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ECOSYSTEMS

Effect of calcium chloride and gelling agents on the physicochemical and sensory characteristics of sugar-free banana preserves

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Abstract: Banana preserve is produced by mixing the puree of the fruit with sucrose and organic acids. However, concerns about body esthetics or health reasons have encouraged the search for low-calorie products. Therefore, the objective of this study was to evaluate the effect of calcium chloride ($CaCl_2$), carrageenan gum, and low methoxyl pectin (LM-pectin) on the physicochemical and sensory characteristics of sugar-free banana preserves. By using a central composite rotational design (CCRD) of 2^3 + 6 axial points + 4 central points, we obtained 18 formulations that were further tested. Lower $CaCl_2$ concentrations (0.54% to 0.61%) resulted in preserves with lower pH and more vivid color. The increased concentration of LM-pectin (1.40% to 1.64%) resulted in formulations with a yellowish-red hue and with lower moisture, thus, reducing the flavor and purchase intention of the product. Higher concentrations of carrageenan gum (1.04% to 1.15%) decreased the perception of banana preserve aroma. Therefore, concentrations of $CaCl_2$ ranging from 0.54% to 0.61%, carrageenan gum ranging from 0.74% to 0.89% and LM-pectin ranging from 1.40% to 1.64% resulted in sugar-free banana preserves with ideal sweetness and consistency and were, therefore, more acceptable.

Key words: Acceptability, diet products, health, response surface methodology.

INTRODUCTION

Banana preserves are a very common product in Brazil. Consumed since the time of colonization, it is a low cost and tasty food that has high energy content, and therefore, is considered a source of energy; it also has a long shelf life (Silva et al. 2017).

Sucrose is one of the main components used in obtaining bulk preserve at the appropriate cut-off, because it establishes gel formation along with pectic substances (Pereira et al. 2019). However, consumers have been adapting to healthier eating habits by consuming less high-caloric foods (Chim et al. 2006) and more foods that are beneficial to health. In this context, there has been an expansion in the supply and diversification of low-calorie (light/diet) products to meet this need (Chim et al. 2006).

The removal of sugar from products that use a significant amount of that ingredient may change sensory and technological characteristics, making it difficult to obtain products similar to the conventional. The use of ingredients that can mitigate the effect of the absence of sugar is essential. For these reasons, the development of sugar-free products requires the inclusion of additives to substitute that ingredient, including sweeteners, body agents, and gelling agents (Goldfein & Slavin 2015).

Numerous sweeteners are available in the market, such as sucralose and acesulfame-k. Acesulfame-k is a nonnutritive sweetener and a potassium salt that has a bitter aftertaste at high concentrations, and because of which is often used in combination with other sweeteners such as sucralose (Chakraborty & Das 2019). Sucralose is characterized by a taste similar to that of sucrose and lacks the unpleasant aftertaste of acesulfame-k. Moreover, sucralose has a sweetness approximately 600 times that of sucrose, and it is stable at high temperatures and in a wide range of pH values (Pereira et al. 2017).

In addition to sweeteners, the use of body agents such as polydextrose is also required. Polydextrose acts on food, improving the texture, working as a thickener, stabilizer, and humectant; it is extremely stable within a wide range of pH, temperature, and processing and storage conditions (Pereira et al. 2017).

Among the gelling agents, carrageenan gum and LM-pectin are notable. At high concentrations, carrageenan gum can form a double helix structure, which can lead to gel formation. Therefore, it, is used as a gelling and stabilizing agent (Spagnuolo et al. 2005). LM-pectin forms a gel in the presence of bivalent metal ions (usually calcium) and does not require sugars (Lima et al. 2019).

Only few studies have discussed the making of sugar-free banana preserves. Thus, the objective of the present study was to evaluate the effect of calcium chloride ($CaCl_2$), LM-pectin, and carrageenan gum on the physicochemical and sensory characteristics of sugar-free banana preserves.

MATERIALS AND METHODS

Ingredients

The ingredients used were banana (Caturra cultivar), polydextrose (Nutramax, Catanduva, Brazil), κ-carrageenan gum (Gastronomy Lab, Distrito Federal, Brazil), LM-pectin (Rica Nata, Piracema, Brazil), sucralose (Nutramax, Catanduva, Brazil), acesulfame-k (Nutramax, Catanduva, Brazil), potassium sorbate (Rica Nata, Piracema, Brazil), and $CaCl_2$ (Rica Nata, Piracema, Brazil).

Methods experimental design

This study evaluated the effects of three factors ($CaCl_2$, carrageenan gum, and LM-pectin). A central composite rotational design (CCRD) with $2^3 + 6 + 4$ points, including axial center points, was applied, totaling 18 formulations. The coded and real values of the factors are specified in Table I.

The polynomial considered in the model adjustment was according to Equation 1.

$$y=\beta_0=\beta_1 x_1+\beta_2 x_2+\beta_3 x_3+\beta_{123} x_1 x_2 x_3+\beta_{11} x^2_1+\beta_{22} x^2_2+\beta_{33} x^2_3+e$$
Eq. 1

where β_0, β_1, β_2, β_3, β_{12}, β_{13}, β_{23}, β_{11}, β_{22}, and β_{33} are the regression coefficients, y is the response,

Table I. Complete factorial design 23 for the optimization of sugar-free banana preserves.

Independent Variables	Symbol	Levels of variables				
		-1.68	-1	0	1	1.68
Calcium chloride ($CaCl_2$) (%)	X1	0.54	0.61	0.71	0.82	0.90
Carrageenan gum (%)	X2	0.64	0.74	0.89	1.04	1.15
LM-pectin (%)	X3	1.4	1.64	1.97	2.3	2.59

x_1, x_2, x_3 are the independent variables ($CaCl_2$, carrageenan gum, and LM pectin), and e is the experimental error.

Processing of banana preserves

Caturra bananas were purchased at a local market in the city of Ouro Preto-MG, Brazil. Bananas at the ripening stages between scales 5 (yellow with green tip) and 6 (all yellow) were used (Loesecke 1950). After sanitization in chlorinated water (2.5%) for 15 min., the bananas were peeled manually and homogenized in an industrial blender (Tron, Tron Master 2L, Catanduva, SP, Brazil) for 60 s to obtain pulp. Subsequently, the pulp was stored at -18 °C in polypropylene pots wrapped in aluminum foil to prevent loss of light and oxygen sensitive nutrients, as well as aroma and taste.

To prepare the preserves, the banana pulp (60%) and polydextrose (35.72%) were added in an open stainless-steel pan. The mixture was subjected to a cooking process and then gelling agents (LM-pectin and carrageenan gum) and $CaCl_2$ were added as shown in Table I. Calcium chloride was added to the mixture solubilized in 3 mL of water. The mixture was then cooked until 65 °Bx, measured using a manual RT-82 refractometer. At the end of the cooking process, sweeteners and potassium sorbate were added. The amount of sweeteners used was according to the methodology described by Souza et al. (2013), who used an acesulfame-k/sucralose blend in a ratio of 3:1. As with acesulfame-k (0.01875%) and sucralose (0.00625%) sweeteners, the potassium sorbate preservative (0.05%) was dissolved in 2 mL of water and added to the mixture at the end of the process. Subsequently, the banana preserves were packed in polypropylene jars and stored in an incubator chamber at 25 °C for further analysis. All formulations were adjusted to 100%.

Physicochemical evaluation of sugar-free banana preserves

Physicochemical analysis was performed in the Sensory Analysis, Bromatology and Multi-user laboratories of the School of Nutrition of the Federal University of Ouro Preto-MG, Brazil. Moisture (%), pH, and soluble solids (°Bx) content, as well as colorimetric parameters (L*, C*, °Hue), were evaluated in quadruplicate.

Moisture, pH, and soluble solids content analysis was performed according to AOAC (2003) protocols. The colors of the banana preserves were evaluated according to the methodology proposed by Curi et al. (2018). The values of L*, C*, and °Hue were determined using a Konica Minolta model CR 400 colorimeter, with D_{65} (daylight) and using CIELAB standards, where L* is luminosity and ranges from 0 (black) to 100 (white), C* is chroma and ranges from 0 (white and/or gray) to 60 (vivid and/or intense colors) and °Hue is hue angle and ranges from 0 (red) to 270 ºh (blue).

Sensorial evaluation of sugar-free banana preserves

Sensory analysis was performed with 100 participants who were among students, university employees and visitors. The evaluation was performed in four sessions (two with five samples and two with four samples), in individual booths. This method was already analyzed and approved by the local Ethics Committee No. 827.360.

The preserve samples, weighing approximately 5.0 g, were served in 50 mL plastic cups and encoded using 3-digit codes in a balanced order (Wakeling & Macfie 1995). Affective tests (acceptance test, ideal scale, and attitude scale) and descriptive tests were performed.

Sensory acceptance of banana preserves was assessed by a 9-point hedonic scale (1 =

dislike extremely and 9 = like extremely). The sensory attributes evaluated were appearance, aroma, taste, and texture.

In order to evaluate the ideal sweetness and consistency of the banana preserves, the ideal scale test was performed using a 9-point hedonic scale (-4 = less sweet/consistent than ideal, 0 = ideal, and +4 = more sweet/consistent than ideal) (Stone & Sidel 1993).

We also evaluated the attitude scale, also called consumers'purchase intention in relation to the product through a 5-point structured scale (1 = certainly would not buy to 5 = certainly would buy).

In the same manner as the sensory acceptance test, the sensory descriptors for each attribute were evaluated using the CATA methodology (check-all-that-apply). The descriptors used were: characteristic appearance of banana preserve, strong brown color, light brown color, lighter than ideal color, darker than ideal color, ideal color, bright, matte, opaque, translucent, lumps, syneresis (presence of water on the surface); strong banana aroma, weak banana aroma, sweet aroma, burnt aroma, pleasant aroma, strange aroma, characteristic banana preserve aroma; strong banana flavor, weak banana flavor, ideal banana flavor, characteristic banana preserve taste, very sweet taste, little sweet taste, ideal sweetness, weird taste, residual sweetener taste, burnt flavor, characteristic banana preserve taste; firm consistency, soft consistency, nice texture, nasty texture, texture uniformity, tackiness, ideal texture, and lumps.

Statistical analysis

The results of all the analyses were evaluated by the response surface methodology using STATISTICA™ software, version 8.0 for Windows (StatSoft®).

In addition, frequency histograms were performed in MS Excel, version 2010 to evaluate the results of ideal sweetness and consistency.

To facilitate the visualization of CATA (attribute frequency) attribute results, the data were analyzed using principal component analysis (PCA). The data were organized in an array of rows (samples) and columns (attributes), standardized (correlation matrix), and PCA was applied using SensoMaker version 1.0 software (Nunes et al. 2011).

RESULTS AND DISCUSSION

Tables II and III show the regression coefficients for measured responses - physicochemical parameters and acceptance.

Non-significant lack of fit ($p > 0.05$) for pH, L, chroma, hue, aroma, flavor, texture, intention to buy, and ideal consistency indicate the accuracy of the statistical model for this result. An R^2 value close to unity indicates closer fitting of model to experimental data. Additionally, a low R^2 value demonstrates that results were not relevant enough to explain behavior variation (Khuri & Mukhopadhyay 2010, Mehmood et al. 2018, 2019); a low p-value indicates a highly significant effect on the response variable (Mehmood 2015).

It was observed that, although the lack of adjustment was not significant and the coefficient of determination was low (0.64), the LM-pectin caused a quadratic effect on moisture (Table II), i.e., increasing the concentration of LM-pectin caused the moisture to decrease. According to Kastner et al. (2020), the LM-pectin, in the presence of ions, interacts with the water present in the gel, reducing the available water. In addition, the increased concentration of LM-pectin makes the gel more rigid and, consequently, with less moisture content (Rahman & Al-Farsi 2005). Moisture is related to food stability (Ma et al. 2020), and so its reduction

Table II. Regression coefficients for measured responses - physicochemical parameters.

Regression coefficients	pH	Moisture (%)	Soluble solids (°Bx)	L	C	°H
Intercept (β_0)	4.53	45.65	51.29	34.97	13.13	67.92
A-$CaCl_2$(β_1)	-0.03	-2.66	2.19	-1.17	-0.25	-0.20
B- Carrageenan gum (β_2)	0.00	-1.26	0.23	-0.23	-0.32	0.68
C- LM-pectin (β_3)	-0.07	-0.23	-0.56	0.42	0.99	0.53
A^2 (β_{11})	0.09*	-3.05	2.78	-2.55	-1.50*	-0.65
B^2 (β_{22})	0.08	-2.74	2.37	-0.07	0.72	-1.40***
C^2 (β_{33})	0.07	-3.20*	2.01	1.57	-1.21	-1.52***
AB (β_{12})	-0.08	-0.91	1.67	0.43	-0.38	-0.76
AC (β_{13})	-0.07	-1.52	1.00	1.9	-0.46	-1.38**
BC (β_{23})	-0.02	-0.17	-0.75	1.57	0.09	-0.14
R^2	0.66	0.64	0.43	0.61	0.69	0.85
Lack of fit	0.37	0.04	0.03	0.50	0.06	0.06

*** Significant at 0.05 level ** Significant at 0.01 level, *** Significant at 0.001 level.**

favors conservation of the product. Thus, this effect caused by LM-pectin may contribute to increasing the shelf life of the product.

The pH was positively significantly affected in a quadratic way by the $CaCl_2$ (Table II). Liu et al. (2014) demonstrated that the bonds formed by divalent calcium are responsible for the interactions that occur in milk gels at high pH, but do not play a significant role at lower pH values. According Fan et al. (2020) many factors could affect pectin emulsifying properties such pH. Günter et al. (2020) report that increasing the pH in gels with calcium chloride increases the swelling of the gel, since, at higher pH values, the carboxylic acid groups were converted into negatively charged carboxylate ions, resulting in electrostatic repulsion between the different polymer chains and network expansion.

It was observed that the study variables did not significantly affect soluble solid content and luminosity (Table II).

$CaCl_2$ caused a negative quadratic effect for parameter C* (Chroma) (Table II). Thus, the increase of $CaCl_2$ concentration resulted in the loss of color vividness of the formulations. According to Pereira et al. (2013), the increase in $CaCl_2$ concentration in preserves with LM-pectin makes the product more rigid, making light transmission difficult (Dervisi et al. 2001, Fan et al. 2020).

According to Emery et al. (2017), based on the values of a* and b*, the °Hue is obtained, which is evaluated on a scale from 0 to 360°. The °Hue defines the correct position of the analyzed sample in the color range, i.e., it reflects the color tone. According to the CIELAB hue sequence, the red hue is indicated by 0 °h, yellow by 90 °h, green by 180 °h and blue by 270 °h. For this parameter, the concentrations of carrageenan gum and LM-pectin caused quadratic effects. The interaction factor β_{13} ($CaCl_2$ and LM-pectin) also had a significant negative effect (Table I and Figure 1). The increase of

Table III. Regression coefficients for measured responses - acceptance attributes.

Regression coefficients	Appearance	Aroma	Flavor	Texture	Intention to buy	Ideal sweetness	Ideal consistency
Intercept (β_0)	5.98	6.59	6.19	6.04	2.97	-0.28	-0.73
A-$CaCl_2$(β_1)	0.05	-0.09	-0.07	-0.06	0.00	0.02	0.06
B- Carrageenan gum (β_2)	0.00	0.00	-0.11	0.03	-0.08	-0.12	-0.05
C- LM-pectin (β_3)	-0.08	-0.05	-0.22*	-0.28	-0.16*	-019	0.01
A^2 (β_{11})	0.17	0.03	-0.07	0.07	-0.03	0.04	0.17
B^2 (β_{22})	0.08	-0.03	-0.19*	-0.08	.0.09	-0.02	0.02
C^2 (β_{33})	0.11	-0.03	-0.04	0.20	0.00	0.00	0.19
AB (β_{12})	-0.11	-0.12	-0.05	0.02	0.02	-0.21	-0.06
AC (β_{13})	-0.06	-0.07	-0.80	-0.10	-0.09	-0.09	0.00
BC (β_{23})	-0.14	-0.09	-0.90	-0.05	-0.02	-0.06	0.07
R^2	0.53	0.60	0.70	0.47	0.64	0.49	0.36
Lack of fit	0.02	0.26	0.11	0.24	0.09	0.01	0.26

***Significant at 0.05 level.**

pectin and carrageenan concentrations causes the elevation of the °Hue to the central point and after this concentration the °Hue decreases (Figure 1a and Figure 1b). Higher values for this angle are found in the formulations that have the combination of carrageenan gum and LM-pectin in their constitution ranging from between 0.74% to 1.04% and 1.64% to 2.3%, respectively. These formulations exhibit orange (red to yellow) hue.

Regarding the acceptance attributes, the carrageenan gum caused a quadratic effect and LM-pectin caused a negative linear effect the on flavor attribute (Table III), i.e., the higher the concentration of these gelling agents, the lower the scores for the flavor attribute. According to Bayarri et al. (2006), the concentrations of gelling agents alter the mechanical characteristics of gels, altering the perception of taste. Chai et al. (1991), when studying sweetened and flavored gels made with alginate, carrageenan, or agar, noted that sensory perceptions depend on both the strength of the gel and the concentration of gelling agents. In addition, cooking, which is one of the main points of the manufacturing process should be a quick procedure to avoid flavor loss (Doi et al. 2019).

For purchase intention, it is noted that the concentration of LM-pectin caused a negative linear effect (Table III). That is, as LM-pectin concentration increases, purchase intent decreases. This effect can be attributed to the decrease in taste as a consequence of the gelling agents concentration, which may affect the perception of this attribute.

The frequency histograms of ideal sweetness and consistency of different banana preserve formulations are presented in Figure 2.

Formulations F1 (0.61% $CaCl_2$, 0.74% carrageenan gum and 1.64% LM-pectin) and F5 (0.61% $CaCl_2$, 0.74% carrageenan gum and 2.30% LM-pectin) were closest to the ideal sweetness (Figure 2a). For optimal consistency, F1 (0.61% $CaCl_2$, 0.74% carrageenan gum and 1.64% LM-pectin) and F3 (0.61% $CaCl_2$, 1.04% gum carrageenan and 1.64% LM-pectin) were the closest (Figure 2b).

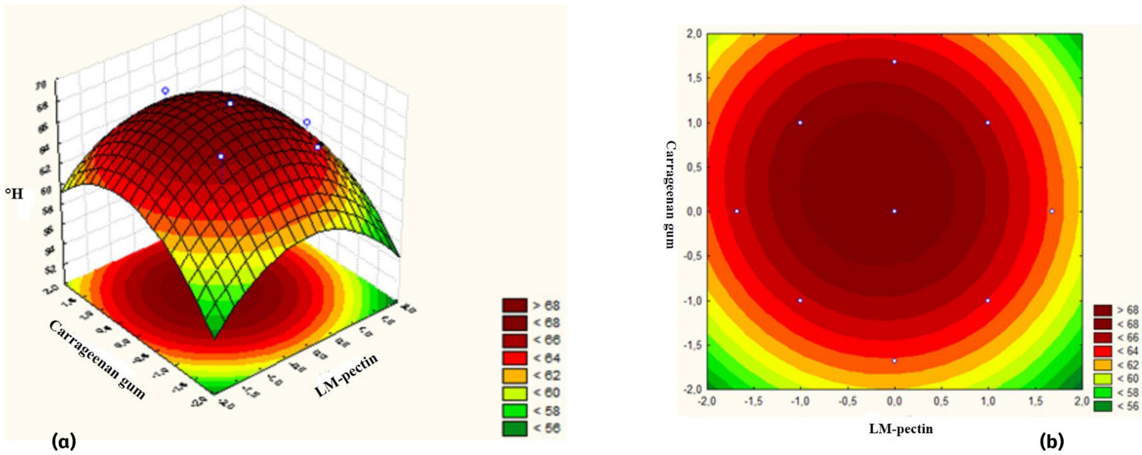

Figure 1. Response surface (a) and contour plot (b) for °Hue of sugar-free banana preserves.

Guichard et al. (1991), investigated the influence of pectin concentration and its methoxylation degree on sensory aspects and volatile compounds in strawberry preserves. The results showed that a 0.6% concentration of LM-pectin provided an adequate texture and ideal taste.

The results of the present study suggest that although the use of gelling agents contributes to both consistency and taste improvements, high concentrations of these additives may mask the sweetness and modify its consistency, resulting in product rejection. Smaller amounts of both carrageenan and pectin provide sweetness and consistency considered ideal by consumers.

Figure 3 shows the principal component analysis (PCA) for the different sugar-free banana preserve formulations in relation to the descriptive terms related to appearance, aroma, flavor, and texture.

Formulations F1 (0.61% $CaCl_2$, 0.74% carrageenan gum and 1.64% LM-pectin), F9 (0.54% $CaCl_2$, 0.89% carrageenan gum and 1.97% LM-pectin), and F13 (0.71% $CaCl_2$, 0.89% carrageenan gum and 1.4% LM-pectin) were described with desirable attributes for banana preserves such as characteristic appearance, aroma, flavor, and consistency, as well as pleasant, uniform, and ideal texture (Figure 3). However, the other formulations were described as having undesirable characteristics for banana preserves, such as a very sweet taste, burnt aroma, presence of syneresis, presence of lumps, lacking luster and opacity, with a weak foreign banana aroma (Figure 3).

Thus, based on the principal component analysis for the different formulations of sugar-free banana preserves (Figure 3), the formulations described with the desirable attributes of banana preserves were those whose concentrations of the studied variables ranged from 0.54% to 0.61% for $CaCl_2$, from 0.74% to 0.89% for carrageenan gum, and from 1.40% to 1.64% for LM-pectin.

CONCLUSION

In the present study, it was found that the different concentrations of $CaCl_2$, carrageenan gum, and LM-pectin influenced the physicochemical and sensory characteristics of sugar-free banana preserves. In general, lower $CaCl_2$ concentrations (0.54% to 0.61%) caused the preserves to have lower pH and more vivid color. The increased concentration of LM-pectin (1.40% to 1.64%) resulted in formulations with a yellowish-red hue and lower moisture, thus,

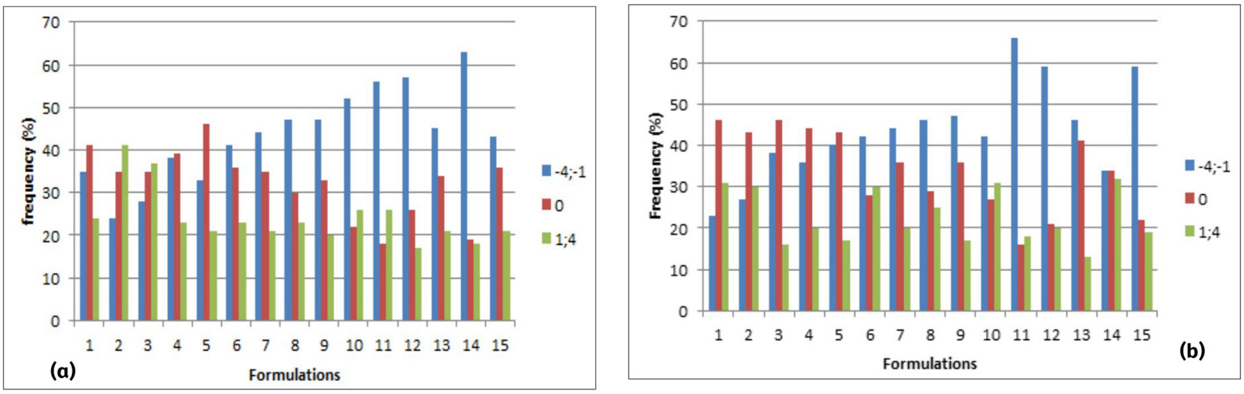

Figure 2. Histograms of sweetness (a) and consistency (b) ideals of different banana preserves formulations. * Formulation 15: average of the values obtained by formulations 15, 16, 17 and 18. (-4;-1 = less sweet/consistent than ideal, 0 = ideal, and 1;4 = sweeter/consistent than ideal).

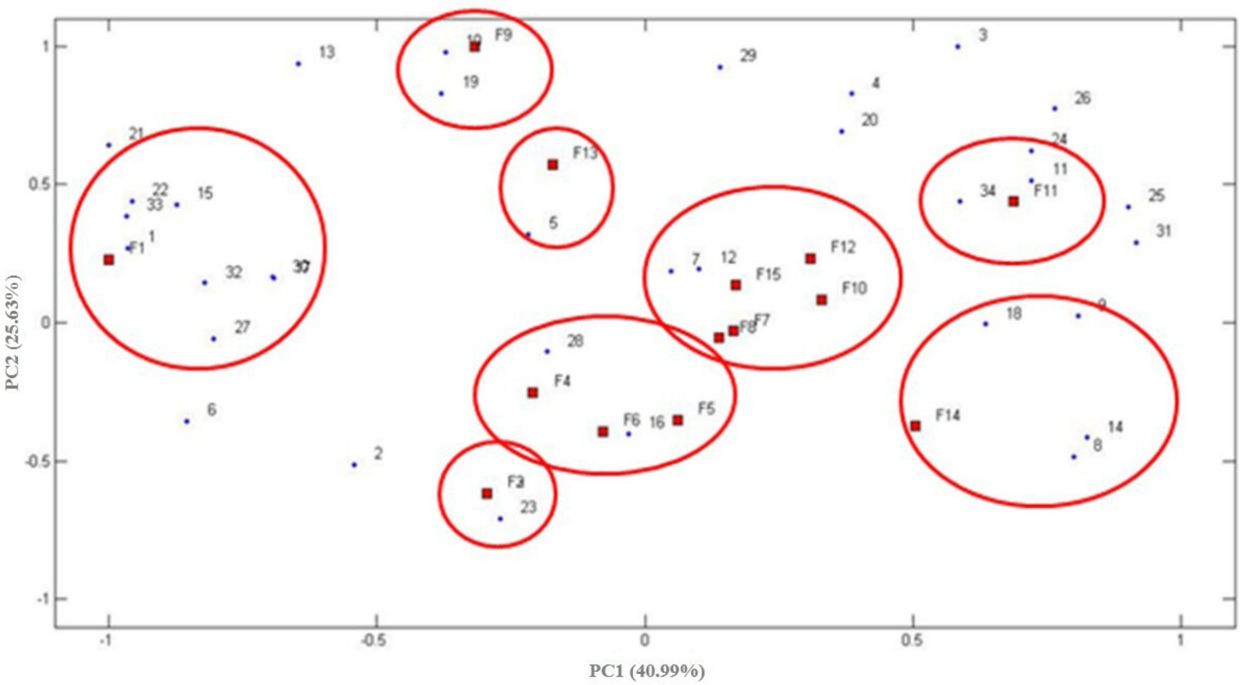

Figure 3. Principal component analysis for the different sugar-free banana preserves formulations. * Formulation 15: average of sensory analysis values obtained by formulations 15, 16, 17 and 18. Descriptors: 1 - characteristic appearance of banana preserve, 2 - strong brown color, 3 - faint brown color, 4 - lighter color than ideal, 5 - darker color than ideal, 6 - ideal color, 7 - bright, 8 - matte, 9 - opaque, 10 - translucent, 11 - lumpiness, 12 - syneresis, 13 - strong banana aroma, 14 - faint banana aroma, 15 - sweet aroma, 16 - burnt aroma, 17 - pleasant aroma, 18 - strange aroma, 19 - characteristic banana preserve aroma, 20 - weak banana flavor, 21 - ideal banana flavor, 22 - characteristic banana preserve taste, 23 - very sweet taste, 24 - slightly sweet taste, 25 - weird taste, 26 - burnt taste, 27 - banana preserve characteristic consistency, 28 - firm consistency, 29 - soft consistency, 30 - nice texture, 31 - nasty texture, 32 - nice texture uniform, 33 - ideal texture, 34 - lumps.

reducing the flavor and purchase intention of the elaborated products.

Carrageenan gum was the variable that least influenced the results; however, the use of higher concentrations of this gum (1.04% to 1.15%) decreases the perception of banana preserve aroma.

Given this, it can be inferred that the use of $CaCl_2$ ranging from 0.54% to 0.61%, carrageenan gum from 0.74% to 0.89% and LM-pectin from 1.4% to 1.64% results in sugar-free banana preserves with ideal sweetness and consistency and are, therefore, more acceptable.

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