# RESEARCH ARTICLE

# Influence of gelling agent concentration on the characteristics of functional sugar-free guava preserves

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## ABSTRACT

Response surface methodology was used to determine the influence of gelling agents and their concentrations on the sensory characteristics and texture of functional sugar-free guava preserves. The gelling agents used were locust bean gum (0.16% to 1.84%), carrageenan (0.16% to 1.84%) and low methoxyl pectin (1.16% to 2.84%). The effects on the sensory and texture characteristics were studied using a central composite design. The results showed that the carrageenan and locust bean gums strongly influenced the texture and sensory characteristics of the functional sugar-free guava preserves. It is recommended that low methoxyl pectin at a concentration of 2.0% can be used together with carrageenan and locust bean gums. The results suggest that for the best scores of the sensory attributes, locust bean gum and carrageenan concentrations ranging from 0.16% to 0.41% must be used. Higher instrumental values of the texture characteristics were achieved with higher concentrations of these two gums.

Keywords: Carrageenan; Locust bean gum; Low methoxyl pectin; Acceptance test; Texture profile test; Stress relaxation test

# INTRODUCTION

The food industry has relied mostly on incremental innovation for its new product launches, but it is becoming increasingly aware that breakthrough, "new to the world" innovations are needed to remain competitive. The modification of food structures to generate novel flavor and texture sensations in products that provide consumers with unique eating experiences is increasing the importance of understanding the relationships between food structure, mastication and sensory perception (Foster et al., 2011). Moreover, due to an alarming increase in diabetes, obesity and other diseases (Hracek et al., 2010; Farias et al., 2019), there is an increased demand for products without added sugar (Acosta et al., 2008) and products that provide health benefits with ingredients, such as bioactive compounds and prebiotics (Foster et al., 2011). However, there are technological problems in the replacement of sugar in food systems because it serves several important functions, such as the development of sweetness and viscosity, contribution to the desired texture, and lowering of the water activity of others functions (Sandrou and Arvanitoyannis, 2000). Therefore, the development of sugar-free products requires the inclusion of many additives, including gelling agents, to provide all the functions of sugar (Hracek et al., 2010; Lima et al., 2019). Among all gelling agents, carrageenan, locust bean gum (LBG) and low methoxyl pectin (LMP) stand out. They act as thickeners, stabilizers and inhibitors of syneresis (Spagnuolo et al., 2005; Ngouémazong et al., 2012). The combined use of these agents have several advantages due to synergistic effects (Ramirez et al., 2002; Mandala et al., 2004), but depending on the type and amount of each gelling agent used, they may change the texture and sensory perception of food (Bayarri et al., 2003, Ishihara et al., 2011, Mesquita et al., 2012). According to Costell et al. (2000) and Bayarri et al. (2007), the higher the concentration of the gelling agents, the lower the perception of taste of the food. This is because increasing the concentration of gelling agents modifies the mechanical properties of the product (Bayarri et al., 2004; Bayarri et al., 2006); thereby, the perception of taste becomes more difficult (Koliandris et al., 2008).

Fruit preserves, such as guava preserves, are largely consumed in Brazil. However, consumption of traditional

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preserves (with sugar added) is decreasing due to the factors previously described (Menezes et al., 2012). Therefore, to retain the historical importance of this product and couple it with new market trends, a new product must be developed with similar properties, both sensory and textural, to the traditional product. It is extremely important to study the interactions between the gelling agents used as well as the optimal concentration of the product. Pereira et al. (2017) evaluated the effect of different additives on the textural properties of functional sugar-free guava preserves. These authors verified that the gelling agents were the additives that most affected the texture of the studied products.

The objective of this study was to evaluate the influence of gelling agent concentration on the characteristics of functional sugar-free guava preserves.

# **MATERIALS AND METHODS**

#### Materials

Was used ripe Pedro Sato guava cultivars from a local market, which were processed in the pilot plant of the Department of Food Science at the Federal University of Lavras.

The ingredients used were as follows: fructooligosaccharides (Beneo P95, São Paulo, SP, Brazil), thaumatin (Nutramax, Catanduva, SP, Brazil), stevioside (Nutramax, Catanduva, SP, Brazil), sucralose (Nutramax, Catanduva, SP, Brazil), gum LBG (Danisco, São Paulo, SP, Brazil), carrageenan (Danisco, São Paulo, SP, Brazil), low methoxyl pectin (LMP) (Danisco, São Paulo, SP, Brazil), polydextrose (Litesse, São Paulo, SP, Brazil), citric acid monohydrate (Nuclear, São Paulo, SP, Brazil), calcium chloride (Vetec, São Paulo, SP, Brazil) and potassium sorbate (Vetec, São Paulo, SP, Brazil).

The use of fructooligosaccharides (FOS) and polydextrose provided body to the product and made them functional because they are not digested or absorbed in the small intestine by modifying the intestinal habitat, thereby causing an increase in stool production and normalization of stool frequency (Rodriguez et al., 2011, Martínez-Cervera et al., 2012).

#### Preparation of guava preserves

The different formulations of guava preserves were processed in open stainless steel pots, according to the methodology proposed by Menezes et al. (2012). A mixture of pulp and polydextrose was heated until a soluble solids content of 45 °Brix was achieved then added to a mixture of gum, pectin LMP and calcium chloride that was previously homogenized under high stirring in water at 80 °C. Cooking was continued until a soluble solids content of 50 °Brix was achieved. FOS diluted 1:1 in water at room temperature was then added, followed by more cooking until a total soluble solids of 65 °Brix was obtained. Citric acid, potassium sorbate and sweeteners (diluted 1:1 in water at room temperature) were added at the end of the cooking process to prevent degradation at high temperatures. The guava preserves were placed in polypropylene containers; the filling process was performed at a high temperature (85 °C). The containers were then sealed and cooled to room temperature before storage in a chamber at 20 °C for later analysis.

All the formulations used the following fixed concentrations: 60.0% guava pulp, 0.025% sucralose, 0.030% stevioside, 0.046% thaumatin, 13.18% FOS, 26.18% polydextrose, 0.2% citric acid, 0.3% calcium chloride and 0.05% potassium sorbate.

#### Sensory evaluation

The acceptance tests for flavor, consistency, sweetness and overall liking of the guava preserves were conducted in a laboratory with 60 consumers using a hedonic scale of 9 points (1 = dislike extremely to 9 = like extremely) (Stone and Sidel, 1993).

The samples, approximately 10.0 g each (Acosta et al., 2008), were served in 50 mL disposable cups at room temperature, following the order of presentation proposed by Wakeling and MacFie (1995). The cups were coded with three-digit numbers taken from a table of random numbers. The tests were performed in individual booths under white light.

#### Texture profile analysis

The texture profile analyses (TPA) were performed in a Stable Micro Systems Model TA-XT2i texturometer (Goldaming, England) at the following speeds over a 20.0 mm distance: pre-test speed of 1.0 mm/s, test speed of 1.0 mm/s, and post-test speed of 1.0 mm/s. Compression was performed with a cylindrical aluminum probe of 6.0 mm. The parameters analyzed were hardness, cohesiveness and gumminess. The tests were performed in quadruplicate. The analyses were conducted in the jars containing the guava preserves.

#### Stress relaxation test

The stress relaxation tests were performed in a texturometer (Stable Micro Systems Model TA-XT2i) according to methodologies proposed by Nobile et al. (2007) and Campus et al. (2010). The samples were cut into cylindrical shapes 2.0 cm in both diameter and height and then compressed to 5.0% of the original height of the sample, with a speed of 1.0 mm/s. The deformation was kept constant for 10.0 minutes, which allowed the stress to reach

equilibrium. During that time, the relaxation of tension was measured at a rate of 1.0 measure per second. A 7.0 cm diameter probe cylinder, which had been lubricated to eliminate the influence of friction between the sample and the equipment, was used. Four measurements were performed for each treatment. The nonlinear regression program R (2011) was used to determine the constants of the Maxwell model.

#### Experimental design and statistical analysis

This study evaluated the effects of three factors (low methoxyl pectin concentration, locust bean gum concentration and carrageenan concentration). A central composite rotational design (CCRD) with  $2^3 + 6 + 4$  points, including axial center points, was applied. The coded and real values of the factors are specified in Table 1. All formulations were adjusted to 100%.

To enable the adjustment of a regression model, axial points were added to make the number of data points greater than the number of estimated parameters. The results of all analyses were evaluated by the response surface methodology using the software STATISTICA<sup>TM</sup>, Version 8.0 for Windows (StatSoft®).

The criterion used to accept the proposed model were the high coefficient of determination  $(R^2)$  and analysis of variance.

To correlate the rheological properties with the sensory attributes, the Pearson correlation in SAS for Windows, version 5.0 was used.

 
 Table 1: Levels and values of the independent variables of the central composite rotational design (CCRD)

Trials	Coc	led Varia	bles	Real Variables			
	<b>X</b> <sub>1</sub>	<b>X</b> <sub>2</sub>	X <sub>3</sub>	<b>X</b> <sub>1</sub> (%)*	X <sub>2</sub> (%)*	X <sub>3</sub> (%)*	
1	-1	-1	-1	1.5	0.5	0.5	
2	+1	-1	-1	2.5	0.5	0.5	
3	-1	+1	-1	1.5	1.5	0.5	
4	+1	+1	-1	2.5	1.5	0.5	
5	-1	-1	+1	1.5	0.5	1.5	
6	+1	-1	+1	2.5	0.5	1.5	
7	-1	+1	+1	1.5	1.5	1.5	
8	+1	+1	+1	2.5	1.5	1.5	
9	-1.68	0	0	1.16	1.0	1.0	
10	+1.68	0	0	2.84	1.0	1.0	
11	0	-1.68	0	2.0	0.16	1.0	
12	0	+1.68	0	2.0	1.84	1.0	
13	0	0	-1.68	2.0	1.0	0.16	
14	0	0	+1.68	2.0	1.0	1.84	
15	0	0	0	2.0	1.0	1.0	
16	0	0	0	2.0	1.0	1.0	
17	0	0	0	2.0	1.0	1.0	
18	0	0	0	2.0	1.0	1.0	

 $x_1$ =pectin LMP;  $x_2$ =locust bean gum;  $x_3$ =carrageenan. \*percentage relative to the whole system

## **RESULTS AND DISCUSSION**

#### Sensory evaluation

The results of the analysis of variance (*F*-test) were shown in Table 2. The complete predicted models were presented through equations 1, 2, 3 and 4. The determination coefficients ( $\mathbb{R}^2$ ) were high and varied between 0.80 and 0.92.

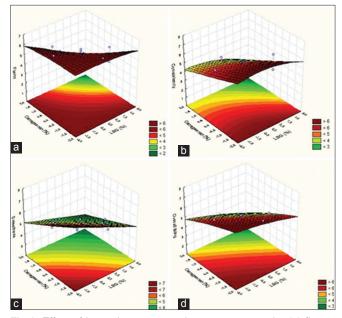
Flavor =  $6.30^{**} - 0.13X_1 - 0.05X_1^2 - 0.55X_2^{**} - 0.05X_2^2 - 0.57X_3^{**} - 0.04X_3^2 - 0.01X_1X_2 + 0.06X_1X_3 - 0.23X_2X_3(1)$ Consistency =  $6.20^{**} - 0.21X_1 - 0.05X_1^2 - 0.37^*X_2 - 0.14X_2^2 - 0.75X_3^{**} + 0.01X_3^2 - 0.13X_1X_2 + 0.17X_1X_3 - 0.08X_2X_3(2)$ Sweetness =  $6.23^{**} - 0.05X_1 - 0.11X_2^2 - 0.50X_2^{**}$ 

$$5weetness = 6.23^{**} - 0.05X_1 - 0.11X_1^2 - 0.50X_2^{**} + 0.00X_2^2 - 0.54X_3^{**} + 0.01X_3^2 + 0.02X_1X_2 + 0.01X_1X_3 + 0.05X_2X_3$$
(3)

 $\begin{aligned} \text{Overall liking} &= 6.22^{**} - 0.14 X_1 - 0.10 X_1^2 - 0.42 X_2^{**} - 0.03 X_2^2 \\ &- 0.63 X_3^{**} - 0.04 X_3^2 - 0.01 X_1 X_2 + 0.10 X_1 X_3 - 0.05 X_2 X_3 (4) \end{aligned}$ 

The effects of LBG and carrageenan on the sensory attributes of the functional sugar-free guava preserves were shown in Fig. 1. The concentration of low methoxyl pectin was fixed at the value of the focal point because it does not show any significant effect on the sensory attributes (Table 2).

Based on the mean hedonic ratings of flavor, consistency, sweetness and overall liking, the preserves with lower levels of carrageenan and locust bean gum were more accepted (Fig. 1).



**Fig 1.** Effect of locust bean gum and carrageenan on the (a) flavor, (b) consistency, (c) sweetness, and (d) overall liking of the functional sugar-free guava preserves.

Table 2: Analysis of variance and predicted models of the sensory attributes of the functional	sugar-free	e guava preserves
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Factors	df	Flavor		Consistency		Sweetness		Overall liking	
		Mean square	F-ratio	Mean square	F-ratio	Mean square	F-ratio	Mean square	<i>F</i> -ratio
1 LMP (L)	1	0.23	0.79	0.60	3.43	0.03	0.30	0.25	2.37
LMP (Q)	1	0.03	0.10	0.03	0.16	0.17	1.52	0.13	1.24
2 LBG (L)	1	4.08	14.00**	1.84	10.44*	3.41	31.32**	2.38	22.41**
LBG (Q)	1	0.03	0.09	0.25	1.43	0.00	0.00	0.01	0.13
3 CAR (L)	1	4.38	15.05**	7.69	43.66**	3.95	36.28**	5.44	51.24**
CAR (Q)	1	0.02	0.07	0.00	0.01	0.00	0.01	0.02	0.16
1x2	1	0.00	0.00	0.13	0.74	0.00	0.04	0.00	0.01
1x3	1	0.03	0.09	0.23	1.31	0.00	0.01	0.08	0.73
2x3	1	0.44	1.50	0.05	0.27	0.02	0.16	0.02	0.20
Error	8	0.29	-	0.18	-	0.11	-	0.11	-
Total	17	-	-	-	-	-	-	-	-
R <sup>2</sup>	-	-	0.80	-	0.88	-	0.90	-	0.91

L- linear; Q- quadratic; R<sup>2</sup>- coefficient of determination; \*p<0.05; \*\*p<0.01

Locust bean gum  $(x_2)$  and carrageenan  $(x_3)$  significantly decreased the flavor attribute. The coefficients of linear regression of both factors were negative (Table 2 and Fig. 1a), indicating that as the concentrations of locust bean gum and carrageenan increased, the flavor notes decreased. The perception of flavor is considered to be a combination (in the brain) of two senses, the sense of smell and the sense of taste. Therefore, flavor can be broken down into two major components, the volatile compounds that are sensed by the olfactory epithelium (aroma) and the non-volatile compounds that are sensed by the taste buds on the tongue (taste). As food is consumed, there are various factors that may influence the release of the volatile components, as well as the tastant. These factors include the breaking of the structure upon mastication and mixing with saliva (Koliandris et al., 2008). It has been reported that the intensity of flavor perception decreases with increased viscosity (Lima et al., 2019). According Bayarri et al. (2004) and Bayarri et al. (2006), the concentration of gelling agents modifies the mechanical properties (diffusion) of the gels, influencing the perception of flavor, ie increasing the concentration of these agents decreases the perception of flavor. Arda et al. (2009) concluded that the use of carrageenan gum and LBG gum mixtures increases gel strength and water binding capacity, as well as modifying gel texture making it more elastic and resistant. Thus, it can be inferred that this mechanism makes the perception of flavor lower. The highest scores for the flavor attribute are achieved with carrageenan concentrations ranging from 0.16% to 0.91% (Fig. 1a). As for the locust bean gum, the variation was from 0.16% to 1.0%.

With regard to consistency, it is observed (Table 2 and Fig. 1b) that increasing the concentration of carrageenan and locust bean gum reduces the sensory evaluation scores for consistency. Carrageenan had a greater effect on this decrease than locust bean gum (Fig. 1b). Formulations with higher carrageenan concentrations exhibited higher values

of hardness (Fig. 2a). According to Cakir et al. (2012), the development of low-calorie products is often perceived by consumers as less texturally attractive because, according to Rogers et al. (2009), an increase in firmness of a product (by the presence of gelling agents) allows a lower degree of decomposition during mastication, thereby reducing its acceptance. According to Fig. 1b, higher scores for the consistency attribute is achieved with concentrations of carrageenan from 0.16% to 0.41% and concentrations of locust bean gum from 0.16% to 1.0%.

The sweetness was affected negatively by factors independent of the linear terms x<sub>2</sub> (locust bean gum) and  $x_{a}$  (carrageenan) (i.e., the concentration of carrageenan and locust bean gum in the functional sugar-free guava preserves decreased sweetness). The highest scores for the sweetness attribute were obtained using concentrations of carrageenan and locust bean gum between 0.16% and 0.41% (Fig. 1c). Several studies have reported that the concentration of gelling agents reduces the perception of the sweetness of the sweetener used (Cook et al., 2003; Bayarri et al., 2003; Bayarri et al., 2007; Koliandris et al., 2008). Bayarri et al. (2003) studied the sweetness of gels made of gellan gum and carrageenan. They reported that there is a greater perception of sweetness with lower concentrations of these gums. According to Gibson (1992), a gel with less firmness disintegrates more easily in the mouth, releasing the sweetener faster. Cook et al. (2003) noted that the sweetness of sucrose was reduced in guar solutions, while that of aspartame was reduced in  $\lambda$ -carrageenan solutions. Both the nature of the thickening agent (carboxymethylcellulose, xanthan and pectin) and viscosity influenced the sweetness of apple juice (Walker and Prescott, 2000). Increased concentrations of the gelling agent increased the firmness (instrumental) and decreased the sweetness perception in pectin gels which were similarly observed for carrageenan, alginate, agar (Chai et al., 1991) and gellan gels (Costell et al., 2000).

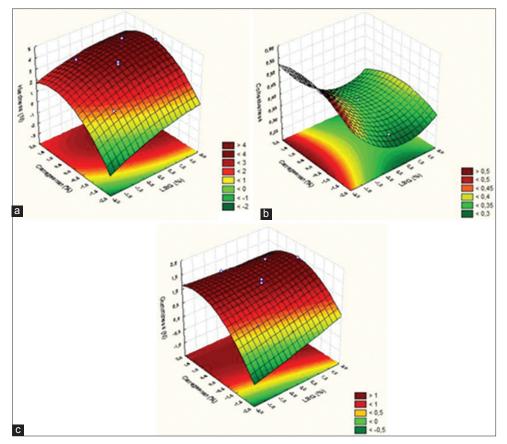


Fig 2. 3D graphic surface optimization of the texture profile parameters of the functional sugar-free guava preserves: (a) hardness (N), (b) cohesiveness, and (c) gumminess (N).

Regarding overall liking, the same response as with the other attributes evaluated was observed; the concentration of the gums in this study reduced overall liking. Higher scores for this attribute were observed with concentrations of locust bean gum and carrageenan ranging between 0.16% and 0.41% (Fig. 1d).

#### Evaluation of the texture profile analysis

The *F*-test results were shown in Table 3. In equations 5, 6 and 7 the complete predicted models were presented. The determination coefficients ( $\mathbb{R}^2$ ) were high and varied between 0.79 and 0.92.

$$\begin{aligned} \text{Hardness} &= 3.23^{**} + 0.32 X_1 + 0.01 X_1^2 + 0.51 X_2^* - 0.12 X_2^2 \\ &+ 0.82 X_3^{**} - 0.48 X_3^{2*} - 0.12 X_1 X_2 + 0.35 X_1 X_3 - 0.08 X_2 X_3(5) \end{aligned}$$

$$Cohesivenes = 0.37^{**} + 0.002X_1 - 0.02X_1^{2*} - 0.03X_2^{**} + 0.05X_2^{2**} + 0.01X_3 - 0.01X_3^{2} + 0.01X_1X_2 + 0.001X_1X_3 - 0.004X_2X_3$$
(6)

 $\begin{aligned} & \text{Gumminess} = 1.19^{**} + 0.13X_1 - 0.05X_1^2 + 0.11X_2 + 0.01X_2^2 \\ &+ 0.32X_3^{**} - 0.19X_3^{2*} - 0.03X_1X_2 + 0.13X_1X_3 - 0.07X_2X_3(7) \end{aligned}$ 

TPA is a method to evaluate sensory properties. The test consists of uniaxially compressing the food (study sample)

twice in a reciprocating motion to mimic the action of the mandible. Therefore, an initial compression and relaxation followed by a second compression were performed during testing. This test yields a graph of force versus time, from which the texture parameters are calculated (Bourne, 2002; Herrero et al., 2007).

Fig. 2 shows the 3D graphic surface optimization of the texture profile parameters of the functional sugar-free guava preserves. The value of low methoxyl pectin was fixed at the center point for the parameters of hardness and gumminess because this independent variable did not significantly affect these parameters (Table 3). For the analysis of cohesiveness, the level of carrageenan was set at the central point because it did not affect this parameter (Table 3).

For hardness, only the linear effects of locust bean gum  $(x_2)$  and carrageenan  $(x_3)$  and the negative quadratic effect of carrageenan  $(x_3)$  were significant (Table 3 and Fig. 2a). That is, the concentration of locust bean gum in the guava preserves increased hardness, and higher hardness values were obtained with LBG concentrations ranging from 1.16% to 1.66% (Fig. 2a). LBG does not form gel

Fatores df		Hardness (N)		Cohesive	ness	Gumminess (N)	
		Mean square	F-ratio	Mean square	F-ratio	Mean square	<i>F</i> -ratio
1 LMP (L)	1	1.44	3.07	0.00	0.14	0.22	2.78
LMP (Q)	1	0.00	0.00	0.00	8.21*	0.03	0.34
2 LBG (L)	1	3.54	7.56*	0.02	42.30**	0.18	2.22
LBG (Q)	1	0.17	0.36	0.01	20.27**	0.00	0.01
3 CAR (L)	1	9.19	19.61**	0.00	3.64	1.37	17.24**
CAR (Q)	1	2.91	6.22*	0.00	4.83	0.47	5.95*
1x2	1	0.11	0.23	0.00	1.92	0.01	0.12
1x3	1	0.99	2.11	0.00	0.01	0.14	1.80
2x3	1	0.05	0.10	0.00	0.38	0.04	0.45
Error	8	0.47	-	0.00	-	0.08	-
Total	17	-	-	-	-	-	-
R <sup>2</sup>	-	-	0.83	-	0.92	-	0.79

Table 3: Analysis of variance and predicted models of parameters of the texture profiles of the functional sugar-free guava preserves

L- linear; Q- quadratic; R<sup>2</sup>- coefficient of determination; \*p<0.05; \*\*p<0.01

on its own (Hamdani et al., 2019). However, when it is combined with other polysaccharides, such as carrageenan and pectin, locust bean gum can form gels (Azero and Andrade, 2006; Bourbon et al., 2010). The increased hardness induced by increasing the amount of locust bean gum in the product may be due to complex interactions involving all the gelling agents used in this study, which make the gel networks more rigid. Similar results were obtained by Alloncle and Doublier (1991), who studied starch gels and hydrocolloids. These authors concluded that the increase in hardness was caused by the increase in locust bean gum concentration, which was due to the modification of the balance between aggregation and separation of the gel. Arocas et al. (2009) studied white sauces with added starch, xanthan gum and locust bean gum, and they observed the same behavior with locust bean gum and that the concentration of starch increased the consistency of the dressing (measured in a rheometer). With regard to carrageenan, it increased hardness up to maximum concentrations between 1.0% and 1.41% (Fig. 2a). Beyond these concentrations, hardness decreases. Spagnuolo et al. (2005) reported that the carrageenan molecule is very flexible and may form a more ordered structure in the form of a double helix, which may lead to gel formation at high concentrations. The gelation process is highly influenced by many factors, such as the type and concentration of salts in the solution, cooling and heating rates, concentration of the hydrocolloid and the presence of other biopolymers. Modifications of these factors greatly affect the gelling and the rheological properties of the gels (Baeza et al., 2002). In this study, was used 0.3% CaCl<sub>2</sub> to promote gelling of both the low methoxyl pectin and carrageenan. Increasing the concentration of carrageenan in the system destructured the double helices of this gelling agent (by binding of the sulphated groups of carrageenan to calcium), thus affecting the balance of the attractive and repulsive forces between the molecules, causing decreased rigidity of the gel (Pérez-Mateos and Montero, 2002). Karim et al. (2009) evaluated the effect of carrageenan in tofu and also observed a decrease in hardness with increasing carrageenan content. These authors attributed this fact to the way protein interacts with calcium and other constituents (e.g., phytic acid) in soy milk and anions that form the microstructure that determines the hardness of tofu, which together with carrageenan, causes the gel strength to decrease.

Low methoxyl pectin  $(x_1)$  had a negative quadratic effect on cohesiveness (Table 3 and Fig. 2b). That is, increasing pectin concentration increased cohesiveness up to a maximum value and then decreased, which was observed at concentrations between 1.16% and 2.0% (Fig. 2b). The decrease may be due to syneresis because the concentration of pectin gels was strong enough to expel water from the system (Thrimawithana et al. 2010), making the gel more prone to disintegration in the first compression cycle (Extralab, 2010). As for locust bean gum ( $x_2$ ), it had a linear and positive quadratic effect on this parameter of the texture profile. As shown in Fig. 2b, higher values of cohesiveness are achieved with lower concentrations of locust bean gum (0.16% to 0.30%).

Gumminess was significantly influenced (positive linear effect and negative quadratic effect) by carrageenan  $(x_3)$  (Table 3 and Fig. 2c). Increasing the concentration of carrageenan in the functional sugar-free guava preserves increased gumminess to a threshold value then subsequently decreased. The concentration of carrageenan in the functional sugar-free guava preserves an optimal region of hardness (Fig. 2c) at concentrations ranging from 1.16% to 1.66%. At higher levels, there was a decrease in hardness, confirming the results obtained for the hardness parameter.

#### Evaluation of the stress relaxation test

The *F*-test results were shown in Table 4. The complete predicted models were presented through equations 8, 9, 10 and 11. The determination coefficients ( $\mathbb{R}^2$ ) were high and varied between 0.79 and 0.83.

$$\begin{split} & \mathrm{E_e} = 15.91^{**} + 2.87\mathrm{X_1} + 4.97\mathrm{X_1}^2 - 0.15\mathrm{X_2} - 0.56\mathrm{X_2}^2 \\ & + 9.08\mathrm{X_3}^{**} - 0.48\mathrm{X_3}^2 - 4.94\mathrm{X_1}\mathrm{X_2} + 2.12\mathrm{X_1}\mathrm{X_3} - 1.67\mathrm{X_2}\mathrm{X_3}(8) \\ & \mathrm{E_1} = 12.54^{**} + 0.05\mathrm{X_1} + 3.53\mathrm{X_1}^2 - 2.25\mathrm{X_2} + 1.61\mathrm{X_2}^2 \\ & + 7.67\mathrm{X_3}^{**} + 0.07\mathrm{X_3}^2 - 0.52\mathrm{X_1}\mathrm{X_2} + 0.24\mathrm{X_1}\mathrm{X_3} - 1.50\mathrm{X_2}\mathrm{X_3}(9) \\ & \lambda = 105.16^{**} + 0.75\mathrm{X_1} - 12.19\mathrm{X_1}^2 - 11.67\mathrm{X_2} + 5.12\mathrm{X_2}^2 \\ & + 22.19\mathrm{X_3}^{**} - 5.10\mathrm{X_3}^2 - 2.78\mathrm{X_1}\mathrm{X_2} + 0.48\mathrm{X_1}\mathrm{X_3} - 0.81\mathrm{X_2}\mathrm{X_3}(10) \\ & \eta = 1271.55^{**} + 2.88\mathrm{X_1} + 81.01\mathrm{X_1}^2 - 496.17\mathrm{X_2} \\ & + 374.23\mathrm{X_2}^2 + 1042.06\mathrm{X_3}^{**} + 107.37\mathrm{X_3}^2 \end{split}$$

$$-153.29X_{1}X_{2} + 92.09X_{1}X_{3} - 189.70X_{2}X$$
 (11)

Fig. 3 shows the 3D graphic surface optimization of the Maxwell model parameters of the functional sugar-free guava preserves. This model was chosen because there was a considerable improvement in R<sup>2</sup> when the generalized Maxwell model of two elements of a spring in parallel was tested (Nobile et al., 2007; Campus et al., 2010). The low methoxyl

pectin concentration was set at the central point for all Maxwell model parameters because this independent variable did not significantly affect any of the parameters (Table 4).

It was observed that the only variable that was linearly independent and positively affected all the Maxwell model parameters was carrageenan ( $x_3$ ). The concentration of this gelling agent increased the rigidity of the material since the parameters  $E_e$  and  $E_1$  quantified this rigidity (Peleg, 1987; Rodriguez-Sandoval, 2009; Palanisamy, et al. 2018). The parameter  $\lambda$  indicated that the greater the relaxation time, the greater the elastic behavior and the firmer the material (Nobile et al., 2007; Campus et al., 2010). The parameter  $\eta$  indicated that the higher its value, the more solid the material (Rodriguez-Sandoval, 2009). These results contradict those obtained from the texture profile analysis. Higher values of  $E_e$ ,  $E_1$  and  $\eta$ were obtained with concentrations of carrageenan between 1.66% and 1.84% and higher values of  $\lambda$  were obtained at concentrations between 1.41% and 1.84% (Fig. 3a-d).

# Correlation between the sensory and rheological parameters

The Pearson correlation coefficient between the sensory and rheological parameters of the functional sugar-free guava preserves was shown in Table 5.

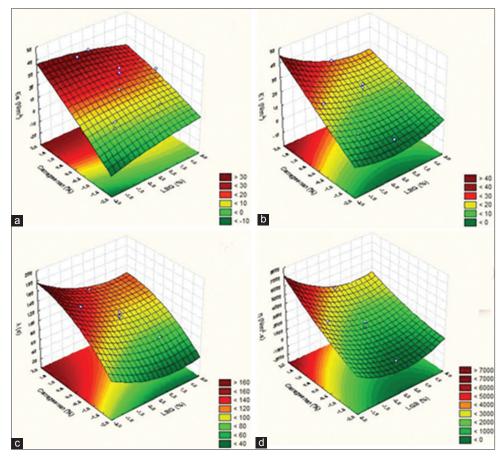


Fig 3. 3D graphic surface optimization of the stress relaxation of the Maxwell model parameters of the functional sugar-free guava preserves: (a) equilibrium elastic modulus ( $E_a$ ); (b) elastic modulus of the ideal elastic body ( $E_i$ ); (c) relaxation time ( $\lambda$ ); and (d) viscosity ( $\eta$ ).

Factors	df	E		Ε,		λ		η	
		Mean square	<i>F</i> -ratio	Mean square	F-ratio	Mean square	F-ratio	Mean square	<i>F</i> -ratio
1 LMP (L)	1	112.67	1.84	0.04	0.00	7.75	0.01	113	0.00
LMP (Q)	1	311.67	5.09	157.14	5.84	1875.48	3.21	82775	0.14
2 LBG (L)	1	0.30	0.00	68.85	2.56	1857.16	3.18	3359196	5.70
LBG (Q)	1	3.92	0.06	32.56	1.21	330.39	0.57	1766286	3.00
3 CAR (L)	1	1124.88	18.39**	803.27	29.86**	6718.77	11.51**	14816775	25.14**
CAR (Q)	1	2.90	0.05	0.07	0.00	327.54	0.56	145380	0.25
1x2	1	194.95	3.19	2.18	0.08	62.03	0.11	187994	0.32
1x3	1	36.12	0.59	0.45	0.02	1.84	0.00	67848	0.12
2x3	1	22.21	0.36	18.00	0.67	5.26	0.01	287877	0.49
Error	8	61.18	-	26.90	-	583.80	-	589364	-
Total	17	-	-	-	-	-	-	-	-
R <sup>2</sup>	-	-	0.79	-	0.83	-	0.71	-	0.81

Table 4: Analysis of variance and predicted models of the parameters of the Maxwell model of the functional sugar-free guava preserves

L- linear; Q- quadratic; R<sup>2</sup>- coefficient of determination; \*p<0.05; \*\*p<0.01

Ee: equilibrium elastic modulus (N/m<sup>2</sup>); E1: elastic modulus of the ideal elastic body (N/m<sup>2</sup>); λ: relaxation time (s); η: viscosity (N/m<sup>2</sup>.s)

Table 5: Pearson correlation coefficient between the sensory
and rheological parameters

			liking
0.66**	-0.71**	-0.75**	-0.74**
0.22	-0.06	0.29	0.17
0.60**	-0.71**	-0.66**	-0.69**
0.53*	-0.57*	-0.54*	-0.63**
-0.34	-0.50*	-0.38	-0.49*
-0.24	-0.40	-0.15	-0.27
-0.27	-0.43	-0.24	-0.37
	0.66** 0.22 0.60** 0.53* -0.34 -0.24 -0.27	0.22         -0.06           0.60**         -0.71**           0.53*         -0.57*           -0.34         -0.50*           -0.24         -0.40	0.22         -0.06         0.29           0.60**         -0.71**         -0.66**           0.53*         -0.57*         -0.54*           -0.34         -0.50*         -0.38           -0.24         -0.40         -0.15

\*p<0.05; \*\*p<0.01

Hardness, gumminess and E<sub>e</sub> were negatively correlated with all the sensory attributes studied, while E<sub>1</sub> was the only negatively correlated attribute with overall liking. Cohesiveness,  $\lambda$  and  $\eta$  were not correlated to any of the sensory attributes studied. These results indicate that increasing the texture parameters (i.e., the concentration of gelling agents in the product) decrease the scores of the sensory attributes decrease, indicating that consumers prefer a functional sugar-free guava preserve with low concentrations of gelling agents. As reported in several studies (Gibson, 1992; Costell et al., 2000; Rogers et al., 2009; Thrimawithana et al., 2010), the increase in gelling agents causes an increase in the rigidity of the gel but makes it more brittle and cohesive, therefore making it difficult to dissolve in the mouth and reduces the product's acceptance.

### CONCLUSIONS

The present study indicated that response surface methodology was a useful experimental technique in the evaluation of the effects and appropriate concentrations of gelling agents on the texture and sensory characteristics of functional sugar-free guava preserves. The results indicated that the independent variables of carrageenan and locust bean gum had the most influence on the texture and sensory characteristics of the functional sugar-free guava preserves and that low methoxyl pectin can be used at a concentration of 2.0% together with locust bean gum and carrageenan. We also conclude that higher sensory scores were achieved at low concentrations of locust bean gum and carrageenan. In relation to the texture parameters, the highest values were obtained with high concentrations of the two gums. Negative correlations were observed between the sensory attributes and texture parameters, indicating that there is greater acceptability of functional guava preserves without added sugar and with concentrations of locust bean gum and carrageenan ranging from 0.16% to 0.41%.

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#### Author contributions

Patrícia Aparecida Pimenta Pereira, Vanessa Rios de Souza and Andressa Alvarenga Silva conducted the experiments and collected the data; Fabiana Queiroz and Soraia Vilela Borges performed the rheological measurements; Ana Carla Marques Pinheiro was involved in sensory analyses, João de Deus Souza Carneiro and Fabiana Queiroz were involved in manuscript preparation and supervised the research project.

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