

Sugar-free guava preserve: influence of additives on textural properties

¹Pereira, P. A. P., ²Souza, V. R., ²Vieira, M. A., ²Queiroz, F., ²Borges, S. V. and ²Carneiro, J. D. S.

¹Department of Food, Federal University of Ouro Preto, Ouro Preto, MG, Brazil.

²Department of Food Science, Federal University of Lavras, Lavras-MG, Brazil.

Article history

Received: 19 August 2016

Received in revised form:

1 October 2016

Accepted: 4 October 2016

Abstract

The development of sugar-free products requires the inclusion of many additives to provide all of the characteristics of sugar products. Thus, the present study aimed to evaluate the effect of different additives (fructooligosaccharide, thaumatin, sucralose, stevioside, maltitol, xanthan gum, locust bean gum, carrageenan, low methoxyl pectin and polydextrose) on the textural properties of functional sugar-free guava preserves. The Plackett and Burman design with 19 tests was used. Texture profile analysis and stress relaxation test were performed. The results were analyzed by analysis of the effects and principal component analysis. The low methoxyl pectin positively affected some parameters of the texture profile (hardness and gumminess) and relaxation test properties (η_2 and k_1). The other ingredients (except maltitol, which did not affect any textural parameters) affected only the texture profile parameters. The results also indicated that higher concentrations of gelling agents in the product resulted in greater influences on the textural properties.

Keywords

Processing

Gelling agents

Sweeteners

Bulking agents

© All Rights Reserved

Introduction

Humans are predisposed to like sweet foods, and this trend along with a sedentary lifestyle has led to an alarming increase in diabetes and obesity, increases, so, the demand for sugar-free products (Acosta *et al.*, 2008; Hracek *et al.*, 2010). However, there are technological problems for the replacement of sugar in food systems because sugar has several important roles, such as developing the sweetness, viscosity and desired texture as well as lowering the water activity (Sandrou and Arvanitoyannis, 2000).

Guava preserves are appreciated in Brazil and result from the processing of the edible parts of healthy guavas combined with sugar; the preserves can be made with or without the addition of water, gelling agents and pH adjusters as well as other ingredients and additives used for proper consistency (Pereira *et al.*, 2013). In producing this product, sugar has an important role because gelation of high methoxyl pectin only occurs in the presence of a co-solution (usually sugar in high concentrations) (Thakur *et al.*, 1997). For these above mentioned reasons, the development of sugar-free products requires the inclusion of many additives, including sweeteners, bulking agents, preservatives and gelling agents, to provide all of the functions of sugar (Hracek *et al.*, 2010).

Gelling agents include xanthan gum, carrageenan, locust bean gum (LBG) and low methoxyl pectin (LMP). Xanthan gum has a high industrial interest mainly in the food, pharmaceutical and petrochemical industries due to its physicochemical properties, particularly its high viscosity in aqueous solutions at low concentrations (0.05 to 1.0%), branched structure, high molecular weight, stability in a wide range of temperatures, pH and shear-thinning behavior (Ramírez *et al.*, 2002). Locust bean gum is often used in the food, pharmaceutical and cosmetic industries acting as a thickener, emulsion stabilizer and syneresis inhibitor as well as providing stability in the pH range of 3.5 to 11.0 (Arda *et al.*, 2009). Carrageenan is a biopolymer galactose that is soluble in water and is classified into the following three main fractions: λ -, ι - and κ -carrageenan. Moreover, carrageenan molecules are flexible but can form a more ordered structure in the form of double helices at high concentrations, which may lead to gel formation; therefore, they are used as gelling agents and stabilizers (Dunstan *et al.*, 2001; Spagnuolo *et al.*, 2005). Low methoxyl pectin (LMP) forms a gel in the presence of divalent metal ions (typically calcium) without the requirement of sugars (Ngouémazong *et al.*, 2012).

There are numerous studies that have demonstrated the advantages of using a combination

*Corresponding author.

Email: patriciaaparecidapimentapereira@yahoo.com.br

of these gelling agents (Dunstan *et al.*, 2001; Ramirez *et al.*, 2002; Mandala *et al.*, 2004; Arda *et al.*, 2009). According to Arda *et al.* (2009), using a mixture of carrageenan and LBG causes an increase in the gel strength and binding ability with water. Moreover, this mixture changes the texture of the gel, making it more elastic and durable, but these effects occur only until a certain concentration of LBG is reached; beyond that concentration, these effects decrease. According to those authors, this phenomenon is called a synergistic peak, and this peak is observed with a carrageenan/LBG ratio of 8:1. The observed synergistic interactions between the locust bean gum (LBG) and xanthan gum are mainly governed by regions free of galactose and mannose chains (Fernandes and Figueiredo, 1995). According to Sandolo *et al.* (2010), gelation occurs through a direct connection between these two polymers rather than a thermodynamic reaction, and this reaction results in a net force that depends on the preparation temperature and the weight ratio between the two components.

The sweeteners used in sugar-free products should have the following characteristics: no aftertaste; ability to promote the functional properties of sucrose, including chemical stability; low calories; sweetening power equal to or higher than that of sucrose; soluble; non-toxic; not carcinogenic; and affordable (Hanger *et al.*, 1996). Sucralose, stevioside and thaumatin are among the many sweeteners found on the market. Sucralose is characterized by a taste similar to sucrose and a lack of unpleasant aftertaste. 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 (Rahn and Yaylayan, 2010). Stevioside is already a natural sweetener that has a sweetness power 150-300 times greater than sucrose, but it has a strong residual bitter taste. Stevioside has a wide application in the food industry due to its stability against heat and a wide pH range (Catharino and Santos, 2011). Thaumatin is a protein that is intensely sweet (300 to 3000 times sweeter than sucrose) (Menu-Bouaouiche *et al.*, 2003) and is used as a sweetener in chewing gum, dairy products and the pharmaceutical industry (Daniell *et al.*, 2000).

In the preparation of sugar-free preserves, it is necessary to use bulking agents, such as maltitol, polydextrose and fructooligosaccharide (FOS). The ability of maltitol to provide "body" is similar to sucrose because it possesses good thermal stability, chemical stability and enzymatic stability. In addition, maltitol leaves no aftertaste or cooling sensation, and it has a sweetness power that is approximately 80-90% that of sucrose (Ronda *et*

al., 2005). Polydextrose improves food texture and functions as a thickener and stabilizer. In addition to moist, it is extremely stable over a wide range of pH values, temperatures, processing conditions and storage conditions (Montenegro *et al.*, 2008). Moreover, polydextrose is considered a functional food because it is fermented in the large intestine but is not digested or absorbed in the small intestine, and it is mostly excreted in feces (Paucar-Menacho *et al.*, 2008). In addition to being an agent that provides body, FOS is also considered a functional ingredient because it is not digested or absorbed in the small intestine and results in modification of the intestinal habitat, thereby causing an increase in stool. The normalization of stool frequency exerts a prebiotic effect (increases the number of bacteria and/or activity of the number of bifidobacteria and lactic acid bacteria in the human intestine) (Cherbut, 2002; Nyman, 2002; Roberfroid, 2007; Rodríguez-Cabezas *et al.*, 2010).

There have been numerous studies evaluating the effect of these components in model systems (Dunstan *et al.*, 2001; Ramirez *et al.*, 2002; Mandala *et al.*, 2004; Spagnuolo *et al.*, 2005; Arda *et al.*, 2009; Ngouémazong *et al.*, 2012), but studies in complex systems, such as in fruit preserves, are scarce. Thus, the aim of this study was to evaluate the effect of additives on the textural properties of a complex system (functional sugar-free guava preserves) and to identify the ingredients that influence these properties.

Materials and Methods

Processing of guava preserves

Ripe guavas (Pedro Sato cultivar) were used from a local market. The guavas were processed in the Pilot Plant Processing Plant Products in the Department of Food Science at the Federal University of Lavras/MG. The fruits were washed in running water, sanitized in a 200 mg L⁻¹ sodium hypochlorite solution for 15 min., selected, pulped in an electrical depulper (sieve of 6.0 mm diameter), packed in polyethylene bags of low density and frozen at -18°C according to the methodology proposed by Menezes *et al.* (2009). The following ingredients were used: fructooligosaccharide (Beneo P95), thaumatin, sucralose, stevioside, maltitol (Nutramax Catanduva, Brazil), xanthan gum, LBG, carrageenan, low methoxy pectin (LMP) (Danisco São Paulo, Brazil), polydextrose (Litesse São Paulo, Brazil), citric acid monohydrate (Nuclear São Paulo, Brazil) and potassium sorbate (Vetec São Paulo, Brazil).

The different formulations of guava preserves

were processed in open stainless steel pots according to the methodology proposed by Menezes (2011). The amount of ingredients were added relative to the amount of guava pulp. The mixture of pulp and polydextrose was heated to 45°Brix, and it was then added to the gum, LMP and sweeteners previously homogenized under high stirring in water at 80°C. The mixture was cooked to achieve a soluble solids content of 50°Brix. The FOS (fructooligosaccharide) was diluted 1:1 in water at room temperature and was then added to the mixture in this step. The process of cooking continued until a total soluble solids content of 65°Brix was obtained. Citric acid and potassium sorbate were added at the end of the cooking process (diluted 1:1 in water at room temperature) to prevent degradation at the high temperature (above 85°C). The guava preserves were placed in polypropylene containers with the filling performed at a high temperature (85°C). The containers were then closed, cooled to room temperature and stored in a BOD at 20°C for later analysis.

No calcium was added in the preparation of the functional sugar-free guava preserves because guava fruit is rich in calcium with calcium contents ranging between 2.7 and 2.78 mg/100 g (dry basis) according to Morgano et al. (1999). Moreover, the gelling ability of carrageenan is enhanced in the presence of 0.5 g calcium/100 g (Montero and Pérez-Mateos, 2002), and gelation of low methoxyl pectin is achieved with 0.4 g of calcium/100 g (Beaulieu et al., 2001). Furthermore, salt was not added to the preserves.

Texture profile analysis

The texture profile analyses (TPA) were performed under the following conditions: pre-test speed of 1.0 mm/s, test speed of 1.0 mm/s, post-test speed of 1.0 mm/s, compression percentage of 30.0% and compression with a cylindrical probe of a 6.0 mm aluminum texturometer (Stable Micro Systems Model TA-XT2i; Goldaming, England). The following parameters were analyzed: hardness, adhesiveness, cohesiveness and gumminess. The test was performed in triplicate.

Stress relaxation test

There are several mathematical models that can explain the behavior of viscoelastic food products, but the Maxwell and Peleg models are used most frequently to describe the behavior of gels and alimentary systems (Morales et al., 2007; Andrés et al., 2008; Bellido and Hatcher, 2009; Khazaei and Mohammad, 2009).

The Maxwell model involves two simple elements combined in a series to represent different

behaviors. These two elements are the ideal elastic element, which can be represented as a spring and has a behavior defined by an elastic constant (E), and the ideal viscous element, which is represented by a dashpot and has a behavior defined by its viscosity (η) (Campus et al., 2010).

In the Maxwell model with a constant strain (ϵ_0), σ describes the tension applied from σ_0 for $\sigma(t)$ after a time t (Nobile et al., 2007) as follows:

$$\sigma(t) = \epsilon_0 \left(E \cdot \exp\left(-\frac{t}{\lambda}\right) + E_e \right) \quad (1)$$

where E is the elastic modulus of the material; E_e is the equilibrium elastic modulus; and λ is the relaxation time given by η/E . Some foods do not follow the Maxwell simplified viscoelastic model. Therefore, the description of their behavior requires more complex models. An example of this case is the generalized Maxwell model, which consists of an infinite number of Maxwell models in parallel over a spring.

The stress relaxation curves (stress versus time) can be adjusted by equation 2, which provides the viscoelastic parameters of the generalized Maxwell model as follows:

$$\sigma(t) = \epsilon_0 \left(E_1 \exp\left(-\frac{t}{\lambda_1}\right) + E_2 \exp\left(-\frac{t}{\lambda_2}\right) + \dots + E_e \right) \quad (2)$$

where $E_1, E_2 \dots$ are the elastic modulus of the ideal elastic body; E_e is the equilibrium elastic modulus; and $\lambda_1, \lambda_2 \dots$ are the relaxation times.

The viscosity of element i can be calculated according to equation 3 as follows:

$$\eta_i = E_i \lambda_i \quad (3)$$

In the Peleg model, stress relaxation data can be interpreted in accordance with the stress normalized according to equation 4 (Peleg and Normand, 1983) as follows:

$$\frac{\sigma_0 t}{\sigma_0 - \sigma(t)} = k_1 + k_2 t \quad (4)$$

where $\sigma(t)$ is the stress at any time during the test; σ_0 is initial relaxation stress; and k_1 and k_2 are constants. The reciprocal k_1 represents the initial decay rate, and the k_2 parameter represents the degree of relaxation of the material (Guo et al., 1999; Bellido and Hatcher, 2009; Rodríguez-Sandoval et al., 2009), i.e., $1/k_2$ represents the equilibrium conditions of the material (Tang et al., 1998; Rodríguez-Sandoval et al., 2009).

The stress relaxation test was performed in a texturometer (Stable Micro Systems Model TA-XT2i). The samples were cut into cylindrical shapes

Table 1. Levels of Real Independent Variables in the Plackett and Burman Design 16

Variables	Variable coded		
	-1	0	1
FOS (%)*	4.93	12.18	19.43
Thaumatococcus (%)*	0	0.0125	0.025
Sucralose (%)*	0	0.003	0.006
Stevioside (%)*	0	0.0045	0.009
Maltitol (%)*	0	0.025	0.05
Xanthan gum (%)*	0	0.1008	0.2016
LBG gum (%)*	0	0.1008	0.2016
Carrageenan (%)*	0	0.1008	0.2016
Low methoxyl pectin (%)*	0	0.504	1.008
Polydextrose (%)*	20.00	30.08	40.16

*relative to the amount of pulp added

(2.0 cm in height and 2.0 cm in diameter) and compressed to 5.0% of their original height 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 cylindrical probe with a diameter of 7.0 cm, which had been lubricated to eliminate the influence of friction between the sample and equipment, was used. Three measurements were performed for each treatment. The nonlinear regression program R (2011) was used to determine the constants of the Maxwell model. The values obtained were used for the Maxwell model (Equation 1). This model was chosen because there was no considerable improvement (increased R^2) when the generalized model of Maxwell's model of two elements and a spring in parallel was tested. Determination of the Peleg model constants was also performed using the nonlinear regression program R (2011).

Experimental design and statistical analysis

An experiment based on the Plackett and Burman theory (Rodrigues and Iemma, 2005) was designed to determine the effect of 10 variables. Each variable was examined in two levels as follows: -1 to +1; and the lower level to the higher level (defined by previous tests). Three repetitions were performed at the central point resulting in 16 trials with three additional tests at the central point totaling 19 trials.

This design is effective to evaluate the effect of a large number of variables using a small number of tests (Rao and Divakar, 2001; Djekrif-Dakhmouche et al., 2006; Zhou et al., 2011). The independent variables were as follows: FOS, thaumatococcus,

sucralose, stevioside, maltitol, xanthan gum, LBG, carrageenan, LMP and polydextrose. Table 1 shows the actual values of the independent variables. The textural properties of the functional sugar-free guava preserves were analyzed using Statistica software 8.0 (2007) and principal component analysis in Matlab software.

Results and Discussion

Texture profile analysis

Texture profile analysis (TPA) is a simple and rapid analytical technique that has been used extensively in the food industry. TPA is also used to some extent in the pharmaceutical industry to characterize semi-solid formulations (Thrimawithana et al., 2010). The mechanical properties, including hardness, cohesiveness, gumminess and adhesiveness, of the functional sugar-free guava preserves were derived from the force vs. time plots (Jones et al., 1996; Jones et al., 1997; Rahman and Al-Farsi, 2005; Ahmed and Ramaswamy, 2006; Kealy, 2006).

Table 2 shows the estimated effects of the texture profile analysis (TPA) of functional sugar-free guava preserves. FOS caused negative effects on the hardness, cohesiveness and gumminess parameters, and it caused a positive effect on adhesion (adhesiveness is a negative magnitude, so its absolute value will be used in the present study to better understand the effects of independent variables on this parameter) (Table 2). This effect of FOS on hardness was not expected because FOS and soluble fiber contribute to the "body" of the product making it firmer. This negative effect with respect to hardness may have been due to the addition of water to solubilize FOS because greater amounts of FOS in the product result in greater amounts of water required to solubilize FOS and, consequently, a longer amount of time required to cook the guava preserves to obtain the same °Brix. When heated to 80°C for a long time in an acid medium, Rasttal (2010) reported that FOS are hydrolyzed to monosaccharides, which are released during in the middle of cooking. Degradation of FOS changes the product's stability in the aqueous phase making the product less firm (Glibowski and Wasko, 2008). With regard to adhesion, increased FOS in the guava preserves made the preserves more adhesive. According El-Nagar et al. (2002), the enhancement of the adhesiveness by increasing the concentration of FOS is due to FOS making the product more viscous because more water is bound to promote the formation of a stable gel. Cohesiveness is a rheological parameter that is correlated with the property that characterizes the ability to swallow

Table 2. Estimated Effects of the Texture Profile Analysis (TPA) of Functional Sugar-Free Guava Preserves

Factor	Hardness (N)	Adhesiveness (N.s)	Cohesiveness	Gumminess (N)
Mean	0.30*	-108.18*	0.46*	0.13*
FOS	-0.14*	57.23*	-0.11*	-0.07*
Th	0.01	-7.36	-0.08*	0.01
Su	0.11*	-15.69	0.05	0.04*
St	0.00	-23.54	0.10*	0.01
Ma	-0.07	23.28	-0.02	-0.02
GXA	-0.18*	54.53*	0.12*	-0.07*
GLBG	0.22*	-26.21	-0.07*	0.07*
CA	0.13*	-69.72*	0.05	0.07*
LMP	0.15*	-44.32	0.00	0.06*
PO	-0.05	37.52	-0.10*	-0.03
R ²	0,89	0,91	0,91	0,90

FOS: fructooligosaccharide; Th: thaumatin; Su: sucralose; St: stevioside; Ma: maltitol; GXA: xanthan gum; GLBG: LBG gum; CA: carrageenan; LMP: low methoxyl pectin; PO: polydextrose; R²: coefficient of determination; *p<0.05

food, especially if the food is solid (Lucas *et al.*, 2002; Ishihara *et al.*, 2011), and cohesiveness is calculated by the ratio between the area under the curve of strength versus time in the second compression cycle and that in the first compression cycle (Bourne, 1982; Gujral *et al.*, 2002). Lower cohesiveness values (observed for higher values of FOS) result in more disintegrated material in the first compression cycle (Extralab 2010), which means that FOS makes the product more easily to disintegrate. The gumminess (force required to chew a semi-solid food) (Oliveira *et al.*, 2009) was negatively affected by FOS because increasing the amount of FOS caused the guava paste to become softer and, consequently, less sticky.

For high intensity sweeteners, the concentration of stevioside and thaumatin in the product caused the cohesiveness to decrease and increase, respectively. Increased sucralose increased the hardness and gumminess of the preserves (Table 2). According to the literature (Bayarri *et al.*, 2004; Bayarri *et al.*, 2006; Bayarri *et al.*, 2007), this result was not expected as high intensity sweeteners do not affect texture parameters because they are added in small quantities. This result may have been due to the chemical interactions between the sweeteners and gel networks, which may have altered the parameters of the texture profile.

Xanthan gum negatively affected the hardness and gumminess, and it positively affected the cohesiveness and adhesiveness. In studies on surimi gels, Ramírez *et al.* (2002) reported negative effects of xanthan gum on the mechanical properties (shear

stress and shear strain) of the gels. These effects occurred because the interaction between xanthan gum, an anionic starch not associated with proteins in surimi (also anionic in nature), and surimi results in repulsion. In sugar-free guava preserves with prebiotics, Menezes (2011) reported the opposite result obtained for cohesiveness when evaluating the effect of a mixture of xanthan gum and locust bean gum (1:1) (addition of 0 to 0.4032%). The result obtained in this present study may have been due to the nature of additives that may have formed an anion-anion interaction resulting in repulsion, thus reducing the hardness and gumminess and increasing the cohesiveness and adhesiveness. According to Jeanes (1974), the properties of molecular interactions and weak gelatinization are manifested in solutions with high concentrations of xanthan gum, thereby confirming the results obtained in the present study, which demonstrated that increased xanthan gum concentration diminished the hardness and gumminess of the guava paste.

The hardness and gumminess were positively affected by LBG (locust), carrageenan and low methoxyl pectin (LMP) (Table 2). Locust bean gum (LBG) alone does not form gel (Fernandes *et al.*, 1991), but when LBG is associated with other polysaccharides, such as carrageenan and pectin, it can form gels (Hernandez *et al.*, 2001; Azero and Andrade, 2006; Bourbon *et al.*, 2010). In their studies Spagnuolo *et al.* (2005) reported that the carrageenan molecule is flexible and may form a more ordered structure in the form of a double helix at high concentrations, which may lead to gel formation, and they reported that the gelation process is highly influenced by the following factors: type and concentration of salts in solution; cooling and heating rates; concentration of the hydrocolloid; and presence of other biopolymers. Modifications of these factors greatly affect the gelling and rheological properties of gels (Baeza *et al.*, 2002). According Peçanha *et al.* (2006) who studied the microbiological, physicochemical and sensorial characteristics of guava preserves produced in the northern state of Rio de Janeiro, and they found that there is a segment of the market that prefers guava preserves with high strength and gumminess. A similar result was obtained by Menezes (2009) when optimizing and evaluating the effects of potassium sorbate and packaging on guava preserves during storage.

Cohesiveness was negatively affected by both LBG and polydextrose indicating that increased concentrations of these at the levels studied caused a decrease in the values of cohesiveness, i.e., they

caused the guava paste to easily disintegrate. In sugar-free guava preserves with prebiotics (FOS), Menezes (2011) using low methoxyl pectin (0 to 1.008%), mixtures of xanthan gum and locust bean gum (1:1) (0 to 0.4032%) and polydextrose (20.0 at 40.16%) observed the same behavior. The author attributed this fact to the contribution of polydextrose to the soluble solids of the product, which is a determining factor for the end of cooking, thus resulting in a weaker gel.

The adhesiveness was negatively affected by carrageenan (Table 2). Increasing the concentration of carrageenan in functional sugar-free guava preserves made them less adhesive (in module) and, therefore, less viscous. Maltitol did not influence any of the parameters studied. This result was not expected because maltitol is considered a bulking agent (Ronda *et al.*, 2005), which would be expected to cause changes in the texture profile parameters. The lack of influence of maltitol on the parameters may have been due to the low concentration of maltitol used in the present study (0 to 0.05%).

Stress relaxation test

When a stress relaxation test is performed, different behaviors can be observed. Ideal elastic materials do not relax, and ideal viscous materials instantaneously show a relaxation over time. Viscoelastic solids gradually relax and reach an equilibrium stress greater than zero. For viscous fluids, however, the residual stress vanishes to zero (Steffe, 1996). To evaluate the relaxation curve, the applied stress is separated into two components as follows: a relaxing stress component and a non-relaxing stress component. The relaxing component represents the viscous property, and the non-relaxing component represents the elastic property (Wu *et al.*, 2011).

Generalized Maxwell model

Table 3 shows the estimated effect on the stress relaxation test using the generalized Maxwell model of functional sugar-free guava preserves. The values obtained for the generalized Maxwell model with two elements and a spring in parallel were used for the analysis of this model. This model was chosen because it presented a better fit (higher R^2) than the Maxwell model. Furthermore, there was a considerable improvement when the generalized Maxwell model of three elements and a spring in parallel was tested.

Only the viscous component (η_2) was affected by an independent variable (LMP) (Table 3), which positively affected the response variable

Table 3. Estimated Effect on the Stress Relaxation Test Using the Generalized Maxwell Model of Functional Sugar-Free Guava Preserves

Factor	E_0 (N/m ²)	E_1 (N/m ²)	λ_1 (s)	η_1 (N/m ² .s)	E_2 (N/m ²)	λ_2 (s)	η_2 (N/m ² .s)
Mean	2.55*	2.17*	5.67*	21.25*	1.71*	119.29*	354.63*
FOS	-1.11	-0.94	0.05	-8.94	-0.75	-1.96	-162.48
Th	0.77	0.68	-0.16	5.75	0.48	-3.07	89.93
Su	0.92	1.05	2.09	9.00	0.86	53.61	183.08
St	-0.23	-0.50	-0.39	-5.87	-0.39	-0.69	-77.84
Ma	0.01	-0.32	0.37	-2.04	-0.24	4.68	-33.60
GXA	-2.01	-1.73	-2.63	-17.20	-1.37	-55.90	-296.15
GLBG	2.31	1.93	4.83	18.62	1.41	106.05	295.40
CA	1.46	1.41	2.78	14.44	1.18	47.68	232.01
LMP	2.48	1.63	5.55	17.98	1.45	110.64	326.79*
PO	0.46	0.22	2.56	2.82	0.32	50.06	64.24
R^2	0,91	0,95	0,93	0,91	0,99	0,94	0,97

FOS: fructooligosaccharide; Th: thaumatin; Su: sucralose; St: stevioside; Ma: maltitol; GXA: xanthan gum; GLBG: LBG gum; CA: carrageenan; LMP: low methoxyl pectin; PO: polydextrose; R^2 : coefficient of determination; * $p < 0.05$

indicating that increases in the low methoxyl pectin concentration caused functional sugar-free guava preserves to behave more solid. According Macku *et al.* (2009), increasing the pectin concentration makes the product more rigid and, therefore, more viscous.

Peleg model

The estimated effects on the stress relaxation test using the Peleg model of functional sugar-free guava preserves is presented in Table 4. According to Tang *et al.* (1998), Sozer and Dalgic (2007), Sozer *et al.* (2008), Rodríguez-Sandoval *et al.* (2009) and Bhattacharya (2010), the application of Peleg's model to describe the relaxation data is a simple way to describe and compare the stress relaxation using rheology data reported in the literature because this model uses only two parameters as follows: initial decay rate ($1/k_1$) and normalized stress (k_2). The k_1 parameter is a measure of the ease with which the material deforms, i.e., higher values of k_1 suggest a harder material, which dissipates less power, thus requiring more force to be compressed (Guo *et al.*, 1999; Rodríguez-Sandoval *et al.*, 2009). The k_1 was positively affected by increasing the low methoxyl pectin concentration (LMP) in the functional sugar-free guava preserves (Table 4), thus resulting in a harder product, which corroborated with previously reported data (Oakenfull, 1987; Fiszman, 1989; Garnier *et al.*, 1994; Nguémazong *et al.*, 2012).

Table 4. Estimated Effects on the Stress Relaxation Test Using the Peleg Model of Functional Sugar-Free Guava Preserves

Factor	k_1 (s)	k_2
Mean	28.86*	0.88*
FOS	-2.02	-0.01
Th	-1.92	-0.02
Su	14.47	0.36
St	0.54	0.05
Ma	1.60	0.03
GXA	-13.71	-0.37
GLBG	23.27	0.76
CA	12.05	0.35
LMP	29.81*	0.82
PO	14.50	0.41
R^2	0,95	0,96

FOS: fructooligosaccharide; Th: thaumatin; Su: sucralose; St: stevioside; Ma: maltitol; GXA: xanthan gum; GLBG: LBG gum; CA: carrageenan; LMP: low methoxyl pectin; PO: polydextrose; R^2 : coefficient of determination; * $p < 0.05$

The k_2 parameter represents the degree of relaxation of the material (Guo *et al.*, 1999; Bellido and Hatcher, 2009; Rodríguez-Sandoval *et al.*, 2009). According to Peleg (1980), $1/k_2$ represents the equilibrium conditions of the material, i.e., the portion of the material that remains without relaxing at equilibrium. The k_2 parameter was not influenced by any variable studied (Table 4), which indicated that the effects of adding the independent variables to the guava preserve study did not influence the degree of relaxation of the material at a 5% significance level.

Principal components analysis

To better understand the differentiation of the tests, a study of the multivariate data was conducted. Principal components analysis was used to evaluate the results when considering the weight of all the measurements obtained experimentally. Figure 1 shows the results of the separation tests and textural properties. The first axis (principal component 1) explained 78.09% of the variation occurring between the tests. Together, principal components 1 and 2 explained 89.10% of the variation occurring between the tests.

In this type of analysis, vectors of small size indicate that the parameters of the trials differ slightly (Cardello and Faria, 1998). Only the cohesiveness vector had reduced size compared to the other vectors indicating that the parameter vectors that have equivalent sizes are those that have similar importance to explain the variations between experiments.

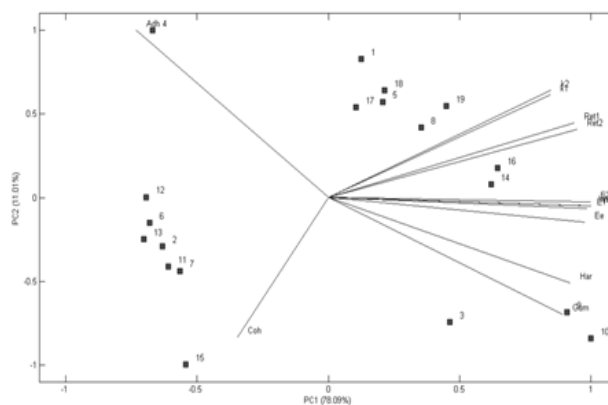


Figure 1. Principal Components Analysis Obtained for the Functional Sugar-Free Guava Preserves

Har: hardness; Adh: adhesiveness; Coh: cohesiveness; Gum: gumminess; Ret1: relaxation time 1; Ret2: relaxation time 2; vis1: viscosity 1; vis2: viscosity 2; E_e : equilibrium elastic moduli; E_1 : elastic moduli of the elastic body ideal 1; E_2 : elastic moduli of the elastic body ideal 2; $1/k_1$: initial decay rate; k_2 : hypothetical value of the asymptotic normalized force

According to Muñoz *et al.* (1992), vectors with steps further from zero correspond to variations with a greater influence on the value of the major component, and vectors closer to zero correspond to a variable with little influence on the major component. Thus, all of the attributes generated for the functional sugar-free guava preserves corresponded to variations with great influence, except for cohesiveness (close to zero).

The adhesiveness (Adh; positively) and cohesiveness (Coh; negatively) parameters contributed the most weight to the variability associated with the second axis (principal component 2). The other rheological parameters had the greatest influence on principal component 1. Most of the tests were influenced by the textural properties. Table 1 and Figure 1 show the major influence of the textural properties of at least two types of gelling agents at levels of 0 to +1, thereby suggesting that the use of gelling agents in functional sugar-free guava preserves changes the textural behavior of the preserves.

Conclusion

The low methoxyl pectin affected several parameters of the texture profile (positive effect on hardness and gumminess) and several relaxation test parameters (positive effect on η_2 and k_1). Half of the other ingredients (except maltitol, which did not affect any rheological parameter) affected only the texture profile parameters. The hardness was positively affected by sucralose, locust bean gum (LBG) and carrageenan, and it was negatively

affected by FOS and xanthan gum. The cohesiveness was positively affected by stevioside and xanthan gum, and it was negatively affected by FOS, thaumatin, locust bean gum and polydextrose. The adhesiveness was positively affected by locust bean gum and negatively affected by carrageenan. The gumminess was positively affected by FOS, sucralose and locust bean gum, and it was negatively affected by carrageenan and xanthan gum. Furthermore, this study demonstrated that higher concentrations of gelling agents in the product results in greater influences on the textural properties.

Acknowledgments

The authors are grateful to FAPEMIG, CAPES and CNPq for their financial support, and the authors are grateful for the donation of sweeteners by Nutramax®.

References

- Acosta, O., Viquez, F. and Cubero, E. 2008. Optimisation of low calorie mixed fruit jelly by response surface methodology. *Food Quality and Preference* 19: 79-85.
- Ahmed, J. and Ramaswamy, H. S. 2006. Physico-chemical properties of commercial date pastes (*Phoenix dactylifera*). *Journal of Food Engineering* 76: 348-352.
- Andrés, S. C., Zaritzky, N. E. and Califano, A. N. 2008. Stress relaxation characteristics of low-fat chicken sausages made in Argentina. *Meat Science* 79: 589-594.
- Arda, E., Kara, S. and Pekcan, Ö. 2009. Synergistic effect of the locust bean gum on the thermal phase transitions of κ -carrageenan gels. *Food Hydrocolloids* 23: 451-459.
- Azero, E. G. and Andrade, C. T. 2006. Characterization of *Prosopis juliflora* seed gum and the effect of its addition to κ -Carrageenan systems. *Journal of the Brazilian Chemical Society* 17: 844-850.
- Baeza, R. I., Carp, D. J., Pérez, O. E. and Pilosof, A. M. R. 2002. κ -carrageenan-protein interactions: effect of proteins on polysaccharide gelling and textural properties. *Food Science and Technology* 35: 741-747.
- Bayarri, S., Durán, L. and Costell, E. 2004. Influence of sweeteners on the viscoelasticity of hydrocolloids gelled systems. *Food Hydrocolloids* 18: 611-619.
- Bayarri, S., Izquierdo, L., Durán, L. and Costell, E. 2006. Effect of addition of sucrose and aspartame on the compression resistance of hydrocolloids gels. *International Journal of Food Science and Technology* 41: 980-986.
- Bayarri, S., Rivas, I., Izquierdo, L. and Costell, E. 2007. Influence of texture on the temporal perception of sweetness of gelled systems. *Food Research International* 40: 900-908.
- Beaulieu, M., Turgeon, S. L. and Doublier, J. L. 2001. Rheology, texture and microstructure of whey proteins/low methoxyl pectins mixed gels with added calcium. *International Dairy Journal* 11: 961-967.
- Bellido, G. G. and Hatcher, D. W. 2009. Stress relaxation behaviour of yellow alkaline noodles: effect of deformation history. *Journal of Food Engineering* 93: 460-467.
- Bhattacharya, S. 2010. Stress relaxation behaviour of moth bean flour dough: product characteristics and suitability of model. *Journal of Food Engineering* 97: 539-546.
- Bourbon, A. I., Pinheiro, A. C., Ribeiro, C., Miranda, C., Maia, J. M., Teixeira, J. A. and Vicente, A. A. 2010. Characterization of galactomannans extracted from seeds of *Gleditsia triacanthos* and *Sophora japonica* through shear and extensional rheology: comparison with guar gum and locust bean gum. *Food Hydrocolloids* 24: 184-192.
- Bourne, M. C. 1982. *Food texture and viscosity*. New York: Academic Press.
- Campus, M., Addis, M. F., Cappuccinelli, R., Porcu, M. C., Pretti, L., Tedde, V., Secchi, N., Stara, G. and Roggio, T. 2010. Stress relaxation behaviour and structural changes of muscle tissues from Gilthead Sea Bream (*Sparus aurata* L.) following high pressure treatment. *Journal of Food Engineering* 96: 192-198.
- Cardello, H. M. A. B. and Faria, J. B. 1998. Quantitative descriptive analysis of sugarcane liquor during aging in oak cask (*Quercus alba* L.). *Food Science and Technology* 18: 169-175.
- Catharino, R. R. and Santos, L. S. 2011. On-line monitoring of stevioside sweetener hydrolysis to steviol in acidic aqueous solutions. *Food Chemistry* 133: 1632-1635.
- Cherbut, C. 2002. Inulin and oligofructose in the dietary fibre concept. *British Journal of Nutrition* 87: S159-S162.
- Daniell, S., Mellits, K. H., Faus, I. and Connerton, I. 2000. Refolding the sweet-tasting protein thaumatin II from insoluble inclusion bodies synthesized in *Escherichia coli*. *Food Chemistry* 71: 105-110.
- Djekrif-Dakhmouche, S., Gheribi-Aoulmi, Z., Meraihi, Z. and Bennamour, L. 2006. Application of a statistical design to the optimization of culture medium for α -amylase production by *Aspergillus niger* ATCC 16404 grown on orange waste powder. *Journal of Food Engineering* 73: 190-197.
- Dunstan, D. E., Chen, Y., Liao, M. L., Salvatore, R., Boger, D. V. and Prica, M. 2001. Structure and rheology of the κ -carrageenan/locust bean gum gels. *Food Hydrocolloids* 15: 475-484.
- El-Nagar, G., Clowes, G., Tudoric, C. M., Kuri, V. and Brennan, C. S., 2002. Rheological quality and stability of yog-ice cream with added inulin. *International Journal of Dairy Technology* 55: 89-93.
- Extralab, B. 2010. Exponent Lite Express Program: 5.0 version. Jarinú: TEE32.
- Fernandes, P. B. and Figueiredo, A. A. 1995. Effect of mesquite seed addition to kappa-carrageenan or xanthan gum mixed systems. *Food Hydrocolloids* 9: 251-255.

- Fernandes, P. B., Gonçalves, M. P. and Doublier, J. L. A. 1991. Rheological characterization of kappa-carrageenan/galactomannan mixed gels: a comparison of locust bean gum samples. *Carbohydrate Polymers* 16: 253-274.
- Fiszman, S. M. 1989. Functional properties of polysaccharide hydrocolloids- gelling mechanisms. *Journal of Agroquímica and Food Technology* 29: 415-429.
- Garnier, C., Axelos, M. A. V. and Thibault, J. F. 1994. Selectivity and cooperativity in the binding of calcium ions by pectins. *Carbohydrate Research* 256: 71-81.
- Glibowski, P. and Wasko, A. 2008. Effect of thermochemical treatment on the structure of inulin and its gelling properties. *International Journal of Food Science and Technology* 43: 2075-2082.
- Gujral, H. S., Kaur, A., Singh, N. and Sodhi, N. S. 2002. Effect of liquid whole egg, fat and textured soy protein on the textural and cooking properties of raw and baked patties from goat meat. *Journal of Food Engineering* 53: 377-385.
- Guo, Z., Castell-Perez, M. E. and Moreira, R. G. 1999. Characterization of masa and low-moisture corn tortilla using stress relaxation methods. *Journal of Texture Studies* 30: 197-215.
- Hanger, L. Y., Lotz, A. and Lepeniotis, S. 1996. Descriptive profiles of selected high intensity sweeteners (HIS), HIS blends, and sucrose. *Journal of Food Science* 61: 456-464.
- Hernandez, M. J., Dolz, J., Dolz, M., Delegido, J. and Pellicer, J. 2001. Viscous synergism in carrageenan (k and l) and locust bean gum mixtures: influence of adding sodium carboxymethylcellulose. *Food Science and Technology International* 7: 383-391.
- Hracek, V. M., Gliemmo, M. F. and Campos, C. A. 2010. Effect of steviosides and system composition on stability and antimicrobial action of sorbates in acidified model aqueous systems. *Food Research International* 43: 2171-2175.
- Ishihara, S., Nakauma, M., Funami, T., Odake, S. and Nishinari, K. 2011. Viscoelastic and fragmentation characters of model bolus from polysaccharide gels after instrumental mastication. *Food Hydrocolloids* 25: 1210-1218.
- Jeanes, A. 1974. Extracellular microbial polysaccharides: new hydrocolloids of interest to the food industry. *Food Technology-Chicago*. 28: 34-40.
- Jones, D. S., Woolfson, A. D., Djokic, J. and Coulter, W. A. 1996. Development and mechanical characterization of bioadhesive semi-solid. Polymeric systems containing tetracycline for the treatment of periodontal diseases. *Pharmaceutical Research* 13: 1734-1738.
- Jones, D. S., Woolfson, D. and Brown, A. F. 1997. Textural, viscoelastic and mucoadhesive properties of pharmaceutical gels composed of cellulose polymers. *International Journal of Pharmaceutics* 151: 223-233.
- Khazaei, J. and Mohammadi, N. 2009. Effect of temperature on hydration kinetics of sesame seeds (*Sesamum indicum* L.). *Journal of Food Engineering* 91: 542-552.
- Kealy, T. 2006. Application of liquid and solid rheological technologies to the textural characterisation of semi-solid foods. *Food Research International* 39: 265-276.
- Lucas, P. W., Prinz, J. F., Agrawal, K. R. and Bruce, I. C. 2002. Food physics and oral physiology. *Food Quality and Preference* 13: 203-213.
- Macku, I., Buňka, F., Voldánová, B. and Pavlínek, V. 2009. Effect of addition of selected solid cosolutes on viscoelastic properties of model processed cheese containing pectin. *Food Hydrocolloids* 23: 2078-2084.
- Mandala, I. G., Savvas, T. P. and Kostaropoulos, A. E. 2004. Xanthan and locust bean gum influence on the rheology and structure of white model-sauce. *Journal of Food Engineering* 64: 335-342.
- Menezes, C. C., Borges, S. V., Cirillo, M. A., Ferrua, F. Q., Oliveira, L. F. and Mesquita, K. S. 2009. Physical and physicochemical characterization of different formulations of guava preserve (*Psidium guajava* L.) of Pedro Sato cultivate. *Food Science and Technology* 29: 618-625.
- Menezes, C. C. 2011. Development of sugar-free guava preserve with prebiotics and its effects on rats's biomarkers carcinogenesis. Lavras, Brazil: Federal University of Lavras, Msc thesis.
- Menu-Bouaouiche, L., Vriet, C., Peumans, W. J., Barre, A., Van Damme, E. J. M. and Rougé, P. A. 2003. Molecular basis for the endo- β 1,3-glucanase activity of the thaumatin-like proteins from edible fruits. *Biochemistry* 85: 123-131.
- Montenegro, F. M., Gomes-Ruffi, C. R., Vicente, C. A., Collares-Queiroz, F. P. and Steel, C. J. 2008. Cassava starch biscuits enriched with soluble and insoluble fiber. *Food Science and Technology* 28: 184-191.
- Montero, P. and Pérez-Mateos, M. 2002. Effects of Na⁺, K⁺ and Ca²⁺ on gels formed from fish mince containing a carrageenan or alginate. *Food Hydrocolloids* 16: 375-385.
- Morales, R., Guerrero, L., Serra, X. and Gou, P. 2007. Instrumental evaluation of defective texture in dry-cured hams. *Meat Science* 76: 536-542.
- Morgano, M. A., Queiroz, S. C. N. and Ferreira, M. M. C. 1999. Minerals determination in juices by inductively coupled plasma optical emission spectrometry. *Food Science and Technology* 19: 344-348.
- Muñoz, A. M., Civille, G. V. and Carr, B. T. 1992. Sensory evaluation in quality control. 1nd ed. New York: Van Nostrand Reinhold.
- Ngouémazong, D. E., Tengweh, F. F., Fraeye, I., Duvetter, T., Cardinaels, R., Loey, A. V., Moldanaers, P. and Hendrickx, M. 2012. Effect of de-methylesterification on network development and nature of Ca²⁺-pectin gels: toward understanding structure-function relations of pectin. *Food Hydrocolloids* 26: 89-98.
- Nobile, M. A., Chillo, S., Mentana, A. and Baiano, A. 2007. Use of the generalized Maxwell model for describing the stress relaxation behavior of solid-like foods. *Journal of Food Engineering* 78: 978-983.
- Nyman, M. 2002. Fermentation and bulking capacity of indigestible carbohydrates: the case of inulin and oligofructose. *British Journal of Nutrition* 87:

- S163-S168.
- Oakenfull, D. 1987. Gelling agents. *Critical Reviews in Food Science and Nutrition* 26: 1-25.
- Oliveira, L. F., Borges, S. V., Nascimento, J., Cunha, A. C., Jesus, T. B., Pereira, P. A. P., Pereira, A. G. T., Figueiredo, L. P. and Valente, W. A. 2009. Use of banana peel in the manufacture of banana preserve-quality assessment. *Food and Nutrition* 20: 581-589.
- Pereira, P. A. P., Souza, V. R., Carneiro, J. D. S., Borges, S. V., Ferreira, L. O., Queiroz, F. 2013. Physical and physicochemical evaluation of different brands of traditional, low calorie and sugar-free guava preserves. *Brazilian Journal of Chemical Engineering* 30: 531-540.
- Paucar-Menacho, L. M., Silva, L. H., Barretto, P. A. A., Mazal, G., Fakhouri, F. M., Steel, C. J. and Collares-Queiroz, F. P. 2008. Development of functional fresh food adding soy protein isolate and polidextrose using paprika as coloring agent. *Science and Food Technology* 28: 767-778.
- Peçanha, D. A., Neves, T. G., Verruma-Bernardi, M. R., Deliza, R., Araújo, K. G. L., Kajishima, S. and Pinheiro, M. S. 2006. Microbiological, Physico-Chemical and Sensory Quality of Thick-Cut Guava Preserve Produced in the Northern Area of Rio de Janeiro State, Brazil. *Brazilian Journal of Food Technology* 9: 25-32.
- Peleg, M. 1980. Linearization of relaxation and creep curves of solid biological materials. *Journal of Rheology* 24: 451-463.
- Peleg, M. and Normand, M. D. 1983. Comparison of two methods for stress relaxation data presentation of solid foods. *Rheologica Acta* 22: 108-113.
- R Development Core Team. 2011. R: A language and environment for statistical computing. Vienna, Austria: R Foundation for Statistical Computing, ISBN 3-900051-07-0, URL. <http://www.R-project.org/>.
- Rahman, M. S. and Al-Farsi, S. A. 2005. Instrumental texture profile analysis (TPA) of date flesh as a function of moisture content. *Journal of Food Engineering* 66: 505-511.
- Rahn, A. and Yaylayan, V. A. 2010. Thermal degradation of sucralose and its potential in generation chloropropanols in the presence of glycerol. *Food Chemistry* 118: 56-61.
- Ramírez, J. A., Barrera, M., Morales, O. G. and Vásquez, M. 2002. Effect of xanthan and locust bean gums on the gelling properties of myofibrillar protein. *Food Hydrocolloids* 16: 11-16.
- Rao, P. and Divakar, S., 2001. Lipase catalyzed esterification of α -terpineol with various organic acids: application of the Plackett- Burman design. *Process Biochemistry* 36: 1125-1128.
- Rastall, R. A., 2010. Functional oligosaccharides: application and manufacture. *Annual Review of Food Science and Technology* 1: 305-339.
- Roberfroid, M. 2007. Prebiotics: the concept revisited. *Journal of Nutrition* 137: 830S-837S.
- Rodríguez-Cabezas, M. E., Camuesco, D., Arribas, B., Garrido-Mesa, N., Comalada, M., Bailón, E., Cueto-Sola, M., Utrilla, P., Guerra-Hernández, E., Pérez-Roca, C., Gávez, J. and Zarzuelo, A. 2010. The combination of fructooligosaccharides and resistant starch shows prebiotic additive effects in rats. *Clinical Nutrition* 29: 832-839.
- Rodrigues, M. I. and Iemma, A. F. 2005. Experimental Design and process optimization: a sequential strategy planning. Campinas: House's Bread.
- Rodríguez-Sandoval, E., Fernández-Quintero, A. and Cuvelier, G. 2009. Stress relaxation of reconstituted cassava dough. *Food Science and Technology* 42: 202-206.
- Ronda, F., Gómez, M., Blanco, C. A. and Caballero, P. A. 2005. Effects of polyols and nondigestible oligosaccharides on the quality of sugar-free sponge cakes. *Food Chemistry* 90: 549-555.
- Sandolo, C., Bulone, D., Mangione, M. R., Margheritelli, S., Di Meo, C., Alhaique, F., Matricardi, P. and Coviello, T. 2010. Synergistic interaction of locust bean gum and xanthan investigated by rheology and light scattering. *Carbohydrate Polymers* 82:733-741.
- Sandrou, D. K. and Arvanitoyannis, I. S. 2000. Low fat/calorie foods: Current state and perspectives. *Critical Reviews in Food Science and Nutrition* 40: 427-447.
- Spagnuolo, P. A., Dalgleish, D. G., Goff, H. D. and Morris, E. R. 2005. Kappa-carrageenan interactions in systems containing casein micelles and polysaccharide stabilizers. *Food Hydrocolloids* 19: 371-377.
- Sozer, N. and Dalgic, A. C. 2007. Modelling of rheological characteristics of various spaghetti types. *European Food Research and Technology* 225: 183-190.
- Sozer, N., Kaya, A. and Dalgic, A. C. 2008. The effect of resistant starch additions on viscoelastic properties of cooked spaghetti. *Journal of Texture Studies* 39: 1-16.
- Statistica. 2007. Statsoft statistica for windows, Release 8.0. Tulsa, OK: Statsoft Inc.
- Steffe, J. F. 1996. *Rheological Methods in Food process Engineering*. 2nd ed. United State: Freeman Press.
- Tang, J., Tung, M. A. and Zeng, Y. 1998. Characterization of gellan gels using stress relaxation. *Journal of Food Engineering* 38: 279-295.
- Thakur, B. R., Singh, R. K. and Handa, A. K. 1997. Chemistry and uses of pectin – A review. *Critical Reviews in Food Science* 37: 47-73.
- Thrimawithana, T. R., Young, S., Dunstan, D. E. and Alany, R. G. 2010. Texture and rheological characterization of kappa and iota carrageenan in the presence of counter ions. *Carbohydrate Polymers* 82: 69-77.
- Wu, Y., Cui, W., Eskin, N. A. M. and Goff, H. D. 2011. Stress relaxation in synergistically associated polysaccharides: galactomanans and a non-pectin polysaccharide fraction from yellow mustard mucilage. *Carbohydrate Polymers* 84: 984-989.
- Zhou, J., Yu, X., Ding, C., Wang, Z., Zhou, Q., Pao, H. and Cai, W. 2011. Optimization of phenol degradation by *Candida tropicalis* Z-04 using Plackett-Burman design and response surface methodology. *Journal of Environmental Sciences* 23: 22-30.