen (with square openings of $0.3\times0.3\text{ cm}$) on top of a funnel that was attached to a cylinder. The sherbet was placed in a controlled temperature chamber at $20\pm1^\circ\text{C}$. The weight of the material that passed through the screen was recorded at 5 min time intervals for 90 min. The dripped weight (g) was plotted against the time (min) (Pereira et al. 2011).

Acceptance test and purchase intent

The sensory evaluation of sherbets was conducted with the participation of 65 untrained panelists. Samples of the eleven formulations were presented in 50 mL plastic cups coded with three digit random numbers. (Kemp, Hollowood, and Hort 2009; Stone and Sidel 2004). Each sample consisted of about 30 g of sherbet at temperatures between -8°C and -10°C . The randomized samples were presented at the same time to the panelists that were instructed to evaluate them initiating from left side. The procedures were conducted in individual booths with white light equivalent to daylight. The panelists evaluated the samples for appearance, flavor, texture, and global acceptance on a structured nine-point hedonic scale (1 = dislike extremely, 9 = like extremely). Purchase intent was analyzed by means of a five-point mixed structured scale (ranging from 1 = definitely will not buy to 5 = definitely will buy), according to Meilgaard, Civille, and Carr (2007). The PARAFAC (*Parallel Factor Analysis*) program (Nunes, Pinheiro, and Bastos 2011) was used for the analysis of sensory parameters.

Results and discussion

Analysis of formulations

Table 2 presents the correlation coefficients, the calculated F value and the regression coefficients for each order with their respective p -values for the significant variables involved in

Table 2 Analysis of the regression coefficients for significant variables in the sherbet formulations

	pH		Total solids		Carbohydrates		Caloric value		Overrun		n(AF)		k(AF)	
	Coef. of regression	p-value	Coef. of regression	p-value	Coef. of regression	p-value	Coef. of regression	p-value	Coef. of regression	p-value	Coef. of regression	p-value	Coef. of regression	p-value
β_0	4.310	0.000	17.113	0.000	11.896	0.000	76.546	0.000	63.517	0.000	0.406330	0.000368	0.413553	0.000052
β_1	-0.079	0.621	0.043	0.231	-0.011	0.657	0.035	0.823	1.195	0.249	-0.085379	0.03137*	-0.001479	0.943456
β_{11}	0.0137	0.476	-0.106	0.037*	-0.065	0.050*	-0.414	0.048*	0.299	0.796	0.033255	0.384783	0.019521	0.447200
β_2	-0.092	0.002*	1.390	0.000*	1.426	0.000*	5.404	0.000*	-1.757	0.113	0.058374	0.102697	0.031118	0.177601
β_{22}	0.0012	0.950	-0.035	0.391	-0.047	0.155	-0.113	0.556	-5.599	0.004*	0.028225	0.455762	-0.000599	0.980791
β_{12}	0.0025	0.910	-0.090	0.099	0.0375	0.311	-0.605	0.035*	1.748	0.235	-0.065000	0.176635	-0.120000	0.007842*
F_{calc}	7.682		393.05		733.02		263.72		7.35		5.23		6.31	
R^2	0.89		0.99		0.99		0.99		0.88		0.76		0.81	

* Significant at the 5 % confidence level

the different formulations applied in the complete codified model shown in Eq. (1).

The usual test of significance of the adjusted regression equation is the null hypothesis test, which involves the calculation of the *F* value and comparing this calculated value with the tabulated value, $F_{\alpha,p-1,N-p}$, where *N* is the number of observations, *p* is the number of adjusted parameters and α is the level of significance. If the calculated *F* value exceeds the tabulated $F_{\alpha,p-1,N-p}$ value, then it is inferred with an α level of significance that the variation accounted for by the model is significantly higher than the unexplained variation. In other words, a higher calculated *F* value indicates a better adjustment. It was observed that practically all of the calculated *F* values for the curve adjustments presented in Table 2 are above the tabulated *F* value, which for this experiment was 5.05, indicating that the parameters are significant (Khuri and Cornell 1996).

Another parameter presented in Table 2 is the coefficient of determination (R^2). The R^2 value is a measure of the proportion of the variation of the values observed around the average explained by the adjusted model. In variance analysis shown in Table 2, the variation percentage explained by the regression is above 75 %, but that value should not be compared to 100 % because of the contribution due to the pure error, which is a measure of the random error that affects the responses (Barros Neto, Scarminio, and Bruns 1996).

Table 2 shows that the concentration of sucrose has a significant negative effect on the linear form of the model proposed to describe the response of the pH variation of the formulations. This behavior can be seen in the response surface in Fig. 1. Figure 1 shows that the pH value decreases with the increasing level of sucrose substitution by sucralose.

Fig. 1 Results of pH as a function of the substitution of sucrose and the iron concentrations in the formulations of fortified sherbets ($R^2=0.885$)

Sucralose (4-chloro-4-deoxy- α -D-galactopyranosyl-1,6-dichloro-1,6-dideoxy- β -D-fructofuranoside) is a chlorinated derivative of the disaccharide sucrose; in which three hydroxyl groups are replaced by chlorine atoms. Sucralose is about 600 times sweeter by weight than sucrose. Sucrose is classified as non-reducing sugars that are carbohydrates which cannot be oxidized by mild oxidizing agents such as Fe(III) or Cu(II). In the work of Sharma et al. 2012, the oxidation kinetics of sucrose and sucralose by ferrate (VI) was studied. The evaluation of pseudo-first-order rate constants, k (s^{-1}) depending on the carbohydrate concentration showed larger values of *k* for sucralose when compared with the sucrose in the same concentration. The increase in the value of *k* is related to the pH value decreased. Therefore, the increase in sucralose concentration reduced pH values of systems.

Figure 1 also shows that the addition of iron decreases the pH. This decrease is related to the acidities of iron solutions and to exchanges between iron ions and micellar bound H^+ (Gaucheron 2000).

Among the chemical parameters of sherbets, the values obtained for the parameters ash, crude protein content and acidity showed significant lack of fit, analysis using the response surface methodology not being possible. The R^2 and $F_{calculated}$ values indicated that less than 75 % of the observed variation in ash content, crude protein content and acidity could be related to variations in the iron and sucrose concentrations.

The results of the regression analysis of total solids values, carbohydrate content and caloric values of the formulations with sucrose and iron fortification are shown in Table 2. Table 2 also shows that sucrose has a significant positive effect on the linear form, iron concentration has a negative effect on the quadratic form and the interaction between these factors has a negative effect on the model proposed to represent the response of the total solids content variation in the tested sherbet formulations. Figure 2a shows the variation of the total solids content as a function of the substitution of sucrose by sucralose and the iron concentrations used in the formulations.

At these concentrations the interactive effects between the factors can be verified, with maximum peaks at the surface extremes and minimum at the central iron concentrations points. Similar behavior to that observed for total solids can be found in the regression analyzes for the carbohydrate content (Fig. 2b). In this case, the higher concentration of sucrose with positive and significant effect on the linear model used to build the response surface and the higher iron concentration with a negative effect on the quadratic model were the obvious factors. Fig. 2c shows the response surface for the caloric value of the formulations whose regression analyzes parameters are shown in Table 2. The significant factors were the substitution level of sucrose by sucralose, the iron concentration and the interaction between these factors. The surface

Fig. 2 **a** Results of total solids content ($R^2=0.990$), **b** carbohydrates ($R^2=0.998$) and **c** the calorific value ($R^2=0.990$), as a function of the sucrose substitution and the iron concentration in formulations of fortified sherbets

of Fig. 2c shows the minimum caloric value points with iron concentrations around 12 mg.

Figure 3a shows a comparative diagram of air incorporation obtained in each formulation in relation to the control.

Figure 3a shows that the average overrun was between 56 and 66 % among formulations that received iron fortification and substitution of sucrose by sucralose, while the control

Fig. 3 **a** Comparison, with the control formulation, of air incorporation into the sherbet formulations fortified with iron and different substitution levels of sucrose by sucralose. **b** Results of the air incorporation (overrun) as a function of the sucrose substitution and the iron concentrations in fortified sherbets ($R^2=0.88$)

sherbet averaged a 28.5 % overrun. This result shows that there were reactive or interactive effects between sucrose, sucralose and iron that favored the formation and emulsion stability. Table 2 shows the regression analysis results in which 11 sherbet formulations fortified with iron and different substitution levels of sucrose by sucralose established for the CCD were considered. The results in Table 2 show that only the substitution level of sucrose by sucralose had a significant effect on the quadratic form of the model proposed to represent the behavior of air incorporation in the formulations. Figure 3b shows that the overrun values had their peaks in formulations that had about 80 % sucrose substituted by sucralose, independent of the iron fortification concentration used. The overrun behavior can be explained by the increased viscosity of the matrix and this viscosity increase enables greater air incorporation. Eisner, Wildmoser, and Windhab

(2005); Chang and Hartel (2002) showed that increasing the viscosity of the unfrozen matrix allows, in addition to a higher overrun, more micro dispersion and stabilization of the air bubbles (Pereira et al. 2011).

The results of the rheological parameters were obtained by the fitting of the experimental data applying the power law model for all base mixture and control formulations before and after freezing. The power law model was a good fit, which indicated that all the formulations tested had a pseudoplastic behavior ($n < 1$). The results of the determination coefficients (R^2) for the flow index (n) ($R^2 = 0.27$) and the consistency index (k) ($R^2 = 0.29$) parameters, of the mixtures before freezing, indicated a significant lack of fit and response surfaces could not be obtained.

The results of the rheological parameters obtained by fitting the experimental data by applying the power law model for all sherbets after freezing, storing and thawing are also shown in Table 2. Observe that in this case the flow index (n) as well as the consistency index (K) were significant, showing that there was a change in rheological behavior caused by the beating and cooling inside the production equipment. Significant factors were the iron concentration for the flow index (n) and the interaction between the iron concentration and sucrose concentration for the consistency index (K). Figure 4a shows the response surface for the flow index (n) and Fig. 4b shows the response surface for the consistency index (K) for the formulations of sherbets after freezing, storage and thawing.

Figure 4a shows that lower flow index (n) values are obtained with iron concentrations of around 12 mg of and with higher levels of sucrose substitution by sucralose. This indicates that in these formulations of thawed sherbets, the systems deviate from a Newtonian fluid behavior ($n = 1$). Figure 4b shows that the consistency index (K) or the viscosity of the system increases with increased concentrations of sucrose and iron in the formulations. Figure 4b also shows that in formulations where around 80 % of the sucrose is substituted by sucralose, the consistency index (K) does not undergo a large variation when the iron concentration increases. This indicates that with 80 % substitution of sucrose by sucralose it is possible to manipulate, i.e. there is freedom for variation in the iron fortification level without changing the rheological properties of the systems.

Optimization of formulation

The optimum condition for formulation of sherbets with iron fortification and sucrose substitution was determined to obtain maximum pH, total solids, overrun, consistency index (KAF) with minimum carbohydrates, caloric value and flow index (n AF). The optimum condition range for the extraction was determined by superimposing the contour surfaces of all the analyzed

Fig. 4 **a** Flow index (n) ($R^2 = 0.76$) values and **b** consistency index (K) ($R^2 = 0.81$) values of sherbets, frozen, stored and thawed, as a function of sucrose level substitution and iron concentrations

results. Figure 5a and b show the overlaying contour plots for the responses which were evaluated as a function of iron concentration at constant sucrose substitution (10.0 %) (Fig. 5a); and as a function of sucrose concentration at constant iron concentration (12 mg) (Fig. 5b).

These graphs show the best combination of factors for the sherbet formulation. Figure 5a demonstrates that the iron concentration of 11.5–15.0 mg is the range with the best combinations of factors. The shaded area in the graph with the six factors is the optimum area of sherbet formulations that results in a higher overrun, lower carbohydrates and caloric value, higher viscosity after freezing and, most importantly, a

Fig. 5 Optimal superposition region of the contour graphs of six responses evaluated as **a** a function of the iron concentration at a constant sucrose substitution level of 10 %, and **b** as a function of sucrose substitution level at a constant iron concentration 12 mg. The shaded area in the graph is the optimum area of extraction conditions

higher iron concentration. Figure 5b shows that the shaded area corresponding to the optimum sucrose substitution conditions is in the range from 0 to 20 %.

Analysis of the optimized formulation

Rheological behavior

Figure 6a shows the rheogram which relates the apparent viscosity and the deformation rate of sherbet base mixtures fortified with 12 mg iron and different substitution levels of sucrose sucralose before freezing. This characteristic is typical of a non-Newtonian pseudoplastic behavior (Chhabra and Richardson 2008; Pereira et al. 2011). In Figure 6 it can be seen that there was no large difference between the formulation viscosities with higher levels of sucrose substitution and

Fig. 6 The relationship between the apparent viscosity and deformation rate of sherbets fortified with 12 mg iron and different levels of sucrose substitution by sucralose **a** before freezing and **b** after freezing

among these and the control, the biggest difference in initial viscosity is attributed to an interaction between iron and sucralose.

Figure 6b shows the relationship between viscosity and deformation rate of the sherbets thawed after freezing and storage. It is noted that the control formulation showed higher shear stress when compared to formulations containing iron fortification and replacement of sucrose by sucralose. In this case the sucrose acts as a bodying agent in the emulsion, increasing its viscosity (Varnam and Sutherland 1994). Figure 6b shows that the apparent viscosity increases with increasing concentration of sucrose, and that this increase is reinforced by the presence of iron, as evidenced by the response surface analysis. Viscosity increases with increasing concentration for two reasons. First, solvent reduction reduces the intermolecular lubrication in such a way that friction increases.

Second, there is hydrate formation by the ions and molecules because the solvent is no longer sufficient for saturating all of the molecules at high concentrations, and they begin to form aggregates (Conceição et al. 2012; Rao 1977).

When comparing the differences between the rheological behavior of the unfrozen and frozen/thawed sherbets, the higher values after thawing can be attributed to the size of suspended particles in the sherberts (Bezerra, Fernandes, and Resende 2014; Pelegrine et al. 2002). With ice as the cooling and structural water separation, the reduction in the temperature leads to a reduction in the molecular distances due to an increase in the intermolecular forces.

Nucleation and thawing temperatures

Figure 7 shows the cooling and heating thermogram of the formulation containing 14 mg of iron and 90 % sucrose substitution by the sucralose. In this thermogram a peak is observed representing nucleation (T_N) during the cooling process and a peak at the thawing temperature (T_D) during the heating process.

Comparing the nucleation temperatures obtained for the control sample $T_N = -13.10$ °C and $T_D = -3.12$ °C with those of the sample with 12 mg of iron fortification and 20 % sucrose substitution $T_N = -14.05$ °C and $T_D = -3.30$ °C, respectively, and the results for all other formulations (data not shown), it was concluded that only sucrose had a significant effect on the nucleation temperature. When a large number of small crystals exist, they interrupt the flow of the matrix because the thawing fluid must flow around more obstacles. The

result is that the sherbet thawing rate occurs more slowly (Pereira et al. 2011).

Thawing behaviors

The thawing of the sherbet involves phenomena of heat transfer and mass. The heat penetrates gradually from outside to inside the sherbet, causing the thawing of the ice crystals. The water produced is diffused in the non-frozen matrix, through which it flows via the complex microstructure and finally dripping occurs (Muse and Hartel 2004; Soukoulis, Chandrinos, and Tzia 2008). The behavior of sherbets produced with iron fortification and partial substitution of sucrose by sucralose during the thawing, compared to the control sherbet, is shown in Fig. 8.

Figure 8 shows that in the first 30 min the sherbet formulations showed behavior similar to the control. From that point on it can be seen that the thawing rate of the control sherbet was more pronounced than that of the other formulations. The effects of the substitution of sucrose by sucralose and iron fortification in uvaia sherbets on reducing the thawing rate compared with the control sample was clearly observed in Fig. 9.

The increased matrix viscosity increases flow resistance and therefore more time is needed for the water to be diffused through the microstructure of the ice cream (Muse and Hartel 2004). The fact that the formulations with iron tri-chelate and the presence of sucralose having shown similar behavior during thawing, of the viscosity increasing and the crystal size decreasing as the sucrose level rises, are related to overrun and microstructural components such as distribution of air bubbles

Fig. 7 Representative thermogram of nucleation (T_N) and thawing (T_D) temperature of 3.370 mg of base sherbet mix containing 14 mg of Fe and 10 % of sucrose substitution

Fig. 8 Thawing behavior of 51 g of the formulations of sherbets fortified with iron and different substitution levels of sucrose by sucralose in relation to the control formulation

Fig. 9 Photographs taken during the thawing to room temperature (20 ± 1 °C) of the control sample (a) and the iron fortified (12 mg) and 80 % sucrose replacement (b) at the times of (1) 20 (2) 50 and (3) 90 min

and the size and destabilization degree of fat globules (Sofjan and Hartel 2004).

Acceptance test and purchase intent

An acceptance and purchase intent test was conducted with 65 panelists between 18 and 60 years of age, 64 % female and 36 % male. Data were analyzed by response surface and using PARAFAC (*Parallel Factor Analysis*) (Nunes, Pinheiro, and Bastos 2011) which is one decomposition methods for multi-dimensional data and can be considered a generalization of principal component analysis (PCA) for higher order data that unites all formulations with all the attributes for result verifications.

Data for appearance, flavor and texture were subjected to regression analyzes according to the CCRD proposed and the results showed that none of the evaluated sensory parameters was significant in the response surface methodology, it not being able to associate the variations in the concentrations of sucrose and iron. The analyze results for the parameters global appearance (GA) and purchase intention (PT) were also evaluated in the sensory analysis indicated a significant lack of fit and response surfaces could not be obtained.

Figure 10 displays the PARAFAC. Figure 10 shows that there is a tendency towards concentration of points around Formulation 4, containing 14 mg of iron and 10 % sucrose.

Each point represents a consumer in relation to all sensory attributes evaluated. It appears that there is a general distribution of panelists being that no attribute achieved a significant clustering of testers in its surroundings.

Fig. 10 PARAFAC sensory attributes regarding uvaia sherbet formulations with substitution of sucrose by sucralose and fortified with iron

Conclusion

The best combination of factors for the sherbet formulation demonstrates that the iron concentration of 11.5–15.0 mg is the range with the best combinations of factors. The formulations that results in a higher overrun, lower carbohydrates and caloric value higher viscosity after freezing and, most importantly, a higher iron concentration corresponding to the optimum sucrose substitution conditions is in the range from 0 to 20 %.

The results for the rheological behavior, overrun, thawing and thermal properties, show that around 80 % of substitution allows to manipulate the fortifying agent within the parameters set by national and international standards, without changing the rheological and thermal properties, significantly improving the thawing rate of sherbets.

Based on the results presented, it is concluded that the substitution of sucrose by sucralose should be around 80 % in order for the iron fortification to reach the maximum set by the norms, obtaining a light product of good acceptance and nutritional value.

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