



# Effect of formulation variables on rheology, texture, colour, and acceptability of apple jelly: Modelling and optimization



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## ARTICLE INFO

### Article history:

Received 18 June 2013

Received in revised form

2 July 2014

Accepted 8 July 2014

Available online 17 July 2014

### Keywords:

Fruit jams

Composition

Gelification

Structure

Sensory evaluation

## ABSTRACT

The objective was to study and model the effect of the main formulation variables on the rheological and mechanical properties, colour and overall acceptability of apple jelly, and to optimize formulation variables in order to maximize overall acceptability. Formulation variables were juice proportion in the initial juice-sugar mix ( $J$ : 350–550 g/kg), product pH (2.8–3.6), concentration of added pectin ( $P$ : 0–10 g/kg), and final content of soluble solids ( $SS$ : 625–725 g/kg). Anova results showed that  $P$  was the main effect on all the rheological and mechanical properties. The strength of the pectin gel network increased at increasing values of  $P$ . Consequently, the jellies were more elastic and firm but more brittle, as well as more adhesive. Also, more work was required to disintegrate the jellies. Besides  $P$ , also  $J$  had a significant positive effect on storage modulus and adhesiveness, while  $SS$  had a significant positive effect on cohesiveness. Colour parameters were mainly affected by  $J$ . Overall acceptability was significantly affected by  $J > pH > SS$ , while  $P$  had no significant effect. The optimum was calculated to be at  $J = 500$  g/kg,  $SS = 700$  g/kg,  $pH = 3.4$ , and  $P = 5$  g/kg.

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## 1. Introduction

Cooking jams, jellies and marmalades from fruits, sugar, pectin and edible acids is one of the oldest food preserving processes known to mankind, allowing fruit consumption in the off-season (Baker, Berry, & Hui, 1996). In this case food stabilization is achieved –besides the thermal treatment– by increasing the soluble solids content (reducing water activity), and increasing the acidity (reducing the pH). Also, these two parameters (soluble solids and pH) are of paramount importance for the texture, structure, and overall quality of fruit jams, since proper gelation of high methoxyl (HM) pectins is only achieved in narrow ranges of pH (2.8–3.5), and sugar content (~600–800 g/kg).

Industrial manufacture of fruit jams requires constant gel strength during production. Consequently, commercial pectin is added (0 to ~10 g/kg of the final product) to minimize the effect of the variability of fruit native pectin. Minimum soluble solids content required for the product depends on the legislation of each country, being 650 g/kg a typical limit (like in Argentine Food

Code). Maximum concentration allowed of added gelling agent also depends on the country, being 5 g/kg (without declaration in the label) in Argentina.

The main difference between jams, jellies and marmalades is the form in which their fruit component is incorporated, namely fruit juice, fruit pulp, pieces of fruit or whole fruit. Jellies are made from strained fruit juice. The minimum proportion of juice in the product also depends on the legislation of each country. In Argentina the legislation states that jellies should be manufactured by heat concentration of no less than 35 parts of filtered fruit juice, with a sweetener. In other words, the minimum ratio of ingredients to be cooked is 35 parts of juice in 65 parts of sugar and others ( $J:S \geq 35:65$  g/g).

Besides determining general physical and chemical properties, many works have also performed sensory analysis of jams (Abdullah & Cheng, 2001; Basu, Shivhare, Singh, & Beniwal, 2011; Grigelmo-Miguel & Martín-Belloso, 1999; Singh, Jain, Singh, & Singh, 2009; Suutarinen et al., 2002), jellies (Acosta, Viquez, & Cubero, 2008; Khouryieh, Aramouni, & Herald, 2005; Moritaka, Naito, Nishinari, Ishihara, & Fukuba, 1999; Royer, Madieta, Symoneaux, & Jourjon, 2006), and marmalades (Egbekun, Nda-Suleiman, & Akinyeye, 1998; Yildiz & Alpaslan, 2012). Some of these studies and others have instrumentally measured the colour of jams (Dervisi, Lamb, & Zabetakis, 2001; Grigelmo-Miguel & Martín-Belloso, 1999; Singh et al., 2009; Suutarinen et al., 2002)

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and marmalades (Yildiz & Alpaslan, 2012). Several works have also studied the mechanical properties (texture) of jams (Basu et al., 2011; Singh et al., 2009; Suutarinen et al., 2002), and jellies (Khouryieh et al., 2005; Moritaka et al., 1999; Royer et al., 2006). A few studies have also determined the flow behaviour of these products (Costell, Carbonell, & Duran, 1993; Griguelmo-Miguel & Martín-Belloso, 1999; Yildiz & Alpaslan, 2012). The only justification of this type of destructive measurement in a structured product is the possibility to establish a quality control method for fruit derived products, based on the estimation of formulation/fruit content of the product from its viscometric properties (Fugel, Carle, & Schieber, 2005).

Remarkably, we just found a couple of works which studied the viscoelastic properties of jams (Basu et al., 2011; Dervisi et al., 2001). On the other hand, pectin gels have been much studied since the 60's and 70's (Barwal & Kalia, 1997; Doesburg & Grevers, 1960; Hinton, 1940; Smit & Bryant, 1968; Walter & Sherman, 1986). There are several studies about the effect of pectin concentration, pH, and type and concentration of cosolute, on the viscoelastic properties of HM pectin gels (e.g., Evageliou, Richardson, & Morris, 2000; Löfgren, Guillotin, Evenbratt, Schols, & Hermansson, 2005; Lopez da Silva, Gonçalves, & Rao, 1995; Tsoga, Richardson, & Morris, 2004). This may be attributed to the more complex structure and composition of jams, jellies and marmalades, compared to model pectin gels (pectin + sugar + acid + water). Genovese, Ye, and Singh (2010) tried to model the former systems by adding fruit particles to HM pectin gels, and studying the effect of particle size and concentration on the rheological and mechanical properties of these composite gels. However and as far as we know, the effect of composition on the viscoelastic properties of fruit jellies has not been studied yet.

The first objective of this work was to study the effect of formulation variables (juice proportion, added pectin concentration, and final soluble solids content), on the colour, rheological and mechanical properties, and overall acceptability of apple jellies. The last objective was to optimize the formulation based on acceptability results, and try to correlate this optimum with the physical properties of the jelly.

## 2. Materials and methods

### 2.1. Materials

Apples (*cv. Granny Smith*) were bought in a local market and stored at 5 °C during 48 h prior to juice extraction. High methoxyl pectin Genu Pectin Type 121 Slow Set (Cp Kelco, Brasil) was donated by Cp Kelco Argentina. Anhydrous citric acid Parafarm (Saporiti, Argentina) was used to regulate the pH. Food grade sucrose and potable bottled water, each from the same batch, were bought in the local market.

### 2.2. Juice extraction process

Only one batch of diluted apple juice was obtained and used to prepare the different jelly samples. To obtain this juice, apples were milled and the pulp mixed with water (1:1 w/w), blanched, press-filtered, and centrifuged. The diluted juice was bottled and frozen until use.

### 2.3. Jelly cook-concentration process

Required amounts of juice and sucrose were mixed in an open pan, heated up to the boiling point, and concentrated by evaporation until the desired concentration of soluble solids, which was monitored with a digital refractometer. The required amount of

pectin was separately dissolved in water with part of the sugar, and allowed to hydrate under agitation during 24 h before addition. In order to minimize pectin hydrolysis, this pectin solution was added to the juice-sugar mix towards the end of the concentration process. The desired pH was adjusted by adding a saturated solution of citric acid. To avoid pre-gelation, this solution was added just before the end of the process, and the pH was monitored with a digital pH meter (Altronix TPX II, Buenos Aires, Argentina) equipped with a high-temperature-resistant electrode (Broadley F-600, Irvine, USA). Finally, each sample was hot-filled at about 100 °C into three sanitized and labelled glass jars, which were sealed with their screw tops and stored for later measurements.

### 2.4. Rheological measurements

Viscoelastic properties of the jellies were determined by small deformation dynamic oscillatory measurements in a Paar Physica rheometer model MCR301 (Anton Paar GmbH, Austria), using a geometry of cone and plate (50 mm diameter, 1° cone angle), with peltier temperature control. Immediately after cook-concentration, an aliquot of the hot jelly was poured on the rheometer's lower plate, previously conditioned at 90 °C. The cone was lowered to the sample, excess sample was removed, and the exposed surface was covered with silicon oil to avoid sample dehydration during measurement. After thermal equilibrium was achieved, the measurement was initiated. Each measurement consisted in three successive steps, performed at an amplitude strain of 0.5%, namely: 1) Gelation: temperature ramp from 90 to 20 °C, at a cooling rate of 1 °C/min and a frequency of 1 rad/s; 2) Curing: time sweep of 180 min, at 20 °C and a frequency of 1 rad/s; and 3) Mechanical spectra: frequency sweep from 0.1 to 100 rad/s at 20 °C. Data obtained in each step were elastic modulus ( $G'$ ), viscous modulus ( $G''$ ), and derived parameters. Immediately after each measurement, a strain amplitude sweep (from 0.01 to 100%, at 20 °C and 1 rad/s) was performed to verify that the measurement was within the linear viscoelastic range (LVR).

### 2.5. Texture analysis

Mechanical properties of the jelly samples were obtained from a texture profile analysis (TPA) test using a TA-Plus texture analyzer (Lloyds Instruments, UK). Two containers (55 mm diameter) of each sample were stored for 1 month at room temperature, and conditioned at 20 °C in a controlled chamber during the last 48 h before measurement. The TPA test consists of two cycles of compression. In each cycle, the sample in each container was penetrated 20 mm by a cylinder probe (2.54 mm diameter), at a crosshead speed of 2 mm/s, and the probe was withdrawn from the sample at the same speed. Time and force exerted by the probe were measured during each test. Test settings followed a test procedure for marmalades (Genovese et al., 2010). Each sample was measured twice, once in each container.

From each force time curve of the TPA test a number of textural parameters can be extracted (Bourne, 2002). Hardness was obtained as the maximum peak force during the first compression cycle ( $H = f_{\max}$ ). Fracturability was obtained as the force at the first significant break in the first compression cycle ( $F = f_{\text{break}}$ ). Adhesiveness was calculated as the negative area under the force curve after the first compression cycle ( $A = a_3$ ). Cohesiveness was calculated as the ratio of the positive force area during the second compression cycle to that during the first compression ( $C = a_2/a_1$ ). Springiness was calculated as the ratio of the time elapsed during positive forces at the second compression, to that of the first compression ( $S = t_2/t_1$ ). And gumminess was calculated as the product of hardness  $\times$  cohesiveness ( $G = H.C$ ).

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