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Rheological properties of aqueous dispersions of chia (Salvia hispanica L.) mucilage



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ABSTRACT

The viscoelastic and flow behavior of aqueous dispersions with different concentrations of chia mucilage (Salvia hispanica L.) from Argentina seeds was characterized. The mucilage was obtained by two methods: (I) soaking–freezing–freeze drying–sieving, and (II) soaking–filtration–concentration–freezing–freeze drying. The effect of mucilage concentration, temperature, pH, ionic strength and presence of sucrose on the rheological properties of the aqueous dispersions with the addition of NaCl or CaCl₂ was also evaluated. All the dispersion samples presented a shear-thinning behavior and a weak elastic gel-like structure because the storage modulus (G') was larger than the loss modulus (G'') in the studied frequency range. The concentration of mucilage was the variable with the most significant effect on k (consistency index), whereas the presence of sucrose had the highest effect on n (flow behavior index) and tan δ . The type of salt and extraction method significantly affected k, not affecting n. Tan δ was affected mainly by the type of salt, and some interaction was observed between both factors. Method II and NaCl provided a higher consistency to the dispersions of chia mucilage. These results suggest that chia mucilage could be applied in the production of foods that require additives with thickening capacity, taking advantage of the zero calorific value of this hydrocolloid.

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1. Introduction

Hydrocolloids are polymers of high molecular weight with a great affinity for water, where they disperse forming colloidal solutions of great viscosity. The term "hydrocolloids" involves all the polysaccharides extracted from plants (cellulose, pectins, starch), seeds (guar gum, locust bean gum, tara gum), seaweeds (agar, carrageenan, alginate) and microbial sources (xanthan gum, gellan gum), and also from plant exudate gums (gum arabic, gum karaya, tragacanth) and chemically or enzymatically modified forms. In addition, gelatin is the only protein that has been accepted into the group of hydrocolloids given its polydispersity and highly hydrophilic character (Dickinson, 2003). Nowadays, hydrocolloids

are used in a wide range of industries as thickening and gelling agents of aqueous solutions, stabilizers of foams, emulsions and dispersions, crystal growth inhibitors, encapsulants, and also in the controlled release of flavors, production of edible films and for texture modification (Williams and Phillips, 2000; Koocheki et al., 2009). A number of studies have been carried out to analyze the rheological characteristics of hydrocolloids individually or as ingredients of food formulations (Sanderson, 1981; Dickie and Kokini, 1983; Stanley, 1990; White et al., 1993; Abdelrahim et al., 1995; Da Silva and Rao, 1995).

The study of rheological properties is important for the design of different processes (such as fluid flow, pumps, processes of extraction, filtration, purification, pasteurization, evaporation, drying) (Tabatabaee and Mirhosseini, 2012). The thickening properties and viscoelastic behavior of hydrocolloids in solution can be significantly affected by variables such as shear rate and time, concentration of the compound, temperature, pressure, ionic strength and pH, among others (Karazhiyan et al., 2009). The analysis of the individual or combined effects of these factors is important, especially when they will be used to modify food texture, and also in the design, evaluation and modeling of processes.

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Chia (Salvia hispanica L.) mucilage is secreted by the seed when it becomes wet, covering it with a transparent hallo. The structural units of this polysaccharide are described as a tetrasaccharide with a main chain consisting of units of $(1 \rightarrow 4)-\beta$ -D-xylopyranosyl- $(1 \rightarrow 4)$ - α -D-glucopyranosyl- $(1 \rightarrow 4)$ - β -D-xylopyranosyl with 4-Omethyl-α-D-glucuronic acid ramifications in the O-2 position of β-D-xylopyranosyl in the main chain. The ratio of β-D-xylose to α-D-glucose monosaccharides to 4-O-methyl-α-D-glucuronic acid is 2:1:1 (Lin et al., 1994). The available data regarding its functional properties are recent, and they indicate that it is a polymer with thickening properties (Marin Flores et al., 2008). Given the high solubility of chia mucilage in water (50 g/mL), it has a potential industrial application, since it is considered that gums and/or mucilages with higher solubility have a better quality (Mhinzi and Mrosso, 1995). The intake of chia mucilage, alone or in combination with the seed, has been shown to affect the metabolism of lipids by reducing the intestinal absorption of fatty acids, cholesterol and bile salts, increasing fecal cholesterol loss, and inhibiting endogenous cholesterol synthesis, slow digestion and absorption of nutrients. In addition, being a type of soluble dietary fiber, mucilage forms gels of high viscosity that produce gastric distension, feeling of satiety and slow stomach emptying, becoming a functional food (Hentry et al., 1990). The aim of the present work was to characterize the rheological properties of aqueous dispersions of chia mucilage obtained from seeds grown in Argentina by two different methods, evaluating the effect of some conditions of the medium such as mucilage concentration, temperature, pH, ionic strength, type of salt, and the presence of sucrose.

2. Materials and methods

2.1. Materials

The commercial chia seeds used in this study were obtained from Salta, Argentina (25°S 65.5°W). The seeds were cleaned manually and the foreign matter, such as stones, dirt and broken seeds, was removed. Then they were packaged in hermetic plastic containers and stored at 5 \pm 1 °C until further use.

2.2. Obtaining the chia mucilage

The mucilage was obtained from whole chia seeds by two methods.

2.2.1. Method I (MI)

Mucilage was obtained by the procedure proposed by Capitani et al. (2013). Samples of 10 g of whole seeds were placed in a tray (9 cm \times 14 cm \times 5 cm), and distilled water was added in a 1:10 (w/v) ratio. They were covered with aluminum foil and maintained at room temperature for 4 h. Then, the samples were frozen at -20 °C, followed by freeze-drying (-50 °C, 0.033 mbar, 4 days). The dried mucilage was separated from the seeds by rubbing over a 20 ASTM mesh screen (840 μ m) for three periods of 15 min each.

2.2.2. Method II (MII)

Mucilage was obtained by the procedure proposed by Marin Flores et al. (2008), with a modified drying method for the mucilage solution. Whole chia seeds were soaked in water (1:20 w/v) for 1 h at room temperature with manual stirring in order to induce the mucilage exudation. The extracted mucilage was separated from the seeds by vacuum filtration through a mesh (100 μ m) at 220 mbar. Then the mucilage solution was concentrated on a rotavapor (Büchi R-215, Switzerland) at 55 °C under vacuum. It was frozen at -20 °C for 96 h followed by freeze-drying (-45 °C, 0.060 mbar, 5 days) (LABCONCO freeze dryer, Freezone 18,

USA). The dried mucilage was ground using a food processor (Moulinex, model 1736249, Spain) to obtain a fine powder.

Both types of mucilage were packaged in hermetically sealed plastic containers, and stored in a desiccator to preserve them from humidity.

2.3. Proximate composition of mucilages

AOCS (1998) procedures were used to analyze moisture (method Ba 2a-38), crude fiber (method Ba 6-84) and ash content (method Ba 5a-49). The oil content was determined following IUPAC standard method 1.122 (1992). Total nitrogen content was determined by Kjeldahl method according to AOAC (1990), and protein content was calculated as % nitrogen \times 6.25. Carbohydrate content was estimated as nitrogen-free extract (NFE) by difference using Eq. (1):

$$NFE = 100 - (oil + protein + crude fiber + ash).$$
 (1)

2.4. Dispersion preparations

Dispersions with four concentrations of mucilage (0.25%, 0.50%, 0.75% and 1.00% w/v) were prepared by hydrating dried mucilage powder in deionized water for 30 min at 60 °C using a magnetic stirrer. The dispersions were then left overnight at 4 °C to ensure a complete hydration prior to the rheological measurements.

2.5. Experimental design

The effect of different variables on the rheological behavior of chia mucilage dispersions was evaluated according to a 25-2 fractional factorial design, analyzing the variables in four blocks, and taking into account the effect of the method used to obtain the mucilage and of the addition of a mono- or divalent salt to the dispersion (Table 1). The studied independent variables were mucilage concentration, temperature, pH, ionic strength and presence of sucrose, and the responses (dependent variables) were the consistency index (k), the flow behavior index (n), and tan δ . The studied variables and their levels (minimum and maximum) are presented in Table 2. After separately weighing the mucilage, salts and/or sucrose (according to the quantities required for each treatment of the design), they were dry blended and dissolved in deionized water under vigorous agitation for 30 min at 60 °C and then cooled to room temperature, and finally the pH was adjusted using 0.1 mol/L NaOH and/or HCl. The dispersions were then left overnight at 4 °C prior to performing the rheological measurements.

Based on the results obtained by means of the fractional factorial design, and taking into account the variables that were significant for k, which was affected by the same variables in the upward and downward curves (and without significant interactions among them), the dispersions were prepared with 1% mucilage, 0.05 M salt, pH 9, without the addition of sucrose, and their properties were determined at 5 °C. Thus the effect of the method used to obtain the mucilage (I and II) and of the type of salt added (NaCl and CaCl₂) were evaluated according to a 2^2 full factorial design, replicated twice.

Table 1 Fractional factorial design blocks.

| Block | Method | Salt |
|-------|--------|---------------------------|
| 1 | I | NaCl |
| 2 | I | CaCl ₂ NaCl |
| 3 | II | NaCl |
| 4 | II | CaCl ₂ |

Table 2 Variables of the flow properties of both types of chia mucilage.

| Variable level | Mucilage concentration | Temperature | рН | Ionic strength | Sucrose presence (%) |
|----------------|------------------------|-------------|----|----------------|----------------------|
| _ | 0.50% (p/v) | 5 °C | 3 | 0 M | 0 |
| + | 1.00% (p/v) | 45 °C | 9 | (0.05 M) | 40 |

where "-" = minimum level, and "+" = maximum level.

An ANOVA test with a confidence interval of 95% was carried out using the software Statgraphics Plus version 5.1 (Statistical Graphic Corporation, Manugistics Inc., Rockville, USA, 2005).

2.6. Rheological measurements

Rheological evaluations were performed using a stress-controlled rheometer (AR-2000, TA Instruments, UK) on its strain mode, with a cone and plate geometry (cone angle = 2° , plate diameter = 40 mm, GAP 52 μm) at constant temperature (25 \pm 1 $^{\circ}$ C) controlled by means of a Peltier system. In all the experiments, the volume of the sample used was 0.59 mL, and the samples were covered with silicone oil to prevent water loss.

2.6.1. Flow behavior

Since previous trials demonstrated that behavior of the sample was shear thinning type it was decided to follow the next conditions: dispersions were subjected to an logarithmic increasing shear rate in continuous ramp from 1 to $500 \, \mathrm{s}^{-1}$ in 2 min, followed by a steady shear at $500 \, \mathrm{s}^{-1}$ for 1 min, and finally a decreasing shear rate from $500 \, \mathrm{to} \, 1 \, \mathrm{s}^{-1}$ in 2 min (Lopes da Silva et al., 1994; Marcotte et al., 2001). The samples were previously left to equilibrate for at least 5 min at $3 \, \mathrm{s}^{-1}$ in order to ensure that sample homogeneity was attained before starting the measurement. The experimental data (shear stress–shear rate) were fitted according to the power-law model using Eq. (2):

$$\tau = k\dot{\gamma}^n \tag{2}$$

where τ is the shear stress (Pa), $\dot{\gamma}$ is the shear rate (s⁻¹), k is the consistency index (Pa sⁿ) and n is the flow behavior index (dimensionless).

Besides previous studies (Vercruysse and Steffe, 1989; Aportela-Palacios et al., 2005; Quek et al., 2013) carried out with still viscometers and others with rheometers, with all kind of foods, still demonstrate that the use of the power law is the one that best explains the behavior even in continuous ramp.

2.6.2. Viscoelastic properties

The storage modulus (G', Pa), loss modulus (G'', Pa) and $\tan \delta$ (G''/G') of each aqueous mucilage dispersion were measured as a function of angular frequency (ω , Hz). Strain sweep tests at 1 Hz for the studied concentrations were previously conducted in order to define the linear viscoelasticity zone, where the shear stress varies linearly with the applied strain. Frequency sweeps were programmed accordingly at a strain value of 5% over a frequency range of 1–10 Hz.

All rheological measurements were carried out in triplicate. The results corresponding to Sections 2.6.1 and 2.6.2 were analyzed by means of ANOVA followed by Tukey's test (p < 0.05) using Infostat software (Infostat Group, Facultad de Ciencias Agrarias, Universidad Nacional de Córdoba, Argentina, 2004).

3. Results and discussions

3.1. Proximate composition

The two studied methods of mucilage extraction exhibited a similar yield of $3.8 \pm 0.1\%$ and $3.7 \pm 0.1\%$ (d.b.) for MI and MII, respectively. The proximate composition of the obtained chia mucilages is presented in Table 3. Both types of mucilage showed a different proximate composition. The content of proteins and residual lipids was significantly higher (p < 0.05) for MI, whereas the crude fiber content was significantly higher for MII. The differences observed between MI and MII could be attributed to the different methods used to separate the seeds from the mucilage liquid, as well as to the differences in the values of the variables of the soaking stage.

3.2. Flow behavior

The variation in apparent viscosity with shear rate at 25 °C is shown in Fig. 1a and b for the different concentrations of chia mucilage obtained by MI and MII, respectively. The viscosity of all the dispersions decreased as the shear rate increased. This fact reveals the pseudoplastic behavior of the samples, since as shear rate increases, the randomly positioned chains of polymer molecules become aligned in the direction of the flow, generating solutions with lower viscosity, resulting in less interaction among adjacent polymer chains (Koocheki et al., 2013). A similar behavior was observed for different dispersion of flaxseed (Mazza and Biliaderis, 1989), Opuntia ficus indica (Medina-Torres et al., 2000), Lepidium sativum (Karazhiyan et al., 2009), tragacanth (Chenlo et al., 2010) and Lepidium perfoliatum gums (Koocheki et al., 2013). On the other hand, the apparent viscosity of the dispersions increased with the increase in mucilage concentration from 0.25% to 1.00% (w/v). This could be attributed to the higher content of total solids in the dispersion, which causes an increase in viscosity due mainly to an increased restriction of intermolecular motion caused by hydrodynamic forces and the formation of an interfacial film (Maskan and Gogus, 2000). This phenomenon was observed in dispersions of xanthan gum (Speers and Tung, 1986), flaxseed (Mazza and Biliaderis, 1989), Opuntia ficus indica (Medina-Torres et al. 2000), tragacanth gum (Chenlo et al., 2010) and Lepidium perfolatium (Koocheki et al., 2013).

The values of the consistency index (k) and flow behavior index (n) of the chia mucilage dispersions for the upward and downward

Table 3 Proximate composition (% d.b.) of chia mucilage obtained by both methods.

| Component | MI | MII |
|----------------------|---|---|
| Moisture Protein* | 9.37 ^a 18.85 ^b | 11.08 ^b 6.79 ^a |
| Crude fiber | 11.42ª | 17.97 ^b |
| Oil Ash | 3.22 ^b 10.27 ^a | 0.88 ^a 9.75 ^a |
| NFE | 56.24 ^a | 64.61 ^b |

Values followed by different letters differ significantly (p < 0.05) according to Tukey's test. Data are means \pm SD of triplicate determinations.

* Factor: 6.25; NFE: nitrogen-free extract.

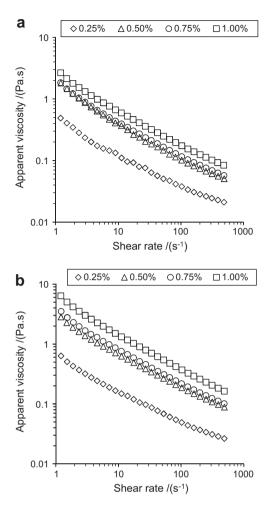


Fig. 1. Flow curves of chia mucilage dispersions for different concentrations (0.25–1.00% w/v) at constant temperature of 25 °C. (a) MI and (b) MII.

curves, respectively, as a function of the polysaccharide concentration are shown in Table 4. The values of n for all the samples were lower than 1, thus confirming the pseudoplastic nature of the dispersions for the studied conditions. From a sensory point of view, this value was also important because it was lower than 0.6, which is considered the limit for a good perception of food in the mouth (Chhinnan et al., 1985; Muller et al., 1994). The determination coefficients (R^2) were close to 1, showing that the power law model was adequate to determine the flow behavior of the chia mucilage dispersions.

The values of k for the dispersions prepared by MII were significantly higher for each concentration (p < 0.05) than those

corresponding to the dispersions formulated by MI, and this is associated with the higher viscosity of the dispersions formulated by MII (see Fig. 1a and b) since the consistency index is indicative of the viscous nature of the system (Ibanoğlu, 2002). The higher viscosity of the dispersions obtained by MII could also be a result of the higher purity of this mucilage, favoring a better interaction among the molecules of the polysaccharide. The larger k values could also be due to the higher solid content (Maskan and Gogus, 2000) (MII mucilage presented higher fiber content and a larger NFE fraction than the MI sample). In addition, k values increased with increasing mucilage concentration, whereas n values decreased significantly (p < 0.05). This confirms that the increase in polysaccharide concentration causes the increase in viscosity and level of pseudoplasticity of the dispersions.

On the other hand, the differences found in the parameters between the upward and downward curves could indicate that the flow behavior of the chia mucilage dispersions exhibited a dependence on shearing time. A similar trend was also observed in dispersions of *L. perfolatium* (Koocheki et al., 2013) and Alyssum homolocarpum seed gum (Koocheki and Razavi, 2009). This could be confirmed by repeating the test again and again, until eventually a constant loop behavior is observed (Barnes, 1997) or with at least two cyclic runs according to the model cited by Barbosa-Canovas and Peleg (1983).

3.3. Viscoelastic behavior

The viscoelastic behavior of the studied chia mucilage dispersions is presented in Fig. 2a and b. It can be observed that for both types of chia mucilage, the storage or elastic modulus (G') and the loss or viscous modulus (G'') showed dependence on frequency, and this effect was more marked in the dispersions prepared by MII. In all the dispersions G' was larger than G'', indicating a prevalent elastic behavior of the samples, similar to that of *psyllium* gum solutions (Farahnaky et al., 2010).

It can also be observed that at higher mucilage concentrations (0.75% and 1.00%), the difference between both moduli was greater, whereas at lower concentrations (0.25% and 0.50%) a cross-over point between G' and G'' occurred. This behavior indicates that at high frequencies and low concentrations, the structure of the mucilage breaks down, showing a slightly viscous behavior, similar to that of *Opuntia ficus indica* mucilage dispersions (Medina-Torres et al., 2000). The values of the elastic and viscous moduli were higher as mucilage concentration increased, presenting the highest values in the dispersions prepared by MII. This would indicate that MII can form stronger structures.

A common method to determine the viscoelastic behavior of a sample is by measuring the tangent of the phase angle (tan δ), which is the ratio of the loss modulus G' to the storage modulus G. Tan δ is directly related to the energy lost per cycle divided

Table 4Power law parameters for chia mucilage dispersions at different concentrations.

| Mucilage concentration (%) | Upward curve | | Downward curv | Downward curve | | | |
|----------------------------|------------------------|-------------------|---------------|------------------------|-------------------|-------|--|
| | k (Pa s ⁿ) | n | R^2 | k (Pa s ⁿ) | п | R^2 | |
| MI | | | | | | | |
| 0.25 | 0.42 ^a | 0.49^{b} | 0.986 | 0.23 ^a | 0.56 ^b | 0.989 | |
| 0.50 | 1.61 ^b | 0.41 ^a | 0.982 | $0.80^{\rm b}$ | 0.53 ^a | 0.999 | |
| 0.75 | 1.62 ^b | 0.44 ^a | 0.991 | 0.93 ^c | 0.52 ^a | 0.999 | |
| 1.00 | 2.43 ^c | 0.44 ^a | 0.993 | 1.44 ^d | 0.52 ^a | 0.999 | |
| MII | | | | | | | |
| 0.25 | 0.56 ^a | 0.48 ^b | 0.991 | 0.30^{a} | 0.56 ^c | 0.998 | |
| 0.50 | 2.55 ^b | 0.44^{a} | 0.993 | 1.41 ^b | 0.54 ^b | 0.999 | |
| 0.75 | 3.18 ^c | 0.42^{a} | 0.993 | 1.82 ^c | 0.52 ^b | 0.999 | |
| 1.00 | 5.80 ^d | 0.41 ^a | 0.994 | 3.50^{d} | 0.50^{a} | 0.999 | |

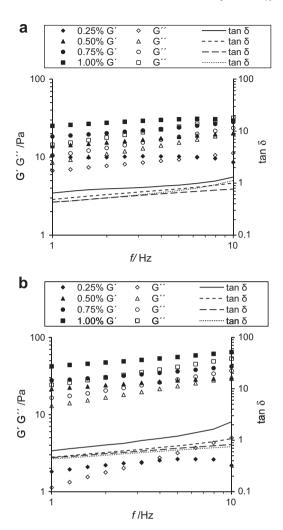


Fig. 2. Effect of chia mucilage concentration on storage (G') and loss (G'') moduli and tan δ as functions of frequency at constant temperature of 25 °C. G', full symbols; G'', empty symbols; tan δ , lines. (a) MI and (b) MII.

by the energy stored per cycle. A tan $\delta < 1$ indicates a predominantly elastic behavior, whereas $\tan \delta > 1$ indicates a predominantly viscous behavior. Polymeric systems can be classified according to their tan δ value into: dilute solutions (high values, usually greater than 3), amorphous polymers (values can oscillate in the 0.2-0.3 range), glassy crystalline polymers and gels (low values, near 0.01) (Steffe, 1996). Fig. 2 shows the variation of tan δ as a function of frequency for the different chia mucilage concentrations tested. In all the dispersions, the values of this parameter exhibited an increasing tendency as frequency increased. The values of tan δ were higher in the dispersions with 0.25% mucilage (0.9 and 1.1 mean values for MI and MII, respectively), whereas in dispersions with 0.50%, 0.75% and 1.00% mucilage the mean values of tan δ were lower (0.6 \leq tan $\delta \leq$ 0.7). Thus, as mucilage concentration increased, the values of tan δ decreased to values below 1, maintaining G' > G'', which would indicate that at high concentrations the samples may display a weak elastic gel-like behavior, defined by Richardson et al. (1989) as the behavior in which G' is higher than *G*" during the measurement interval, presenting small variations with frequency. This result is similar to that obtained for other gums such as L. perfoliatum (Hesarinejad et al., 2014), psyllium (Farahnaky et al., 2010) and xanthan gum (Chen et al., 2002). As mentioned above, as the concentration of chia mucilage increases, the solid nature of the dispersions can be expected to be larger, indicating the possible formation of gels at concentrations >1%.

3.4. Effect of various factors on the rheological properties of chia mucilage

The design of the experiments and the consistency index (k) and flow behavior index (n) responses corresponding to the upward and downward curves obtained by fitting the experimental data to the power law model are shown in Table 5.

All the dispersions showed a non-Newtonian fluid behavior of a pseudoplastic nature, since n values were lower than 1 for all the studied conditions. When comparing the k and n parameters for the upward and downward curves of all the experiments, it can be observed that k values decreased, whereas n values increased. These differences would show that the dispersions could exhibit a thixotropic behavior. Thus, at a constant shear rate, apparent viscosity decreased with shearing time, a fact that could be attributed to the progressive structural breakdown of this type of hydrocolloid (Abu-Jdayil and Mohameed, 2004). A similar behavior was observed in solutions of hydrocolloids obtained from L sativum seed extract (Karazhiyan et al., 2009), salep tuber (Orchismascula) and Balangu seeds (Lallemanti aroyleana) (Razavi and Karazhiyan, 2009).

The effect of the variables and their interactions on the consistency index (k) of the upward and downward curves is shown in Fig. 3a and b, respectively. Both in the upward and the downward curves, k was significantly affected by mucilage concentration,

Table 5Power law parameters corresponding to the experimental design for different chia mucilage dispersions.

| Trial | Block | Α | В | С | D | Е | Upward curve | | Downward curve | |
|-------|-------|----|----|----|----|----|------------------------|------|------------------------|------|
| | | | | | | | k (Pa s ⁿ) | n | k (Pa s ⁿ) | n |
| 1 | 1 | -1 | -1 | -1 | -1 | -1 | 0.63 | 0.47 | 0.27 | 0.60 |
| 2 | 1 | +1 | +1 | -1 | -1 | -1 | 1.44 | 0.40 | 0.61 | 0.51 |
| 3 | 1 | +1 | -1 | +1 | +1 | -1 | 2.22 | 0.44 | 1.06 | 0.55 |
| 4 | 1 | -1 | +1 | +1 | +1 | -1 | 0.40 | 0.47 | 0.19 | 0.55 |
| 5 | 1 | +1 | -1 | +1 | -1 | +1 | 2.88 | 0.50 | 1.60 | 0.60 |
| 6 | 1 | -1 | +1 | +1 | -1 | +1 | 0.68 | 0.52 | 0.36 | 0.61 |
| 7 | 1 | -1 | -1 | -1 | +1 | +1 | 0.50 | 0.61 | 0.27 | 0.70 |
| 8 | 1 | +1 | +1 | -1 | +1 | +1 | 1.00 | 0.51 | 0.67 | 0.56 |
| 9 | 2 | +1 | -1 | -1 | -1 | -1 | 2.53 | 0.42 | 1.21 | 0.54 |
| 10 | 2 | -1 | +1 | -1 | -1 | -1 | 0.39 | 0.47 | 0.19 | 0.54 |
| 11 | 2 | -1 | -1 | +1 | +1 | -1 | 0.32 | 0.40 | 0.20 | 0.38 |
| 12 | 2 | +1 | +1 | +1 | +1 | -1 | 0.97 | 0.34 | 0.64 | 0.35 |
| 13 | 2 | -1 | -1 | +1 | -1 | +1 | 1.09 | 0.56 | 0.65 | 0.65 |
| 14 | 2 | +1 | +1 | +1 | -1 | +1 | 2.49 | 0.46 | 1.40 | 0.55 |
| 15 | 2 | +1 | -1 | -1 | +1 | +1 | 1.59 | 0.51 | 0.75 | 0.63 |
| 16 | 2 | -1 | +1 | -1 | +1 | +1 | 0.20 | 0.57 | 0.10 | 0.65 |
| 17 | 3 | +1 | -1 | +1 | -1 | -1 | 7.14 | 0.43 | 4.76 | 0.50 |
| 18 | 3 | -1 | +1 | +1 | -1 | -1 | 1.23 | 0.45 | 0.73 | 0.53 |
| 19 | 3 | -1 | -1 | -1 | +1 | -1 | 0.78 | 0.48 | 0.37 | 0.59 |
| 20 | 3 | +1 | +1 | -1 | +1 | -1 | 1.71 | 0.41 | 0.80 | 0.52 |
| 21 | 3 | -1 | -1 | -1 | -1 | +1 | 0.85 | 0.58 | 0.45 | 0.67 |
| 22 | 3 | +1 | +1 | -1 | -1 | +1 | 3.11 | 0.39 | 1.35 | 0.53 |
| 23 | 3 | +1 | -1 | +1 | +1 | +1 | 3.83 | 0.50 | 2.22 | 0.59 |
| 24 | 3 | -1 | +1 | +1 | +1 | +1 | 1.08 | 0.42 | 0.41 | 0.57 |
| 25 | 4 | -1 | -1 | +1 | -1 | -1 | 1.55 | 0.48 | 0.90 | 0.57 |
| 26 | 4 | +1 | +1 | +1 | -1 | -1 | 4.75 | 0.40 | 2.75 | 0.48 |
| 27 | 4 | +1 | -1 | -1 | +1 | -1 | 2.57 | 0.45 | 1.25 | 0.57 |
| 28 | 4 | -1 | +1 | -1 | +1 | -1 | 0.30 | 0.49 | 0.15 | 0.54 |
| 29 | 4 | +1 | -1 | -1 | -1 | +1 | 4.49 | 0.48 | 2.32 | 0.58 |
| 30 | 4 | -1 | +1 | -1 | -1 | +1 | 0.98 | 0.45 | 0.39 | 0.60 |
| 31 | 4 | -1 | -1 | +1 | +1 | +1 | 0.27 | 0.69 | 0.26 | 0.66 |
| 32 | 4 | +1 | +1 | +1 | +1 | +1 | 2.97 | 0.36 | 1.01 | 0.53 |

⁽A) Mucilage concentration; (B) temperature; (C) pH; (D) ionic strength; and (E) sucrose presence.

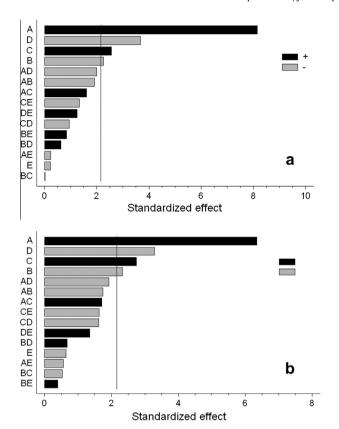


Fig. 3. Pareto chart of the consistency index (k). The black line indicates the critical level above which the variables presented a significant effect (p < 0.05). (A) Mucilage concentration; (B) temperature; (C) pH; (D) ionic strength; and (E) sucrose presence. (a) Upward flow curve and (b) downward curve.

ionic strength, pH and temperature, but concentration was the variable with the most significant effect. The consistency index (k)increased with increasing mucilage concentration and at alkaline pH, whereas it decreased in a medium with ionic strength and when temperature increased. These results are in agreement with previous studies (Marcotte et al., 2001; Vardhanabhuti and Ikeda, 2006; Farhoosh and Riazi, 2007) that reported that the consistency index of different hydrocolloid solutions increased with increasing concentration of the polysaccharide due to the greater hydrodynamic interactions among the molecules of the polysaccharide, whereas it decreased with increasing temperature due to the increase in molecular energy dissipation of the polysaccharide; therefore, intermolecular interactions decreased and the solutions showed a lower flow resistance. On the other hand, the decrease of k in a medium with the presence of salts could be associated with the presence of sulfate groups and carboxylic acids in the structure of the polysaccharide (Lin et al., 1994). The salts cause the contraction of the polysaccharide molecules, and thus viscosity decreases. In addition, the presence of these groups in the molecules of the polysaccharide increases k with increasing pH, associated with an increase in charge density (Huei Chen and Yuu Chen, 2001). This phenomenon was reported for mucilage dispersions of Opuntia ficus indica (Medina-Torres et al., 2000). Significant differences were detected between blocks (p < 0.05), which could be due to the type of salt used (mono- or divalent salt) or the mucilage extraction method (I and II). But the interactions between the factors were not significant (p > 0.05).

The effect of the variables and their interactions on the flow behavior index (n) of the upward and downward curves is shown in Fig. 4a and b, respectively. It can be observed that sucrose

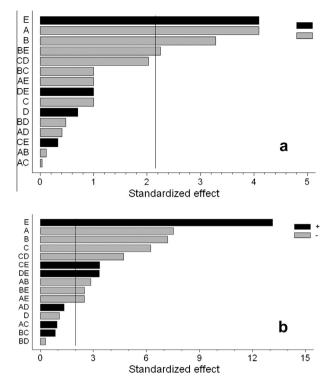


Fig. 4. Pareto chart of the flow behavior index (n). The black line indicates the critical level above which the variables presented a significant effect (p < 0.05). (A) Mucilage concentration; (B) temperature; (C) pH; (D) ionic strength; and (E) sucrose presence. (a) Upward flow curve and (b) downward curve.

concentration was the variable with the greatest effect on both curves, followed by mucilage concentration and temperature. At higher mucilage concentration, n decreased and the dispersions exhibited an increase in pseudoplasticity, whereas the addition of sucrose produced less pseudoplastic dispersions (Table 5). Regarding the addition of a disaccharide into hydrocolloid dispersions, a similar behavior was observed in solutions of Balangu seed (Lallemantia royleana) gum formulated with the addition of glucose (Salehi et al., 2014), and sodium alginate with the addition of sucrose (Yanes et al., 2002). However, Elfak et al. (1980) and Salehi et al. (2014) observed a decrease in the flow behavior index (n) of solutions of guar gum and Balangu seed gum, respectively, formulated with the addition of sucrose. Thus, the effect of the sucrose-hydrocolloid interaction on the flow behavior of actual products depends on the composition of the product itself, on the ingredient concentration, as well as on the rheological properties of the particular hydrocolloid (Yanes et al., 2002). In is worth noting that there was a significant interaction between temperature and the presence of sucrose in the upward curve, and a significant effect of pH was observed in the downward curve. No significant effect was detected among blocks; therefore, for the studied experimental conditions, n was not affected by the type of salt or the method of mucilage extraction.

Regarding tan δ , Fig. 5 shows that, with the exception of ionic strength, the rest of the variables significantly affected this property, being the presence of sucrose the one with the greatest effect. Tan δ increased in solutions with presence of sucrose, but it decreased with increasing temperature, mucilage concentration and alkaline pH. Significant interactions (p < 0.05) were also observed between different variables (pH-ionic strength, mucilage concentration-presence of sucrose, pH-presence of sucrose).

The effect of the blocks was significant (p < 0.05), which would indicate that this parameter behaved differently in a medium with an added mono- or divalent salt for dispersions formulated with mucilage extracted by MI or MII.

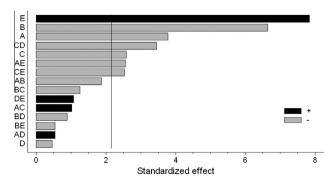


Fig. 5. Pareto chart of $\tan \delta$. The black line indicates the critical level above which the variables presented a significant effect (p < 0.05). (A) Mucilage concentration; (B) temperature; (C) pH; (D) ionic strength; and (E) sucrose presence.

Table 6Power law parameters obtained for chia mucilage dispersions with the addition 0.05 M of a monovalent or divalent salt.

| Trial | Repetition | Method | Salt | Upward curve | | Downward curve | |
|-------|------------|--------|-------------------|-------------------------------------|------|-------------------------------------|------|
| | | | | k (Pa s ^{n}) | n | k (Pa s ^{n}) | n |
| 1 | 1 | I | NaCl | 1.81 | 0.45 | 0.96 | 0.55 |
| 2 | 2 | I | NaCl | 1.87 | 0.43 | 0.90 | 0.55 |
| 3 | 1 | I | CaCl ₂ | 0.72 | 0.46 | 0.44 | 0.51 |
| 4 | 2 | I | CaCl ₂ | 0.73 | 0.44 | 0.35 | 0.52 |
| 5 | 1 | II | NaCl | 3.82 | 0.44 | 2.12 | 0.54 |
| 6 | 2 | II | NaCl | 4.20 | 0.43 | 2.46 | 0.52 |
| 7 | 1 | II | CaCl ₂ | 2.38 | 0.46 | 1.32 | 0.55 |
| 8 | 2 | II | CaCl ₂ | 1.88 | 0.44 | 0.93 | 0.57 |

The power law parameters of the upward and downward curves of dispersions of chia mucilage obtained by different methods (I and II) with the addition of a mono- or divalent salt are presented in Table 6.

The dispersions prepared with mucilage obtained by MII presented a higher consistency index than those formulated with mucilage obtained by MI (Table 6). The values of k were lower when a divalent salt (CaCl₂) was added, compared with those when a monovalent salt (NaCl) was added, in the dispersions prepared with hydrocolloids obtained by both methods. These results suggest that the molecular structure of the chia mucilage is negatively charged, behaving like a polyelectrolyte, and thus the addition of positive ions reduces the repulsion and molecular expansion, causing a significant decrease in viscosity, and this effect is more pronounced when using divalent ions (Medina-Torres et al., 2000). A similar behavior was observed in dispersions of xanthan, locust bean (Higiro et al., 2007), L. perfoliatum (Koocheki et al., 2013) and Balangu gum (Mohammand and Razavi, 2012).

For the set of analyzed samples, the values of tan δ were in the range of 0.5–0.8 (MI-CaCl $_2$ and MI-NaCl, respectively), which could indicate a weak elastic gel-like behavior in the frequency range studied for chia mucilage dispersions with a 1% concentration.

4. Conclusions

The analyzed chia mucilage dispersions presented a shear-thinning behavior (n < 1). Mucilage concentration was the variable that had the most significant effect on the consistency index (k), whereas the presence of sucrose was the variable with the most significant effect on the flow behavior index (n) and $tan \delta$, and an increase in those rheological properties was observed when the corresponding variables increased (mucilage concentration and presence of sucrose, respectively). The type of salt and mucilage extraction method significantly affected k, although they did not affect n. The values of $tan \delta$, suggest that chia mucilage can form

weak elastic gels for the frequency range tested. The extraction method II (soaking-filtration-concentration-freezing-freeze drying) and the addition of NaCl provided a higher consistency to chia mucilage dispersions. It would be interesting to take these results into account for the application of chia mucilage as a food additive, particularly in the production of food that requires certain rheological behaviors, for example as stabilizer and thickener of yogurt, mayonnaise and ketchup, among others.

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