

Rheological behavior of mixed nectars of pineapple skin juice and tropical fruit pulp

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Abstract

This study aimed to evaluate the rheological behavior at different temperatures of mixed nectars containing pineapple skin juice and fruit pulp (cupuaçu, taperebá, and guava), which then underwent microbiological and physicochemical analyses according to the current legislation. The rheological data were obtained using a rotational viscometer with concentric cylinder geometry. The formulations had low pH at 3.44 to 3.85, acidity between 0.71 and 0.87 (% citric acid), °Brix/acidity ratio ranging from 12.60 to 15.41, vitamin C content between 1.23 and 7.56 mg/100 g, and fiber content between 0.46 and 0.68%. The microbiological analyses showed no coliforms at 35°C or Salmonella sp. The mixed nectars showed a non-Newtonian behavior at the temperature range studied. The Power Law model fitted the experimental data and was shown to be appropriate to describe the behavior of the mixed nectars according to the statistical fitting parameters assessed (R², x², and RSS). The products showed behavior indices below 1, which characterizes the pseudoplasticity of the beverages. Formulation F1 had the highest consistency index (K), however, the index of F2 and F3 decreased as temperature increased. Temperature impacted the nectars' shear stress and apparent viscosity with the lowest values being observed at 60°C. The effect of temperature on the rheological behavior of the nectar was described by an equation analogous to Arrhenius equation and discussed in terms of activation energy (Ea). Ea values were 3.92 (F1), 4.61 (F2), and 5.19 kcal.g.mol⁻¹(F3), which match the data in the literature for juices and nectars.

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Keywords

Mixed nectar
Viscosity
Tropical fruits
X² Residue

Introduction

Fruits are sources of several vitamins and minerals and contribute to an appropriate diet (Françoso *et al.*, 2008). However, during the processing of juices, frozen pulps, nectars, and jams, among other products, the nutritious substances found in the skins, seeds, and bagasse are not used. Given the constant expansion in industrialization, agro-industrial waste generation considerably increases. Preparing products with pineapple generates 78% of residues (skin and crown) which are rich in fibers and nutrients that can be employed in human diets. The great challenge industries face is finding viable ways of using these residues so they yield financial gains to the industry and minimize the impact on the environment (Botelho *et al.*, 2002).

Increasingly demanding consumers have driven the development of new products, thus contributing to the betterment of agro-industrial product processes and quality and leading to the adaptation of systems

and equipment (Granjeiro *et al.*, 2007). Mixed fruit nectars can be formulated aiming to improve the nutritional and sensory characteristics of certain products by complementing the nutrients provided by fruits or industrialization residues, which enables the creation of new products from the combination of different aromas and flavors (Mattietto *et al.*, 2007).

Hence, the fruit juice industries have been concerned about improving and automating the preparation of such products. During the processing of fruit juices as well of other fruit-derived processing (nectars, sweets, ice cream), the products are exposed to several types of tensions caused by the flow through pipes, pumps, heat exchangers, mixers, filters, and other such processing equipment. Knowing the rheological parameters of fruit nectars is crucial for the proper equipment sizing, operation, and control of the production process (Cabral *et al.*, 2007).

The effect of temperature on the rheological behavior must be known to understand and size unit operations such as thermal treatment and

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concentration (Ferreira *et al.*, 2002). The rheological behavior of mixed fruit nectars can be described by several empirical models that relate shear stress and strain rate.

Apparent viscosity is impacted by countless factors such as the amount of soluble and insoluble solids, the size and shape of particles, and the process variables (temperature, pressure, etc.). According to their viscous rheological behavior, fluids may be classified as Newtonian and non-Newtonian. In Newtonian fluids, viscosity remains constant as the strain rate changes and is impacted only by temperature and pressure. In non-Newtonian fluids, viscosity changes with the strain rate and may or may not depend on the shear time (Ferreira *et al.*, 2005).

Few researches in Brazil have been developed on the rheological behavior of mixed tropical fruit nectars, particularly with the addition of fiber-rich fruit residues. Given the lack of rheological data of mixed fruit nectars, the present research aimed to evaluate the rheological behavior, at different temperatures, of three mixed nectar formulations prepared with pineapple skin juice and cupuaçu, taperebá, and guava pulps.

Material and Methods

Mixed nectar preparation

Residues from the depulping of pineapple (*Ananas comosus* L. Merril) and pulps of cupuaçu (*Theobromagrandiflorum*), taperebá (*Spondias mombin* L.), and guava (*Psidium guajava*) were used. The material was provided by the Cooperative of Fruit Producers of Abaetetuba (COFRUTA) in the city of Abaetetuba, PA, Brazil.

The pineapple skins were heated in stainless steel pans (90°C for 10 s) and then chopped, ground in a blender (Philips Walita) for 80 s, and the resulting material was screened with filtered water at a ratio of 300 mL/L ground skin. The resulting juice was bottled and refrigerated at 5°C.

Nine nectar formulations were prepared with the different juice/pulp proportions (30/70, 50/50, and 70/30) combining pineapple juice/cupuaçu pulp, pineapple juice/taperebá pulp, and pineapple/guava pulp based on the work by Arruda *et al.*, (2006). These formulations underwent preliminary sensory tests to choose the ones with the highest mean acceptance and/or highest pineapple skin juice concentration aiming at the lowest cost while using pineapple depulping residue. The acceptance test employed 30 untrained judges, who used a nine-point structured hedonic scale (Stone and Sidel, 1993).

Nectar was prepared by mixing pineapple skin

juice with the fruit pulps at the pre-defined proportions and adding water at the same amount (1:1) and saccharose until 11°Brix soluble solids content was reached (MAPA, 2014). The formulations were pasteurized in stainless steel pans at 85°C for 60 s, cooled under stirring down to 65°C, and stored under refrigeration (5°C) in clear 1,000 mL polyethylene terephthalate (PET) containers.

Physicochemical characterization of the mixed nectars

The following analyses were performed: soluble solids content (°Brix) using a digital refractometer (INSTRUTHERM, model RTD-95), pH through direct reading in a pH meter (M. S. TECNOPON, model mPA - 210), and titratable acidity expressed in % of citric acid (AOAC, 1997). The ascorbic acid content was determined through titration based on the reduction of indicator 2,6-dichlorophenolindophenol by ascorbic acid, expressed in mg vitamin C/100 g sample (Pearson, 1976). The total fiber content was determined through the acid detergent method according to Goering and Van Soest (1970). All physicochemical parameters were analyzed statistically using analysis of variance (ANOVA) and Tukey's test to compare the means at a 5% significance level using the statistical software Assistat (version 7.6 beta).

Rheological measures

Viscosity was determined according to Vidal, Pelegri and Gasparetto (2004) using a concentric cylinder viscometer (Brookfield Engineering Laboratories, model LVDV-II, USA) with a DIN-85, spindle that measures the strain rate ($\dot{\gamma}$) between 0 and 25 s⁻¹. The measurements were taken at 10, 20, 30, 40, 50, and 60°C, controlled using a thermostatic bath (LAUDA RE206), which include the final product's storage and pasteurization temperatures (Ferreira *et al.*, 2008). The data of viscosity, shear stress (T), and strain rate ($\dot{\gamma}$) were obtained using the software Win Gather®. The spindle's rotational velocities were between 12 and 180 rpm while the strain rate was between 15.5 and 232.0 s⁻¹. All samples were measured in triplicate and the shear stress values relative to the strain rates applied were obtained, in a range from 15.5 to 232.0 s⁻¹. Each repetition used a new sample to prevent possible effects of time. The final value of the parameters was the average of three readings.

In order to correlate the shear stress and strain rate data, the empirical model by Ostwald-de-Waele (Power Law) (Equation 1) was employed with the aid of the software ORIGIN 8.0.

$$T=Ky^n \quad (1)$$

Where: T = shear stress (Pa); y = strain rate (s^{-1}); K = Power Law consistency index ($Pa \cdot s^n$); and n = behavior index (non-dimensional).

The Power Law (PL) model's fit was analyzed based on the correlation coefficient R^2 , defined as the ratio between the total quadratic sum (Equation 2), on chi-squared (χ^2), which expresses the difference between the calculated values and those obtained experimentally (Equation 3), and on the residual sum of squares (RSS), which identifies the error in the model's fit (Equation 4).

$$R^2 = \frac{\sum(y_{pred} - \bar{y})^2}{\sum(y_{avg} - \bar{y})^2} \quad (2)$$

$$\chi^2 = \sum_{i=1}^n \left[\frac{(y_{avg} - y_{pred})}{y_{pred}} \right]^2 \quad (3)$$

$$RSS = \sum_{i=1}^n (y_{avg} - y_{pred})^2 \quad (4)$$

Where: y_{pred} = the value predicted by the model, y_{avg} = the experimental value, and \bar{y} = the sampling average.

The effect of temperature on apparent viscosity was described by an equation analogous to Arrhenius equation (Equation 5), which indicates the apparent viscosity's downward trend as temperature increases. The higher the activation energy, the greater the effect of temperature on viscosity (Silva *et al.*, 2005).

$$na = n_0 \exp\left(\frac{E_a}{RT}\right) \quad (5)$$

Where: y_{pred} = apparent viscosity ($Pa \cdot s$); n_0 = pre-exponential factor ($Pa \cdot s$); R = universal gas constant ($1.987 \times 10^{-3} \text{ kcal} \cdot \text{gmol}^{-1} \cdot \text{K}^{-1}$); E_a = activation energy for viscous flow ($\text{kcal} \cdot \text{gmol}^{-1}$); and T = absolute temperature (K).

The activation energy value was determined from the variation in apparent viscosity with temperature by fitting Equation 5 using linear regression. Apparent viscosity, as a function of the PL model, was calculated from the data of consistency index (K), fluid behavior index (n), and shear rate (Equation 6), which, rearranged with Equation 1, yielded Equation 7.

$$na = \frac{\tau}{\gamma}$$

$$na = K(\gamma)^{(n-1)}$$

Microbiological analyses

Coliforms were counted at 35°C and presence of *Salmonella sp.* was investigated in 25 mL of sample

according to Resolution RDC no. 12/2001 (Brasil, 2001) for fruit beverages, juices, and nectars using the methodologies described by Downes and Ito (2001)

Sensory evaluation

A nine-point structured hedonic scale, ranging from "disliked very much" (1) to "liked very much" (9) was used to assess the acceptability of the products (Stone and Sidel, 1993). The tests were performed with 40 untrained judges, who received samples randomly coded with three digits. The results were analyzed statistically by ANOVA and Tukey's test to compare the means at 5% probability using the statistical software Assisat (version 7.6 beta).

Results and Discussion

The result of the preliminary sensory tests to select the mixed nectars with good acceptance and higher pineapple skin juice concentration indicated the following formulations: F1 (70% pineapple skin juice + 30% cupuaçu pulp), F2 (70% pineapple skin juice + 30% taperabá pulp), and F3 (30% pineapple skin juice + 70% guava pulp).

Physicochemical characterization of the mixed nectars

Table 1 shows the results of the physicochemical parameters of the mixed nectars with pineapple skin juice combined with cupuaçu, taperebá, and guava pulp. pH and titratable acidity were in accordance with the identity and quality standards for tropical juices (MAPA, 2014). pH and acidity of formulation F2 significantly differed ($p < 0.05$) from F1 and F3, which impacted the total soluble solids ($^{\circ}\text{Brix}$)/total titratable acidity ratio. This result is in agreement with that found by Lima *et al.*, (2008) to develop the mixed drink of coconut water and acerola juice. The total soluble solids contents, which were standardized at 11 $^{\circ}\text{Brix}$, did not vary after pasteurization. Pinheiro *et al.* (2006) found values of pH, vitamin C, total titratable acidity in citric acid, and total soluble solids for whole pineapple juices close to those in the present research.

Formulation F3 had higher vitamin C content ($p < 0.05$) compared to F1 and F2 since guava is considered a fruit rich in this nutrient (USDA, 2006). Higher values were observed by Castro *et al.* (2007) when analyzing whole guava juices (37.05 to 48.09 mg/100 g) and by Matsuura and Rolim (2002) when they use whole pineapple juice and acerola for formulation of mixed nectars. Because, as vitamin C is very sensitive, it may have been affected by the

Table 1. Mean and standard deviation of the physicochemical analyses of the mixed nectars

Analyses	Formulations		
	F1	F2	F3
pH	3.84 ^a ± 0.02	3.44 ^b ± 0.04	3.85 ^a ± 0.08
Titratable acidity (% citric acid)	0.86 ^a ± 0.03	0.71 ^b ± 0.01	0.87 ^a ± 0.03
TSS/titratable acidity ratio	12.80 ^a ± 0.37	15.44 ^b ± 0.12	12.64 ^a ± 0.36
Vitamin C (mg/100 mL)	1.40 ^a ± 0.30	1.23 ^a ± 0.30	7.56 ^b ± 0.36
Total fibers (%)	0.68 ^a ± 0.05	0.49 ^b ± 0.09	0.46 ^c ± 0.02

Means followed by the same letters on the same row do not differ according to Tukey's test at 5% probability. F1: 70% pineapple skin juice + 30% cupuaçu pulp. F2: 70% pineapple skin juice + 30% taperebá pulp. F3: 30% pineapple skin juice + 70% guava pulp. TSS: total soluble solids

pasteurization and/or storage process in the present research.

Fiber content differed ($p < 0.05$) among the samples analyzed, with the highest concentration in sample F1 and the lowest, in F3, which contained only 30% of pineapple skin juice. This result is similar to that reported by Bezerra et al. (2013) when studying the fiber content of mixed juice of acerola, passion fruit, and taperebá.

Rheological behavior

Figure 1 shows the effect of temperature on the rheological behavior of the mixed nectars (F1, F2, and F3) with their respective fits to PL model. It can be seen that, for constant strain rate, shear stress decreases as temperature increases. According to Alparslanand Hayta (2002), this effect can be explained by the structural collapse of the pulp due to the hydrodynamic force generated and the greater alignment of the constituting molecules.

Along with an increase in temperature, the slope gradient decreases as strain rate increases, which indicates that apparent viscosity decreases as strain rate increases. This behavior can be justified by the sample's structural change due to the hydrodynamic forces generated, which makes the particles rearrange in parallel directions and the smaller particles break. The latter more easily flow in the direction of the tension applied (Rao, 1999; Chinnet et al., 2009; Shamsudin et al., 2013).

The parameters related to the PL model satisfactorily fit all temperature ranges studied, with low RSS and X^2 values and high determination coefficient (R^2) values at over 0.96 (Table 2), indicating a strong correlation to the shear stress and strain rate parameters. The behavior index (n) at all temperature analyzed had values below 1, which indicates a non-Newtonian and typically pseudoplastic behavior for all mixed nectar samples.

Branco and Gasparetto (2003) have identified

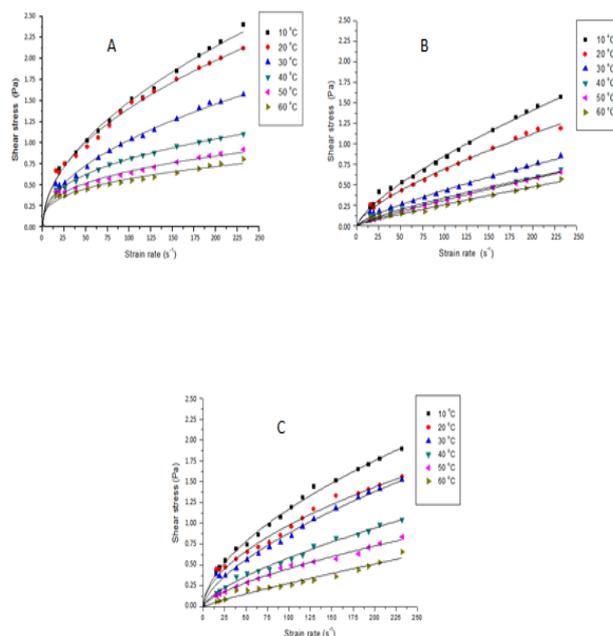


Figure 1. Relation between shear stress and strain rate adjusted fitted by the PL model: a) Formulation F1; b) Formulation F2; c) Formulation F1

pseudoplastic behavior so ternary mixtures of mango pulp and orange and carrot juice, by model of Ostwald-de-Waele. Silva et al., (2012a) found flow behavior index (n) values below 1 in samples of cashew, mango, and acerola pulps. This behavior (pseudoplasticity) has also been reported by Braga et al., (2013), Guedes et al., (2010), and Silva et al., (2005), respectively, for pineapple juice, watermelon pulp, and acerola juice.

Formulation F1 had the highest consistency index (K) values and its high fiber content might have raised the product's consistency. In formulations F2 and F3, higher temperatures lowered the consistency index (K) (Table 2). This behavior was expected for all samples since higher temperatures tend to make the product less consistent.

Some authors have also reported higher consistency indices at lower temperatures. Vandresen et al., (2009), when analyzing the rheological parameters of carrot juice using the PL model at 8 and 25°C, found that, at the lower temperature, the samples had higher consistency indices, which was also observed by Bezerra et al., (2013) when analyzing the rheological parameters of mixed juice of acerola, passion fruit, and taperebá between 10 and 60°C.

Figure 2 shows that apparent viscosity decreased as temperature and strain rate increased. In this case, higher temperature lowers the viscosity of the liquid phase due to the greater particulate matter mobility (Vidal et al., 2006). For formulation F1 (Figure 2A),

Table 2. Parameters of the PL model's fit for samples F1, F2, and F3

Parameter	Temperature (°C)					
	10	20	30	40	50	60
F1						
K (Pa.s ⁿ)	0.130	0.147	0.104	0.144	0.132	0.127
n	0.529	0.490	0.498	0.374	0.349	0.326
x ²	0.003	0.002	0.001	2.27 x 10 ⁻²	8.00 x 10 ⁻²	9.79 x 10 ⁻²
RSS	0.0483	0.0271	0.0178	0.00341	0.0120	0.0147
R ²	0.992	0.995	0.994	0.997	0.985	0.973
F2						
K (Pa.s ⁿ)	0.030	0.028	0.013	0.009	0.006	0.005
N	0.729	0.695	0.765	0.788	0.874	0.875
x ²	9.41 x 10 ⁻⁴	8.55 x 10 ⁻⁴	4.70 x 10 ⁻⁴	3.57 x 10 ⁻⁴	3.13 x 10 ⁻⁵	4.78 x 10 ⁻⁴
RSS	0.0141	0.0128	0.0071	0.0054	4.69 x 10 ⁻⁴	0.0072
R ²	0.996	0.994	0.992	0.991	0.999	0.982
F3						
K (Pa.s ⁿ)	0.081	0.076	0.045	0.025	0.020	0.005
N	0.581	0.556	0.646	0.688	0.676	0.872
x ²	0.001	0.003	0.002	6.76 x 10 ⁻⁴	4.93 x 10 ⁻⁴	0.0012
RSS	0.0167	0.0382	0.0283	0.0101	0.0074	0.0185
R ²	0.996	0.987	0.990	0.993	0.991	0.962

F1: 70% pineapple skin juice + 30% cupuaçu pulp. F2: 70% pineapple skin juice + 30% taperebá pulp. F3: 30% pineapple skin juice + 70% guava pulp

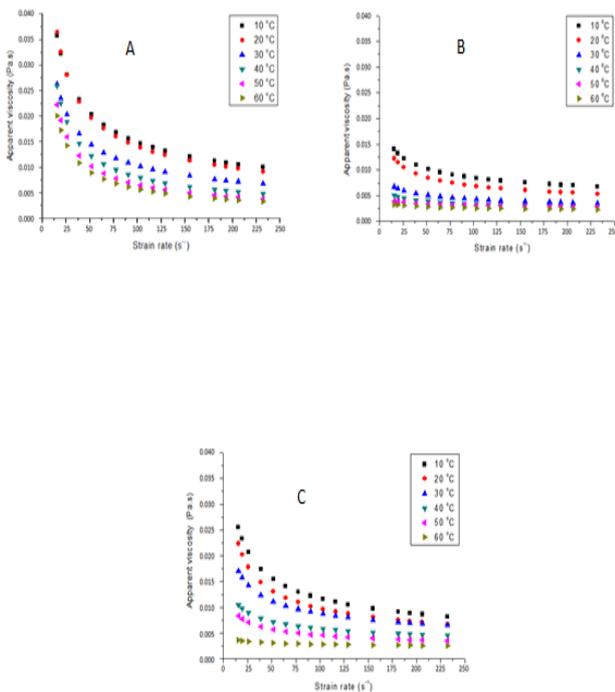


Figure 2. Relation between apparent viscosity and strain rate: A) Formulation F1; B) Formulation F2; C) Formulation F1

apparent viscosity decreased as strain rate increase at all temperatures studied, which is more evident in the strain rate points below 103 s⁻¹, which is typical of pseudoplastic behavior. Silva *et al.*, (2012b) analyzing the rheological behavior of mixed drinks of caja and mango with addition the prebiotics found that all samples presented non-Newtonian behavior with pseudoplastic character. Bezerra *et al.*, (2009) found similar results when analyzing the effect of temperature on the rheological properties of

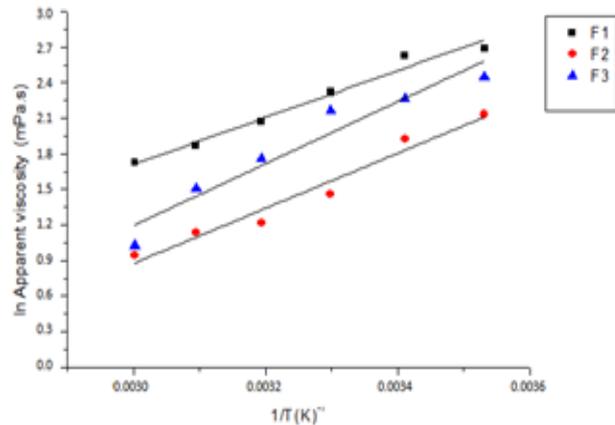


Figure 3. Effect of temperature on apparent viscosity of mixed nectar samples fitted by the Arrhenius equation

strawberry pulp.

However, Figure 2B shows that apparent viscosity in formulation F2 was little affected by the higher strain rate, with a slight decrease at the early strain rate values, at 10 and 20°C. At higher temperatures, the nectar's apparent viscosity tends to remain constant likely because of the matrix breakdown due to shear time. In studies with peach puree and grape juice, respectively, Toralles *et al.*, (2006) and Oliveira *et al.*, (2009) also found greater pseudoplasticity at lower temperatures.

Figure 2C shows that formulation F3 has a similar behavior as F2, with a more marked decrease in apparent viscosity at lower temperatures. At 10, 20, and 30°C, two very discrete regions are formed, corresponding to well-differentiated structural behaviors. In the first strain rate region, below 129

s-1, the binding forces start being overcome by shear forces due to the Brownian movement, with induces particle alignment and makes the mixed juice's apparent viscosity decrease exponentially. In the second region, with strain rate above 129 s⁻¹, the particle alignment state and orientation is established and the juice's apparent viscosity tends to remain constant, which indicates a Newtonian behavior.

A similar behavior was observed in other studies on rheological characterization of fruit fluids (Silva *et al.*, 2005; Haminiuket *et al.*, 2006). According to Braga *et al.*, (2013), this can be related to the smaller size of colloidal aggregates caused by the higher strain rate. Still regarding formulation F3, at higher temperatures (40, 50, and 60°C), apparent viscosity remained virtually constant at all strain rates, which indicates a Newtonian behavior for the samples analyzed at those temperatures.

The values of τ calculated at a strain rate of 103 s⁻¹ decreased as temperature increased for the mixed nectar samples analyzed (Figure 3). A similar behavior was observed by Oliveira *et al.*, (2011) when studying the rheology of gabioba (*Campomanesia xanthocarpa*) and by Toralles *et al.*, (2006) when analyzing the effect of temperature and concentration on the rheology of homogenized peach puree. Bezerra *et al.*, (2013) and Silva *et al.*, (2013) analyzed the effect of temperature on the rheology of mixed tropical fruit juice and mixed nectars cashew, mango and acerola, respectively, and also found a strong influence of temperature on apparent viscosity, which decreased as temperature increased, matching the results in the present research.

From an industrial standpoint, the lower viscosity facilitates pulp flow and heat exchange during processing. The lower the viscosity, the lower is the load loss during flow, which cuts down the pumping power needed and, consequently, energy costs (Lucena *et al.*, 2005; Braga *et al.*, 2013; Bezerra *et al.*, 2013). Arrhenius equation (Equation 5) satisfactorily represented the effect of temperature on the apparent viscosity of the mixed nectars at the strain rate verified (103 s⁻¹), with R² values above 0.92, as seen in Figure 3.

According to Braga *et al.*, (2013), high Ea values in a system indicate a quicker change in apparent viscosity with temperature. A comparison of the samples show that the highest Ea value was found for formulation F3, hence, this sample's molecular structure is more influenced by temperature compared to the others. The Ea and R² values obtained in the present study for the mixed nectar formulations were: F1: Ea = 3.92 kcal.g.mol⁻¹ and R² = 0.97; F2: Ea = 4.61 kcal.g.mol⁻¹ and R² = 0.96; and F3: Ea = 5.19 kcal.g.mol⁻¹ and R² = 0.92, which match the values

found in the literature for juices and nectars. Silva *et al.*, (2005), when analyzing the rheological behavior of acerola juice, found Ea values ranging from 1.79 to 3.50 kcal.g.mol⁻¹. Bonomoet *et al.*, (2009), when studying the rheological behavior of cashew juices, found Ea values between 2.99 and 4.46 kcal.g.mol⁻¹. Ea values close to those obtained in the present study were also reported by Braga *et al.*, (2013) at 4.54 and 4.89 kcal.g.mol⁻¹ for natural and depectinized pineapple juice, respectively.

Microbiological analysis

The mixed nectar samples analyzed were within the ranges set by the Brazilian legislation (ANVISA, 2001), which mandates no coliforms at 35°C or *Salmonella* sp. in 25 mL of the product.

Sensory evaluation

Formulations F2 (70% pineapple skin juice + 30% taperebá pulp) and F3 (30% pineapple skin juice + 70% guava pulp) obtained acceptability indices (AI) of 84.44% and 82.00%, respectively. Nectar F1 (70% pineapple skin juice + 30% cupuaçu pulp) significantly differed (p<0.05) compared to the other formulations with AI of 65.33%. According to Teixeira *et al.* (1987), a product must have AI above 70% for it to be sensorily acceptable.

Researches carried out with mixed nectars of acerola, passion fruit, and taperebá (Bezerra *et al.*, 2013), umbu and cajá (Mattietto *et al.*, 2007), passion fruit and sugar apple (Morzelle *et al.*, 2009), and ambarella with mint (Damiani *et al.*, 2011) obtained good sensory acceptability, which indicates the tasters are interested in healthy products with exotic flavors.

Conclusion

The PL model satisfactorily described the rheological behavior of the samples analyzed according to the parameters R², X², and RSS. The nectars had non-Newtonian behavior with pseudoplastic-fluid characteristics. Apparent viscosity values decreased as strain rate and temperature increased. The Arrhenius equation represented the effect of temperature on apparent viscosity of the mixed nectars with R² values above 0.92. Knowing these properties is important for quality and process control, to design production lines, and develop these products at an industrial level. The highest AI was obtained by formulation F2, which contains 70% pineapple skin juice + 30% taperebá juice, which suggests the possible use of pineapple industrialization by-products and, thus, decrease its environmental impact.

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