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Antioxidant properties, technological and physicochemical characteristics of milk ice cream with addition of camu-camu pulp

Propriedades antioxidantes, características tecnológicas e físico-químicas de sorvetes de leite com adição de polpa de camu-camu

Propiedades antioxidantes, características tecnológicas y fisicoquímicas del helado de leche con adición de pulpa de camu-camu

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Abstract
Camu-camu is an exotic fruit, known for having high concentrations of vitamin C and bioactive compounds. In order to diversify the consumption of this fruit, this study developed milk ice cream formulations and evaluated the effect of the addition of different concentrations of camu-camu pulp (20 to 26%), sugar (12 to 14%) and defatted dry extract (DDE) (12 to 16%) through a mixture design. Ice creams were evaluated by analyses of *overrun*, density, melting time, *ratio*, and cost. Three formulations were defined based on maximum melting time and *ratio* and minimum cost. The ice creams formulations defined by the experimental design consisted of the following proportions of pulp, sugar and DDE (%): 26:12:12, 20:14:16 and 24:14:12. These formulations were evaluated in relation to their physicochemical and technological characteristics, antioxidant and reducing capacity and sensory acceptance. The results showed that all formulations had high concentrations of antioxidant compounds and vitamin C and the formulation with the addition of camu-camu pulp lower than 24% had greater sensory acceptance. Therefore, it is concluded that the addition of camu-camu pulp in milk ice cream is interesting from a nutritional point of view, as it can increase the content of nutrients and minerals in the final blend.
Keywords: Food ingredients; Milk and dairy products; Antioxidants; Bioactive compounds; Sensory analysis.

Resumo
O camu-camu é uma fruta exótica, conhecida por possuir altas concentrações de vitamina C e compostos bioativos. Visando diversificar o consumo dessa fruta, o presente estudo desenvolveu formulações de sorvete de leite e avaliou o efeito da adição de diferentes concentrações de polpa de camu-camu (20 a 26%), açúcar (12 a 14%) e extrato seco desengordurado (ESD) (12 a 16%) através de um planejamento de mistura. Os sorvetes foram avaliados pelas análises de *overrun*, densidade aparente, tempo de derretibilidade, *ratio* e valor do custo estimado. Três formulações foram definidas com base no maior tempo de derretibilidade, *ratio* e custo mínimo. As formulações de sorvetes definidas pelo delineamento experimental consistiram nas seguintes proporções de polpa, açúcar e ESD (%): 26:12:12, 20:14:16

e 24:14:12. Essas formulações foram avaliadas quanto às suas características físico-químicas e tecnológicas, capacidade antioxidante e redutora e aceitação sensorial. Os resultados mostraram que todas as formulações apresentaram altas concentrações de compostos antioxidantes e vitamina C e a formulação com adição de polpa de camu-camu inferior a 24% apresentou maior aceitação sensorial. Portanto, conclui-se que a adição de polpa de camu-camu ao sorvete de leite é interessante do ponto de vista nutricional, uma vez que pode aumentar o teor de nutrientes e minerais na mistura final.
Palavras-chave: Ingredientes para alimentos; Leite e laticínios; Antioxidantes; Compostos bioativos; Análise sensorial.

Resumen
Camu-camu es una fruta exótica, conocida por tener altas concentraciones de vitamina C y compuestos bioactivos. Con el fin de diversificar el consumo de esta fruta, este estudio desarrolló formulaciones de helado de leche y evaluó el efecto de agregar diferentes concentraciones de pulpa de camu-camu (20 a 26%), azúcar (12 a 14%) y extracto seco desgrasado (ESD) (12 a 16%) mediante una planificación mixta. Los helados se evaluaron mediante análisis de esponjamiento, densidad aparente, tiempo de fusión, *ratio* y valor de costo estimado. Se definieron tres formulaciones basadas en el tiempo de fusión más largo, el *ratio* y el costo mínimo. Las formulaciones de helado definidas por el diseño experimental consistieron en las siguientes proporciones de pulpa, azúcar y ESD (%): 26:12:12, 20:14:16 y 24:14:12. Estas formulaciones fueron evaluadas por sus características fisicoquímicas y tecnológicas, capacidad antioxidante y reductora y aceptación sensorial. Los resultados mostraron que todas las formulaciones tenían altas concentraciones de compuestos antioxidantes y vitamina C y la formulación con la adición de pulpa de camu-camu por debajo del 24% mostró una mayor aceptación sensorial. Por tanto, se concluye que la adición de pulpa de camu-camu al helado de leche es interesante desde el punto de vista nutricional, ya que puede incrementar el contenido de nutrientes y minerales en la mezcla final.
Palabras clave: Ingredientes alimentarios; Leche y productos lácteos; Antioxidantes; Compuestos bioactivos; Análisis sensorial.

1. Introduction

Camu-camu (*Myrciaria dubia* (H.B.K.) McVaugh) is an exotic fruit found mainly on the banks of rivers and lakes in the Amazon rainforest. Peru is the leading country of production and commercialization, however, neighboring countries such as Venezuela, Brazil and Colombia have expanded the area of cultivation of this fruit (Kaneshima et al., 2017).

The growing interest of the scientific community in camu-camu is justified by its high concentrations of ascorbic acid (vitamin C) and other antioxidant compounds such as anthocyanins, carotenoids, and phenolic compounds. Camu-camu fruits also have considerable amounts of minerals such as potassium, calcium and zinc and various types of amino acids such as valine, leucine, and serine (Aguiar & Souza, 2018; Cunha-Santos et al., 2019; Fujita et al., 2017).

The consumption of foods rich in antioxidant compounds is important in the prevention of several diseases such as arthritis, cancer, and coronary heart disease. In addition to having antioxidant activity, camu-camu has been associated with other protective effects, such as anti-hyperglycemic, antimicrobial, and anti-hypertensive (Azevedo et al., 2019). Despite the nutritional value of camu-camu, this fruit has high perishability and acidity, factors that make its consumption in natura limited. Thus, the camu-camu fruits are commonly processed in the form of frozen pulp, which facilitates their commercialization to distant locations, in addition to increasing the income of producers and diversifying their use as an ingredient in the production of other food products (Conceição et al., 2020; Fidelis et al., 2020).

New forms of processing and applications of camu-camu have been explored in the food industry in order to diversify its consumption and meet the growing demand for products with functional appeal, however, there are few reports of its application in the production of dairy products (Fidelis et al., 2020). Ice cream is a dairy product with wide acceptance by all age groups, being the most consumed product in the dessert category. Currently, the ice cream market aims to offer more nutritious products, with reduced caloric value and with diversified flavors, among which involve new raw material options and technological innovations (Gremski et al., 2019).

In this context, the objective of this study was to develop milk ice cream formulations with the addition of camu-camu pulp through extreme vertex mixing design, combining different proportions of defatted dry extract, sugar and camu-camu pulp.

2. Methodology

2.1 Samples

The camu-camu pulp (*Myrciaria dubia* (H.B.K.) Mc Vaugh) was donated by the Docefrutas® Company. The fruits were harvested in the State of Pará/Brazil, at the degree of ripeness between intermediate and ripe. The pulp was kept frozen at -18 ºC until the moment of use. Other ingredients for ice cream preparation, such as powdered milk (Nestlé®), skim milk powder (Nestlé®), refined sugar (União®), emulsifier (Emustab - Selecta®) and stabilizer (Liga Neutra - Selecta) ®) were obtained in the local market (Londrina/Brazil).

2.2 Preparation of camu-camu ice cream

Ice cream formulations were obtained using extreme vertex design, with variation in the concentration of camu-camu pulp, sugar, and defatted dry extract (DDE). The concentrations of the other ingredients used were kept constant. The fat content of ice cream was standardized at 3%, which was adjusted by mixing whole and skim milk powder. The maximum and minimum limits adopted in the experimental design were determined based on preliminary tests and resulted in values for camu-camu pulp from 20 to 26% (w/w), sugar from 12 to 14% (w/w) and DDE from 12 to 16% (w/w). The nine formulations determined are shown in Table 1.

Table 1 - Mixing design of camu-camu ice cream formulations with variation in the concentrations of camu-camu pulp, sugar and defatted dry extract (DDE) content.

Formulation	Pulp (x_1)	Sugar (x_2)	DDE (x_3)	Pulp (w/w)	Sugar (w/w)	DDE (w/w)
S1	0.40	0.28	0.32	20	14	16
S2	0.52	0.24	0.24	26	12	12
S3	0.48	0.28	0.24	24	14	12
S4	0.44	0.24	0.32	22	12	16
S5	0.48	0.24	0.28	24	12	14
S6	0.44	0.28	0.28	22	14	14
S7	0.50	0.26	0.24	25	13	12
S8	0.42	0.26	0.32	21	13	16
S9 (CP*)	0.46	0.26	0.28	23	13	14
S10 (CP*)	0.46	0.26	0.28	23	13	14

*CP = Central point. Source: Authors.

The model coefficients were evaluated using F test for the analysis of variance ANOVA ($p < 0.05$), the adjusted determination coefficients (R^2adj) were considered with the p-value.

The extreme vertex design allowed to test two models, a linear model, and a quadratic model according to the equations:

$$\hat{y} = b_0 + b_1 x_1 + b_2 x_2 + b_3 x_3 \qquad (1)$$

$$\hat{y} = b_0 + b_1 x_1 + b_2 x_2 + b_3 x_3 + b_{12} x_1 x_2 + b_{13} x_1 x_3 + b_{23} x_2 x_3 \quad (2)$$

with restriction $x_1 + x_2 + x_3 = 1$

The manufacture of camu-camu ice creams followed the methodology proposed by Clark (2015). Initially, all dry, liquid, and doughy ingredients were weighed (Table 2). Then, the liquid ingredients were mixed, heated to 60 ºC and added to the dry ingredients, resulting in a mixture called syrup. The previously heated syrup was homogenized in a domestic blender (Steel II 227, Faet®, Brazil) for 5 min. Then, the mixture was pasteurized at 75 ºC for 15 min and cooled in an ice bath until reaching 4 ºC. The resulting syrup was matured for 12 h at 4 ºC. After maturation, the camu-camu pulp was added and homogenized in a mixer (BPA, ARNO®, Brazil) for 10 min. The container was kept in an ice bath to guarantee the syrup temperature at 10 ºC. Finally, the syrup was frozen in an ice cream maker (R. Camargo®, Pasta producer, São Carlos - SP) under stirring until it reached a temperature of -7 ºC. The ice cream was immediately packaged in polypropylene pots with a capacity of 1.5 L. The ice cream pots of each formulation were kept in a freezer (Metalfrio®, DA420, Brazil) at -18 ºC.

Table 2 - Proportion of ingredients used in camu-camu ice cream formulations obtained through extreme vertex design with variation in the concentrations of camu-camu pulp, sugar and defatted dry extract (DDE) content.

Ingredients	S1	S2	S3	S4	S5	S6	S7	S8	S9
Camu-camu pulp (%)	20	26	24	22	24	22	25	21	23
Sugar (%)	14	12	14	12	12	14	13	13	13
Powdered milk (%) Skim milk powder (%)	16	12	12	16	14	14	12	16	14
Emulsifier (%)	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7
Stabilizer (%)	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7
Water (%)	48.6	48.6	48.6	48.6	48.6	48.6	48.6	48.6	48.6
Total	100	100	100	100	100	100	100	100	100

Source: Authors.

2.3 Technological characterization of ice cream

The determination of air incorporation (*overrun*), density and melting time was performed for the nine ice cream formulations. The *overrun* analysis was performed as described by Goff (2015). The procedure consisted of weighing the ice cream syrup before (M mixing) and after (M ice cream) freezing. The ratio between the difference in the mass of the syrup before and after freezing in relation to the weight of the ice cream determined the *overrun* (Equation 3).

$$\%\ overrun = \frac{M\ mixing - M\ ice\ cream}{M\ ice\ cream} \times 100 \quad (3)$$

Density was determined by the ratio of mass and volume of 100 mL of ice cream, expressed in g. mL-1. The melting time was determined according to Granger et al. (2005). The melted dough was monitored every 5 min until the total melting of ice cream. The results were expressed in time (min) necessary to melt 100 g of sample. The *ratio* was determined by the ratio of soluble solids content and acidity.

Considering the results of the experimental design, technological and *ratio* analyses, three ice cream formulations were selected for physicochemical analyses, determination of antioxidant and reducing capacity and sensory acceptability.

2.4 Physicochemical characterization

The physicochemical characterization was performed for the three selected formulations, with measurements in triplicate.

The moisture, proteins, acidity (% lactic acid), lipids and ash contents were determined according to AOAC (2019). The carbohydrate content was calculated by difference (100% minus the percentages of moisture, proteins, lipids, and ash).

The pH was measured using a digital pH meter (GEHAKA, model PG2000, Brazil) and the concentration of total soluble solids (ºBrix) was measured using a refractometer (Mettler Toledo, model LiquiPhysics ™ Excellence RM40, Brazil).

The quantification of vitamin C was determined by the method of Tillmans, modified by Benassi & Antunes (1988). The energy value was calculated using the conversion factors of 4 kcal g-1 for carbohydrates and proteins, and 9 kcal g-1 for lipids, with results expressed in Kcal 60 g-1 (Marshall et al., 2003).

2.5 Determination of antioxidant and reducing capacity

The analyses to determine the antioxidant and reducing capacity consisted of the determination of FRAP (ferric ions that reduce the antioxidant power), described by Benzie & Strain (1996), DPPH (2,2-diphenyl-1-picryl-hydrazyl) described by Casagrande et al. (2007) and ABTS [2,2'-azino-bis acid (3-ethylbenzothiazoline-6-sulfonic acid)] described by Re et al. (1999). The procedure for determining reducing compounds were performed according to Singleton et al. (1999).

The extraction of antioxidant compounds was performed according to Rufino et al. (2011). The absorbance readings were read on a spectrophotometer (Thermo ScientificTM, model GENESYS 10S UV-Vis, United States) at wavelengths of 595 nm for FRAP, 517 nm for DPPH, 734 nm for ABTS and 765 for the determination of compounds reactive to Folin-Ciocalteu.

For FRAP, DPPH and ABTS analyses, standard Trolox curves (+/-) (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) were used with purity greater than 98%, the results were expressed in μmol TEAC (Trolox equivalent antioxidant capacity) per 100 g of sample. For the analysis of compounds reactive to Folin-Ciocalteu, gallic acid (3,4,5 hydroxybenzoic - Sigma G7384) was used to construct the standard curve and the result was expressed in mg of equivalent gallic acid (GAE) per 100 g of sample. All analyses were performed in triplicate.

2.6 Sensory analysis

The sensory analysis of the three selected ice cream samples was performed as described by Villanueva et al. (2005). Thirty-two grams of each sample were served. The samples were evaluated on a scale of 1-10 in relation to appearance, aroma, color, flavor, texture, and overall appearance. Additionally, the evaluator was also invited to answer a questionnaire regarding the frequency of consumption of ice cream. Seventy untrained adults participated in the sensory test. Before the evaluation, the ice creams were subjected to microbiological analysis to verify the safety of the samples. The project was approved by the ethics and research committee - UTFPR, according to CAAE 90720018.8.0000.5547.

2.7 Statistical analysis

The Software Statistica® 10.0 program (StatSoft, 2010) was used to estimate the models of extreme vertex design, *ratio* and cost.

The results of physicochemical analysis, antioxidant and reducing capacity, and sensory analysis were subjected to analysis of variance (ANOVA), with means compared by the Tukey test at the level of 5% significance, using the Software Statistica® 10.0 program (StatSoft, 2010).

3. Results and Discussion

3.1 Analysis of experimental design and choice of ice cream formulations

Table 3 shows the results of the analyses of *overrun*, density, melting time and *ratio* of the nine formulations.

Table 3 - Average values of *overrun*, density, melting time and *ratio* of camu-camu ice cream formulations obtained from the effect of the mixture of camu-camu pulp (*x1*), sugar e (*x2*) and defatted dry extract (DDE) content (*x3*) using the extreme vertex design.

Formulation	Pulp (x_1)	Sugar (x_2)	DEE (x_3)	*Overrun (%)*	Density (g/ mL)	Melting time (min)	*Ratio*
S1	0.40	0.28	0.32	45.73 ± 8.39	0.729 ± 0.04	47 ± 5.77	53.60
S2	0.52	0.24	0.24	52.93 ± 4.14	0.707 ± 0.01	63 ± 2.89	44.09
S3	0.48	0.28	0.24	47.79 ± 6.47	0.715 ± 0.00	53 ± 2.89	53.15
S4	0.44	0.24	0.32	53.11 ± 3.56	0.728 ± 0.01	47 ± 2.89	54.43
S5	0.48	0.24	0.28	40.04 ± 6.75	0.786 ± 0.05	53 ± 2.89	49.00
S6	0.44	0.28	0.28	44.20 ± 3.03	0.755 ± 0.02	45 ± 0.00	49.63
S7	0.50	0.26	0.24	50.87 ± 1.75	0.726 ± 0.01	48 ± 2.89	42.64
S8	0.42	0.26	0.32	40.27 ± 6.99	0.767 ± 0.03	42 ± 2.89	56.39
S9	0.46	0.26	0.28	39.02 ± 1.59	0.768 ± 0.01	43 ± 2.89	48.91
S10	0.46	0.26	0.28	39.23 ± 1.53	0.788 ± 0.02	43 ± 2.89	50.18

Source: Authors.

The results presented for the analyses of *overrun*, density, melting time and *ratio* (Table 3), suggests that these analyses do not depend on an isolated factor, but depend on the balance of the proportions of the ingredients used.

All samples showed density in accordance with Brazilian legislation, where ice cream must have a minimum density of 475 g L-1 (Brasil, 2005). According to Marshall et al. (2003), the density of the ice cream mixture varies according to the volume and its composition. However, in this study, not all treatments showed density values proportionally inverse to *overrun* after freezing, possibly due to the heterogeneous dispersion of air bubbles incorporated in the ice cream mass, when larger, dispersion can occur and consequently provides an increase in density during storage.

The melting time is represented by the ratio of the amount of ice cream and the total time it takes to melt. This analysis depends on several factors such as size, distribution of ice crystals, distribution of air bubbles, and fat destabilization (partial coalescence and agglomeration at the air bubble interface). Bahramparvar & Tehrani (2011) emphasize that the amount and size of air bubbles are important factors that influence the melting time and are related to the presence of stabilizers, which contribute to the delay in the melting time. In addition, low-fat ice creams tend to melt faster. The proportions and interactions of components such as fat, proteins, stabilizers, and ice crystals form a network around air bubbles and tend to increase the product's resistance to melting (Muse & Hartel, 2004). We can observe that acidity is also related to melting since acidic ice creams demand lower freezing temperatures, making melting difficult. Formulations S2 and S7 that received higher amounts of pulp (more acidic) showed a lower value for the *ratio* and consequently longer melting time.

The parameters *overrun* and density did not generate significant models through the statistical treatment adopted in this study, since they did not allow observing the effects of the interactions among the factors (ingredients) on the response of these parameters. Thus, to determine the optimal formulations for camu-camu ice creams, the models of melting time, *ratio* and cost (Table 4) were adopted, the first of which was fitted to a quadratic model and the others were fitted to a linear model.

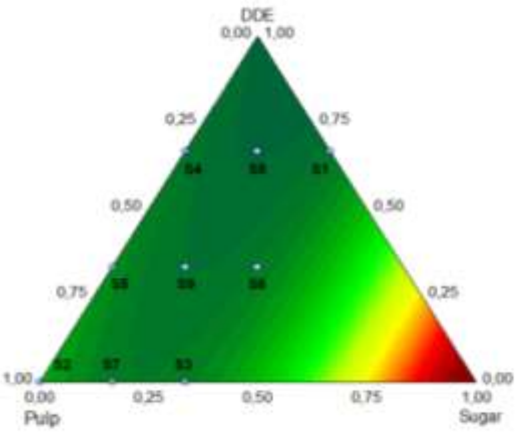
Table 4- Model equations for melting time, *ratio* and cost of camu-camu ice cream.

Parameters	Equation	p-value	R^2 (%)	$R^2_{aj.}$ (%)
Melting time	$Y = 61.90(\pm 2.11)x_1 + 202.20(\pm 39.28)x_2 + 39.50(\pm 3.45)x_3 - 260.40(\pm 56.75)x_1x_2 - 215.40(\pm 60.85)x_2x_3$	0.0055	92.24	86.03
Ratio	$Y = 44.63(\pm 1.94)x_1 + 53.49(\pm 6.21)x_2 + 56.90(\pm 2.85)x_3$	0.0251	65.08	55.11
Cost[a]	$Y = 11.64(\pm 0.00)x_1 + 10.44(\pm 0.00)x_2 + 12.68(\pm 0.00)x_3$	0.0001	99.99	99.99

x1 camu-camu pulp; x2 sugar; x3 defatted dry extract; calculated in Brazilian money (reais) per liter (R$/L of ice cream). The values in parentheses indicate the standard error for each coefficient (β). Source: Authors.

All models generated were significant ($p \leq 0.05$), with an adjusted R_2 greater than 55%. Analyzing the model (Table 4 and Figure 1), it is observed that sugar had a greater effect on the total melting time, followed by camu-camu pulp and powdered milk. On the other hand, the binary interactions had antagonistic effects between pulp and sugar, as well as sugar and powdered milk, which had a strong effect in reducing the total melting time. Sugar influences the lowering of the water freezing point, therefore, there is lower formation of ice crystals, increasing the resistance to melting, that is, increasing the melting time (Almeida et al., 2016). However, the pulp and sugar interactions (*x1x2*) contribute to reducing the melting time (Table 5), that is, they decrease the stability of the ice cream mass, as well as the sugar and DDE interaction (*x2x3*).

Figure 1 - Level curves obtained through the extreme vertex design for total melting time of camu-camu ice cream formulations.

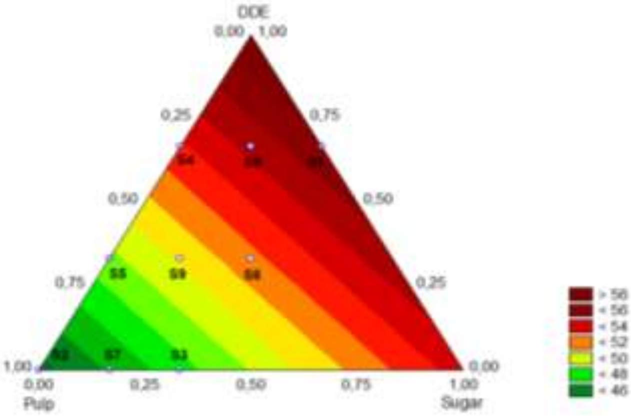


Source: Authors.

The behavior of camu-camu ice cream formulations during melting was also analyzed by monitoring the time *versus* the volume of ice cream drained, which allowed to calculate the total melting time. It was observed that the ice cream formulations started the melting process close to 10 min, which is recommended. According to Soler & Veiga (2001), an ideal melting begins to occur between 10 and 15 min, and should form a homogeneous liquid, with good fluidity and little foam.

The model determined for the *ratio* variable was linear, significant and with an adjusted R_2 of 55.11% (Table 4). The ingredients that showed the greatest effect were DDE followed by sugar. Camu-camu pulp had the lowest effect among the ingredients used (Table 4 and Figure 2).

Figure 2 - Level curves obtained through the extreme vertex design for *ratio* of camu-camu ice cream formulations.

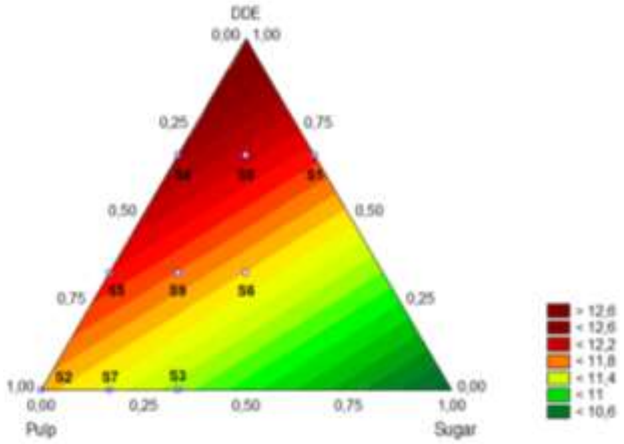


Source: Authors.

Within the limitations worked for camu-camu pulp (0.40 - 0.60), sugar (0.24 - 0.28) and DDE (0.24 - 0.32), the variations obtained were 35.17 to 40.87% for soluble solids and 0.60 to 0.83 g 100-1 (lactic acid) for titratable acidity, resulting in the *ratio* of 44.11 and 56.56. The greater reduction in acidity and the increase in soluble solids contribute to the increase in the *ratio* of titratable acidity/soluble solids. The lower the *ratio* value, the higher the total acidity concentration, which is desirable for processing some products (liqueurs). On the other hand, the higher the *ratio*, the higher the sugar content, which is desirable for other types of products (juices, ice cream, sweets, among others).

The model determined for the cost was linear, with high significance and adjustment, 99.99% (Table 4). The cost was based on the commercial value; therefore, the contribution of each component was homogeneous in the model. However, DDE contributed most to the cost, followed by camu-camu pulp and sugar (Table 4 and Figure 3).

Figure 3 - Level curves obtained through the extreme vertex design for cost (real) of camu-camu ice cream formulations.



Source: Authors.

The cost variations ranged from R$ 11.24 to R$ 12.34 for each liter of camu-camu ice cream. In order to obtain ice cream formulations with longer melting time, maximum ratio and lower cost, the desirability test was applied (Figures S1, S2 and S3 of the supplementary material) and resulted in the following formulations: S11 (26% camu-camu pulp, 12% sugar and 12% DDE), S12 (20% camu-camu pulp, 14% sugar and 16% DDE) and S13 (24% camu-camu pulp, 14% sugar and 12% DDE).

3.2 Characterization of the selected ice creams

Table 5 presents the results of the physicochemical analysis of the selected formulations (S11, S12 and S13).

Table 5 - Physicochemical characteristics of camu-camu ice cream formulations.

Sample	Moisture (%)	TDE[a] (%)	Ash (%)	Lipids (%)	Proteins (%)	Carb[b] (%)	pH	Acidity (g 100^{-1})	TSS[c] (ºBrix)	AA[d] (mg 100^{-1})
S11	64.98[a] ± 1.09	33.97[c] ± 0.39	1.57[a] ± 0.02	3.03[a] ± 0.05	4.55[b] ± 0.11	25.86[b] ± 1.00	5.79[b] ± 0.35	0.79[a] ± 0.01	35.38[c] ± 0.22	202.35[a] ± 11.23
S12	57.96[b] ± 0.81	41.49[a] ± 1.18	1.71[a] ± 0.07	3.00[a] ± 0.25	6.02[a] ± 0.07	31.31[a] ± 0.92	6.07[a] ± 0.00	0.75[b] ± 0.01	40.95[a] ± 0.52	169.36[b] ± 2.62
S13	62.60[a] ± 1.23	36.27[b] ± 0.33	1.63[a] ± 0.07	3.03[a] ± 0.14	4.57[b] ± 0.10	28.18[b] ± 1.36	5.84[b] ± 0.01	0.68[c] ± 0.01	36.60[b] ± 0.17	201.74[a] ± 0.80

[a]TDE = total dry extract, [b]Carb=carbohydrates, [c]TSS = Total soluble solids, [d]AA= Ascorbic acid. Mean ± Standard deviation, on a dry basis; values with equal letters, in the same column, did not differ statistically at the 5% level of significance. Source: Authors.

The moisture content of the formulation S11 (26% pulp, 12% sugar and 12% DDE) and S13 (24% camu-camu pulp, 14% sugar and 12% DDE) showed the highest averages, due to the addition of higher concentrations of camu-camu pulp, which has a high moisture content (92.46%).

The total soluble solids content of all formulations showed a statistical difference, in which formulation S12 had the highest average (41.49%), justified mainly by the addition of the higher concentrations of sugar and powdered milk. Total soluble solids are the sum of all non-aqueous components of ice cream and are responsible for improving the texture and creaminess of the product (Soler & Veiga, 2001). However, its excess (above 42%) is not recommended since the final product can be dense and gummy. Oliveira et al. (2005) observed a decrease in total soluble solids, accompanied by an increase in moisture, in their studies with ice cream with addition of fruit pulp. This effect was also observed in this study with the formulation with higher camu-camu pulp content and lower DDE and sugar (S11). For the analysis of lipids, there was no significant difference among the samples, since there was a standardization of fat in ice cream. The ash content also had no difference among the samples. The high values for the ash content can be justified by the high content of mineral (calcium, sodium, potassium, magnesium, among others) present in milk and in the camu-camu pulp (Aguiar & Souza, 2018; Lamounier et al., 2015).

For the protein and carbohydrate content, the S12 formulation presented the highest concentrations, 6.02% and 31.31%, respectively. The main source of carbohydrate in milk-based ice cream is lactose (milk sugar) and sucrose. Sucrose is the most common sugar in the manufacture of milk ice cream. Milk ice creams, in addition to being a source of carbohydrates, are also a source of protein (Marshall et al., 2003). According to the Brazilian legislation (RDC N °54 of November 12, 2012), a food is considered a source of protein if it supplies the minimum amount of 6.0 g. 100-1. The S12 formulation reached the content of this nutrient and can be considered a protein source.

Formulations S11 and S13 showed lower pH values and higher acidity, due to the addition of higher concentrations of camu-camu pulp that has acidic pH (2.06). All formulations showed high levels of ascorbic acid. S11 and S13 ice cream had the highest averages (202.3 and 201.74 mg of ascorbic acid (AA) 100 g-1, respectively). According to the World Health Organization (WHO), the recommended daily intake of ascorbic acid is 60 mg day -1 for an adult. The consumption of 29.65 g of formulation S11, 35.43 g of formulation S12 and 29.74 g of formulation S13 g provide the daily intake of ascorbic acid recommended.

Camu-camu ice creams showed caloric values compatible with the products available in local markets, with the same characteristic, that is, milk ice cream with (partial) addition of fruit pulp or fruit juice. The energy value of S11, S12 and S13 was 89.35 Kcal. 60 g -1, 105.79 Kcal. 60 g -1 and 94.96 Kcal. 60 g-1 respectively. The development of ice cream using exotic fruit pulp may require the addition of other ingredients to ensure desirable acceptability among consumers. It is necessary to add more sugars and/or essences to mask the pulp's flavor. The greater addition of sugar impacts the caloric value. In this study, the proportions of the ice cream balance did not result in a high energy value.

The technological parameters analyzed for the camu-camu ice cream (Table 6) showed that there was no significant difference for *overrun* and density at the level of 5% among the formulations studied.

Table 6 - Technological parameters of camu-camu ice cream formulations.

Sample	*Overrun* (%)	Density (g/ mL)	Melting time (min)	*Ratio*
S11	$51.33^a \pm 2.57$	$0.706^a \pm 0.00$	$65^a \pm 0.00$	$44.78^c \pm 0.01$
S12	$46.91^a \pm 3.85$	$0.730^a \pm 0.02$	$48^b \pm 2.89$	$54.60^a \pm 0.01$
S13	$46.62^a \pm 4.42$	$0.714^a \pm 0.00$	$53^b \pm 2.89$	$53.82^b \pm 0.01$

Mean ± Standard deviation, on a dry basis; values with equal letters, in the same column, did not differ statistically at the 5% level of significance. Source: Authors.

Formulation S11 had a longer melting time compared to formulations S12 and S123. *Overrun* analysis can affect the melting time, as the air acts as a thermal insulator and reduces the heat transfer rate, however, in this study, it was not

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observed.

The melting time of the samples can be related to other factors such as fat content, acidity, protein content and stabilizers, as previously discussed (Muse & Hartel, 2004; Soukoulis et al., 2009).

Table 7 shows the antioxidant and reducing capacity of the three formulations.

Table 7 - Antioxidant and reducing capacity of camu-camu ice cream formulations.

Sample	DPPH[a]	ABTS[a]	FRAP[a]	Reducing compounds[b]
S11	260.11[a] ± 7.87	711.56[b] ± 65.29	581.46[b] ± 4.33	121.48[a] ± 12.15
S12	270.76[a] ± 13.04	652.63[b] ± 20.71	438.53[c] ± 3.46	97.70[a] ± 6.90
S13	238.82[a] ± 20.38	818.01[a] ± 12.36	727.58[a] ± 3.32	121.38[a] ± 14.07

Mean ± Standard deviation; values with equal letters, in the same column, did not differ statistically at the 5% level of significance. [a]ABTS; FRAP and DPPH were expressed in µmol TEAC 100 g-1. [b]Reducing compounds were expressed in mg GAE 100 g-1. Source: Authors.

All samples showed antioxidant capacity and the presence of reducing compounds. The analysis of DPPH and reducing compounds showed no significant difference, the values ranged from 238.82 to 270.76 µmol TEAC. 100 g -1 and 97.70 to 121.48 mg GAE. 100 g-1, respectively. For the ABTS method, sample S13 had a significant higher value compared to other formulations, with an average of 818.01 µmol TEAC. 100 g -1. All samples showed a significant difference using the FRAP method. In general, the antioxidant properties did not show a direct relationship with the increase in 6% in the amount of camu-camu pulp, thus, the 6% variation in the amount of the pulp was not enough to increase the antioxidant capacity of the samples.

The functionality of reducing compounds when in contact with dairy products is based on their ability to interact with milk proteins (Omenn et al., 1996). According to Sarker et al. (1995), catechin increases the volume and improves the stability of β-lactoglobulin foams, intentionally destabilizing them. Therefore, the presence of polyphenols may have improved the stability of ice cream. Furthermore, the reducing compounds in camu-camu ice cream formulations indicate that they have several components that are health-promoting, such as antioxidant and anticarcinogenic agents.

3.3 Sensory analysis

The evaluators who participated in the sensory analysis were aged 18 to 60 years, of these, 48.57% were 18 to 20 years old, 32.86% 21 to 25 years old, 9% 26 to 30 years old. Only 4% declared to be older than 30 years. Most participants were male (61.43%).

The acceptability indices are presented in Table 8.

Table 8 - Acceptability indices of camu-camu ice cream formulations obtained through sensory analysis.

Sample	Aroma	Appearance	Color	Flavor	Texture	Overall appearance
S11	7.58[a] ± 2.11	7.59[a] ± 2.09	7.13[a] ± 1.93	7.31[b] ± 2.27	7.74[a] ± 1.52	6.97[b] ± 2.07
S12	7.78[a] ± 1.85	7.96[a] ± 1.47	7.71[a] ± 1.59	8.61[a] ± 1.47	8.36[a] ± 1.43	8.24[a] ± 1.46
S13	7.55[a] ± 1.64	7.52[a] ± 1.74	7.54[a] ± 1.51	7.43[b] ± 2.01	8.04[a] ± 1.50	7.41[b] ± 1.58

Mean ± Standard deviation; values with equal letters, in the same column, did not differ statistically at the 5% level of significance. Source: Authors.

All samples presented satisfactory scores in all attributes, with averages above 7.00. S12 ice cream had the highest average for flavor (8.61), differing from other ice creams. It is assumed that the greatest contribution to this score was the lowest addition of camu-camu pulp and the largest addition of sugar and powdered milk.

The same occurred with the overall appearance, in which the S12 formulation obtained the highest average (8.24). It is worth mentioning that all ice creams had low fat content, but only S12 was considered a source of protein.

The variation in the concentration of camu-camu pulp, DDE, and sugar, did not result in significant differences in sensory perception in relation to the aroma, appearance, color, and texture attributes.

In the test, the frequency of ice cream consumption was also evaluated and none of the interviewees reported consuming it daily or not consuming it. However, 18.57% said they consume ice cream eventually, 37.14% weekly and 44.29% monthly.

4. Conclusion

The variations in the contents of camu-camu pulp, sugar and DDE resulted in changes in the technological characteristics, *ratio* and cost of ice cream. Sugar and camu-camu pulp had greater effects in increasing the total melting time of the ice cream while DDE had a greater effect on the *ratio* and cost. Sensory analysis showed that consumers prefer ice cream with higher sugar and DDE contents and with the addition of pulp in concentration lower than 24%. The three selected formulations showed antioxidant properties and high levels of vitamin C. For future research, we suggest evaluating the effect of substituting sugar for another ingredient such as brown sugar and sweetener.

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