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Rheological characterization of prickly pear juice (Opuntia Ficus Indica)

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Abstract: The quality factors of prickly pear juice *(Opuntia ficus indica)*, such as the flow index, consistency index, and yield stress index, proved to be important in the rheological characterization and acceptance of this food; reducing the challenges for technologies related to this product. The objectives were to characterize the rheology of prickly pear juice, determine the flow index, yield stress threshold, consistency index (K) and Pearson regression coefficients (r) and determination ($r^2 \times 100$) for a temperature of 20 °C and concentrations of 5, 15, 25, and 40 °Brix. The methodology was based on the rheology of natural prickly pear juice, red-violet variety, mixed with 25% cold boiled water (p/p), with a pH of 3.64 and acidity of 0.85%. Measurements were carried out using a concentric cylinder viscometer, with a thermostatic bath for temperature control within a 0.5 °C range, from 15 °C to 70 °C, obtaining the shear stress (τ) corresponding to the strain rate gradients (γ). To determine the yield stress (τ_0) of the Herschel-Buckley rheological model $(\tau = K(\gamma)^n + \tau_0)$ the procedure followed the theory of Bronshtein, et al. [1]. The flow index (n) and yield stress (τ_0) for prickly pear juice at concentrations of 5, 15, 25, and 40 °Brix were 0.5826, 0.7302, 0.7901, and 0.8611; and 58.0935, 65.2047, 73.3424, and 94.2155, respectively, which were progressive and directly proportional to the concentrations; concluding that the consistency index decreases and increases within the working concentration range, while the yield stress and flow index increase, and the regression (r) and determination $(r^2 \times 100)$ coefficients show a perfect correlation between shear stress (τ) and strain rate gradient (γ).
Keywords: *Consistency and flow indices, Prickly pear juice, Rheological models, Yield stress.*

1. Introduction

The difficulties regarding quantity and quality in the production of fruit juices are related to the rheological parameters of different liquid foods, with the flow index, consistency index, and yield stress being particularly significant in the case of prickly pear juice (*Opuntia ficus-indica*). Matos and Aguilar [2] mention that these rheological indices contribute to the quality and acceptance of the juice, aiding in its rheological classification before, during, and after control processes, transformation, and conservation of liquid foods. Ibarz, et al. [3] highlight that fruit juice often involves the addition of a concentrate from the same fruit, which undergoes a despectinization process, followed by clarification and concentration through evaporation.

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Ayala Bendezú [4]points out that the functional properties of both fruits and pads, along with their juices, offer health benefits, with notable nutritional characteristics such as fiber, hydrocolloids, pigments, calcium, potassium, and vitamin C, all of which have antioxidant properties and are vital compounds for a healthy diet. Costell and Durán [5] as well as Torre [6] mention that fruit juices exhibit highly variable rheological behavior, which plays a crucial role in technological processes.

Méndez, et al. [7] indicate that the prickly pear fruits have a pH of 5.2; soluble solids of 13.1 °Brix; and acidity of 0.085%, whereas the aguaymanto fruits have a pH of 3.6; soluble solids of 14.1 °Brix; and acidity of 1.49%. Torre [6] and Flores [8] indicate that rheology is the study of the deformation and flow of raw materials, intermediate products, semi-processed products, and finished products. Torre [6] and Whorlow [9] mention that plastic products correspond to the Bingham model, where for certain practical purposes, plastic materials differ from liquids in that they do not flow when only gravity acts on them. Matos and Aguilar [2] mention that the prickly pear fruit (*Opuntia ficus indica*) is ovoid and native to Peru, Bolivia, and Mexico; growing in various climates from sea level up to 3,000 meters, cultivated in both the mountains and coast at temperatures ranging from 12 to 34 °C, according to [10].

Torre [6] and Wrusch [11] mention that liquid foods based on fruits, such as juices, nectars, and concentrates, describe different rheological models, as each behaves differently due to its chemical composition and proximate composition.

Cheftel, et al. [12] state that the yield point is evaluated to measure, assess, and model, rheologically, liquid and pseudoplastic foods.

Torre [6] and Sherman [13] indicate that, to obtain adequate rheological characteristics of the final product, in the final processing of prickly pear juice, concentrated prickly pear juice is added, which has undergone a despectinization process, clarification, and finally a concentration by evaporation.

Torre [6] mentions that one of the first tasks in the study of the rheological properties of fluid foods is to determine the rheological model that best represents a set of experimental data on shear stress (τ) and strain rate (γ). Kokini [14] mentions that one of the most used models, of general application for fitting experimental data and quantitatively expressing the flow behavior of time-independent inelastic fluids, is the model proposed by Herschel-Bulkley, given by the expression: $\tau = K(\gamma)^n + \tau_0$

Were,

τ : Shear stress [$Pa.s$].

τ_0: Yield stress [$Pa.s$].

K : Consistency index.

γ: Deformation rate [s^{-1}]

Fruit juices are highly sought after by city dwellers in any country, due to their exquisite taste, in which the rheological behavior of prickly pear juice (*Opuntia ficus indica*) plays a valuable role in acceptance, control, process improvement, and in the design and manufacturing of the different equipment used to treat the prickly pear juice, thus reducing technological problems.

Abdullah, et al. [15] state that if a material deforms but does not flow when stress is applied, it is considered a solid; if the material flows when a very small stress is applied (in mathematical terms: a differential stress), then it is considered a fluid.

Barreiro, et al. [16] mentions that the rheological properties of a food are closely related to its quality and acceptance by the consumer, and the determination of the effects of concentration and temperature on the rheological properties is essential for carrying out calculations related to the flow of non-Newtonian fluids with simultaneous heat transfer, which occurs during food processing, particularly in the concentration and sterilization processes of tomato paste (*Solanum lycopersicon*).

The minerals present in prickly pear (*Opuntia ficus indica*) are potassium, phosphorus, and magnesium; it is highly recommended for individuals who care about their aesthetics due to its very low

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caloric content. It also provides a significant amount of fiber, ideal for individuals with constipation, as indicated by [17].

Barboza-Mejía and Velásquez-Barreto [18] mention that viscosity varies considerably with temperature changes, and that they have an inversely proportional relationship, and that rheological models and measurements exhibit unpredictable and highly variable changes.

Rheology is defined as the science of flow that studies the deformation of a body subjected to external stress, and its study is essential in many industries, including plastics, paints, food, printing inks, detergents, and lubricating oils. A formal definition of the term "rheology" can be derived as part of mechanics that studies the elasticity, plasticity, and viscosity of matter, according to [19].

Chen and Wu [20] identified that the increase in the concentration of soluble solids in Newtonian juices significantly elevates viscosity, affecting the stability of the product and its sensory acceptance. This highlights the importance of maintaining strict control over concentration levels during processing to ensure product consistency.

Andia [21] mentions that creep is a phenomenon that describes the slow and continuous deformation of a material under a constant load over time. This occurs in both solid and liquid foods when they are subjected to constant stress for an extended period and is particularly relevant for foods that can deform, where the rate of deformation changes over time. The flow of food fluids is the continuous movement that occurs over time, and the fundamentals of fluid flow are derived from various disciplines, beginning with the most appropriate language. Volumetric flow rate or fluid flow rate is the volume of fluid passing through a given surface over a specified time, and fluid flow refers to the movement of a fluid subjected to different unbalanced forces, corresponding to a part of fluid mechanics. Fluid flow dynamics is concerned with the fluid's behavior under these conditions [18].

The consistency index refers to the thickness of a liquid, where thickened liquids can be described as slightly thick, moderately thick, or moderately viscous. In which dynamic viscosity in fluids is determined to describe the consistency of fluids, and viscosity is defined as the frictional resistance developed by a fluid subjected to shear or compressive stress through deformation [20].

The yield point or yield threshold indicates the maximum stress that can be developed in a material without causing plastic deformation, and the yield point, or yield strength, is the point on a stress-strain curve where elastic behavior ends and plastic behavior begins. Therefore, creep describes the onset of fiber rupture in the sample being analyzed [20].

2. Materials and Methods

Methodology: This study is based on natural fruit juices and nectars (without the addition of thickeners, clarifiers, or any other inputs). The prickly pear juice (*Opuntia ficus indica*) was chosen due to its seasonal nature and its increasing demand in society, especially for its nutritional and health benefits in liquid form. The spatial scope is defined by the technologies used to process the prickly pear juice, while the temporal scope is determined by the seasonality of the raw material, which could be a challenge to address through sustainable technology.

The prickly pear juice (*Opuntia ficus indica*), a variety red violet, was considered for this study. The prickly pear fruit was received, washed, classified (manually by color), peeled, and dreaded in a stainless-steel container. The deseeded pulp was mixed with 25% cold boiled water (w/w) and then blended to obtain the prickly pear juice, which was despectinized. Suspended particles were removed from the despectinized juice, with the sole purpose of obtaining clarified and homogeneous prickly pear juice. The final sample volume was neither greater than one liter nor smaller than 0.25 liters. The soluble solids content of the prickly pear juice was determined using an ABBE-ZEISS refractometer at a temperature of 20 °C. The pH of the prickly pear juice concentrate was measured using a pH meter, yielding a pH of 3.64 at 20 °C. Acidity was evaluated by titration with a NaOH solution to the phenolphthalein endpoint, using a diluted solution of prickly pear juice, resulting in an acidity of 0.85%.

Materials and Packaging: Thermometer, stopwatch, 125 ml and 250 ml Erlenmeyer flasks, 100 ml and 250 ml beakers, 10 ml, 25 ml, 50 ml, and 100 ml flasks, funnels, digital thermometer, stainless steel tables, fan with power source, 10 L cooler, wash bottle, stopwatch, plastic crates (10-20 kg), plastic containers, and other materials as specified by the techniques and procedures in the laboratory manual. All rheological measurements were performed using a Roto visco RV 12 concentric cylinder viscometer (Hooke), with specifications detailed in the work of [19].

Once the deformation gradients corresponding to the different shear stress values were obtained, the appropriate flow curve could be constructed, from which the equation describing the rheological behavior of the fluid under study could be determined [16]. The prickly pear juice (Opuntia ficus indica), previously treated, was divided into two portions of 0.5 liters each. The first portion was used as a reference or auxiliary sample. The second portion was subjected to different temperature and concentration conditions as used in this study. It is important to note that the temperatures and concentrations used were 20°C and 5, 15, 25, and 40°Brix, respectively.

The shear stress (τ) data versus deformation gradient (γ) were directly read from the equipment used. The observed and collected data were tabulated, plotted, and graphed, describing the behavior of the prickly pear juice subjected to various processing conditions.

2.1. Procedure

All rheological measurements were performed using a concentric cylinder viscometer, Roto Visco. For this process, a thermostatic bath was necessary to maintain the temperature control within a range of 0.5 °C, from 15 °C to 70 °C. After obtaining the deformation gradient values corresponding to the different shear stress values, they were graphed to produce the respective rheograms, through which it was possible to characterize the prickly pear juice and determine the rheological model. Nikolidaki, et al. [22] mention that rheological models can be used for the rheological characterization of fluid products and all types of foods, and they can be created with mathematical models that express, through an equation, the relationship between shear stress (τ) and shear rate (γ).

2.2. Rheological Model

The rheological model that most closely approximated the study was that of Herschel-Bulkley, given by the expression: $\tau = K(\gamma)^n + \tau_0$

Were,

τ : Shear stress [$Pa.s$].

τ_0: Yield stress [$Pa.s$].

K : Consistency index.

γ: Shear rate [s^{-1}].

n: Flow index.

To determine the rheological model, we applied the procedure outlined by Bronshtein, et al. [1]. The method for calculating the yield stress (τ_0) involves considering three independent random values and their corresponding dependent values from the database, and using the relation: $\tau_0 = \frac{A \times B - I^2}{A + B - 2I}$; 2) The first value ($A$) is the dependent variable corresponding to the first value of the independent variable (γ_1); the second value (B) is the dependent variable corresponding to the last value of the independent variable (γ_2) and the third value (I), is the dependent variable corresponding to the geometric mean of the independent variables γ_1 and γ_2: $\gamma_3 = \sqrt{\gamma_1 \times \gamma_2}$; 3) If the value of I, corresponding to γ_3, is not found in the table, it must be interpolated using the Lagrange method with four points, considering two points before and two points after the value of γ_3; 4) The value of τ_0 is then replaced into the Herschel-Bulkley rheological model: $\tau = K(\gamma)^n + \tau_0$; 5) The Herschel-Bulkley rheological model is mathematically linearized, and the least squares method (LSM) is applied, taking the form: $\ln(\tau - \tau_0) =$

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$lnK + n \times ln\gamma$; This is a linear equation of the form: $y = A + Cx$; where: $y = \ln(\tau - \tau_0)$, $x = ln\gamma$ and $A = lnK$; 6) The linear regression statistical process can be performed on a computer or scientific calculator by entering the ordered pairs (x, y) in the form: $[\, ln\gamma, \ln(\tau - \tau_0)\ ]$; 7) After entering all the ordered pairs, the values of lnK and n, are obtained. 8) The value of n is the slope of the linear equation, i.e., the value of "C" in the linear equation $y = A + Cx$; the value of A is lnK and, and therefore $K = e^A$, from the regression analysis, we also evaluate the Pearson correlation coefficient, r, and the coefficient of determination ($r^2 \times 100$); thus determining the Herschel-Bulkley rheological model.

3. Results

After obtaining the juice of prickly pear (*Opuntia ficus indica*), partially pectinized and standardizing its concentrations (5, 15, 25, and 40 °Brix), rheological tests were performed using a concentric cylinder rheometer or viscometer (Rheotest) at a temperature of 20 °C. Prior to the rheological tests of the despectinized prickly pear juice (*Opuntia ficus indica*), the equipment was calibrated with a 5% sugar solution, which yielded viscosity values very close to those reported in the literature. Preliminary tests were conducted, where deformation data $\gamma(s^{-1})$ and shear stress $\tau\ (Pa)$, were measured, as shown in Tables 1, 2, 3, and 4, and their behavior in Figures 1, 2, 3, and 4, respectively.

Table 1.
Rheological data of prickly pear juice (Opuntia ficus indica) at a concentration of 5 °Brix.

$\gamma(^1/_s)$	$\tau(Pa)$
410.21	90.54
1970.04	151.91
3450.32	196.72
4970.84	234.70
8020.62	303.27
11150.72	365.47
14172.84	419.62
17419.24	475.66
20405.32	523.53
23374.42	569.16
24782.64	590.25
26201.64	610.89

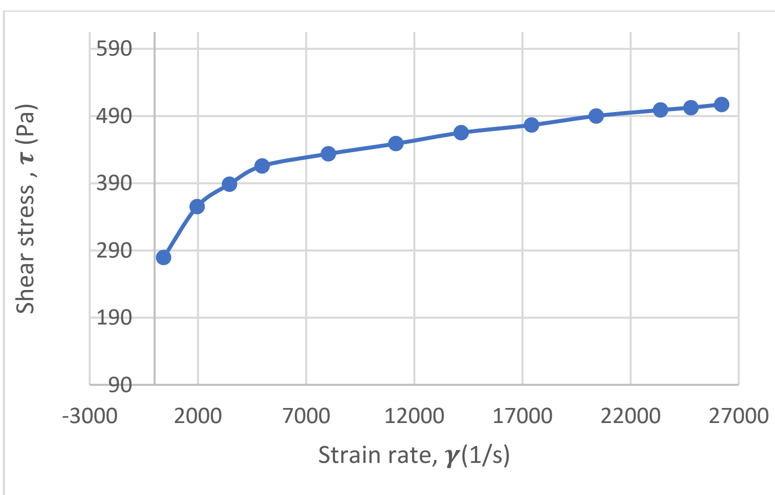

Figure 1.
Behavior of shear stress (τ) vs strain rate (γ) of prickly pear juice (*Opuntia ficus indica*) at a concentration of 5 °Brix.

Table 2.
Rheological data of prickly pear juice (Opuntia ficus indica) at a concentration of 15 °Brix.

$\gamma(^1/_s)$	$\tau(Pa)$
408.71	111.75
1976.74	212.72
3452.55	287.00
4968.92	354.50
8019.62	475.62
11148.55	589.12
14122.04	684.13
17421.15	780.45
20398.92	872.13
23371.33	958.42
24779.57	1002.41
26204.05	1043.78

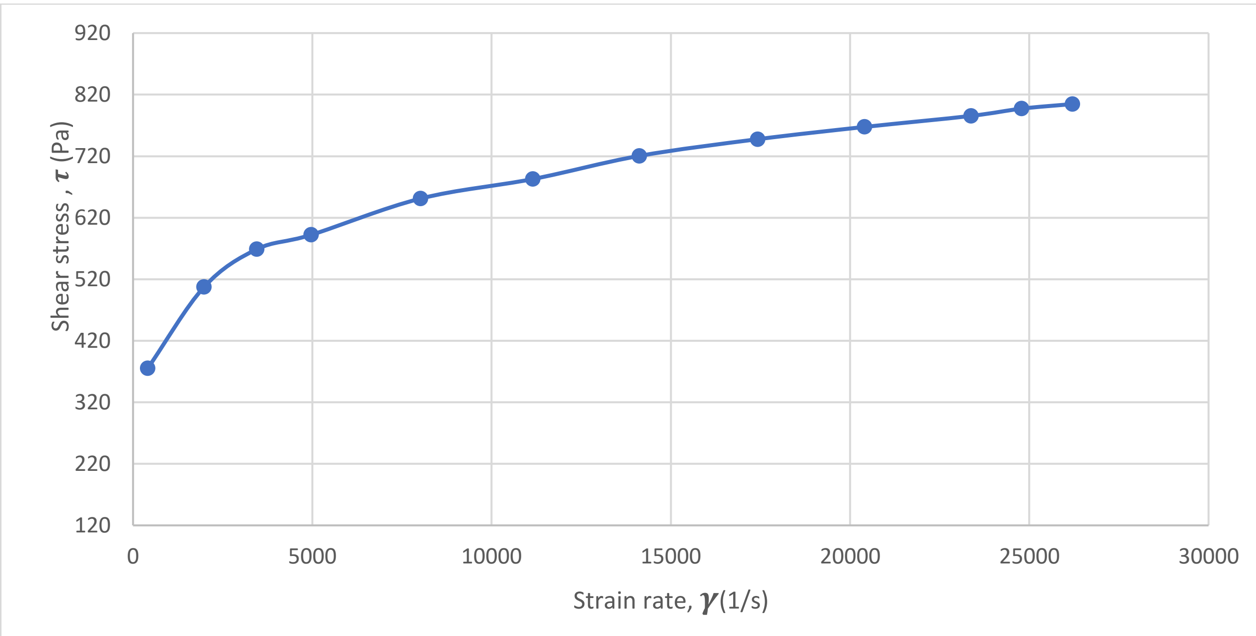

Figure 2.
Behavior of shear stress (τ) vs strain rate (γ) of prickly pear juice (Opuntia ficus indica) at a concentration of 15 °Brix.

Table 3.
Rheological Data of Prickly Pear Juice (Opuntia ficus indica) at a Concentration of 25 °Brix.

$\gamma(^1/_s)$	$\tau(Pa)$
412.28	159.62
1976404	370.62
3453.45	535.27
4975.84	689.60
8023.12	972.31
11154.48	1239.01
14174.23	1482.52
17425.11	1734.18
20412.23	1956.42
23372.35	2175.23
24780.45	2262.13
26210.21	2363.22

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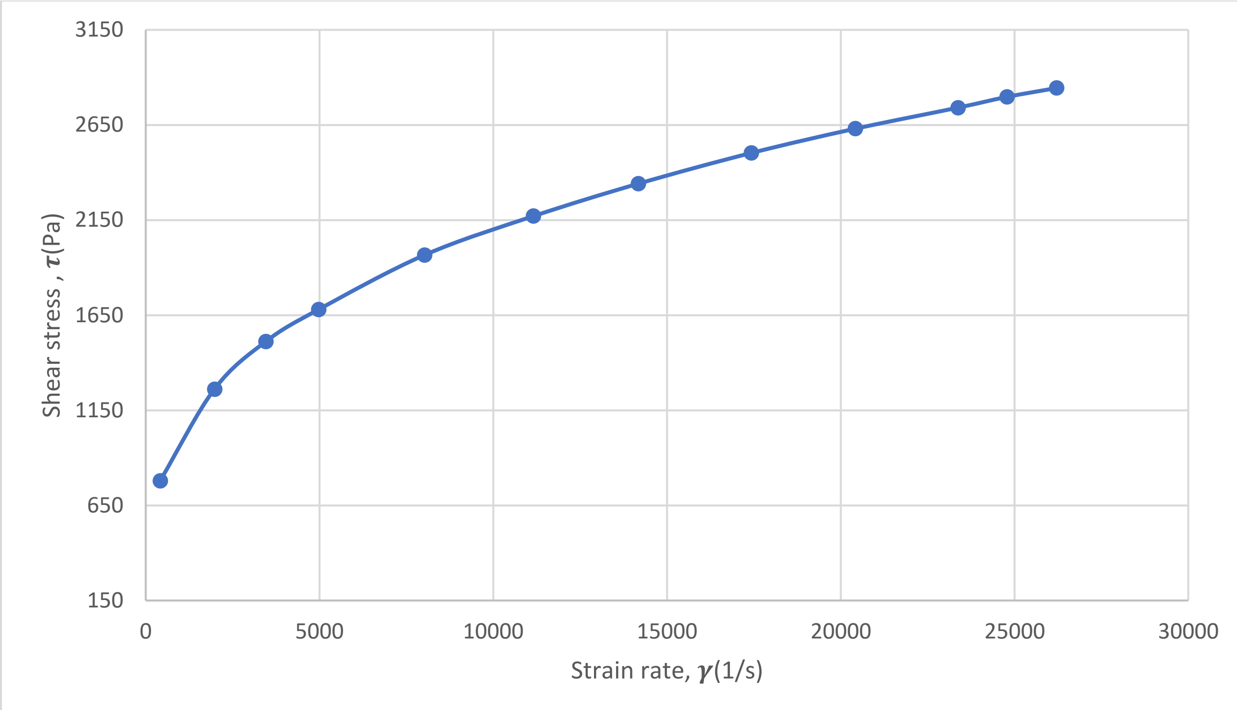

Figure 3.
Behavior of Shear Stress (τ) vs Strain Rate (γ) of Prickly Pear Juice (Opuntia ficus indica) at a Concentration of 25 °Brix.

Table 4.
Rheological data of prickly pear juice (Opuntia ficus indica) at a concentration of 40 °Brix.

$\gamma(^1/_s)$	$\tau(Pa)$
411.55	249.30
1969.54	692.15
3449.23	1060.82
4977.89	1423.60
8022.36	2094.76
11149.89	2754.32
14177.56	3365.76
17424.01	4000.76
20411.12	4570.30
23375.12	5123.10
24784.56	5383.70
26208.02	5632.12

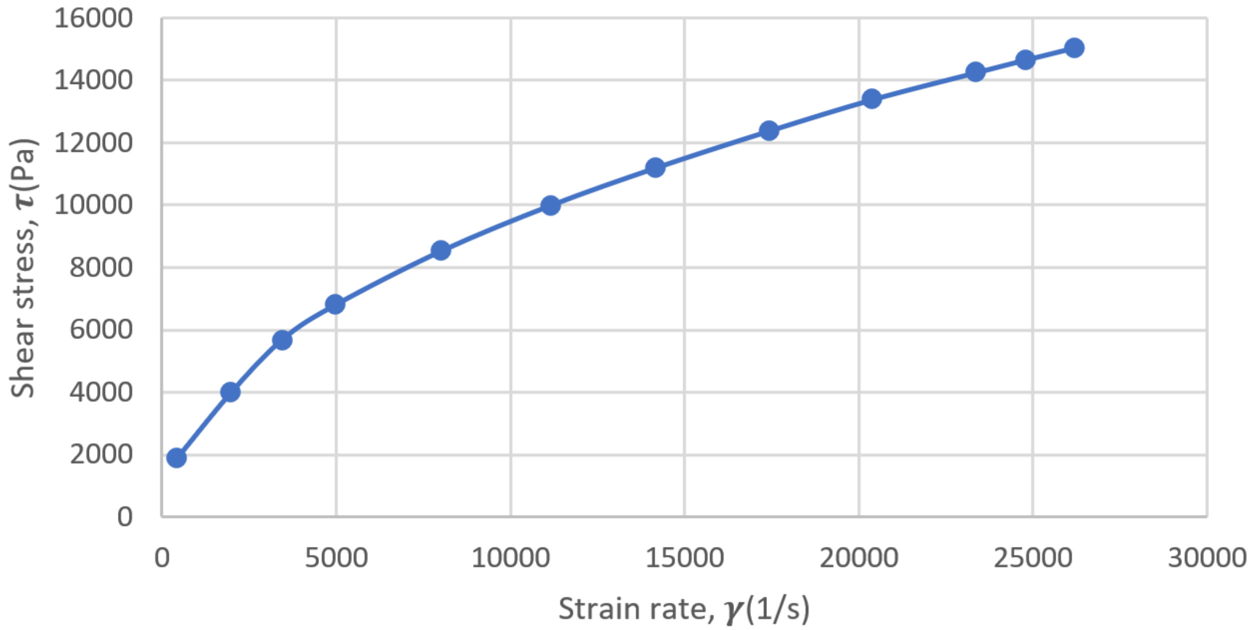

Figure 4.
Behavior of Shear Stress (τ) vs Strain Gradient (γ) of Prickly Pear Juice (Opuntia ficus indica) at a Concentration of 40 °Brix.

3.1. Obtaining Rheological Models and Statistical Coefficients

Calculation of Yield Point $\left(\tau_0 = \frac{A\times B - I^2}{A+B-2I}\right)$, Consistency Index (K), Flow Index (n), Pearson's Regression Coefficient (r) and Coefficient of Determination ($r^2 \times 100$).

- For the temperature of $T = 20\ °C$ and the concentration $C = 5\ °Brix$

$$\gamma_1 = 410{,}21;\ A = 90{,}54$$
$$\gamma_2 = 26\ 201{,}64;\ B = 610{,}89$$
$$\gamma_3 = \sqrt{410{,}21 \times 26\ 201{,}64} = 3\ 278{,}44$$

For this value of $\gamma_3 = 3\ 278{,}44$; the interpolation (by Lagrange) gives: $I = 192{,}02$. Replacing the corresponding values, we get:

$$\tau_0 = \frac{90{,}54 \times 610{,}89 - 192{,}02^2}{60{,}54 + 610{,}89 - 2(192{,}02)} = 58{,}0935$$

The value of τ_0 is replaced in the rheological model: $\tau = K(\gamma)^n + 58{,}0935$, it is linearized as $\ln(\tau - 58{,}0935) = lnK + n \times ln\gamma$ and linear regression is performed; obtaining the values of $K = 1{,}3742$; $n = 0{,}5826$; $r = 0{,}9883$ and $r^2 \times 100 = 97{,}67\ \%$; thus determining the Herschel Buckley rheological model: $\tau = 1{,}3742(\gamma)^{0{,}5826} + 58{,}0935$

- For the temperature of $T = 20\ °C$ and the concentration $C = 15\ °Brix$

$$\gamma_1 = 408{,}71;\ A = 111{,}75$$
$$\gamma_2 = 26\ 204{,}05;\ B = 1\ 043{,}78$$
$$\gamma_3 = \sqrt{408{,}71 \times 26\ 204{,}05} = 3\ 272{,}5218$$

For this value of $\gamma_3 = 3\ 272{,}5218$; the interpolation (by Lagrange) gives: $I = 278{,}6246$. Replacing the corresponding values, we get:

$$\tau_0 = \frac{111{,}75 \times 1043{,}78 - 278{,}6246^2}{111{,}75 + 1043{,}78 - 2(278{,}6246)} = 65{,}2047$$

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The value of τ_0 is replaced in the rheological model: $\tau = K(\gamma)^n + 65{,}2047$, it is linearized as $\ln(\tau - 65{,}2047) = lnK + n \times ln\gamma$ and linear regression is performed; obtaining the values of $K = 0{,}5776$; $n = 0{,}7302$; $r = 1{,}00$ and $r^2 \times 100 = 100{,}00\ \%$; thus determining the Herschel Buckley rheological model: $\tau = 0{,}5776(\gamma)^{0{,}7302} + 65{,}2047$

- For the temperature of $T = 20\ °C$ and the concentration of $C = 25\ °Brix$

$$\gamma_1 = 412{,}28;\ A = 159{,}62$$
$$\gamma_2 = 26\ 210{,}21;\ B = 2\ 363{,}22$$
$$\gamma_3 = \sqrt{412{,}28 \times 26\ 210{,}21} = 3\ 287{,}2398$$

For this value of $\gamma_3 = 3\ 287{,}2398$; the interpolation (by Lagrange) gives: $I = 517{,}8254$. Replacing the corresponding values, we get:

$$\tau_0 = \frac{159{,}62 \times 2\ 363{,}22 - 517{,}8254^2}{159{,}62 + 2\ 363{,}22 - 2(517{,}8254)} = 73{,}3424$$

The value of τ_0 is replaced in the rheological model: $\tau = K(\gamma)^n + 73{,}3424$, it is linearized as $\ln(\tau - 73{,}3424) = lnK + n \times ln\gamma$ and linear regression is performed; obtaining the values of $K = 0{,}7401$; $n = 0{,}7901$; $r = 1{,}00$ and $r^2 \times 100 = 100{,}00\ \%$; thus determining the Herschel Buckley rheological model: $\tau = 0{,}7401(\gamma)^{0{,}7901} + 73{,}3424$

- For the temperature of $T = 20\ °C$ and the concentration of $C = 40\ °Brix$

$$\gamma_1 = 411{,}55;\ A = 249{,}30$$
$$\gamma_2 = 26\ 208{,}02;\ B = 5\ 632{,}12$$
$$\gamma_3 = \sqrt{411{,}55 \times 26\ 208{,}02} = 3\ 284{,}1910$$

For this value of $\gamma_3 = 3\ 284{,}1910$; the interpolation (by Lagrange) gives: $I = 1\ 020{,}9534$. Replacing the corresponding values, we get:

$$\tau_0 = \frac{249{,}30 \times 5\ 632{,}12 - 1\ 020{,}9534^2}{249{,}30 + 5\ 632{,}12 - 2(1\ 020{,}9534)} = 94{,}2155$$

The value of $\boldsymbol{\tau_0}$ is replaced in the rheological model: $\tau = K(\gamma)^n + 94{,}2155$, it is linearized as $\ln(\tau - 94{,}2155) = lnK + n \times ln\gamma$ and linear regression is performed; obtaining the values of $K = 0{,}8700$; $n = 0{,}8611$; $r = 1{,}00$ and $r^2 \times 100 = 100{,}00\ \%$; thus determining the Herschel Buckley rheological model: $\tau = 0{,}8700(\gamma)^{0{,}8611} + 94{,}2155$

Table 5.
Rheological and statistical parameters of the models obtained for the prickly pear juice at a temperature of 20°C.

°Brix	Yield stress (τ_0)	Consistency index (K)	Flow index (n)	Regression coefficient (r)	Coefficient of determination ($r^2 \times 100$)
5	58.0935	1.3742	0.5826	0.9883	97.67
15	65.2047	0.5776	0.7302	1.0000	100.00
25	73.3424	0.7401	0.7901	1.0000	100.00
40	94.2155	0.8700	0.8611	1.0000	100.00

4. Discussion

The prickly pear juice (Opuntia ficus indica) exhibited variable rheological behavior, which is of great importance in food engineering processes, and has gained even greater importance in human consumption due to its inherent qualities, in agreement with the findings reported by Costell and Durán [5] and Torre [6]. The growing interest in consuming prickly pear juice increases the need to meet nutritional and protein requirements while providing relevant health benefits for the population across all social strata, races, and conditions, consistent with the reports by Osorio and Mejía-España [10]. The Herschel-Buckley model proved to be ideal for fitting the experimental data, characterizing the rheology of prickly pear juice, and quantitatively expressing the flow behavior of the juice, behaving as an inelastic, time-independent food fluid, as noted by Kokini [14]. The determination of the temperature effect on the rheological properties of prickly pear juice is essential for calculating the flow of non-Newtonian fluids using the flow index (n) with values for concentrations of 5, 15, 25, and 40 °Brix being 0.5826, 0.7302, 0.7901, and 0.8611, respectively, in agreement with the findings of Barreiro, et al. [16]. The flow index of prickly pear juice increased progressively and was directly proportional to the concentration, in accordance with the findings of Barboza-Mejía and Velásquez-Barreto [18]. The yield stress (τ_0) was directly proportional to concentrations of 5, 15, 25, and 40 °Brix, with values of 58.0935, 65.2047, 73.3424, and 94.2155, respectively, in line with the results proposed by Chen and Wu [20]. It is concluded that the consistency index of prickly pear juice decreases and increases within the concentration range studied, that yield stress and flow index increase, and that the correlation coefficients (r) and determination ($r^2 \times 100$) show a perfect correlation between shear stress (τ) and deformation gradient (γ). Further studies on different concentrations of prickly pear juice are recommended, using temperature intervals of up to 0.5 °C, utilizing other types of equipment to determine additional parameters beyond those found in this study, and with juices from other fruits contributing different structural components. More frequent measurements across temperature intervals should be conducted to obtain a more detailed profile of how rheological patterns change with temperature and concentration, which would improve the accuracy of mathematical modeling and the prediction of prickly pear juice behavior.

Transparency:

The authors confirm that the manuscript is an honest, accurate, and transparent account of the study; that no vital features of the study have been omitted; and that any discrepancies from the study as planned have been explained. This study followed all ethical practices during writing.

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