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Physicochemical properties, Steady and Dynamic Rheological Measurements of Sour Guava (*Psidium Araca*) Pulp

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Abstract

Sour guava (*Psidium araca*) is a tropical fruit of the tropical region, recognized for its typical taste and nutritional composition. This work aimed to investigate how temperature affects the physicochemical and rheological characteristics of the sour guava pulp subjected to controlled scalding conditions. Physicochemical properties of fresh and scalded pulp were analyzed. Rheological analyses of steady shear rate in function of temperature (5 - 80 ºC) and viscoelastic properties were done. Scalding process did not affect the physicochemical properties (total acidity, soluble solids, pH, and maturity index) of the pulp, nevertheless, a 30% decrease in total phenolic compounds was observed. Pulps exhibit a non-Newtonian behavior type shear thinning described by using the *Herschel-Bulkley* model ($R^2 > 0.970$). The influence of temperature was described by an Arrhenius-type equation on the pulp consistency index. The pulps displayed characteristics of a weak gel, where the storage modulus surpassed the loss modulus. A power function of the oscillatory frequency accurately described the storage and loss moduli, indicating elastic properties. The results showed that the sour guava pulp is suitable for use as a raw material for the development of processes for the manufacture of food products.

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Keywords

Rheology;
Sour Guava (Psidium Araca);
Shear Thinning;
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Introduction

Sour guava (*Psidium araca*) is a fruit of *Myirtaceae* family which has around 142 genera and 4620 species, being *Psidium* genus the most found, specifically distributed in tropical regions such as Panama, Brazil, Peru, Ecuador, and Colombia. The fruit contains 83% of moisture, 0.83% of protein, 6.8% of carbohydrates, 0.52% of fat, and 7.9% of fiber, vitamin C (400mg/100g),[1] and high percentages of antioxidants and other beneficial characteristics for consumption.[2] However, its consumption is fresh and juice, and it is necessary to know the rheological

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properties to develop manufactured products such as nectar, smoothies, jellies, preserves, ice cream compotes, jams and purees.

The rheological properties of complex fluids are essential in the design and development of food products in the industry, in the characterization of textural characteristics, and in quality control.[3] In the same way, the study of rheological properties has been considered an analytical tool for providing fundamental information about the structural organization of food. Various rheological studies have affirmed the varied flow behaviors observed in food systems.[4–8] Due to the complex interactions between soluble sugars, pectic substances and suspended solids, fruit pulps are commonly classified as non-Newtonian fluids,[9] and in addition, the rheological behavior of fruit pulp can be influenced by several factors, including particle size[10] and concentration of soluble solids concentration.[11] However, temperature is one of the factors that most affect the viscosity of fruit pulp[12] the knowledge of rheological behavior as a function of temperature is essential to provide a better knowledge during processing at elevated temperatures.[13] Therefore, this study aims to investigate how changes in temperature affect the physicochemical and rheological properties of the pulp of sour guava (*Psidium araca*).

Materials and Methods

Pulp Preparation

The sour guava (*Psidium araca*) fruits were obtained in a commercial state of maturity from the Cartagena Food Supply Center. After washing, the fruit was peeled and seeded by hand, and the remaining pulp was homogenized. The resulting pulp was then scalded for 2 minutes at 70°C, and subsequently stored at 4°C.

Determination of Total Soluble Solids (TSS)

Total soluble solids (TSS) of the sour guava juice were measured using the AOAC method 932.12 (Association of Official Analytical Chemists[14]) in conjunction with a hand refractometer. To determine the TSS, a homogenized sample of 1 mL of sour guava juice was placed on the prism of the refractometer and the reading was recorded in ºBrix.

Determination of pH

The pH values of the fruits were measured using the AOAC[14] method 981.12 with the use of a digital pH meter (Model HANNA HI 9124). The pH meter was calibrated using buffer solutions of pH 4.0 and 7.0.

Determination of Titratable Acidity (TTA)

The determination of titratable acidity (TTA) in the samples was carried out using the AOAC [14] method number 942.15. For this method, 10 g of the sample was diluted with 250 mL of distilled water. Then, a 50 mL aliquot was mixed with 0.2 mL of phenolphthalein indicator and titrated with 0.1 N NaOH. The endpoint was determined by the appearance of the first pink color. The TTA was expressed as a percentage of citric acid.

Determination of the maturity index (MI)

The MI value was determined using equation (1) that relates the titratable acidity and the total soluble solids.[15]

$$MI= TSS / TA \qquad ...(1)$$

Total Phenolic Compounds

The total phenolic content of the samples was determined using the Folin-Ciocalteu method.[16] First, a methanol extraction was performed by mixing 1 g of pulp with 50 mL of methanol in an ultrasound bath at 25 °C, followed by centrifugation at 4000 rpm for 5 minutes. For the subsequent steps, the methodology used by Quintana *et al*.[17] was followed with some modifications. The results were reported as mg gallic acid equivalents/g of sample (GAE).

Rheological Evaluations

The pulp rheological characteristics were evaluated following the procedures described by Quintana *et al*., (2017) [8] using a Haake Mars 60 controlled stress rheometer (Thermo Scientific, Germany) equipped with plate-plate geometry (35 mm in diameter and 1 mm gap). To ensure consistency, the temperature was maintained using a Peltier system at different temperatures ranging from 5 to 80 °C. A 300-second equilibration period was applied to each sample prior to the rheological test to standardize its thermal and mechanical histories.

- Steady-state viscous flow tests were performed in the range of 0.001 to 1000 s^{-1}.
- A stress sweep from 0.001 to 1000 Pa was done at 1 Hz to determine the lineal viscoelastic region (LVR).

- The frequency sweep was performed on the LVR within a frequency range of 0.01 to 100 rad s^{-1}.

Statistical Analysis

Data collected were analyzed using Statgraphics software (centurion XVI version) to perform a one-way analysis of variance (ANOVA) and identify statistically significant differences ($p < 0.05$) between samples. The experimental analysis was conducted in triplicate.

Results and Discussion

Physicochemical Characterization of Sour Guava Pulp

The physicochemical characterization of fresh and scalding treated sour guava pulp is presented in Table 1. The fresh pulp has a pH value of 3.50 ± 0.04, acidity of 1.79 ± 0.31 % citric acid, soluble solids of 8.04 ± 0.05 ºBrix, a maturity index of 4.49 ± 0.06 and 279.50 ± 0.41 mg acid gallic/100 g of pulp. This result is consistent with the finding by Lara Mantilla *et al.*, (2007)1, which present similar values of acidity (1.764), pH (2.68) and soluble solids (8.0 ºBrix)

Then the acidity values of the sour guava pulp (1.79% citric acid) and the pH values (3.50) of the sour guava pulp are above the common guava (*Psidium guajava*), with values between 0.42 and 0.96% expressed as citric acid and pH 3.90 – 4.60[18] associated with the chemical composition of fruits and the degree of maturation; this is considered an advantage in food processing because it would reduce the amount of acid added to the products to control the growth of microorganisms,[1] transforming this pulp into a raw material with a high potential for industrialization.

Table 1: Physicochemical properties and total phenolic compounds of guava pulp

Treatment	Fresh pulp	Scalded fruit
pH	3.50 ± 0.04^{a}	3.67 ± 0.04^{a}
TTA (% citric acid)	1.79 ± 0.31^{a}	1.67 ± 0.24^{a}
TSS (ºBrix)	8.04 ± 0.05^{a}	8.08 ± 0.04^{a}
MI	4.49 ± 0.06^{a}	4.83 ± 0.08^{a}
TPC (mg acid gallic/100 g sample)	279.50 ± 0.41^{a}	196.48 ± 11.68^{b}

The data presented are expressed as the mean ± standard deviation.
Different letters in the same column indicate statistically significant differences ($p < 0.05$).

The percentages of soluble solids of sour guava were lower than those reported for the "common guava", with values of 11.9 - 13.2 ºBrix, indicating that this variety has a higher percentage of simple carbohydrates such as sucrose, glucose, and fructose.[18] The maturity index of the sour guava pulp in this case was 4.49 and 4.83 for the fresh and scalded pulp, respectively. The value obtained is slightly lower than the values documented by Arrázola *et al.*,[19] *Psidium guajava* L. from Córdoba with values between 9.38 and 17.94. It should be noted that this index is influenced by factors such as harvest time and environmental conditions, and industrial processes can also have an impact on it, as they can involve adjustments in acidity and °Brix through methods that alter the concentration of acids or total solids in the final product.[20]

Then, scalded fruits present 3.67 ± 0.04 of pH, 1.67 ± 0.24 % of citric acid, 4.83 ± 0.08 of maturity index, and 196.48 ± 11.68 of mg acid gallic/100 g sample. The pH did not vary with the thermic treatment in comparison with fresh pulp, due to the time and temperature of exposure in blanched process that caused some organic acids to be degraded in fruits; however, a nonsignificant difference ($p > 0.05$) was observed in both cases. Significant differences in total phenolic compounds (TPC) were observed ($p < 0.05$) (Table 1); Fresh pulp contains 279.50 mg of acid gallic/100 g of pulp, while scalded pulp contains 196.48 mg of acid gallic/100 g of pulp, showing a 30% decrease associated with degradation of thermolabile phenolic compounds or their polymerization during heat treatment.[21] Similarly, these values were higher than the content reported for fruits of fruits of high consumption in the country, such as guava, apple, banana, mango and papaya.[22,23] This indicates that the sour guava pulp is rich in phenolic compounds compared to the pulps of different fruits, which gives us an indication of how rich this dietary matrix is in antioxidants, considering that different studies show that these parameters are closely related[24] and that the scalded guava pulp is an interesting raw material for processing food products.

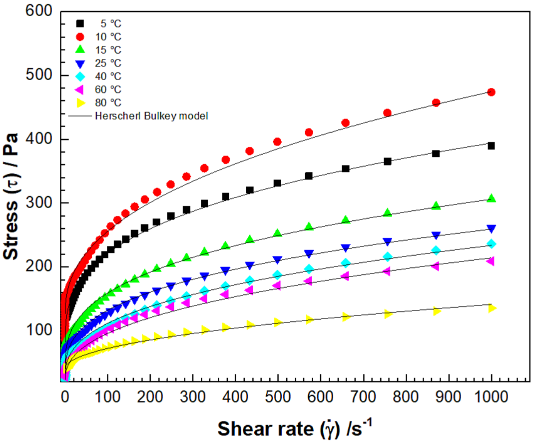

Fig. 1: Flow curves of sour guava pulps at different temperatures (5, 10, 15, 25, 40, 60 and 80 ºC) adjusting to the Herschel Bulkley model

Rheological Properties

Steady State Shear Properties

To analyze the rheological properties of the sour guava pulp, the relationship between shear stress (τ) and shear rate ($\dot{\gamma}$) was examined and the results are presented in Figure 1, which shows the flow behavior of the samples at different temperatures. The scalded guava pulps exhibited shear thinning behavior with yield stress (τ_o), a typical characteristic of multiphase materials, such as pulps and juices[25] and indicate that there is a cross-linked structure or other interactive structure that must be decomposed before flow can occur at an adequate speed. Such materials show a shear stress curve that does not originate from the origin of the shear stress/strain rate graph and is downward concave.[26,27]

Table 2: The parameters of the *Herschell Bulkley* model steady shear rheological parameters of sour guava pulp

Temp.	τo	k		
ºC	Pa	Pa·s^n	n	R2
5	123.66 ± 1.97^a	30.29 ± 0.22^a	0.42 ± 0.02^a	0.98
10	75.98 ± 1.81^b	25.27 ± 0.21^b	0.34 ± 0.01^b	0.99
15	36.94 ± 1.68^c	22.14 ± 1.44^c	0.34 ± 0.01^b	0.99
25	25.73 ± 1.52^d	18.75 ± 1.33^d	0.34 ± 0.01^b	0.99
40	23.94 ±1.31^d	15.36 ± 1.05^d	0.37 ± 0.01	0.99
60	21.16 ± 1.43^d	11.20 ± 0.08^{de}	0.41 ± 0.01	0.99
80	16.55 ± 1.51^e	5.19 ± 0.65^e	0.50 ± 0.03^d	0.97

The data presented are expressed as the mean ± standard deviation.
Different letters in the same column indicate statistically significant differences ($p < 0.05$).

The flow behavior of pulps could be well described by the Herschel-Bulckley model (Equation 2):

$$\tau = \tau_o + k\dot{\gamma}^{n} \qquad \text{...(2)}$$

Where, k (Pa·s^{-1}) is the consistency index, τ_o is the yield stress and n are the flow index. The Herschel-Bulckley parameters obtained are summarized in Table 2 with $R^2 > 0.970$. The mean values obtained for the parameters (τ_o k and n) showed temperature dependence ($p < 0.05$).

The temperature significantly decrease the yield stress (τ_o) from 123.66 to 16.55 Pa and the consistency index (k) from 30.29 a 3.19 Pa.s^n, of sour guava pulp while the with flow index (n) showed no significant difference in the temperature range between 10 - 40 ºC and present similar values between 0.340 – 0.490, although significant difference were obtained between 5 and 80 ºC, all n values were less than 1, confirming that the pulp present a shear thinning behavior.[3,28] Consequently, viscosity is related to the thermally activated process.[29] Then, the microstructure of complex fluids and the temperature dependence of rheological parameters can be characterized.[29] Consequently, the natural logarithm of the consistence index versus the absolute temperature of the pulp is shown in Figure 2. The dependence of the consistence index on temperature supported an Arrhenius equation (Equation 3):

Table 3: Values for the power law model for storage (G') and modulus (G") as a function of frequency (ω) for guava pulps at different temperature (5, 10, 25, 40, 60 and 80 ºC)

Temp. ºC	*K'*	*n'*	R²	*K"*	*n"*	R²
5	2619.81 ± 21.14^{a}	0.19 ± 0.002^{a}	0.997	835.84 ± 11.55^{a}	0.23 ± 0.004^{a}	0.995
10	2158.86 ± 13.63^{a}	0.21 ± 0.001^{b}	0.998	735.20 ± 5.56^{a}	0.23 ± 0.002^{a}	0.998
25	1593.65 ± 6.58^{ab}	0.22 ± 0.001^{bc}	0.999	560.96 ± 3.94^{b}	0.25 ± 0.002^{b}	0.999
40	846.54 ± 19.09^{b}	0.22 ± 0.006^{bc}	0.987	296.15 ± 3.97^{c}	0.28 ± 0.003^{c}	0.997
60	2309.08 ± 88.00^{a}	0.23 ± 0.010^{c}	0.986	1195.15 ± 55.25^{d}	0.32 ± 0.013^{d}	0.963
80	30159.10 ± 1428.47^{c}	0.25 ± 0.014^{d}	0.938	6008.69 ± 232.34^{e}	0.37 ± 0.007^{e}	0.948

The data presented are expressed as the mean ± standard deviation.
Different letters in the same column indicate statistically significant differences ($p < 0.05$).

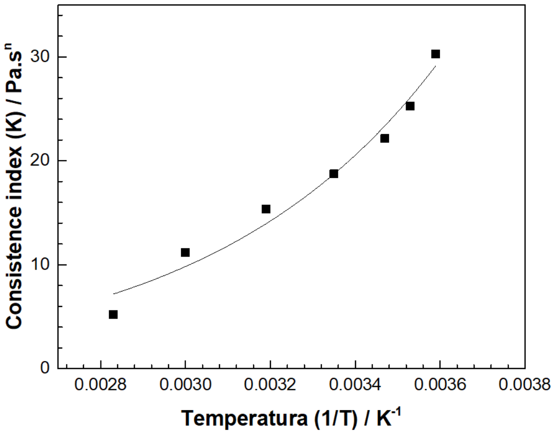

Fig. 2: Variation of K values as a function of temperature and fit to the Arrhenius equation

$$k = Aexp\left(\frac{E_a}{RT}\right) \qquad ...(3)$$

the absolute temperature (T, K), the gas constant (R, 8.314 J/mol K), and the activation energy (Ea, J/mol). In addition, there is a material parameter A that depends on the consistency index.

where, T is the absolute temperature (K), the gas constant R (8.314 J/mol K), and the activation energy Ea (J/mol). In addition, there is a material parameter A that depends on the consistency index. The coefficient of determination R^2 was 0.9689, concluding that the Arrhenius equation adequately describes the effect of temperature on the consistency of the fruit with an activation energy (Ea) of sour guava pulp of 15.319 kJ/mol. This value is slightly higher than that reported for jabuticaba pulp (Ea = 13.00 kJ/mol)[30] and the *Carica papaya* pulp (Ea=10.55 kJ/mol).[8] Therefore, the internal structure of the *Psidium araca* pulp is less susceptible to temperature changes compared to the pulps of the fruits mentioned above.

The activation energy, Ea, is essential to facilitate molecular movement when the temperature is increased. As a result, at high temperatures, liquids with higher Ea flow more smoothly.[31] Furthermore, Karwowski *et al.*, (2013)[32] noted that activation energy values can be associated with insoluble solid content, where a low insoluble solid content results in high activation energy values.

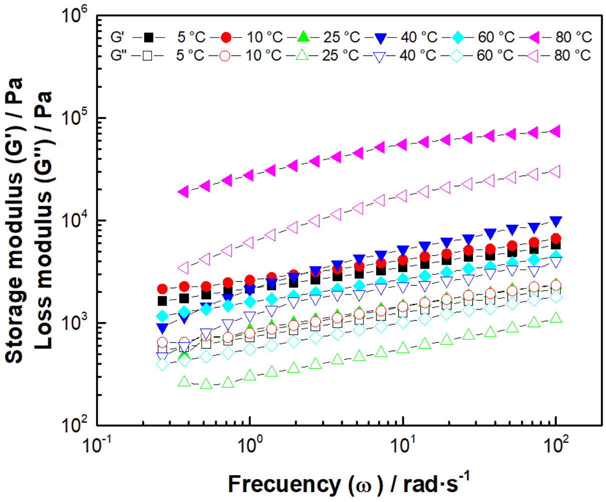

Fig. 3: The storage modulus (G′) and modulus (G″) as a function of frequency (ω) for *P. araca* pulps at different temperatures (5, 10, 25, 40, 60 and 80 ºC)

Viscoelastic Properties

Figure 3 shows the storage (G') and loss (G'') modulus in function of angular frequency (ω) of the sour guava pulp at different temperatures in the linear viscoelastic range. G' was greater than G'' and increase with ω indicating an elastic behavior; then, the pulp can be categorized as a weak gel.[28] This behavior is typically observed in suspensions with net-like structures,[33] which has been reported for various fruit and vegetable products such as quince,[34] umbu[35] and squash[36] pulp. Therefore, G' and G'' were model with power law equations (4) and (5):

$$G'=K'\cdot \omega n' \qquad ...(4)$$

$$G''=K''\cdot \omega n'' \qquad ...(5)$$

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The data presented are expressed as the mean ± standard deviation.
Different letters in the same column indicate statistically significant differences ($p < 0.05$).

where K′(Pa · s^n/rad^n) is the elastic constant, K″(Pa · s^n/rad^n) is the viscous constant, n′ and n″ (rad·s^{-1}) are indices of the elastic and viscous effect.

The viscoelastic behavior of pulps was well described by the power law model ($R^2 > 0.938$), as presented in Table 3. The elastic and viscous components of the pulps were found to increase considerably with increasing temperature, with n′ and n″ indicating that the elastic component was more dominant than the viscous component at higher frequencies. Additionally, K′ and K″ decreased from 5 to 40 ºC; and increased from 60 to 80 °C, indicating that the viscoelastic properties of the pulp of guava (*Psidium araca*) pulp are quite dependent on the temperature in this last range, reflecting changes in the internal structure of the pulp.

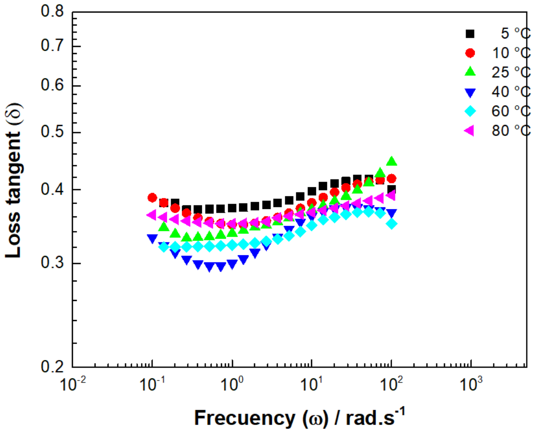

Fig. 4: Phase angle tangent (δ) as a function of frequency (*ω*) for sour guava pulps at different temperatures (5, 10, 25, 40, 60 and 80 ºC)

To determine whether the elastic or viscous property dominates, the loss tangent (Tan δ) compares the amount of energy lost during an oscillatory test with the amount of energy stored during the same period. This value is obtained by calculating the ratio of the viscous component (G″) to the elastic component (G′). The resulting value, which ranges from 0 to 1, indicates the predominance of the elastic character.[37] This analysis can be used to assess the behavior of a material.

Figure 4 shows that as a function of frequency and with increasing temperature, the value of tan δ of the *Psidium araca* pulp presents a brief drop, while as the frequency value increases, the value of tan δ for all temperatures, which confirms the predominance of elastic properties for high temperatures.

Conclusions

The physicochemical properties of sour guava pulp are not significantly affected by the scalding process, except for total phenolic compounds. The rheological properties of the sour guava pulps showed a shear thinning behavior, showing the yield stress in the steady shear measurement, when the flow behavior of the product could be well described by the Herschel–Bulkley model $R^2 > 0.970$. The effect of temperature on the viscosity of the sour guava pulp was observed, leading to the possibility of consistency parameters that correspond to the Arrhenius equation.

Weak gel behavior of the viscoelastic properties was observed, and it was found that these gels were stable under varying temperatures, with notable differences depending on the temperature. Moreover, Tanδ confirms the predominance of elastic properties. The results of this project promote the use of the flow properties of the pulp of sour guava (*Psidium araca*), raw materials of national interest to be used for the preparation of new products and the development of unit processes.

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Conflict of interest

The authors declare no conflict of interest.

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