DOI: https://doi.org/10.1590/fst.38417



Different formulations of camu-camu popsicle: characterization, vitamin C and sensorial analysis of an opportunity to family agroindustry

Maria Luiza GRIGIO^{1*}, Maria Fernanda Berlingieri DURIGAN², Edvan Alves CHAGAS²

Abstract

The high levels of vitamin C in camu-camu have stimulated the interests of extractivists, farmers and consumers. The aim of this study was to characterize five accessible for family agroindustry different formulations of camu-camu popsicles and assess their acceptance. A completely randomized design was used and the treatments consisted of five different popsicle formulations: camu-camu pulp (T1 and T2: 27%, T3 and T4: 20.8%; T5: 61%), water (T1 and T2: 43.5%, T3 and T4: 20.8%; T5: no water), whole milk powder (T1 and T2: 14.7%, T3 and T4: 29.2%; T5: 18.3%), sugar (T1 and T2: 14.7%, T3 and T4: 29% and T5: 20.6%), neutral alloy and emulsifier. The formulations were processed and the popsicles were evaluated for pH, soluble solids, titratable acidity, vitamin C, acceptability index (SS/TA) and macro and micro nutrients were identified. In the sensorial analysis, the popsicles were evaluated for appearance, color, flavor, texture and purchase intent by 40 untrained tasters. The acceptance of the camu-camu popsicles was lower when the formulation was more diluted. Formulations T3, T4 and T5 showed the most wanted qualitative attributes and these preparations received top scores in the sensory analysis.

Keywords: Amazon; Myrciaria dubia; ice cream; caçari.

Practical Application: The present study shows the use of camu-camu for making popsicle. Evidentiating the great potential of the fruit, where the popsicle that is a food of routine consumption, can be transformed and exploited commercially like natural source of vitamin C and bioactive compounds. Demonstrating formulations of greater sensory acceptance and with higher concentrations of vitamin C.

1 Introduction

The camu-camu (*Myrciaria dubia* (H.B.K.) McVaugh), also known as caçari, araçá d'água, or sarão, is a species belonging to the *Myrtaceae* Family, native to the swamps and lakes of Amazonia. It has great economic potential, mainly because of its nutritional characteristics resulting from its high vitamin C content (Maeda et al., 2007).

The increasing interest in camu-camu fruit is due to its remarkable ascorbic acid or vitamin C content, and it is known as "King of Vitamin C" or "Super Fruit". In the state of Roraima, Brazil, the fruit is known as 'caçari', presenting mean values of 3.571 to 7.355 mg 100 g¹ pulp (Aguiar & Souza, 2016; Chagas et al., 2015; Grigio et al., 2015, 2016). However, its consumption is still restricted because of its high acidity, bitterness and astringency in the skin so that specific technologies are needed for its use and consumption. According to Maeda et al. (2006) the bitterness is related to the phenolic compound content, which is high in the camu-camu skin, and the type of processing used to obtain the pulp determines its perception.

In Amazonia these fruits are mainly collected by local farms and used to prepare juices, conserves, sweets and liqueurs (Viegas et al., 2004), mainly by small family farmers and agroindustries. However, the camu-camu is a fruit that still requires research especially to adapt conventional technologies

and develop new technologies to process the fruit for lucrative use and better aggregation of value to the product.

According to the Agência Nacional de Vigilância Sanitária (ANVISA), resolution RDC number 266 (Brasil, 2005), ice cream or edible ice is

[...] a food product obtained from an emulsion of fat and proteins, with or without the addition of other ingredients and substances, or from a mixture of water, sugars and other ingredients and substances that have been submitted to freezing, under conditions that guarantee the conservation of the product in the frozen or partially frozen state, during storage, transport and delivery to the consumer [...].

The popsicle, further according to Brazilian legislation (ANVISA) regulation number 379, April 26th 1999 (Brasil, 1999), is characterized by individual servings of edible ice of several compositions, generally supported by a stick, obtained by chilling the homogeneous or not mixture of food ingredients until frozen, with or without beating.

The popsicle can be processed with easily obtained ingredients in the Amazonia region, using pulp from native fruits that aggregates value and improves the exploitative potential of these fruits. However, due to the variability of formulations and interactions among the ingredients used, a sensory assessment in

Received 17 Nov, 2017

Accepted 15 Oct., 2018

¹Programa de Pós-graduação em Agronomia, Centro de Ciências Agrárias, Universidade Federal de Roraima – UFRR, Campus Cauamé, Boa Vista, RR, Brasil

²Empresa Brasileira de Pesquisa Agropecuária – Embrapa Roraima, Boa Vista, RR, Brasil

^{*}Corresponding author: luizagrigio@hotmail.com

addition to the technological assessment is necessary to obtain greater information on the potential of the resulting products. Thus, the objective of the present study was to characterize the physical, chemical and physical chemical properties of different camu-camu popsicle formulations and assess their acceptance.

2 Materials and methods

To obtain the camu-camu pulp used in popsicle manufacture, ripe fruits were collected from a native population situated on the banks of the Branco River in Boa Vista-RR. After collection, the fruits were transported to a local family agroindustry, where they were selected for absence of damage, washed in running water and cleaned with 0.02% sodium hypochlorite (NaClO) for 30 min following the recommendations by ANVISA. They were then de-pulped, the seeds and peel were removed in the process, without adding water, and the final product from the pulping was packed and frozen.

The experiments were carried out in May 2015 in a complete randomized design. The treatments consisted of five different popsicle formulations and each treatment consisted of four replications, and each popsicle was considered a replication.

The pulp used to formulate the popsicles was characterized with the following analyses: vitamin C content = 403.44 mg ascorbic acid 100 mL⁻¹ pulp; pH = 2.88; Soluble Solids (SS) = 7.4 °Brix; Titratable Acidity (TA) = 1.90 citric acid 100 g⁻¹ pulp; SS/TA ratio = 3.9191 and Crude Protein = 2.72%. In addition, the following macro and micro nutrients were just identified, whitout statical representativeness: 0.08% phosphorous (P), <0.02% sodium (Na), 0.87% potassium (K), 0.32% calcium (Ca), 17.71 mg/Kg iron (Fe), 34.05 mg/Kg zinc (Zn) and 0.47 mg/Kg selenium (Se).

The popsicles were formulated using the ingredients and concentrations shown in Table 1.

According to each treatment, the formulations were processed and the resulting mixture was placed in popsicle molds and frozen at -12 to -15 °C. After two days freezing, the popsicles

were taken to the Laboratory of Embrapa Roraima, where the following were assessed:

The pH, was determined by reading on a PM 608 pH meter.

Soluble solid content, determined by direct reading on a RTD-45 portable refractometer and the results expressed in $^{\rm o}Brix.$

Titratable acidity content, determined by titrimetry (Instituto Adolfo Lutz, 2008), considering 10 g of each of the formulations, which were diluted in distilled water to 100 mL. For reading, after addition of the phenolphthalein indicator, the solution was titrated with NaOH solution (0.1M), and the results expressed in grams of citric acid 100 $\rm g^{-1}$ sample.

Vitamin C content by the Tillmans titrimetry method (Instituto Adolfo Lutz, 2008). This method is based on the reduction of the 2,6 dichlorophenol indophenol dye (DCFI) by the acid solution of ascorbic acid. 10 g of the sample was diluted in 10 mL of oxalic acid, filtered and titrated with the Tillmans solution. The reaction is fast and the final change is indicated by the dye, which in acidic environment and once oxidized by ascorbic acid changes color to a pink color. The results were expressed in milligrams of ascorbic acid $100~\rm mL^{-1}$ sample.

The acceptability index ("Ratio") calculated by the SS/TA ratio.

Samples of each treatment and the pulp, each one containing three replications, were taken for quantification analysis of nutrients and crude protein, carried out in the Animal Nutrition Laboratory of the São Paulo State University (FCAV/UNESP), Jaboticabal Campus, SP. The analysis method used to quantify phosphorous was nitro-perchloric digestion and colometry of the ammonium phosphomolybdate (Lott et al., 1956); for sodium and potassium, flame photometry (Skoog et al., 2002); atomic absorption spectrophotometry (Perkin-Elmer Corp., 1966) was used to analyze calcium, iron, zinc and selenium. The results for characterization only, are shown in Table 2.

Table 1. Different formulations tested for camu-camu (Myrciaria dubia) popsicle. Boa Vista, Roraima, 2015.

Treatments	Camu-camu pulp (%)	Water (%)	Whole poder milk (%)	Sugar (%)	Red Coloring	
T1	27	43.5	14.7	14.7	no	
T2	27	43.5	14.7	14.7	yes	1 11 1
T3	20.8	20.8	29.2	29	no	+ neutral alloy and emulsifier
T4	20.8	20.8	29.2	29	yes	emuismei
T5	61	-	18.3	20.6	yes	

Table 2. Analyses of nutrients and crude protein in different formulations tested for camu-camu (*Myrciaria dubia*) popsicle. Boa Vista, Roraima, 2015.

Treatments	Crude Protein	P	Na	K	Ca	Fe	Zn	Se
							mg kg ⁻¹	
T1	7.20	0.21	0.13	0.45	0.27	34.20	10.57	0.12
T2	6.81	0.21	0.13	0.46	0.27	41.32	9.14	0.04
Т3	6.42	0.20	0.12	0.37	0.26	49.65	9.14	0.02
T4	6.34	0.19	0.12	0.37	0.25	26.04	8.06	0.12
T5	7.54	0.21	0.11	0.40	0.28	4.57	9.50	0.18

For the sensorial analysis, the samples were placed in disposable cups and codified with random numbers. Each assessor received five popsicle formulations and a sheet of paper with a questionnaire and a hedonic scale to assess appearance, color, flavor and texture ranging from nine to one (9 - I liked it very much, 8 - I enjoyed it, 7 I - liked it regularly, 6 - I liked it a little, 5 -indifferent- I didn't like or dislike it, 4 - I disliked a little, 3 - I regularly disliked it to moderately disliked it, 2 - I disliked it, and 1 - I extremely disliked it). Another score was used to assess the purchase intention (1 - definitely would buy, 2 - would probably buy, 3 - perhaps yes/perhaps no, 4 - would probably not buy, 5 definitely would not buy). The assessors drank water between assessments so there was no confusion between the formulations analyzed.

After submission and approval by the ethic committee of Plataforma Brasil (CAAE: 39610314.3.0000.5302), the sensorial analysis was carried out at Embrapa Roraima by 40 untrained testers.

The data obtained were submitted to analysis of variance and the means tested by the Tukey test at 5% statistical probability.

3 Results and discussion

All the variables presented statistical differences among the tested treatments. The T5 popsicle formulation had the highest SS content (Table 3). This value was higher than all the other formulations and almost 7 times greater than that detected in the fruit pulp. This may have been because more pulp was added (61%) instead of water, not added, that with the milk powder and sugar in the formulation resulted in a greater soluble solids concentration. The T5 formulation was followed for SS content, in decreasing order, by T4 and T3, that did not differ significantly. Their formulations presented differences only from the addition (T4) or not (T3) of coloring.

The highest sugar percentages detected at these treatments were counter balanced with the addition of water. The T1 and T2 formulations presented the lowest SS content, but also did not differ significantly. They were the most diluted formulations, that presented lower values than those reported by Santana et al. (2003) when they tested bred papaya genotypes in the form of ice cream.

Regarding the titratable acidity, the lower pulp concentration and water addition in the T1, T2, T3 and T4 formulations reduced the acidity in these treatments, which did not differ significantly. Similarly to the SS content, the TA was higher in the T5 formulation, also because of dilution in its formulation and the greater camu-camu pulp concentration. In its natural form has a very high acidity, that justifies the development of technologies to minimize this flavor and develop ways of industrial use (Maeda & Andrade, 2003).

The results showed that the camu-camu popsicle formulas have lower acidity compared to the natural fruit pulp, a fact confirmed by the lower in pH, around pH 4.0, considered moderately acid. The percentage of whole milk powder also may have influenced the pH in the popsicle formulations. Greater acidity was observed in T1 and T2 which, although they were more diluted and had lower pulp content, presented the lowest milk powder percentage. The highest pH value, consequently the least acid, was observed in treatments T3 and T4, which presented the highest milk powder percentage in their formulations. The pH value of T5 treatment was placed between these treatments, probably because of the whole milk percentage, also intermediate to the percentage in the other treatments. Its formulation also presented the highest camu-camu pulp percentage. However, similar performance was reported by Santana et al. (2003) when they testing bred papaya genotypes in the form of ice cream, they added milk and detected a rise in the pH values.

For the camu-camu popsicle, greater value of the SS/TA ratio was observed for the T3 and T4 formulations, because they presented the greatest difference between the proportion of SS compared to the titratable acids, which resulted in popsicles more sweet than acid. These two formulations did not differ between then, but were differed significantly from all the other treatments (T1, T2 and T5) which also did not differ statistically from each other. Although it had the highest SS content, the T5 formulation also presented the highest TA content, that resulted in a smaller difference in the SS/TA ratio and consequently in a popsicle that was neither very sweet nor very acid. The opposite occurred with the T1 and T2 popsicles. Although they had low TA they also had a low SS content, which resulted in a smaller difference in the SS/TA ratio, they consequently was neither sweet nor acid.

The SS/TA ratio is an important qualitative attribute, because it indicates the inherent flavor of the product, that is the result of the contribution of the components responsible for acidity and sweetness (Chitarra & Chitarra, 2005).

For the vitamin C content, the only formulation statistically different from the others was T5, with the highest vitamin C

Table 3. Physico-chemical, chemical and nutrient analysis of different formulations tested for camu-camu (*Myrciaria dubia*) popsicle. Boa Vista, Roraima, 2015.

Treatments	Soluble Solids (°Brix)*	Titratable acidity (g 100 g-¹)*	pH*	SS/TA*	Vit. C (g 100 g ⁻¹)*
T1	24.67 a	0.58 a	3.91 a	42.32 a	84.04 a
T2	24.63 a	0.53 a	3.71 a	48.10 a	80.70 a
Т3	47.19 b	0.56 a	4.41 c	81.04 b	94.07 a
T4	48.00 b	0.58 a	4.42 c	85.29 b	90.40 a
T5	51.30 с	1.01 b	4.02 b	51.05 a	149.90 b
CV (%)	1.50	7.44	1.27	7.95	6.22

^{*}Means followed by the same letters in the columns do not differ by the Tukey test at 5% probability.

Table 4. Sensory analysis of different formulations tested for camu-camu (Myrciaria dubia) popsicle. Boa Vista, Roraima, 2015.

Treatments	Appearence (scores)*	Color (scores)*	Flavor (scores)*	Texture (scores)*	Purchase intention (scores)*
T1	5.60 a	5.55 a	5.72 a	5.95 a	3.07 b
T2	7.12 bc	7.82 b	6.20 ab	6.55 ab	2.52 ab
Т3	6.57 ab	6.55 a	7.62 c	7.00 ab	2.30 ab
T4	7.72 c	7.82 b	7.75 c	7.42 b	2.05 a
T5	6.77 bc	6.55 a	6.90 bc	6.82 ab	2.27 ab
CV (%)	26.14	24.74	26.09	28.65	54.56

^{*}Means followed by the same letters in the columns do not differ by the Tukey test at 5% probability.

contents, around 149.90 mg $100~g^{-1}$ sample, probably due to the higher percentage of camu-camu pulp (61%) in its formulation. The other treatments (T1, T2, T3 and T4) did not differ statistically but still presented high vitamin C content for a processed product (84,70 mg $100~g^{-1}$). However, the concentration and stability of vitamin C vary with the species, ripeness stage, processing time and temperature, pH and presence of oxygen and enzymes (Maeda et al., 2007).

The appearance of the products, in decreasing order, the best score was attributed to T4 formulation, which did not differ statistically from T2 and T5, which did not differ from T3 formulation that, in turn, did not differ from the T1 (Table 4). The addition of red coloring in the T4, T2 and T5 formulations contributed to the higher scores for appearance. However, only the T4 and T2 formulations presented the highest scores for the color variable, confirming the importance of coloring for the appearance of camu-camu popsicles. However, T5 formulation, to which coloring was also added, did not present good color and received a score statistically similar to the formulations where coloring was not added.

The highest scores for flavor were attributed to the T4, T3 and T5 formulations, which received greater quantities of sugar and corresponded, in decreasing order, respectively, to the highest SS contents and highest SS/TA ratio values. This demonstrated the preference of the tasters for the sweeter formulations. According to Silva & Lannes (2011) sugars are responsible for the flavor in ice cream, but also affect the freezing performance and viscosity of the mixtures.

The T4 treatment received the highest score for texture, but differed statistically only from the T1, which received the lowest score. It may occur probably because of it had a greater percentage of water and a lower percentage of milk in its formulation, which resulted in a less creamy texture. T4 had an inverse proportion of the ingredients water and milk to the T1 formulation, resulting in a highest creamy popsicle. The fat present in milk may be acting as an emulsifier giving greater preference for texture.

The T4 formulation received the highest score for the purchase intention variable. It was statistically different only when compared to T1, and, respectively, the greater probability of purchase of the T4, T5, T3 and T2 formulations.

Disagreeing with the results observed in the present study, the sensorial assessment by Maia et al. (2008) for ice cream based on xylitol reported that the best scores for appearance were received by the treatment where there was no the presence of this sweetener.

Dilution and coloring were considered determining factors in this choice. Once the formulation was more diluted and did not have coloring, there was less purchase interest.

4 Conclusions

The use of coloring was determinant for a better acceptance of the formulations. The T3, T4 and T5 formulations presented the best qualitative attributes and were the formulations that received the best scores for sensorial analysis. Being the camu-camu popsicle acceptance was lower when the dilutions were greater. Further studies should be performed to develop other formulations from camu-camu popsicle with greater acceptability.

Acknowledgements

The authors thank the Conselho Nacional de Desenvolvimento Científico e Tecnológico, Fundação Estadual do Meio Ambiente e Recursos Hídricos and Coordenação de Aperfeiçoamento de Pessoal de Nível Superior for the financial support and J.G. Ribeiro Agroindustry for making its infrastructure available to carry out the study.

References

Aguiar, J. P. L., & Souza, F. C. A. (2016). Camu-Camu super fruit *Myrciaria dubia* (H.B.K) Mc Vaugh) at different maturity stages. *African Journal of Agricultural Research*, 11(28), 2519-2523. http://dx.doi.org/10.5897/AJAR2016.11167.

Brasil. Ministério da Saúde. Agência Nacional de Vigilância Sanitária. (1999). Portaria nº 379, de 26 de abril de 1999. Aprova o regulamento técnico referente a gelados comestíveis, preparados, pós para o preparo e bases para gelados comestíveis. *Diário Oficial [da] República Federativa do Brasil*.

Brasil. Ministério da Saúde. Agência Nacional de Vigilância Sanitária. (2005). Resolução RDC nº 266, de 22 de setembro de 2005. Aprova o "regulamento técnico para gelados comestíveis e preparados para gelados comestíveis". Diário Oficial [da] República Federativa do Brasil.

Chagas, E. A., Lozano, R. M. B., Chagas, P. C., Bacelar-Lima, C. G., Garcia, M. I. R., Oliveira, J. V., Souza, O. M., Morais, B. S., & Araújo, M. C. R. (2015). Intraspecific variability of camu-camu fruit in native populations of northern Amazonia. *Crop Breeding and Applied Biotechnology*, 15(4), 265-271. http://dx.doi.org/10.1590/1984-70332015v15n4a44.

- Chitarra, M. I. F., & Chitarra, A. B. (2005). *Pós-colheita de frutos e hortaliças: fisiologia e manuseio* (785 p.). Lavras: ESAL/FAEPE.
- Grigio, M. L., Chagas, E. A., Berlingieri Durigan, M. F., Andrade Sousa, A., Mota, A. B. Fo., & Chagas, P. C. (2016). Determination of harvest time and quality of native camu-camu fruits (*Myrciaria dubia* (Kunth) Mc Vaugh) during storage. *Fruits*, 71(6), 373-378. http://dx.doi.org/10.1051/fruits/2016029.
- Grigio, M. L., Durigan, M. F. B., Chagas, E. A., Chagas, P. C., Nascimento, C. R., & Almeida, M. S. (2015). Post-harvest conservation of camucamu fruits (*Myrciaria dubia* (Kunth) Mc Vaugh) using different temperatures and packages. *Food Science and Technology (Campinas)*, 35(4), 652-658. http://dx.doi.org/10.1590/1678-457X.6788.
- Instituto Adolfo Lutz IAL. (2008). *Métodos físico-químicos para análise de alimentos* (pp. 1020). São Paulo: IAL.
- Lott, W. L., Nery, J. P., Gallo, J. R., & Medcalf, J. C. (1956). A técnica de análise foliar aplicada ao cafeeiro (No. 79, Boletim). Campinas: Instituto Agronômico.
- Maeda, R. N., & Andrade, J. S. (2003). Aproveitamento do camu-camu (*Myrciaria dubia*) para produção de bebida alcoólica fermentada. *Acta Amazonica*, 33(3), 489-498. http://dx.doi.org/10.1590/S0044-59672003000300014.
- Maeda, R. N., Pantoja, L., Yuyama, L. K. O., & Chaar, J. M. (2006). Determinação da formulação e caracterização do néctar de camucamu (*Myrciaria dubia* McVaugh). *Food Science and Technology* (*Campinas*), 26(1), 70-74. http://dx.doi.org/10.1590/S0101-2061200600100012.

- Maeda, R. N., Pantoja, L., Yuyama, L. K. O., & Chaar, J. M. (2007). Estabilidade de ácido ascórbico e antocianinas em néctar de camucamu (*Myrciaria dubia* (H. B. K.) McVaugh). Food Science and Technology (Campinas), 27(2), 313-316. http://dx.doi.org/10.1590/S0101-20612007000200018.
- Maia, M. C. A., Galvão, A. P. G. L. K., Della Modesta, R. C., & Pereira Júnior, N. (2008). Avaliação sensorial de sorvetes à base de xilitol. Food Science and Technology (Campinas), 28(1), 146-151. http:// dx.doi.org/10.1590/S0101-20612008000100021.
- Perkin-Elmer Corp. (1966). *Analytical methods for atomic absorption spectrophotometry*. Connecticut: Perkin-Elmer Corp.
- Santana, L. R. R., Matsuura, F. C. A. U., & Cardoso, R. L. (2003). Genótipos melhorados de mamão (*Carica papaya* L.): avaliação tecnológica dos frutos na forma de sorvete. *Food Science and Technology (Campinas)*, 23, 151-155. http://dx.doi.org/10.1590/S0101-20612003000400028.
- Silva, E. D. Jr., & Lannes, S. C. S. (2011). Effect of different sweetener blends and fat types on ice cream properties. Food Science and Technology (Campinas), 31(1), 217-220. http://dx.doi.org/10.1590/ S0101-20612011000100033.
- Skoog, D. A., Holler, F. J., & Nieman, T. A. (2002). *Princípios de análise instrumental* (5. ed.). Porto Alegre: Bookman.
- Viegas, I. J. M., Thomaz, M. A. A., Silva, J. F., Conceição, E. O., & Naiff, A. P. M. (2004). Efeito da omissão de macronutrientes e boro no crescimento, nos sintomas de deficiências nutricionais e na composição mineral de plantas de camucamuzeiro. *Revista Brasileira de Fruticultura*, 26(2), 315-319. http://dx.doi.org/10.1590/S0100-29452004000200032.