

http://periodicos.uem.br/ojs ISSN on-line: 1807-8664 Doi: 10.4025/actascitechnol.v45i1.64193



# Standardization of processing parameters and color of gels aiming to replace citric pectin by mesocarp flour or pectin of passion fruit

Juliana Resende Gonçalves Silva<sup>1</sup>, Jhone Gleison de Oliveira<sup>2</sup>, Ricardo Augusto Mendonça Vieira<sup>2</sup>, Eder Dutra de Resende<sup>1\*</sup>

<sup>1</sup>Laboratório de Tecnologia de Alimentos, Universidade Estadual do Norte Fluminense, Av. Alberto Lamego, 2000, 28013-602, Campos dos Goytacazes, Rio de Janeiro, Brasil. <sup>2</sup>Laboratório de Zootecnia, Universidade Estadual do Norte Fluminense, Campos dos Goytacazes, Rio de Janeiro, Brasil. \*Author for correspondence. E-mail: eresende@uenf.br

**ABSTRACT.** The processing of passion fruit generates a huge quantity of wastes, reaching 50% in mass of rind. The main use of the rinds is to produce rind flour or to be source of pectin as a gelling agent. This work develops a standard procedure to evaluate jelly products subjected to the same temperature histories and content of soluble solids along the processing time of the gel-based product, using different concentrations of mesocarp flour and pectin. The gels were evaluated by color patterns aiming to verify the applicability of mesocarp flour as substitute of commercial pectin in jelly of fruits. The quantitative analysis of the data for temperature histories and content of soluble solids enabled to confirm the standardization of the parameters used in the production of the gels, which is important to warrant the same processing conditions during the elaboration of the different types of gels. The gels made with passion fruit's mesocarp flour have less translucent shades and a more yellowish color than the gels produced with commercial citric pectin, however, these small differences may not interfere in the final color of fruit jelly.

Keywords: Passiflora edulis; rind flour; pectin extraction; gel processing; jelly ingredients.

Received on june 30, 2022 Accepted on november 03, 2023

## Introduction

The sustainability of food systems requires the optimum utilization of all produced raw materials of the food chain (Galanakis, 2020). Fruit co-products are formed by a mixture of peels, endocarps, seeds, inner parts of the fruit and even pulp. The predominant classes of macromolecules found in plant co-products are polysaccharides such as cellulose, hemicellulose and pectin (Sabino et al., 2020).

The industrial processing of passion fruit generates a huge amount of rind residues. According to the Brazilian Institute of Geography and Statistics (IBGE, 2022), the national production of passion fruit in 2020 was 690,364 t, and the major part of it goes to the processing industry (López-Vargas, Fernández-López, Pérez-Álvarez, & Viuda-Martos, 2013).

The rind of passion fruit is composed by epicarp and mesocarp fractions. Removal of the epicarp allows the pure white mesocarp flour to be obtained after drying. According to Talma, Regis, Ferreira, Mellinger-Silva and Resende (2019), ripe fruits have 50% rind and the mesocarp fraction represents the largest proportion of the rind (70.06%).

Passion fruit mesocarp is considered an excellent source of pectin that can be used to prepare food products, either in the form of pure pectin extracted from the rind, or by direct application of mesocarp flour as a source of pectin. The importance of pectin in the food industry is attributed to its gelling, stabilizing, thickening and emulsifying properties (Oliveira, Giordani, Gurak, Cladera-Olivera, & Marczak, 2015).

The passion fruit mesocarp flour has a high fiber content (58.8 g 100 g<sup>-1</sup>), low lipid (0.63 g 100 g<sup>-1</sup>) and ash contents (1.95 g 100 g<sup>-1</sup>), and considerable levels of minerals, such as calcium (1363.94 mg 100 g<sup>-1</sup>), magnesium (314.27 mg 100 g<sup>-1</sup>) and potassium (87.53 mg 100 g<sup>-1</sup>) (Silva, Silva, Silva, Lopes, & Gusmão 2016).

Acid extraction is the cheaper and the most industrially useful method to obtain pectin (Vriesmann, Teófilo, & Petkowicz, 2012). According to Talma et al. (2019), with the use of nitric acid, the yield of pectin extraction from passion fruit mesocarp is 26.6%.

Page 2 of 12 Silva et al.

Apple and citrus fruits are the main sources used by industry to obtain commercial pectin, however, new sources of pectic materials have been studied (Lara-Espinoza, Carvajal-Millán, Balandrán-Quintana, López-Franco, & Rascón-Chu, 2018). The replacement of commercial pectin by passion fruit mesocarp flour may be an alternative way to reduce the cost of making sweets, jellies or other products, as pectin extraction is a high-cost technique for this important ingredient for the agroindustry.

There are few studies in the literature that evaluate the pectin gels obtained by the conventional process of jelly production, however, they do not adequately control the processing variables, in order to standardize the heat effect on the physical and rheological characteristics of different gels. This work shows a standard procedure to control the parameters of pH, content of soluble solids, temperature and processing time. The objective was to promote the same heat processing conditions necessary to evaluate the quality characteristics of gels produced with passion fruit mesocarp flour and its pectin, compared to commercial citrus pectin. The work also evaluates the color of gels made with different types of pectin, as well as the one from passion fruit mesocarp flour.

#### Material and methods

#### Fruit processing and mesocarp flour production

The yellow passion fruits (*Passiflora edulis* f. *flavicarpa* Deg.) were harvested manually in the ripe stage at the orchard of the Fluminense Federal Institute located in Bom Jesus de Itabapuana (RJ, Brazil). The fruits were washed under tapping water, sanitized with  $0.2 \text{ mL L}^{-1}$  hypochlorite solution, and dried with paper towels.

First, the fruits were manually peeled with a knife to remove the epicarp, and then cut off in half to scrap the endocarp and obtain the pure mesocarp fraction. The mesocarp was washed in distilled water and chopped into 2 cm pieces, and left to dry in a tray dryer (Pardal, Brazil) with forced air circulation at 60 °C for 24 hours. The dried material was ground in an analytical mill (Quimis, Brazil), and the flour was standardized by using a 60 Mesh sieve. The obtained material was packed in low density polyethylene bags, and kept in a desiccator until the analyze procedure.

#### Yield of pectin extraction

The pectin extraction was performed according to Fertonani et al. (2009), with modifications. About 1 g of mesocarp flour was weighed (Gehaka, model AG 200) and dissolved in 50 mL of 1 mol  $L^{-1}$  nitric acid, previously heated to 80 °C, at a solid-liquid ratio of 1:50 (m/v). The sample was kept under stirring in a water bath (Nova Técnica, model NT 232, Brazil) for 40 min at 80 °C, and then cooled in an ice bath. The acidic solution was filtered through synthetic fabric and the filtrate was added to three volumes of 95% ethanol and kept at 4 °C for 15 h (overnight). The precipitated pectin was filtrated and dried for 6 hours at 60 °C (Pardal, Brazil). The dried pectin was ground in an analytical mill (Quimis, Brazil) and standardized in a 60-Mesh sieve. The pectin yield (%) was obtained according to Torralbo, Batista, Di-Medeiros and Fernandes (2012), by considering the ratio in weight of pectin to mesocarp flour, standardized to 100 g of dry matter (db).

## Physicochemical characterization of pectin and flour

The mesocarp flour and its pectin were evaluated for color, moisture and ash content. For a comparative study, a fast-gelling commercial citrus pectin, CPKelco® (Genu 105) was used as a reference.

## Color analysis of pectin and rind flour

The color measurements were performed in the Hunter colorimeter (MiniScan Spectrophotometer Plus, HunterLab, USA), illuminant D65,  $10^{\circ}$  observation angle, calibrated with black and white reflective plate, with the results expressed by the CIE L\* a\* b\* parameters. The readings were taken in a crystal cuvette ( $40 \times 60 \text{ mm}$ ) coupled to a black opaque cup ( $65 \times 75 \text{ mm}$ ) using a 20 mm sample height. Readings were taken in clockwise rotational cuvette positions every  $45^{\circ}$  until reaching  $360^{\circ}$  of rotation, taking the average of eight readings for each sample. The same procedure was performed with the empty cuvette to correct the color readings of the samples.

#### Moisture and ash content

Moisture analysis of mesocarp flour and pectin was performed by direct drying of 1 g of the sample in a drying oven (Quimis, Brazil) at 105 °C for 24 h, according to the Association of Official Analytical Chemists [AOAC] method (1995). The ash content of pectin was determined by the residue incineration method in a

muffle oven (Quimis, model Q318M24, Brazil) at 550 °C, according to the AOAC methods. The measurements were performed in triplicates with four replicates, and data were expressed in percentage.

#### **Gel elaboration process**

The gelling process was evaluated in gel-models at concentrations of 1.0, 2.0 and 3.0% (w/v), which cover the concentration range normally employed in the commercial gelling process, using commercial pectin, the flour of passion fruit mesocarp and pectin extracted from it. The initial amount of sucrose added to the water mixture was the same applied in the production of commercial jellies (60%), using crystal sugar from local market (Neve brand).

The gel solution was prepared with 180 g of sugar dissolved in 300 mL of distilled water in a 1 L Beaker. The pectic material was previously dissolved in this water volume according to its own concentration in the gel, using a mechanical stirrer (Fisatom, model 713, Brazil). The beaker with gel solution was heated on a heating plate (Fisaton, model 752 A) inside a 2 L Beaker containing 40% ethylene glycol solution to a temperature of 105 °C (Figure 1). The processing was performed under stirring at 400 rpm, until the soluble solids content reached 65 °Brix, and the acidity (pH 3.0) was adjusted with 25% citric acid solution or 0.1 mol L-1 NaOH. Gel samples with the same volume were quickly placed in 25 mL glass Beaker or Petri plate using an ultrasonic bath (Clifton, model UM-22) to remove air microbubbles from the gel. Then, the samples were cooled at room temperature and kept refrigerated at 4 °C for further analysis. Processing parameters were monitored by a temperature sensor (Incoterm digital thermometer, model 6132) and a digital refractometer (Atago, model PAL-3), at regular intervals of 10 min.



**Figure 1**. Assembly of the equipment used in production of gels inside a 1 L Beaker supported by cork stoppers inside a 2 L Beaker containing ethylene glycol solution at 40%, coupled on a heating plate, and to a mechanical stirrer.

#### Gel color analysis

The color measurements of gels were performed in the Hunter colorimeter (MiniScan Spectrophotometer Plus, HunterLab, USA). The readings were taken in a Petri plate ( $60 \times 15 \text{ mm}$ ) with 10 mm of sample height, coupled to a black opaque cup ( $65 \times 75 \text{ mm}$ ). The passion fruit pectin analysis' protocol was the same used for color analysis of commercial pectin powder.

#### Statistical analysis

The experiment was set up in a completely randomized design with four replications, using analysis of variance (ANOVA) and Tukey test at 5% significance level, in order to evaluate the physicochemical characteristics of the passion fruit pectin and commercial pectin, as well as the color data of gels containing 1%, 2% and 3% of these ingredients. Results were processed using the STATISTICA program, version 7.0.

The histories of bath temperature (Tb), temperature of gel (Tg) and soluble solids contents of gel (SSg) were fitted by sigmoid regression models. The sigmoid models were as follows:

$$\mu = \mu_0 + \mu_g \left( 1 - (\theta^N \exp(-\kappa t) + \exp(-\lambda t) \sum_{i=0}^{N-1} (1 - \theta^{N-i}) (\lambda t)^i / i!) \right)$$
 (1)

$$\mu = \mu_0 + \mu_g (1 - \exp(-\lambda t) \sum_{i=0}^{N-1} (\lambda t)^i / i!)$$
 (2)

$$\mu = \mu_f \exp(-\kappa_0 \exp(-\delta t)/\delta) \tag{3}$$

Page 4 of 12 Silva et al.

$$\mu = \mu_0 \mu_f / (\mu_0 + (\mu_f - \mu_0) \exp(-\kappa_1 t)) \tag{4}$$

$$\begin{cases} \mu = \mu_f \exp(-\kappa_0 \exp(-\delta t)/\delta), 0 \le t \le t_{knot} \\ \mu = \mu_f \exp(-\kappa_0 \exp(-\delta t_{knot})/\delta) + \beta_1 (t - t_{knot}), t > t_{knot} \end{cases}$$
(5)

$$\begin{cases}
\mu = \mu_f \exp(-\kappa_0 \exp(-\delta t)/\delta), 0 \le t \le t_{knot} \\
\mu = \mu_f \exp(-\kappa_0 \exp(-\delta t_{knot})/\delta) + \beta_1 (t - t_{knot}) + \beta_2 (t - t_{knot})^2, t > t_{knot}
\end{cases}$$
(6)

The parameters  $\mu_0$  and  $\mu_f$  represent the Tb, Tg (°C) and SSg (dimensionless concentration) at time zero and close to the asymptotic stages (at t =  $t_{knot}$ ), and the constant  $\theta = \lambda/(\lambda - \kappa)$ . Parameter  $\mu_g$  represents the amount added to  $\mu_0$  until the asymptote reach,  $\lambda$  is the first-order delay constant for changes in the dependent Tb, Tg and SSg over time,  $\kappa$  is the specific rate of decline of changes in those variables, and N is the order of time dependency due to change delays in both equations (1) and (2). In the Gompertz equation (Eq. 3),  $\mu_f$  is the asymptotic value,  $\delta$  is the specific rate of decline of the initial specific rate at time zero ( $\kappa_0$ ), and in the logistic equation (Eq. 4), the specific rate of decline of variable changes is  $\kappa_1$ . As the behavior of Tb, Tg and SSg was close to the asymptotic phase, that is, there was a curvature in the observed behavior, an additional solution was used, by adjusting Eq. (5) for this variable. Equations (5) and (6) are segmented models about the knot ( $t_{knot}$ ), so that the behavior prior to the knot is sigmoid, and after the knot it could be a smooth decreasing straight line or a parabola. The parameters of the Gompertz (Eq. 3) have the same meanings, and  $\beta_1$  and  $\beta_2$  are the scale parameters of the quadratic parabola.

The models were fitted with the nlme function of R, by introducing gradually fixed and random parameters, as well as by challenging the traditional homogeneous and independence assumptions for residual variances and errors. Therefore, the models under the independence and homoscedasticity assumptions were fitted with an autoregressive correlation function (corCAR1) to challenge independence, and with a power-of-the-mean variance function (VarPower) to challenge homoscedasticity, or even by both (Pinheiro & Bates, 2000). The fit quality of the models was evaluated based on the information-theoretic (I-T) approach (Burnham, Anderson, & Huyvaert, 2011), with special reference to a nonlinear model selection framework (Vieira et al., 2020).

The graphical representations containing the 99% confidence intervals for predictions of Tg, Tb, and SSg values for each material and pectin level were drawn. The NLMIXED procedure of SAS University Edition (SAS Systems, Inc.) was used to predict the confidence intervals of those variables, according to the quantitative methods described by Vieira et al. (2018).

# **Results and discussion**

#### Physicochemical characterization of mesocarp flour and pectin

The moisture content of mesocarp flour  $(9.4 \pm 0.3 \%)$  was similar to the commercial pectin  $(9.9 \pm 0.5 \%)$ , but slightly higher than the pectin extracted from mesocarp flour  $(6.9 \pm 0.6 \%)$ . Oliveira and Resende (2012) also obtained similar results for mesocarp flour moisture (9.7%). Pectin moisture depends on its source and drying conditions. Linares-García, Ramos-Ramírez and Salazar-Montoya (2015) found moisture content of 10.3% in commercial citrus pectin.

The pectin extracted from mesocarp flour presented 24.9% yield (dry weight). This content is similar to the findings of Talma et al. (2019) (25.5%) and Oliveira and Resende (2012) (26.4%), using the same extraction conditions. According to Talma et al. (2019), the yield of pectin from mesocarp flour does not change with the maturation stage of the passion fruit, but in the epicarp flour it decreases from 16.7% (green stage) to 12.6% (ripe stage).

The high content of pectin in the passion fruit mesocarp flour indicates its great applicability when compared to other materials. Apple pomace contains 10-15% of pectin on a dry matter basis. Citrus peel contains relatively higher pectin (30-35%), when compared to that of apple (Lara-Espinoza et al., 2018).

The ash contents found in the pectin extracted from mesocarp flour (PM)  $(2.28 \pm 0.04 \%)$  and commercial citric pectin (PC)  $(2.25 \pm 0.04 \%)$  samples did not differ statistically (p < 0.05). According to Kar and Arslan (1999), low ash content indicates the purity of the samples.

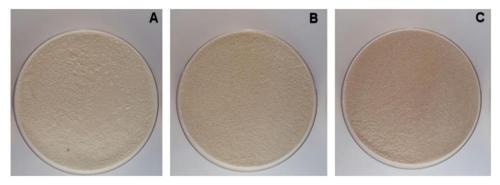
The color parameters of pectin and mesocarp flour are exhibited in Table 1. Mesocarp flour and PM pectin presented higher luminosity values (CIE L\*), with a lighter shade than the commercial pectin evaluated. Talma et al. (2019) and López-Vargas et al. (2013) found lower CIE L\* values (82.08 and 79.91, respectively) in mesocarp flour (50-Mesh) of ripe passion fruit. The smallest particle size used in our study (60-Mesh) may have

contributed to make more clear materials. According to Talma et al. (2019), the passion fruit mesocarp flour is bright and clear, which do not change with the maturation stage. In contrast, the color of epicarp flour presents lower luminosity (CIE L\* values) and it has a greener index (CIE a\*) at the early maturation stage of the fruit.

**Table 1**. Color parameters of the yellow passion fruit mesocarp flour (FAR), pectin extracted from mesocarp flour (PM) and commercial citrus pectin (PC). The data are represented by the mean values and standard deviations.

Color parameters	FAR	PM	PC
CIE L*	88.1 <sup>a</sup> ± 0.1	82.0 <sup>b</sup> ± 0.3	$75.4^{c} \pm 0.4$
CIE a*	$0.9^{c} \pm 0.1$	$1.2^{\rm b} \pm 0.1$	$2.9^{a} \pm 0.1$
CIE b*	$18.6^{a} \pm 0.2$	$16.3^{\rm b} \pm 0.5$	$16.1^{c} \pm 0.3$
C*	$18.6^{a} \pm 0.2$	$16.3^{\rm b} \pm 0.5$	$16.4^{b} \pm 0.3$
h	$87.3^{a} \pm 0.2$	$85.6^{b} \pm 0.4$	$79.7^{c} \pm 0.2$

CIE L\* - luminosity index; CIE a\* - greenness index; CIE b\* - yellowness index; C\* Chroma and h Hue angle. Values followed by the same letter on the same row do not differ significantly by Tukey's test at 5% probability.



**Figure 2**. Photographic records of yellow passion fruit mesocarp flour (A); pectin extracted from the yellow passion fruit mesocarp (B); commercial citric pectin (C). Materials with 60-Mesh sieve particle size.

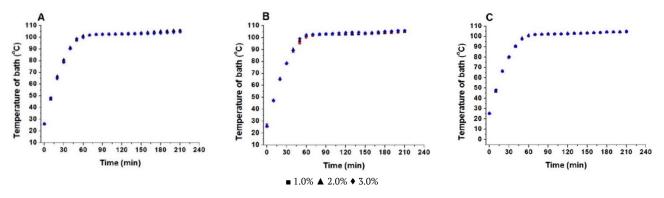
Figure 2 shows that the mesocarp flour and PM pectin presented higher luminosity, as indicated by the CIE L\* values in Table 1. The higher value of CIE b\* and Hue angle of the flour (FAR) is due to the aggregation of pulp traces in the mesocarp. The extraction of pectin from the flour drags the pigments, which may cause a smaller yellow color index (CIE b\*) in PM pectin. The mesocarp flour has lower CIE a\* value, however, values close to zero indicate neutral shade for this parameter.

The color differences in Hue angles for the two pectin (PM and PC) are due to the slightly higher value of CIE b\* for PM pectin (Table 1). The color similarity is highlighted by the Chroma parameter.

## Histories of temperatures, soluble solid content, and pH of gels

Figure 3 presents the histories of bath temperatures (Tb) measured during the elaboration of gels made with increasing content of mesocarp flour (FAR), pectin extracted from mesocarp flour (PM) and commercial citric pectin (PC).

The bath temperature profiles were similar during the elaboration of gels with different contents of materials (Figure 3). In the first one hour, the bath temperature increased steadily to 100 °C, however, after that, there was only a slight increase in temperature up to 105 °C, until the end of the gelling process.

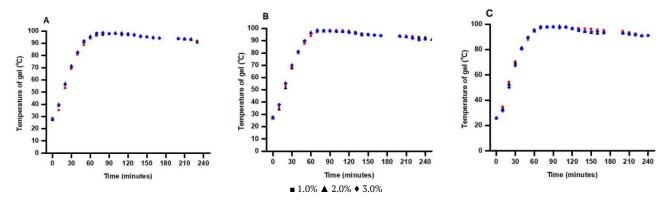


**Figure 3.** Histories of the bath temperatures (°C) over time (min) during elaboration of gels with different concentrations of mesocarp flour (A), pectin extracted from mesocarp flour (B) and commercial citric pectin (C).

Page 6 of 12 Silva et al.

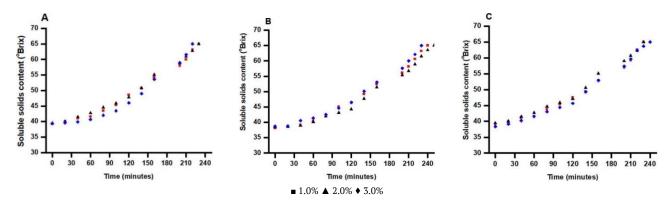
The temperature histories (Tg) of gels made with increasing content of mesocarp flour, PM pectin and PC pectin are presented in Figure 4. The temperature histories of the gels were similar for the different contents of materials (FAR, PM and PC). The gels reached 97 °C in the first one hour of processing, remaining at stable temperatures for approximately 2 hours, maintaining temperatures above 90 °C until reaching the 65 °Brix gelling endpoint.

According to Kastner, Einhorn-Stoll and Senge (2012), low temperatures favor hydrogen bonds, while hydrophobic interactions are more dominant at higher temperatures. In this study, we tried to maintain a similarity in the temperature histories, in order to standardize the heat effect on the change of physical and rheological properties of gels. The heating causes losses of sensorial and nutritional quality attributes. For these reasons, it is desirable to keep thermal treatment conditions at a least possible level to minimize quality losses (Deng et al., 2015).



**Figure 4.** Histories of the gel temperatures (°C) over time (min) during elaboration of gels with different concentrations of mesocarp flour (A), pectin extracted from mesocarp flour (B) and commercial citric pectin (C).

Figure 5 shows the histories of total soluble solids (SSg) content of gels made with different content of mesocarp flour, pectin extracted from mesocarp flour and commercial pectin.



**Figure 5**. Histories of soluble solids contents of the gel (SSg) (°Brix) over time (min) during elaboration of gels with different concentrations of mesocarp flour (A), pectin extracted from mesocarp flour (B) and commercial citric pectin (C).

The change in soluble solids along the time seems to be dependent of the gelling agent concentration, since soluble solids value is specific for each concentration of gelling material, as identified in the fitting of the models presented below. However, through data evaluation, it is noted that all the models could be accommodated inside the confidence intervals (p < 0.05) (Figure 5). A change in soluble solids would be expected with higher concentration of the gelling agent, and also with the increase in the mixture viscosity, thus affecting the heat transfer and the mass transfer coefficient of water vapor at the surface of the gel. (Welty, Wicks, Wilson, & Rorrer, 2008). However, at the stirring conditions and temperatures used in the processing of the gels, no consistent difference was found among the models that describe de profiles of soluble solids along the processing time of the gels.

Table 2 shows the pH measurements at the beginning and after the elaboration of the gels. In the beginning of the process, the differences in pH values indicate the influence of the acidity of the flour or pectin used in the preparation of gels.

**Table 2.** Values of pH at the beginning and after elaboration of gels with mesocarp flour (FAR), pectin extracted from mesocarp flour (PM) and commercial citric pectin (PC) in different concentrations.

	pH before ge	l preparation	
	1.0%	2.0%	3.0%
FAR	$4.7^{\mathrm{bA}} \pm 0.01$	$4.8^{aA} \pm 0.02$	$4.8^{aA} \pm 0.07$
PM	$3.1^{aC} \pm 0.03$	$2.8^{bC} \pm 0.09$	$2.4^{\text{cC}} \pm 0.02$
PC	$3.4^{aB} \pm 0.03$	$3.2^{\mathrm{bB}} \pm 0.02$	$3.1^{\text{cB}} \pm 0.02$
		pH after gel elaboration	
FAR	$3.0^{aA} \pm 0.02$	$3.1^{aA} \pm 0.03$	$3.1^{aA} \pm 0.02$
PM	$3.1^{aA} \pm 0.01$	$3.1^{aA} \pm 0.02$	$3.1^{aA} \pm 0.06$
PC	$3.1^{aA} \pm 0.01$	$3.0^{aA} \pm 0.04$	$3.1^{aA} \pm 0.04$

Data followed by equal lowercase letters in the same row and equal uppercase letters in the same column do not differ significantly from each other by the Tukey test at 5% probability.

The increase in the concentration of pectin in the formulations promotes a significant decrease of pH values. The pH of the pectin-containing solutions was lower than the pH of the mesocarp flour-containing formulation (FAR), and this difference is attributed to the composition of the pectin that contains high concentrations of galacturonic acid. The lowest pH values found in the solutions with PM pectin may be related to the acid used in the pectin extraction, as well as to the acid group content present in the pectin molecule.

In contrary, the pH of solutions containing mesocarp flour (FAR) increased with increasing levels of FAR, probably because of other flour components (cellulose and hemicellulose) which promote higher pH values. López-Vargas et al. (2013) and Coelho et al. (2018) found pH values of 4.36 and 4.25 in passion fruit mesocarp flour and passion fruit rind flour, respectively.

Analysis of the pH data after gel elaboration indicate similarity among all the gels, since the pH values were adjusted to 3.0 at the end of the cooking process (Table 2). According to Basu and Shivhare (2010), the gelling process is ideal when the pH is between 3.0 and 3.4.

#### Model fitting and quality of fit

The fits by pectin gel materials and levels were discriminated based on the I-T approach (Table I – Supplementary file). Therefore, the model solutions for each variable were down ranked according to the estimates of the Akaike information criterion corrected for small samples (Cavanaugh, 1997) and its derived measures, namely the Akaike differences ( $\Delta_m$ ), model probabilities ( $w_m$ ), and evidence ratios ( $ER_m$ ). The most suitable solution for Tb was Eq. (6), that is, the Gompertz-Quadratic function, fitted with the interaction effects of pectin gel material and level, a random batch factor accounting for variation in parameter  $\delta$ , as well as with the VarPower and corCAR1 functions. The most suited model to represent SSg was Eq. (2) fitted with VarPower and corCAR1 functions. Nonetheless, the fit of the model was insensitive to the gel level, and only the fixed effect of gel material was the most likely solution. The model that best suited variable Tg was the dual pool Gompertz-Linear segmented (Eq. 5), but only the corCAR1 function was fitted to reduce information loss; the VarPower function was ineffective to improve the model fit. There was no suitable solution for Tg and SSg with random factors accounted for. Since the solutions that suited better were those with  $w_m > 0.90$ , a model averaging process was not necessary (Burnham, Anderson, & Huyvaert, 2011).

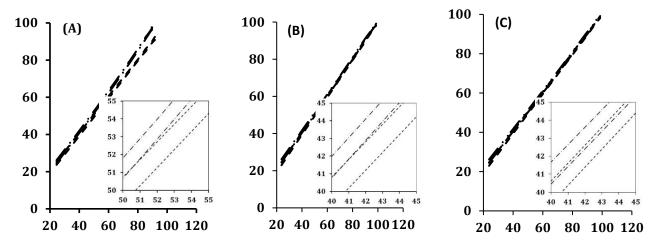
#### Graphical analysis based on confidence intervals

As showed in Table I (Supplementary material), the best fit to the bath temperature (Tb) was accomplished by the Gompertz-Quadratic function, fitted with the interaction effects of pectin gel material and level (Eq 6). Surprisingly, the interacting fixed effects model best described the bath temperature (Tb). This means that each treatment combination resulted in empirical conditions with specific parametric estimates. Nevertheless, the confidence intervals for the predicted Tb for each gel source and level overlapped (graphical results not shown). In this way, the production of gels with different materials at different concentrations was performed under the same conditions of bath temperatures (Figure 3), enabling to warrant a similar thermal treatment that allows to standardize the heat effect on the maintenance of quality characteristics of the gels.

The best fit to the temperature of the gels (Tg) was accomplished by the dual pool Gompertz-Linear segmented (Equation 5), as indicated in the Table I (Supplementary material). This model is capable to predict the gel temperatures (Tg) for the different materials and concentrations (Figure 4) with 99% of confidence.

Figure 6 presents the confidence intervals (99 % probability) of the model for prediction of the whole range of gel temperatures (Tg).

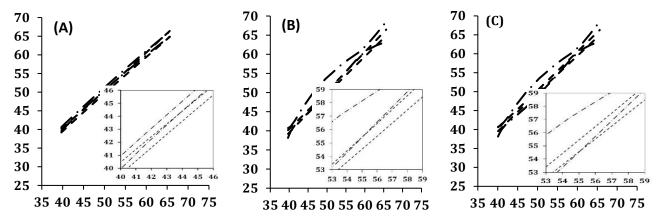
Page 8 of 12 Silva et al.



**Figure 6.** Comparison of 99% confidence intervals for predictions of gel temperature (Tg) (°C). In panel (A) are confidence intervals for predictions of FAR (dashed lines) and PC (dashed and dotted lines), panel (B) FAR (dashed lines) and PM (dashed and dotted lines), and on panel (C) PC (dashed lines) and PM (dashed and dotted lines).

When comparing the confidence intervals for predictions of Tg of FAR (dashed lines) and PC pectin (dashed and dotted lines), the intervals became disjoint for Tg at values approximately greater than 51 °C (small box in Figure 6, panel A), but it overlapped throughout the entire scale for Tg of pectin (PC), presenting only 7% overestimating of Tg for the gel of FAR, at the end of the heating process. Despite this small divergence in the confidence intervals, those differences might not interfere on changes of the quality characteristics of gels, in practice.

In the same way, when comparing the confidence intervals for predictions of Tg of FAR (dashed lines) and pectin PM (dashed and dotted lines), the intervals became disjoint for Tg at values approximately greater than 42 °C (Figure 6, panel B), but it overlapped throughout the entire scale for Tg of pectin PM, presenting only 6.6% overestimating of Tg for the gel of FAR, at the end of the heating process. However, in case of comparing the Tg of gels made with PC pectin (dashed lines) and PM pectin (dashed and dotted lines), the temperature of both gels overlapped throughout the entire scale for Tg (Figure 6, panel C), which make possible to standardize the effect of temperature Tg on the change of quality characteristics of gels.



**Figure 7.** Comparison of 99% confidence intervals for predictions of soluble solids (SSg) (°Brix). In panel (A) are confidence intervals for predictions of FAR (dashed lines) and PC (dashed and dotted lines), panel (B) FAR (dashed lines) and PM (dashed and dotted lines), and on panel (C) PC (dashed lines) and PM (dashed and dotted lines).

The gels with different pectic materials also presented models with specific parameters for predicting the SSg content, but indicating part of the confidence intervals overlapping and part disjoint. Thus, when comparing the confidence intervals for predictions of SSg of FAR (dashed lines) and PC pectin (dashed and dotted lines), the intervals became disjoint for SSg at values approximately greater than 44 °Brix (small box in Figure 7, panel A), but it overlapped throughout the entire scale for SSg of pectin (PC), presenting only 1,4% overestimating of SSg for the gel of FAR, at the end of the heating process. Despite this small divergence in the confidence intervals, those differences might not interfere on changes of the quality characteristics of gels, in practice.

In the same way, when comparing the confidence intervals for predictions of SSg of FAR (dashed lines) and PM pectin (dashed and dotted lines), the intervals became disjoint for SSg at values approximately greater than 55 °Brix (Figure 7, panel B), but it overlapped throughout the entire scale for Tg of pectin PM, presenting only 1.7% overestimating of SSg for the gel of FAR, at the end of the heating process. In case of PC and PM pectin gels, the confidence intervals overlapped throughout the entire scale of SSg (Figure 7, panel C).

Despite the need of specific parameters for each model, there is an intersection in the variability of the models which is intrinsic of the process, thus, it generates the same results for whole range of SSg and Tg, which means that the gels with different material concentrations were processed under standard conditions during the heating time.

### Color of the gels

Table 3 shows the color parameters CIE L\* a\* b\*, Chroma (C\*) and Hue angle (h) of gels made with different concentrations of mesocarp flour (FAR), pectin extracted from mesocarp flour (PM) and commercial citric pectin (PC). The increase in the concentration of each pectic material promotes an increase of all color parameters of gels. The more translucent gels have the lowest values of brightness and intensity of the color parameters, because the color measurements are based on the reflectance of the light passing throughout the gel from the bottom of the black opaque glass. Thus, the gels made with mesocarp flour and PM pectin showed the higher values of color parameters, being less translucent and more yellowish than gels made with PC pectin (Figure 8, 9 and 10). It must be emphasized that all gels were processed under the same standard conditions of heating, so, the differences in color of gels are intrinsic of the pectic materials.

The sugar caramelization during the heating process makes the gel more yellowish. This is enhanced by the higher concentration of pectin and mesocarp flour that makes the solution more viscous, which impacts on light reflectance index and color parameters.

The incorporation of fiber-rich products into food systems is one of the industry challenges in face of the changes in product color and texture (López-Vargas et al., 2013). The mesocarp flour of yellow passion fruit has been proposed as a thickening, stabilizing and gelling agent, and also as a functional ingredient in foods (Bezerra et al., 2019). It is a good source of high methoxyl pectin, that provides an alternative application as a rheological modifier in a wide range of products (Abboud, Yacomini, Simas, & Cordeiro, 2020). In an *in vitro* toxicity analysis of the polysaccharide fraction of passion fruit peel, it was not identified cytotoxic effect on epithelial cell culture of mice (Sabino et al., 2020).

**Table 3.** Color parameters, CIE L\* a\* b\*, Chroma (C\*) and Hue angle (h), of gels made with different concentrations of mesocarp flour (FAR), pectin extracted from mesocarp flour (PM) and commercial citric pectin (PC)

Color	Gelling material			
parameters	FAR	PM	PC	
		Concentration (1.0%)		
CIE L*	$19.5^{aC} \pm 0.3$	$19.4^{aC} \pm 0.7$	$7.1^{bC} \pm 0.5$	
CIE a*	$-0.9^{cA} \pm 0.1$	$-1.5^{aA} \pm 0.1$	$-1.4^{bB} \pm 0.1$	
CIE b*	$6.0^{aC} \pm 0.7$	$5.3^{bC} \pm 0.8$	$1.0^{cC} \pm 0.2$	
C*	$6.1^{aC} \pm 0.7$	$5.5^{bC} \pm 0.8$	$1.8^{cC} \pm 0.1$	
h	$-81.0^{aC} \pm 1.0$	$-73.2^{bC} \pm 3.1$	$-36.0^{cC} \pm 4.0$	
	Concentration (2.0%)			
CIE L*	$20.9^{\mathrm{bB}} \pm 0.6$	$23.3^{aB} \pm 0.5$	$10.7^{\text{cB}} \pm 0.2$	
CIE a*	$-0.6^{\text{cB}} \pm 0.3$	$-1.5^{aA} \pm 0.2$	$-1.4^{bB} \pm 0.1$	
CIE b*	$12.7^{aB} \pm 0.7$	$8.3^{\mathrm{bB}} \pm 0.5$	$1.9^{cB} \pm 0.2$	
C*	$12.7^{aB} \pm 0.7$	$8.4^{\mathrm{bB}} \pm 0.4$	$2.4^{cB} \pm 0.1$	
h	$-87.0^{aB} \pm 1.0$	$-79.0^{\mathrm{bB}} \pm 1.0$	$-53.0^{cB} \pm 3.0$	
	Concentration (3.0%)			
CIE L*	$22.9^{bA} \pm 0.2$	29.9 <sup>aA</sup> ± 0.5	$13.2^{cA} \pm 0.5$	
CIE a*	$-0.3^{bC} \pm 0.1$	$-0.3^{\text{cB}} \pm 0.1$	$-1.7^{aA}\pm0.2$	
CIE b*	$16.1^{aA} \pm 0.5$	$15.5^{\text{bA}} \pm 0.3$	$3.3^{cA} \pm 0.8$	
C*	$16.1^{aA} \pm 0.5$	$15.5^{\text{bA}} \pm 0.3$	$3.7^{cA} \pm 0.7$	
h	$-88.8^{aA} \pm 0.3$	$-89.1^{aA} \pm 0.2$	$-62.0^{bA} \pm 6.0$	

The values of each parameter at different concentrations followed by equal lowercase letters in the same row and equal uppercase letters in the same column do not differ significantly from each other by the Tukey test at 5% probability.

Page 10 of 12 Silva et al.

High methoxyl pectin usually form thermo-irreversible gels, since the heating and cooling process may strongly affect the intra- and intermolecular interactions that are important to maintain the gel network. The thermal treatment that is usually applied in the pasteurization process of gel products does not change the structure of the gel (Abboud, Yacomini, Simas, & Cordeiro, 2020). The polysaccharide fraction of the yellow passion fruit peel is mainly composed of pectin with arabinose residues. This polysaccharide presents high molecular weight with heterogeneous molecules with different molecular weights. Under thermogravimetric analysis (TGA), this polysaccharide exhibits high thermal stability with the first decomposition stage starting at 200 °C (Sabino et al., 2020).

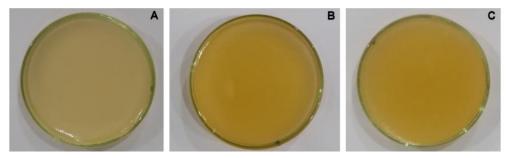


Figure 8. Photographic record of gels made with passion fruit mesocarp flour (FAR) in three concentrations: 1% (A), 2% (B) and 3% (C).

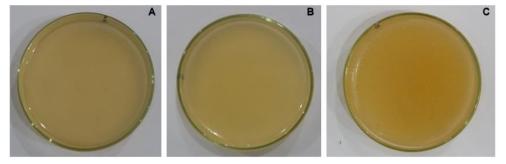


Figure 9. Photographic record of gels made with pectin extracted from mesocarp flour (PM) in three concentrations: 1% (A), 2% (B) and 3% (C).

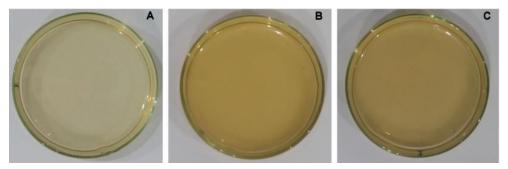


Figure 10. Photographic record of gels made with commercial citric pectin (PC) in three concentrations: 1% (A), 2% (B) and 3% (C).

#### Conclusion

The quantitative analysis of data for temperature and content of soluble solids along the processing time confirmed the standardization of parameters used in the production of the gels. In this way, the pectic materials can be evaluated according to its intrinsic properties, without interference of the processing conditions of gels.

Mesocarp flour (FAR) and PM pectin powder present more clear shade colors than PC pectin. However, the gels made with mesocarp flour (FAR) and PM pectin have less translucent shades and a more yellowish color than gels produced with PC pectin. However, these small differences may not interfere in the color of fruit jelly.

As far as we know, this is the first study that has standardized the processing conditions of gel-made products, ensuring the same heat effect on the quality characteristics of gels.

## Acknowledgements

The authors are grateful to the Fundação Carlos Chagas de Amparo à Pesquisa do Estado do Rio de Janeiro – Faperj; Conselho Nacional de Desenvolvimento Científico e Tecnológico – CNPq; and to the Instituto Federal Fluminense (Bom Jesus do Itabapoana, RJ/Brazil) for supplying the raw material.

## References

- Abboud, K. Y, Yacomini, M., Simas, F. F., & Cordeiro, L. M. C. (2020). High methoxyl pectin from the soluble dietary fiber of passion fruit peel forms weak gel without the requirement of sugar addition. *Carbohydrate Polymers*, *246*, 116616. DOI: https://doi.org/10.1016/j.carbpol.2020.116616.
- Association of Official Analytical Chemists [AOAC]. (1995). *Methods of analysis of the Association of Official Analytical Chemistry*. Arlington, VA: AOAC International.
- Basu, S., & Shivhare, U.S. (2010). Rheological, textural, micro-structural and sensory properties of mango jam. *Journal of Food Engineering*, 100(2), 357-365. DOI: https://doi.org/10.1016/j.jfoodeng.2010.04.022.
- Bezerra, P. Q. M., Matos, M. R. F., Ramos, I. G., Magalhães-Guedes, K. T., Druzian, J. I., Costa, J. A. V., & Nunes, I. L. (2019). Innovative functional nano dispersion: Combination of carotenoid from *Spirulina* and yellow passion fruit albedo. *Food Chemistry*, *285*, 397-405. DOI: https://doi.org/10.1016/j.foodchem.2019.01.181.
- Burnham, K.P., Anderson, D.R., & Huyvaert, K.P. (2011). AIC model selection and multimodel inference in behavioral ecology: some background, observations, and comparisons. *Behavioral Ecology and Sociobiology*, *65*(1), 23-35. DOI: https://doi.org/10.1007/s00265-010-1029-6.
- Cavanaugh, J. E. (1997). Unifying the derivations for the Akaike and corrected Akaike information criteria. *Statistics & Probability Letters*, *33*(2), 201-208. DOI: https://doi.org/10.1016/S0167-7152(96)00128-9
- Coelho, E. M., Azevêdo, L. C., Viana, A. C., Ramos, I. G., Gomes, R. G., Lima, M. S., & Umsza-Guez, M. A. (2018). Physico-chemical properties, rheology, and degree of esterification of passion fruit (*Passiflora edulis* f. Flavicarpa) peel flour. *Journal of the Science of Food and Agriculture*, *98*(1), 166-173. DOI: https://doi.org/10.1002/jsfa.8451
- Deng, Q., Zinoviadou, K. G., Galanakis, C. M., Orlien, V., Grimi, N., Vorobiev, E., ... Barba, F. J. (2015). The Effects of Conventional and Non-conventional Processing on Glucosinolates and Its Derived Forms, Isothiocyanates: Extraction, Degradation, and Applications. *Food Engineering Reviews*, 7(3), 357-381. DOI: https://doi.org/10.1007/s12393-014-9104-9
- Fertonani, H. C. R., Scabio A., Carneiro E. B. B., Canteri-Schemin M. H., Nogueira, A., & Wosiacki G. (2009). Extraction model of low methoxyl pectin from apple pomace effects of acid concentration and time on the process and the product. *Brazilian Archives of Biology and Technology, 52*(1), 177-185. DOI: https://doi.org/10.1590/S1516-89132009000100023
- Galanakis, C. M. (2020) The Food Systems in the Era of Coronavirus (COVID-19) Pandemic Crisis. *Foods*, *9*(4), 523. DOI: https://doi:10.3390/foods9040523.
- Instituto Brasileiro de Geografia e Estatística [IBGE]. (2022). B*anco de dados agregados. Produção Agrícola em 2020.* Retrieved on January, 2022 from https://cidades.ibge.gov.br/brasil/pesquisa/15/0
- Kar, F., & Arslan, N. (1999). Effect of temperature and concentration on viscosity of orange peel pectin solutions and intrinsic viscosity–molecular weight relationship. *Carbohydrate Polymers*, *40*(4), 277-284. DOI: https://doi.org/10.1016/S0144-8617(99)00062-4.
- Kastner, H., Einhorn-Stoll, U., & Senge, B. (2012). Structure formation in sugar containing pectin gels Influence of Ca<sup>2+</sup> on the gelation of low-methoxylated pectin at acidic pH. *Food Hydrocolloids*, *27*(1): 42-49. DOI: https://doi.org/10.1016/j.foodhyd.2011.09.001.
- Lara-Espinoza, C., Carvajal-Millán, E., Balandrán-Quintana, R., López-Franco, Y., & Rascón-Chu, A. (2018). Pectin and Pectin-Based Composite Materials: Beyond Food Texture. *Molecules*, 23(4), 942. DOI: https://doi.org/10.3390/molecules23040942.
- Linares-García, J. A., Ramos-Ramírez, E. G., & Salazar-Montoya, J. A. (2015). Viscoelastic properties and textural characterization of high methoxyl pectin of hawthorn (*Crataegus pubescens*) in a gelling system. *International Journal of Food Science & Technology, 50*(6), 1484-1493. DOI: https://doi.org/10.1111/ijfs.12792

Page 12 of 12 Silva et al.

López-Vargas, J. H., Fernández-López, J., Pérez-Álvarez, J. A, & Viuda-Martos, M. (2013). Chemical, physico-chemical, technological, antibacterial and antioxidant properties of dietary fiber powder obtained from yellow passion fruit (*Passiflora edulis* var. Flavicarpa) co-products. *Food Research International*, *51*(2), 756-763. DOI: https://doi.org/10.1016/j.foodres.2013.01.055

- Oliveira, C. F., Giordani, D., Gurak, P. D., Cladera-Olivera, F., & Marczak, L. D. F. (2015). Extraction of pectin from passion fruit peel using moderate electric field and conventional heating extraction methods. *Innovative Food Science & Emerging Technologies*, *29*, 201-208. DOI: https://doi.org/10.1016/j.ifset.2015.02.005.
- Oliveira, E. M. S., & Resende, E. D. (2012). Yield of albedo flour and pectin content in the rind of yellow passion fruit. *Food Science and Technology, 32*(3), 492-499. DOI: https://doi.org/10.1590/S0101-20612012005000067
- Pinheiro, J. C., & Bates, D. M. (2000). Mixed-effects models in S and S-PLUS. Springer New York, NY: USA.
- Talma, S. V., Regis, S. A., Ferreira, P. R., Mellinger-Silva, C., & Resende, E. D. (2019) Characterization of pericarp fractions of yellow passion fruit: density, yield of flour, color, pectin content and degree of esterification. *Food Science and Technology*, *39*, 683-689. DOI: https://doi.org/10.1590/fst.30818
- Torralbo, D. F., Batista, K. A., Di-Medeiros, M. C. B., & Fernandes, K. F. (2012). Extraction and partial characterization of *Solanum lycocarpum* pectin. *Food Hydrocolloids*, *27*(2), 378-383. DOI: https://doi.org/10.1016/j.foodhyd.2011.10.012
- Sabino, L. B. S., Gonzaga, M. L. C., Oliveira, L. S., Duarte, A. S. G., Silva, L. M. A., Brito, E. S., ... Sousa, P. H. M. (2020). Polysaccharides from acerola, cashew apple, pineapple, mango and passion fruit co-products: Structure, cytotoxicity and gastroprotective effects. *Bioactive Carbohydrates and Dietary Fibre, 24*. DOI: https://doi.org/10.1016/j.bcdf.2020.100228.
- Silva, E. C. O., Silva, W. P., Silva, E. T., Lopes, J. D., & Gusmão, R. P. (2016). Obtenção e caracterização da farinha do albedo de maracujá (Passiflora edulis f. Flavicarpa) para uso alimentício. *Revista Verde de Agroecologia e Desenvolvimento Sustentável, 11*(3), 69-74. DOI: https://doi.org/10.18378/rvads.v11i3.4062
- Vieira, R. A. M., Rohem Júnior, N. M., Gomes, R. S., Oliveira, T. S., Bendia, L. C. R., Azevedo, F. H. V., ... Rodrigues, M. T. (2018). The ontogenetic allometry of body morphology and chemical composition in dairy goat wethers. *Animal*, *12*(3), 538-553. DOI: https://doi.org/10.1017/s1751731117001884
- Vieira, R. A. M., Rohem Júnior, N. M. Abreu, M. L. C., Silva, M. C., Oliveira, J. G., Tedeschi, L. O., & Gloria, L. S. (2020). The transit of external markers throughout the ruminant digestive tract: 2. The estimation of fiber digestibility, ruminoreticular fill, and related biases. *Animal Feed Science and Technology, 261*, 114420. DOI: https://doi.org/10.1016/j.anifeedsci.2020.114420.
- Vriesmann, L. C., Teófilo, R. F., & Petkowicz, C. L. O. (2012). Extraction and characterization of pectin from cacao pod husks (*Theobroma cacao* L.) with citric acid. *LWT Food Science and Technology, 49*(1), 108-116. DOI: https://doi.org/10.1016/j.lwt.2012.04.018
- Welty, J. R., Wicks, C. E., Wilson, R. E., & Rorrer, G. L. (2008). *Fundamentals of Momentum, Heat and Mass transfer* (5th ed.). Chichester, EN: John Wiley & Sons.