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Morphological characterization of hydrocolloids using scanning electron microscopy and evaluation of their effects in a model system of low-calorie fruit jelly

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Highlights: The effects of hydrocolloids in a model system of low-calorie fruit jelly" and is important because "to know the behavior of the different hydrocolloids s used in order to produce a product with low-calorie similar to the conventional one.

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ABSTRACT - Companies and individual entrepreneurs which takes the milk from dairy storage points and dairy farms to distribute in the city centers, use milk cooling units mounted on pickup chassis. Cooling units are manufactured using stainless steel, 1st class Tungsten Inert Gaswelded, with a capacity of 200 to 5000 L, designed to remain no liquids inside. Cooling units are required for milk to be delivered to the customer without losing its freshness, without spoiling on the road, hundreds of kilometers on the vehicle. In this study, a milk cooling and storage unit based on a Peltier element with a capacity of 1000 L was designed. Energy calculations and finite element method was used to analyze the design of the developed model. As a result of the analyzes, it has been concluded that the milk can be cooled by three times less energy consumption with the Peltier Centric Cooling Unit developed.

Keywords: Milk tank, peltier, finite element method, ANSYS, milk cooling, energy harvesting, economic analysis

INTRODUCTION

Water-soluble gums (also called hydrocolloids), particularly the naturally occurring macromolecular proteins and polysaccharides, constitute the food structuring agents that play a critical role in imparting structure and stability to food (Gao et al., 2017). Such hydrocolloids are widely used in the food industry as a thickening and gelling aqueous solutions, stabilizing foams, emulsions and dispersions to inhibit the formation of ice and sugar crystals and allow controlled release of flavors (Das, Raychaudhuri & Chakraborty, 2015; Kang et al., 2015; Ferrero, 2017; Wei et al., 2019). The interactions of the hydrocolloids with each other or other components present in the food formulation determine their molecular distribution and consequently the final structure of the foods. Thus, hydrocolloids have a great impact on the texture and nutritional and functional aspects of foods, highlighting the need for the evaluation of their morphologies, chemical composition, and applications (Banerjee & Bhattcharya, 2012; Ai et al., 2015; Li and Nie, 2016).

According to Van de Velde et al. (2003), the products with reduced soluble solids, such as low-calorie fruit jellies, are characterized by the susceptibility to syneresis, brittle texture, lack of clarity, and loss of color and taste. Hence, it is necessary to improve our understanding about the use of appropriate hydrocolloids, to improve the rheological characteristics and alleviate the problems associated with the reduction in solids in these products, and to facilitate the use of sweeteners while maintaining the flavor. Among the most frequently used hydrocolloids in the food industry are guar gum, kappa-carrageenan, and low-methoxylated pectin (LM-pectin) (Mudgil, Barak & Khatkar, 2014; Wei et al. 2019).

According to Wu et al. (2016), the properties of polysaccharides in solution are also affected by their chain conformations other than chemical structures.

Guar gum, is compatible with use in conjunction with other agents such as carrageenan and pectin, is commonly used in the food industry for its ability to form viscous solutions and high moisture retention capacity (Mirhosseini & Amid, 2012). It may be used to enrich oral tactile sensation and texture and to modify and control the behavior of water in food (Dodi et al., 2016). It is described as a galactomannan isolated from the seeds of a leguminous plant, Cyamopsis tetragonolobus or Cyamopsi psoraloides, and comprises a linear long chain of β-(1-4) mannose with subunits of α-(1-6)-galactopyranosides, resulting in the formation of a highly branched polysaccharide (Patel, Karve & Patel, 2014; Szopinski & Luinstra, 2016; Mudgil, Barak & Khatkar, 2018). According to Liu et al. (2020) the rod-like morphology is intertwined more easily to form a network than the bean-like morphology thus leading to the lowest values in viscosity of guar gum so it has no gelling power.

Pectin, widely used in food and pharmaceutical applications, possesses great structural diversity. It is an anionic biopolymer (Li et al., 2013) with a complex structure comprising three domains as follows: (i) homogalacturonane (HG) consisting of α-d-galacturonic acid units attached to 1,4-(GalA); (ii) rhamnogalacturonan-I (RG-I) consisting of the repeated disaccharide [-4]-α-d-GalA-(1-2)-α-L-Rha-(1); and (iii) RG-(II) having an HG backbone with complex side chains attached to GalA residues (Kang et al., 2015). Mainly extracted from citrus peels and apple bagasse, commercial pectin may be classified as high-methoxylated (HM-pectin) pectin with usually possessed a methoxylation degree (DM) of 60–80% and low-methoxylated (LM-pectin) with DM less than 60% (Agoda-Tandjawa et al., 2012). Both forms can form physical gels. LM-pectin may form

gels at acidic pH or in the presence of bivalent ions such as calcium (Ca2+) through the formation of “egg-box” structures (Ormus et al., 2015), while HM-pectin forms gels in the presence of more than 55% sugar or a similar co-solute at pH below 3.5 (Ström, Schuster & Goh, 2014). According to Dupuis, Chambin, and Génelot (2006), this structure causes the water to be withdrawn in the LM-pectin network.

Carrageenan is a generic term for linear sulfated polysaccharides produced from alkaline extraction of red algae (Mahdavinia, Etemadi, & Soleymani, 2015). Among the carrageenans, the most commonly used and of commercial importance are kappa (κ), iota (ι), and lambda (λ). The primary differences between the carrageenan types are based on the content and distribution of sulfate ester groups with repetitive sequences of linear alternating disaccharides of 1,3- and 1,4-linked residues (Evageliou, Ryan & Morris, 2019). Higher levels of sulfate ester imply lower gelling force and lower solubilization temperature. kappa-carrageenan forms firm and brittle gels in water and milk and exhibits a certain syneresis and good water retention properties (Andrade et al., 2016), due to its structure, forms thermoreversible hydrogels, which has been widely used as an agent food and pharmaceutical industries (Salgueiro et al., 2013). According to Dewi, Ibrahim, and Suharto (2015) the commercial of carrageenan has granular form and well distributed.

The study of the interactions between hydrocolloids and other major components in food formulations may help explore the microstructure and characterize the thickening systems. The use of two or more gums in the formulation of a product is extensive in the food industry, owing to the synergistic effect observed with the combination. Certain gelling agents, when used in isolation, do not form gels or form fragile gels, favoring the syneresis process. However, in combination with other hydrocolloids, these agents form resistant gels (He et al., 2017).

According to López-Méndez et al. (2018) model systems are used because they are structurally and mechanically similar to many gel-like food products such as meats, cheese, gelatin desserts, jellies, frankfurters, yogurt, among others. Moreover, they represent a convenient way to avoid the inherent variability of raw materials in characteristics such as composition and texture, thus allowing the study of relevant features such as shrinkage and shape change in controlled scenarios (Waje et al., 2005).

The objective of this work was to evaluate the morphological structure of gelling agents, LM-pectin, kappa-carrageenan, and guar gum using scanning electron microscopy (SEM) and to study the effects of these gelling agents in low-calorie fruit jelly model systems.

MATERIAL AND METHODS

a) Materials

A commercial sample of *kappa*-carrageenan was supplied by Gastronomy Lab (Distrito Federal, DF, Brazil), while that of guar gum and LM-pectin was provided by PrymeFoods (Sorocaba, SP, Brazil) and Rica Nata (Piracema, MG, Brazil), respectively.

b) Scanning electron microscopy (SEM)

The powdered samples were placed under a carbon tape and then loaded onto a Q150R S Aname (Spain) metallizer for carbon deposition (100 to 200 nm) under vacuum, following the procedures of Sales (2016). The morphologies of the gum samples were analyzed with SEM of the Oxford Instruments Nordlys (UK) model TESCAN VH3. The acceleration voltage of 15 Kev was used, and the images were obtained at different magnifications (200×, 5,000×, and 10,000×).

c) Preparation of low-calorie fruit jelly model systems

To prepare model systems mixture samples were used LM-pectin (X1), guar gum (X2), and *kappa*-carrageenan (X3) at different proportions corresponding to each treatment (Table 1).

Table 1. Compositions of model system mixture samples in a simplex lattice mixture design.

Mixtures	Ingredient proportions		
	X1	X2	X3
1	1	0	0
2	0	1	0
3	0	0	1
4	0.5	0.5	0
5	0.5	0	0.5
6	0	0.5	0.5
7	0.68	0.16	0.16
8	0.16	0.68	0.16
9	0.16	0.16	0.68
10	0.33	0.33	0.34

X1: LM-pectin; X2: guar gum; X3: *kappa*-carrageenan

First, 60% distilled water, 20% crystal sugar (50% reduction in sugar quantity compared to conventional jelly), and 18.925% polydextrose were mixed and the mixture was cooled at a temperature of approximately 80 °C until the value of 30 ° Brix was reached. Concentration occurred at atmospheric pressure under stirring. Gelling agents (LM-pectin, guar gum, and *kappa*-carrageenan) were then added to the mixture according to the experimental design (Table 1) at 1% of the total mass of the mixture; these were dissolved in water at 40 °C. The endpoint of the processing of the model system was determined by the method of the measurement of the index of refraction at 65 °Brix. In the subsequent step, acesulfame-K and sucralose sweeteners were added at concentrations of 0.01875% and 0.00625%, respectively, followed by the treatment with potassium sorbate at 0.05% of the total blend (Souza et al., 2013). The samples were hot while packaged in sterilized glass containers and were sealed and cooled to room temperature.

d) Determination of rheological behavior of low-calorie fruit jelly model systems

Rheological properties of low-calorie fruit jelly model systems samples were determined using a rheometer (cone/plate type; Brookfield, model RV-III, USA) coupled to a Software Rheocalc version V 3.0 using spindle CP52. A total of 0.5 g of sample was used and the analysis was performed at 25 °C. The measurements were made in triplicates using a rotation speed of 3.0 to 15.0 rpm, with a variation in the range of 3.0 at 3.0 rpm, to obtain an upward curve. The procedure was repeated in the reverse direction with progressively decreasing velocities (15.0-3.0 rpm) to obtain the descending curve. The experimental values of shear stress and strain rate were adjusted by the Ostwald-de-Waele rheology model (Power Law) and consistency coefficient and flow behavior index values were calculated according to the following model (Equation 1).

$$\eta_a = K\gamma^{n-1} \quad \textbf{(1)}$$

where η_a is the apparent viscosity (Pa.s), K is the consistency coefficient (Pa.s^n), γ is the shear rate (s^{-1}) and n is the flow behavior index (dimensionless).

e) Experimental design, statistical analysis, and modeling of experimental data

In the present study, the simplex lattice mixture design was used to evaluate the effect of LM-pectin (X1), guar gum (X2), and *kappa*-carrageenan (X3) on the rheological characteristics of the samples (Table 1). The 10 points were 3 single-ingredient treatments, 3 two-ingredient mixtures, and

4 three-ingredient mixtures.

The following polynomial equation of function x_i was fitted for each factor assessed at each experimental point. This polynomial model differs from full polynomial models because it does not contain a constant term (intercept equal to zero). The polynomial model equation (Equation 2) used was:

$$Y = \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 \quad \textbf{(2)}$$

where Y is the estimated response; β_1, β_2, β_3, β_{12}, β_{13} and β_{23} are constant coefficients for each linear and non-linear (interaction) term produced for the prediction models of rheological parameters.

The computational work, including ternary contour graphical presentations of the models, was performed using Statistica 6.0 software (StatSoft Inc., U.S.A., 2007). The general regression model was adjusted to the values of the response variables. For the parameters that did not fit the model, the Scott-Knott test at 5.0% probability was used in Sisvar software (Ferreira, 2014).

RESULTS AND DISCUSSION

a) Scanning electron microscopy (SEM)

Figure 1 shows the micrographs of the gelling agents at 200× magnification.

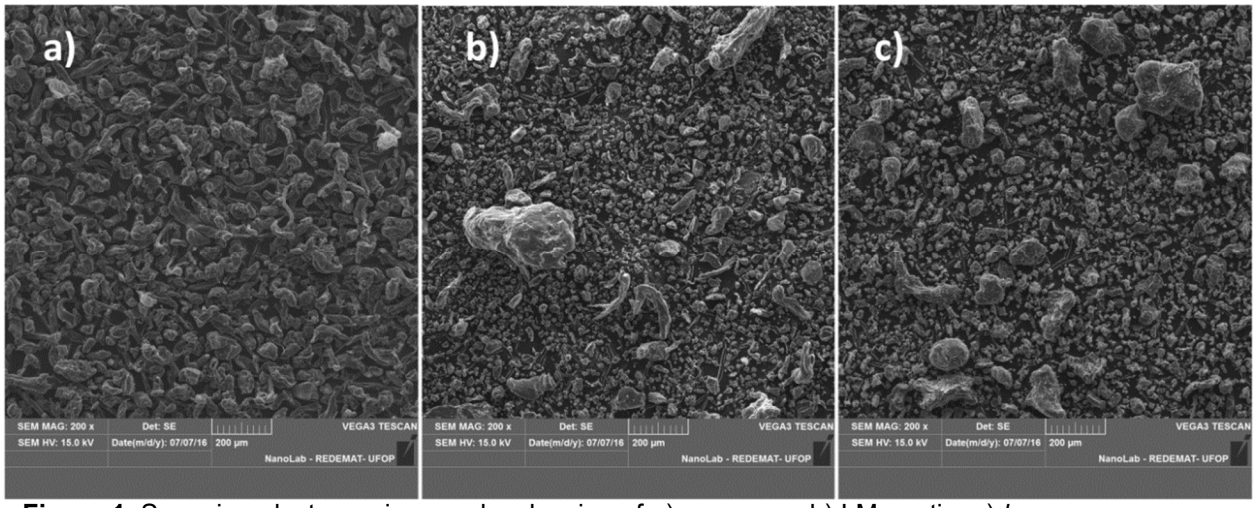

Figure 1. Scanning electron micrographs showing of a) guar gum; b) LM-pectin; c) *kappa*-carrageenan (200× magnification)

The particles were observed to have different shapes and sizes. Samples of LM-pectin (Figure 1b) and *kappa*-carrageenan (Figure 1c) showed similarity, consistent with the results of a study by Chomto & Nunthanid (2017) that evaluated the physicochemical characteristics of LM-pectin for pharmaceutical applications. Sadeghi (2012) reported similar results using pure commercial *kappa*-carrageenan, which showed fewer pores and smaller structures than derivatized copolymers. The physical characterization of the guar gum sample also revealed differences in the grain sizes, as shown in Figure 1a. The presence of fillets of different sizes and distribution was observed. Besides, the spherical nature of the particles was confirmed. The same result was observed for guar gum powder at different concentrations in the study by Wang, Ellis, and Ross-Murphy (2003), wherein the possible differences between particle size and specific surface areas related to the hydration behavior of these samples were evaluated.

Figures 2 and 3 show images acquired at 5.000× and 10.000× magnification, respectively, and reveal that *kappa*-carrageenan had a more granular surface with less smooth regions compared to guar gum, while LM-pectin had high roughness index on the surfaces of its granules.

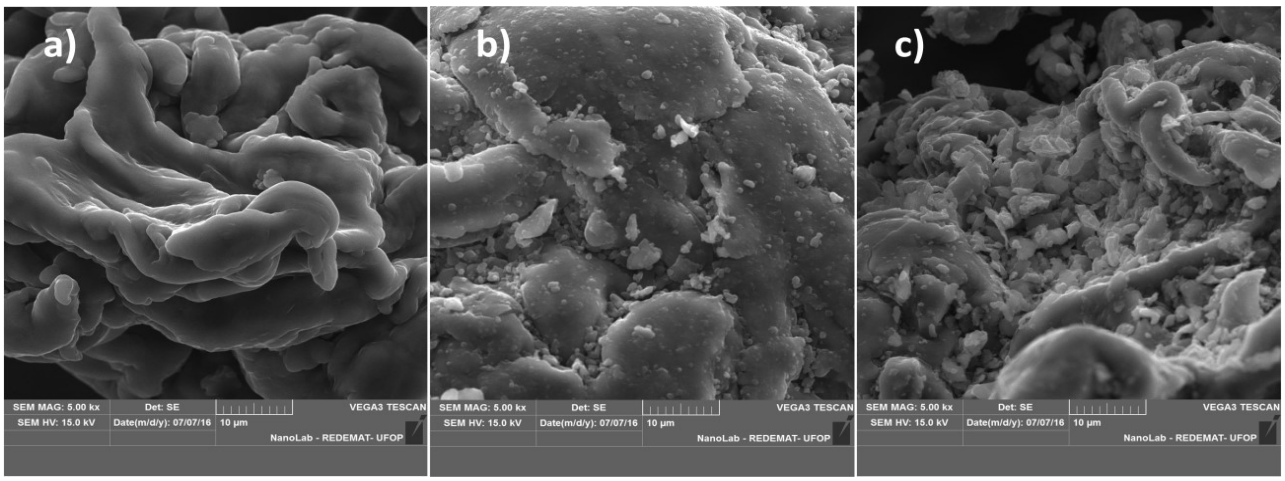

Figure 2. Scanning electron micrographs showing of a) guar gum; b) LM-pectin; c) *kappa*-carrageenan (5.000× magnification)

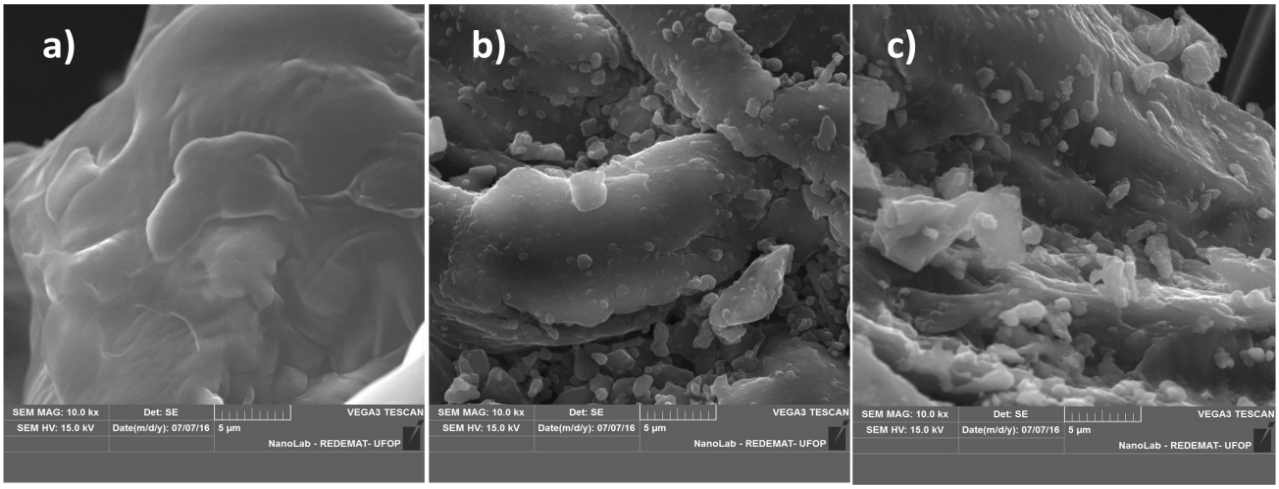

Figure 3. Scanning electron micrographs showing of a) guar gum; b) LM-pectin; c) *kappa*-carrageenan (10.000× magnification)

The high scoring and roughness and the shortest distance between the lamellae in LM-pectin and *kappa*-carrageenan samples may suggest the presence of the necessary structures for the formation of the secondary joints in the process of gel formation, thereby providing viscosity and maintaining the structure of the system. The rough and cracked surface favors the gelation process by imprisoning the aqueous contents in these spaces and delaying their release (Daud et al., 2015). According to Furmaniak, Terzyk, and Gauden (2007), water may be bound to the pectin powder through physical immobilization on the surfaces of the particles by sorption or via entrapment in microcapillaries or voids between the particles. Murrieta-Pazos et al. (2012) reported that several properties related to dust and particles are crucial to these effects. Both depend very much on the generation of the particles, that is, the processes and technological conditions during the extraction and modification of the gum. As a result, the particles differ in shape, size distribution, surface properties, pores, and physical state. The properties of the particles determine the parameters of the powder, such as density and porosity, which have an impact on water absorption (Cuq, Rondet & Abecassis, 2011). Large and coarse rounded particles often exhibit late water absorption and bonding as compared to more homogeneous particle surfaces or smaller, fibrous particles; however, on a large scale, the former causes larger voids between particles, wherein additional water may be immobilized (Einhorn-Stoll, Hatakeyama & Hatakeyama, 2012).

The micrographs corresponding to the guar gum revealed its discrete, elongated, and irregular granular structure. The granules were separated from each other and had irregular but smooth surface (flawless). This observation was in line with that reported by Chandrika et al. (2014). Guar gum does not exhibit gel formation and is commonly used as a thickening agent or in combination with other gums. According to Panchev et al. (2010), the crystalline and vitreous

materials restrict the binding of water to the hydrophilic groups on the surface of the particle during the initial phase of water absorption. These require time for swelling, softening, and plastification until their transformation into a rubberized state, which makes the inner groups more accessible for hydration. In contrast, water molecules may permeate the amorphous materials more quickly and reach the inner hydrophilic groups. In general, the more resistant the material is to swelling, the lower is the total water absorption (Ping et al., 2001).

b) Rheological evaluation of low-calorie fruit jelly model systems

The contour area of the consistency coefficient parameter is shown in Figure 4.

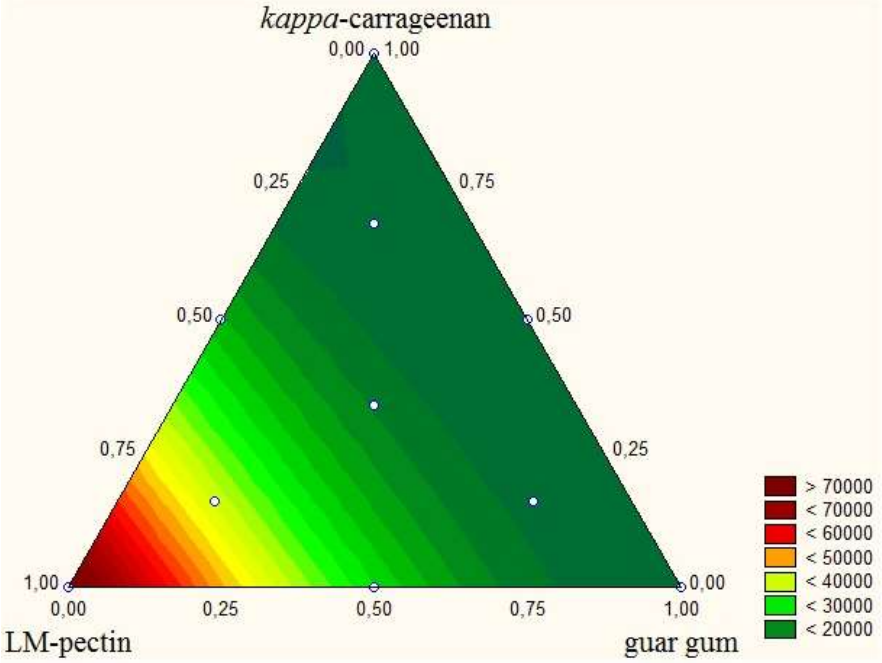

Figure 4. Mixture contour plots consistency coefficient parameter

The consistency coefficient (K) is directly related to viscosity and the Power Law Model, which provides the simplest representation of rheological behavior and uses this parameter as a measure of fluid consistency. Another parameter provided by the Law of Power is the flow behavior index (n) that defines the behavior of the Newtonian or non-Newtonian fluid and evaluates the degree of fluid pseudoplasticity (Steffe, 1996).

The model systems that presented the highest values of the consistency coefficient (Figure 4) were obtained in the regions with LM-pectin concentrations of greater than 75%, whereas the lowest values of the consistency coefficient were observed in the regions that had concentrations of greater than 50% for guar gum and *kappa*-carrageenan. Thus, the use of gelling agents (LM-pectin, guar gum, and *kappa*-carrageenan) has influenced the values of consistency index; the presence of LM-pectin tended to increase the values of this coefficient.

The factors that influence the gel formation behavior of LM-pectin include the degree of esterification as well as the percentage of calcium and sucrose added (Dickinson, 2003). Although the gel-forming ability of LM-pectin in the absence of sucrose is known, the addition of this compound increases the gel strength and set temperature. This is attributed to the specific effects of sucrose on water activity and hydrophobicity. In this way, sucrose helps to stabilize the structure of the junction zones during the formation of gel (Fu & Rao, 2001).

Fu & Rao (1999) studied the influence of sucrose and sorbitol on gel-sol transition in pectin gels with Ca^{2+} ions. The results showed that the concentration of sucrose influenced the melting temperature, and the increase was related to the increase in sucrose concentration. The authors related this observation to the hypothesis that sucrose may provide the hydroxyl groups that stabilize the structure of the junction zones and promote hydrogen bonds, which may immobilize the free water and prevent the melting of the gel.

In their study with pectin obtained from pear orange peel (*Citrus sinensis* L. Osbeck), Dias &

Pulzatto (2009) prepared yogurt samples without pectin, with LM-pectin extracted in the laboratory, and with HM-pectin and commercial LM-pectin. These authors observed that the viscosity of the yogurt samples increased with the addition of LM-pectin. The samples treated with LM-pectin showed lower syneresis as compared with those prepared with HM-pectin and without any pectin addition. The presence of pectin did not affect the physicochemical parameters of the yogurt samples.

In the present study, gelation was affected by the presence of sufficient amounts of metal ions (Ca^{2+}) in the gelling agent used to make the gel (data not shown). It has been reported that the gelation mode of pectin depends on the calcium concentration and that the nets in the pectin gels may be transformed from semi-flexible to flexible with a decrease in the level of calcium, as determined by effective calcium concentration over the free carboxyl group in pectin molecules (Vincent & Williams, 2009).

Fraeye et al. (2010), while evaluating the influence of pectin estrus on the texture of pectin-calcium gels, reported that the increase in Ca^{2+} concentrations led to an increase in the elasticity modulus and, hence, resulted in more rigid gels.

LM-pectin had the highest positive effect among the gelling agents evaluated in the model systems. Although statistically significant ($p \leq 0.05$), guar gum exerted a more discrete effect as compared to LM-pectin. Yaseen et al. (2005) evaluated the rheological behavior of 12 solutions of gums in food systems and found that guar gum showed higher values of viscosity than other gums when used at concentrations around 0% to 2% at 25 °C.

The mixture of guar gum and *kappa*-carrageenan had no significant synergistic effect on the consistency coefficient. A similar result was found by Maruyama et al. (2006) while evaluating the influence of different gums on petit-suisse cheese texture. These authors concluded that the mixture of guar and *kappa*-carrageenan gums failed to achieve the necessary firmness and presented the lowest values of viscosity as compared with the mixture containing xanthan gum. In a previous paper, Maruyama et al. (2006) found a decrease in firmness throughout the storage period in petit-suisse cheeses treated with carrageenan gum alone.

Gelation with *kappa*-carrageenan requires a significant ordering of molecules even before viscosity construction occurs (Gladkowska-Balewicz, Norton & Hamilton, 2014). According to Campo et al. (2009), *kappa*-carrageenan molecules in solution exist as random unstructured balls above a certain temperature as a result of the electrostatic repulsion between the chains. Reduction in temperature induces the formation of double helices. The intermolecular association between the double helices is confined with the formation of small independent domains involving a limited number of such structures. However, in the presence of cations such as K^+, Ca^{2+}, and Na^+, aggravation of helices occurs, eventually causing a long-range crosslinking that may lead to the formation of a gel. As shown by Liu & Li (2016), the low concentrations of ions present in the medium are insufficient to facilitate the binding to *kappa*-carrageenan helices to the point of reducing the charge density of these ions, the aggregation of the propellers.

Table 2 shows the results of the flow behavior index (n) of low-calorie fruit jelly model systems.

Table 2. Flow behavior index (n) of low-calorie fruit jelly model systems

Mixtures	Flow behavior index (n)
1	0.215 ± 0.09^{b}
2	0.290 ± 0.07^{b}
3	0.525 ± 0.18^{a}
4	0.510 ± 0.04^{a}
5	0.395 ± 0.09^{b}
6	0.560 ± 0.04^{a}
7	0.530 ± 0.01^{a}
8	0.610 ± 0.16^{a}
9	0.240 ± 0.07^{b}
10	0.515 ± 0.02^{a}

n=3. Mean values with common letters in the same column indicate that there is not a significant difference between samples ($p > 0.05$) from Scott-Knott's mean test.

According to Chenlo, Moreira, and Silva (2010), the flow behavior index measures the deviation from the Newtonian behavior and indicates the degree of pseudoplasticity of the fluids such that the further away the "n" value is from the unit, the greater is the pseudoplasticity of the material. Although it was not possible to generate a model for flow index, all the values found were smaller than 1 ($n < 1$), suggesting that all the formulations showed pseudoplastic behavior (Table 2). Similar behavior was also observed by Kayacier & Dogan (2006) who prepared solutions with different gums, including xanthan and guar, and determined the rheological properties of these solutions using the Law of Power model.

According to Funami (2011) the steady shear rheological properties are associated to the flow velocity of the food bolus (or viscosity), is that the flow index (n) values are related to sliminess perceived in the mouth. The lowest n values of low-calorie fruit jelly model systems indicate that it is likely to facilitate swallowing and reduce organoleptic viscosity (slimy feel) due to its higher pseudoplasticity, providing a pleasant and light mouthfeel (Vieira et al., 2020).

CONCLUSION

It was possible to evaluate the morphology of the gelling agents and delineate the relationship between gel formation capacity. The gelling agents LM-pectin and kappa-carrageenan have rough surfaces with slits or micro-capillaries that favor the gelation process by imprisoning the aqueous content. Guar gum, on the other hand, presented an irregular but smooth surface without any porosity, decreasing the water absorption and making its internal groups less accessible to hydration; hence, guar gum shows no gel formation and is suitable only as a thickener.

The interaction between the gelling agents was influenced by the rheological characteristics of the low-calorie fruit jelly model systems. The obtained values indicated that the model systems that presented the highest values of consistency coefficient were obtained in the regions with higher concentrations of LM-pectin, while the lowest values for consistency coefficient were observed in the regions with higher concentrations of guar gum and kappa-carrageenan. Thus, the use of gelling agents (LM-pectin, guar gum, and kappa-carrageenan) influences the values of the consistency coefficient, and the presence of LM-pectin tended to increase the values of this coefficient. As for the flow behavior index, all the formulations presented pseudoplastic behavior.

Based on all the information gathered through the analysis of low-calorie fruit jelly modeling systems using the gelling agents studied, we suggest that the best combinations among the formulations tested were those containing concentrations higher or equal than 75% LM-pectin.

The results of this study are extremely important for the fruit processing industries, given that new demands from consumers for products with reduced sugars should produce new products similar to conventional ones. Thus, the elucidation of the mechanisms of gelation of these agents in fruit jellies makes the food industries more subsidized in their choices.

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