



Rheological behavior of concentrated tucupi

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Abstract

Tucupi, which is widely enjoyed in the North region of Brazil, is a fermented liquid derived from cassava (*Manihot esculenta* Crantz) and has been taken abroad for its exotic characteristics. This study aimed to assess the rheological behavior of tucupi with 30, 35, and 40% solids and concentrated at 50, 70, and 90 °C. The rheological data were obtained at 25, 40, 60, and 80 °C with increasing and decreasing shear rates. Rheology at 25 °C indicated that the partial gelification of starch during concentration causes a decrease in the product's viscosity and, if the concentration is carried out at a temperature that favors total starch gelification, the product's viscosity increases. Concentrated tucupi behaved as a pseudoplastic fluid, but at 60 and 80 °C with increasing shear rates, the product behaved as a dilatant fluid. Hysteresis were observed in flow curves starting at 40 °C, which characterizes concentrated tucupi as a rheopectic fluid. The Ostwald-de Waele model predicted the product's flow curves and an Arrhenius-like equation described the dependence of temperature on apparent viscosity for the rheological data obtained with increasing shear rates. The product's activation energy (E_a) values ranged from 16.86 to 25.23 kJ/mol as a function of concentration.

Keywords: *Manihot esculenta*; cassava wastewater; concentration; temperature; rheopectic fluid.

Practical application: Concentration and temperature have influence on the rheological behavior of tucupi.

1 Introduction

Tucupi, a fermented product obtained from cassava (*Manihot esculenta* Crantz) roots, is produced and widely consumed in the North region of Brazil as an ingredient to prepare several foods and as sauces (Costa et al., 2017a; Costa et al., 2017b; Pires & Silva Pena, 2017). Although tucupi has low pH (3.0 - 3.4) and high acidity (3.9 - 10.7 meq NaOH/100 mL), it is a liquid product and, hence, virtually entirely water (94.6 - 97.5% moisture), which makes it prone to biochemical and microbiological spoilage processes (Chisté et al., 2007).

Liquid foods are often concentrated as a preservation method and this is a key step for the industry when dealing with fluid products with low solids content, particularly as a pre-treatment for drying. The concentration process decreases the product's volume and weight, which results in lower storage, packaging, and shipping costs. Water activity also decreases, which increases microbiological and biochemical stability (Brennan et al., 1999; Silva et al., 2005; Vasconcelos & Melo Filho, 2010). Concentrating tucupi is an alternative for better product preservation and for enabling its use in new food products.

Knowing the physical phenomena associated with the flow (plastic deformation) of fluid foods is of uttermost importance for designing and sizing equipment such as pumps and pipes, stirrers, heat exchangers, homogenizers, and extruders, among others. These parameters are also important for quality control, for defining conditions to process and store the product, and

for establishing the product's shelf life (Steffe, 1996; Rao, 1999; Abu-Jdayil et al., 2002; Silva et al., 2005).

The rheological behavior of a fluid can be Newtonian or non-Newtonian, whether dependent or independent of time, given its origin, composition, and structural behavior (Rao, 2014). During processing, storage, shipping, and consumption, a fluid food's concentration and temperature may vary, which makes it important to know its rheological properties as a function of these parameters (Ibarz & Cánovas, 2002). Such information is important for the sizing of unit operations such as thermal treatment and concentration (Silva et al., 2005).

Tucupi is a product frequently used in the form of concentrated sauces. Thus, since the scientific literature carries no information on the rheology of concentrated tucupi, this research aimed to investigate the product's rheological behavior at different concentrations and obtained at different concentration temperatures, as well as to assess the effect of temperature on the product's rheology.

2 Material and methods

2.1 Sample

The tucupi used in the research was obtained from a producer in the city of Acará (1° 57' 39" S and 48° 11' 48" W), Pará, Brazil. The study used boiled tucupi with no added spices or sodium chloride.

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2.2 Tucupi concentration

The tucupi was concentrated in a rotary evaporator (Marconi, São Paulo, Brazil) coupled to a vacuum pump (Quimis, 60 LPM, São Paulo, Brazil). The product was concentrated at low pressure at 50, 70, and 90 °C until the solids contents were 30, 35, and 40%. After the samples were concentrated, they were stored in amber flasks under refrigeration (≈ 5 °C) until the analyses.

2.3 Concentrated tucupi characterization

In order to characterize the concentrated tucupi, total solids, pH, total titratable acidity, and starch content were determined according to the Association of Official Analytical Chemists (2010). All analyses were carried out in triplicate.

2.4 Rheological measures

The rheological data of concentrated tucupi were obtained with a rheometer (Brookfield, R/S PLUS – SST, Massachusetts, USA) coupled to a temperature-controlled bath (Lauda Ecoline, RE 200, New Jersey, USA) and connected to a computerized system for data acquisition. The experimental data were obtained using the CR (Controlled-Rate) mode with the shear rate ranging from 0 to 450 s⁻¹ (ascending curve – up ramp) and from 450 to 0 s⁻¹ (descending curve – down ramp). A cone-plate system with C50-1 spindle and 1 mm spacing was used. The rheological curves were obtained at 25, 40, 60, and 80 °C and all assays were carried out in triplicate. Both tucupi and products based on tucupi are, in general, preserved at room temperature ($T > 25$ °C), which has motivated the use of temperatures starting at 25 °C in this study.

2.5 Mathematical modeling

In order to predict tucupi's rheological behavior in the different conditions studied, the goodness-of-fit of the Newton model (Equation 1) and Ostwald-de Waele model (Equation 2) to the experimental data was assessed. Only these models were used since the flow curves showed no initial stress for the product to start flowing within the experimental domain. The fits were performed by non-linear regression analysis, using Levenberg-Marquardt methodology and convergence criterion of 10⁻⁶. The coefficient of determination (R²), the reduced chi-squared value (χ^2), and the root mean square error (RMSE) were the parameters used to evaluate the fits. The values for the parameters of the fits of the rheological models to the experimental data underwent analysis of variance (ANOVA) and a complementary Tukey's test to compare the means at 5% significance ($p \leq 0.05$).

$$\tau = \eta \cdot \dot{\gamma} \quad (1)$$

$$\tau = k \cdot \dot{\gamma}^n \quad (2)$$

where, τ = shear stress (Pa); $\dot{\gamma}$ = shear rate (s⁻¹); η = viscosity (Pa.s); k = consistency index (Pa.sⁿ); and n = behavior index.

The apparent viscosity (η_a) values were calculated by Equation 3 using the values of τ calculated with the model that presented the best fit to the flow experimental data. The effect of temperature on apparent viscosity was assessed by an Arrhenius-like equation (Equation 4) (Steffe, 1996). From the angular coefficient of the

linear regression of η_a versus 1/T, the product's activation energy (E_a) value was obtained. The regression used η_a values determined for a shear rate of 100 s⁻¹ for every work temperature (25 - 80 °C).

$$\eta_a = \frac{\tau}{\dot{\gamma}} \quad (3)$$

$$k = A \cdot e^{\frac{-E_a}{RT}} \quad (4)$$

where, η_a = apparent viscosity (Pa.s); A = Arrhenius constant (non-dimensional); E_a = activation energy (kJ/mol); R = universal gas constant (8.314 J/mol.K); and T = absolute temperature (K).

3 Results and discussion

3.1 Concentrated tucupi characterization

The concentrated products, with 30 and 40% total solids, had the following characteristics, respectively: pH of 3.94 ± 0.01 and 3.24 ± 0.01, total acidity of 72.74 ± 0.87 and 90.18 ± 0.05 meq NaOH/100 mL, and 5.82 ± 0.31 and 7.76 ± 0.42% starch.

3.2 Rheological behavior of concentrated tucupi

The flow curves (τ versus $\dot{\gamma}$) and viscosity curves (η versus $\dot{\gamma}$) obtained at 25 °C for the concentrated tucupi are presented in Figure 1, where the effect of concentration and of concentration temperature on the product's rheological behavior can be seen. According to the behavior of the curves, both the increase in concentration and in concentration temperature of tucupi impacted the product's rheological behavior at 25 °C. Overall, concentration presented a greater effect on the product's rheological behavior, but the increase in concentration become more evident the effect of concentration temperature.

As a whole, concentrated tucupi's stress (Figure 1A) and viscosity (Figure 1B) decreased as concentration temperature increased for a constant $\dot{\gamma}$ value. However, the flow and viscosity curves obtained for the product concentrated at 90 °C are above those obtained for the product concentrated at 70 °C, which suggests higher stresses and viscosities for the former throughout the shear rate range studied. This behavior can be attributed to the gelification and downgrading of the starch in the product. According to Hoover (2001), cassava starch gelification occurs between 57 and 84 °C. That allows stating that no starch gelification took place in the product concentrated at 50 °C, while partial starch gelification took place in the concentration at 70 °C and full gelification took place in the concentration at 90 °C. Gelification occurs when the starch granules are heated in water, which causes structural disorganization and an irreversible swelling of the granules (Singh et al., 2003). As the granules expand, the amylase inside the starch structure goes to the aqueous phase, which increases the system's rheological properties (Tharanathan, 2002). When maximum swelling is reached, the granules begin to rupture and, thus, viscosity decreases, a process that can be intensified at higher shear rates. Downgrading occurs during cooling, when solubilized polymers from starch associate into an ordered structure once again. Depending on the type and concentration of the starch, the final structure may be a thick solution or a gel (Singh et al., 2003; Eliasson, 2006). Therefore,

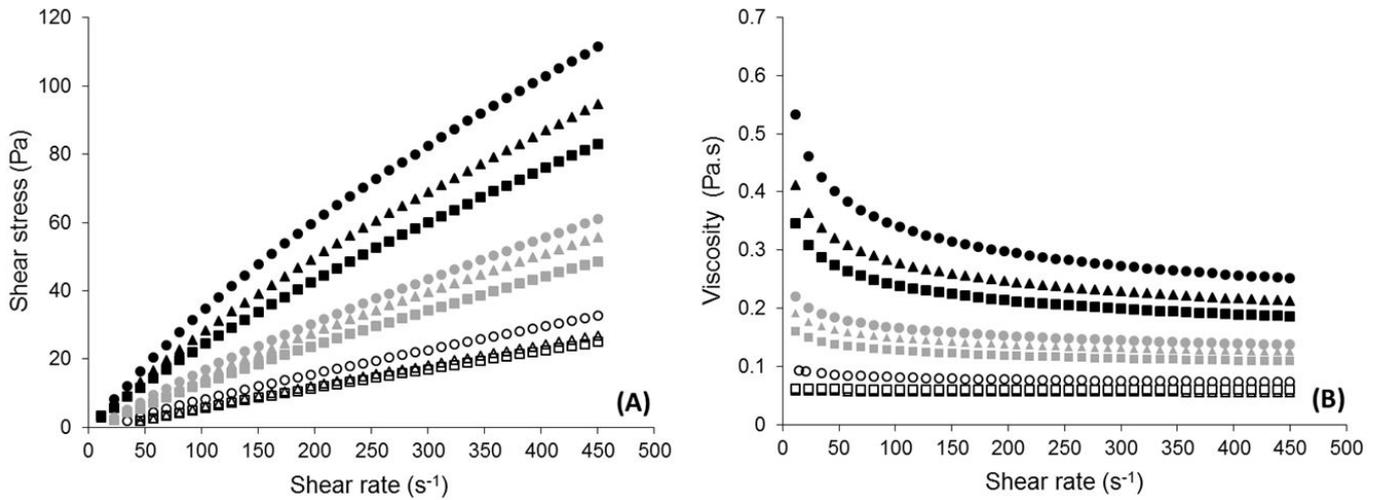


Figure 1. Effect of tucupi concentration and concentration temperature on the ratio between shear stress and shear rate (A) and between viscosity and shear rate (B). 30% solids (○ 50 °C, □ 70 °C, △ 90 °C); 35% solids (● 50 °C, ■ 70 °C, ▲ 90 °C); and 40% solids (● 50 °C, ■ 70 °C, ▲ 90 °C).

as full starch gelification occurred in the tucupi concentrated at 90 °C, its downgrading was also more marked, which justifies the behavior observed.

Irrespective of the concentration and concentration temperature, all concentrated tucupi samples showed typical non-Newtonian fluid behavior. The decreases in the tangent of the stress curves (Figure 1A) and of the viscosity curves (Figure 1B) as the shear rate applied increased classify the product as a pseudoplastic fluid (Schramm, 2000). Such effect was more visible as the product's concentration increased. Tecante & Doublier (1999), Zimeri & Kokini (2003), and Genccelep et al. (2015) observed a pseudoplastic fluid behavior for products with starch in their composition. This type of behavior was also observed by Horne (1998) and Lucey (2002) for protein-rich products. The pseudoplastic behavior can be explained by the weaker interactions among the molecules that make up the product as the shear rate applied increases, which leads to lower molecular interaction energy (Pasephol et al., 2008; Gozzo et al., 2009).

Figure 2 shows the flow curves (τ versus γ) and viscosity curves (η versus γ) for tucupi with 40% solids, where the effects of concentration temperature (50, 70, and 90 °C) and of the temperature at which the rheological analysis was carried out are assessed. According to the flow curves, when the rheological analysis was carried out starting at 40 °C, hysteresis, i.e., the area between the up-ramp and down-ramp curves (Holdsworth, 1993), were observed, which considerably increased with higher rheological analysis temperature. The fact that the down-ramp flow curves are above the up-ramp curves characterizes an increase in the product's viscosity over the shear time and shows an effect opposite to that of thixotropy, which is characteristic of rheopectic fluids (Holdsworth, 1993; Rao, 1999; Saravacos & Maroulis, 2001). Starch suspensions may have thixotropic characteristics at low concentrations and rheopectic characteristics as the starch concentration in the suspension increases (Tattiyakul & Rao, 2000).

Thixotropy is common for many fluids, while rheopecty is, indeed, very rare (Rao, 1999; Schramm, 2000). Rheopectic behavior is not commonly observed in foods, but has been observed in suspensions and concentrated starch pastes. Tárrega et al. (2005) observed a thixotropic behavior for gels with 4% modified corn starch and cassava starch, while a gel with 6% corn starch had rheopectic behavior. Tecante & Doublier (1999) and Nayouf et al. (2003) observed rheopectic behavior for reticulated waxy corn starch pastes. Those authors suggest that the rheopectic behavior can be explained by a denser packaging of the starch granules as shear rates increase.

3.3 Mathematical modeling

Tables 1 and 2 present the values for the parameters of the fits of the rheological models to the experimental data obtained for concentrated tucupi in the different conditions studied. According to the results, the Ostwald-de Waele model had better fits ($R^2 > 0.998$; $\chi^2 < 1.466$; RMSE < 0.194) than the Newton model ($R^2 > 0.969$; $\chi^2 < 30.491$; RMSE < 0.884) for the rheological data obtained at 25 °C (Table 1). Under this condition, no hysteresis effect was observed. In the experimental conditions under which this phenomenon was observed (40 - 80 °C) (Table 2), the Ostwald-de Waele model had excellent fits to the data obtained with increasing (up-ramp) shear rates ($R^2 > 0.997$; $\chi^2 < 1.005$; RMSE < 0.163). For the data observed with decreasing (down-ramp) shear rates, the fits of the Ostwald-de Waele model were also very good, however, they were impaired by higher temperature during rheological data acquisition: 40 °C ($R^2 > 0.996$; $\chi^2 < 1.183$; RMSE < 0.176), 60 °C ($R^2 > 0.988$; $\chi^2 < 3.492$; RMSE < 0.299), and 80 °C ($R^2 > 0.953$; $\chi^2 < 17.185$; RMSE < 0.672).

The values of the fluid behavior index (n) obtained by the Ostwald-de Waele model (Tables 1 and 2) confirm that, overall, concentrated tucupi behaved as a pseudoplastic fluid ($n < 1$) regardless of the product's concentration or of the rheological analysis temperature. However, the increase in rheological

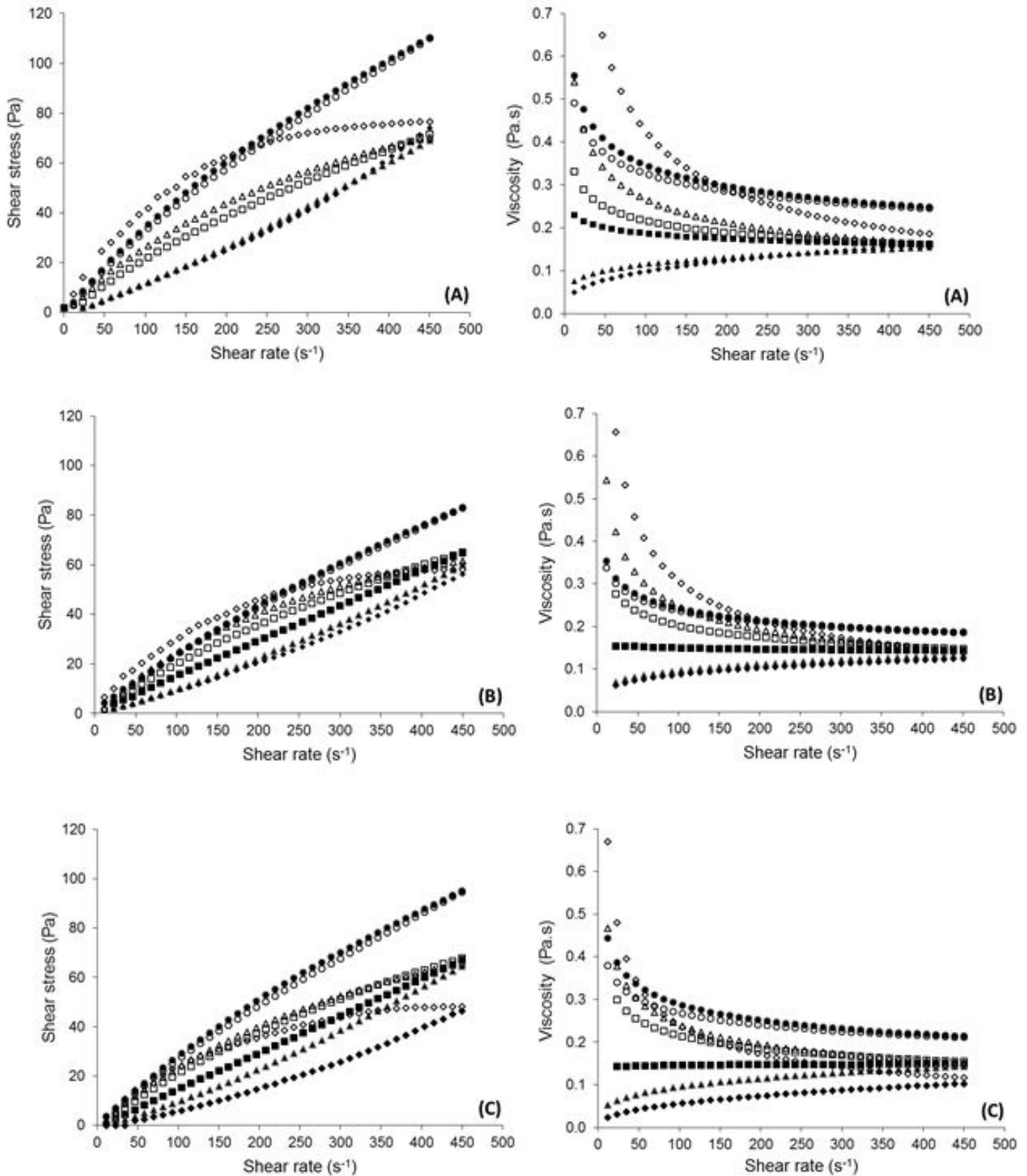


Figure 2. Ratio between shear stress and shear rate and between viscosity and shear rate for tucupi with 40% solids. Concentration temperature: (A) 50 °C, (B) 70 °C, and (C) 90 °C. Rheology temperature: (●) 25 °C (up ramp); (○) 25 °C (down ramp); (■) 40 °C (up ramp); (□) 40 °C (down ramp); (▲) 60 °C (up ramp); (△) 60 °C (down ramp); (◆) 80 °C (up ramp); (◇) 80 °C (down ramp).

Table 1. Values of modeling parameters for the rheological data obtained at 25 °C with the effect of tucupi concentration and concentration temperature.

Model	Parameter	Tucupi concentration/Concentration temperature								
		30%			35%			40%		
		50 °C	70 °C	90 °C	50 °C	70 °C	90 °C	50 °C	70 °C	90 °C
Newtonian	η (Pa.s)	0.075 ^f	0.056 ^g	0.060 ^g	0.143 ^d	0.113 ^e	0.130 ^{de}	0.269 ^a	0.196 ^c	0.226 ^b
	R ²	0.996 ^{ab}	0.998 ^a	0.998 ^a	0.988 ^d	0.992 ^{bc}	0.990 ^{cd}	0.969 ^f	0.981 ^e	0.978 ^e
	χ^2	0.356 ^g	0.077 ^h	0.080 ^h	3.356 ^d	1.398 ^f	2.342 ^e	30.491 ^a	10.519 ^c	16.126 ^b
	RMSE	0.098 ^f	0.046 ^f	0.047 ^f	0.297 ^d	0.192 ^e	0.248 ^{de}	0.884 ^a	0.519 ^c	0.643 ^b
Ostwald-de Waele	k (Pa.s ⁿ)	0.113 ^f	0.061 ^g	0.062 ^g	0.300 ^d	0.208 ^e	0.255 ^{de}	0.879 ^a	0.525 ^c	0.640 ^b
	n	0.929 ^b	0.986 ^a	0.997 ^a	0.871 ^d	0.894 ^c	0.883 ^{cd}	0.795 ^f	0.830 ^e	0.819 ^e
	R ²	0.999 ^a	0.998 ^a	0.998 ^a	0.999 ^a	0.999 ^a	0.999 ^a	0.998 ^a	0.999 ^a	0.999 ^a
	χ^2	0.105 ^{de}	0.074 ^e	0.082 ^e	0.286 ^c	0.125 ^d	0.268 ^c	1.466 ^a	0.276 ^c	0.687 ^b
	RMSE	0.053 ^d	0.045 ^d	0.048 ^d	0.087 ^c	0.057 ^{cd}	0.084 ^c	0.194 ^a	0.084 ^c	0.133 ^b

Mean values of the replicates with the same letters on the same row do not significantly differ among themselves ($p \leq 0.05$) according to Tukey's test.

Table 2. Values of modeling parameters for the rheological data obtained for tucupi at 40% solids for the different concentration temperatures and rheological analysis temperatures.

Model	Parameter	Rheological analysis temperature					
		40 °C		60 °C		80 °C	
		Up ramp	Down ramp	Up ramp	Down ramp	Up ramp	Down ramp
Concentration at 50 °C							
Newtonian	η (Pa.s)	0.166 ^{bc}	0.172 ^b	0.145 ^c	0.181 ^b	0.145 ^c	0.224 ^a
	R ²	0.994 ^a	0.968 ^a	0.986 ^a	0.893 ^b	0.966 ^a	0.370 ^c
	χ^2	2.559 ^d	11.833 ^c	5.719 ^d	41.009 ^b	15.220 ^c	228.697 ^a
	RMSE	0.256 ^d	0.558 ^{cd}	0.388 ^d	1.025 ^b	0.633 ^c	2.453 ^a
Ostwald-de Waele	k (Pa.s ⁿ)	0.293 ^d	0.536 ^c	0.047 ^e	1.206 ^b	0.022 ^e	5.348 ^a
	n	0.902 ^c	0.803 ^d	1.194 ^b	0.671 ^e	1.324 ^a	0.450 ^f
	R ²	0.998 ^a	0.998 ^a	0.998 ^a	0.994 ^a	0.998 ^a	0.955 ^b
	χ^2	0.240 ^d	0.759 ^c	0.022 ^d	2.562 ^b	1.005 ^c	17.185 ^a
	RMSE	0.079 ^d	0.141 ^c	0.024 ^d	0.256 ^b	0.163 ^c	0.672 ^a
Concentration at 70 °C							
Newtonian	η (Pa.s)	0.145 ^c	0.158 ^c	0.124 ^d	0.163 ^b	0.114 ^d	0.169 ^a
	R ²	0.998 ^a	0.962 ^b	0.982 ^a	0.854 ^c	0.980 ^a	0.506 ^d
	χ^2	0.152 ^e	11.855 ^c	5.208 ^d	43.122 ^b	5.368 ^d	110.788 ^a
	RMSE	0.063 ^e	0.559 ^c	0.370 ^d	1.052 ^b	0.376 ^d	1.685 ^a
Ostwald-de Waele	k (Pa.s ⁿ)	0.170 ^d	0.539 ^c	0.035 ^e	1.318 ^b	0.028 ^e	3.335 ^a
	n	0.973 ^b	0.787 ^c	1.218 ^a	0.637 ^d	1.240 ^a	0.482 ^e
	R ²	0.998 ^a	0.998 ^a	0.998 ^a	0.988 ^b	0.998 ^a	0.968 ^c
	χ^2	0.022 ^e	0.791 ^c	0.034 ^e	3.492 ^b	0.190 ^d	7.676 ^a
	RMSE	0.024 ^e	0.144 ^c	0.030 ^{de}	0.299 ^b	0.071 ^d	0.444 ^a
Concentration at 90 °C							
Newtonian	η (Pa.s)	0.148 ^b	0.166 ^a	0.132 ^c	0.168 ^a	0.091 ^d	0.137 ^c
	R ²	0.998 ^a	0.958 ^b	0.976 ^a	0.910 ^c	0.951 ^b	0.648 ^d
	χ^2	0.069 ^e	14.785 ^c	8.900 ^d	30.504 ^b	9.120 ^d	59.261 ^a
	RMSE	0.043 ^d	0.624 ^c	0.478 ^c	0.884 ^b	0.503 ^c	1.233 ^a
Ostwald-de Waele	k (Pa.s ⁿ)	0.137 ^d	0.601 ^c	0.027 ^e	0.989 ^b	0.009 ^f	2.149 ^a
	n	1.013 ^c	0.777 ^d	1.275 ^b	0.693 ^e	1.406 ^a	0.523 ^f
	R ²	0.998 ^a	0.996 ^a	0.998 ^a	0.994 ^a	0.998 ^a	0.976 ^b
	χ^2	0.039 ^d	1.183 ^c	0.067 ^d	2.116 ^b	0.065 ^d	4.019 ^a
	RMSE	0.032 ^c	0.176 ^b	0.041 ^c	0.233 ^b	0.043 ^c	0.321 ^a

Mean values of the replicates with the same letters on the same row do not significantly differ among themselves ($p \leq 0.05$) according to Tukey's test.

analysis temperature had a greater impact on this behavior for the flow curves obtained with decreasing (down-ramp) shear rates. On the other hand, $n > 1$ was observed for the flow curves obtained with increasing (up-ramp) shear rates when the rheological assays were carried out at 60 °C and 80 °C. Under these conditions, the product behaved as a dilatant fluid and this behavior became more evident as temperature increased both in the product concentration and in the rheological analysis.

Overall, the fluid's consistency index (k) decreased as the product's concentration temperature and rheological analysis temperature increased for the flow curves obtained with increasing (up-ramp) shear rates. In turn, the value of k increased as the rheological analysis temperature increased for all flow curves obtained with decreasing (down-ramp) shear rates regardless of the concentration temperature.

From a process point of view, operations such as pumping and filling of concentrated tucupi or sauces based on tucupi will be facilitated by conditions in which the product behaves as a pseudoplastic fluid. On the other hand, under conditions in which the product behaves as a dilatant fluid, these operations will be hampered (Rao, 2014).

3.4 Activation energy

The Arrhenius-like equation (Equation 4) represented very well the effect of temperature on apparent viscosity calculated by the Ostwald-de Waele model for the rheological data obtained with increasing (up-ramp) shear rates ($R^2 > 0.96$). On the other hand, for the data obtained with decreasing (down-ramp) shear rates, the fits were very poor ($R^2 < 0.21$). Thus, only the rheological data obtained with increasing shear rates can be used to calculate the activation energy (E_a).

The E_a values calculated for a shear rate of 100 s^{-1} were: 20.11 kJ/mol for the product concentrated at 50 °C; 16.86 kJ/mol for the product concentrated at 70 °C, and 25.23 kJ/mol for the product concentrated at 90 °C. Coutinho & Cabello (2005) observed an E_a value of 19.48 kJ/mol for a gel with 10% cassava starch. According to Rao (1999), polysaccharide gels can have E_a values between 10 and 273 kJ/mol. E_a values indicate the sensitivity of a rheological parameter as a function of temperature and the higher the E_a values, more sensitive a product is to rheological changes (Steffe, 1996). Therefore, the viscosity of the product concentrated at 90 °C showed the highest thermal sensitivity while the viscosity of the product concentrated at 70 °C was less susceptible to the effect of temperature.

4 Conclusion

The rheological study of concentrated tucupi showed that the product concentration at a temperature above 70 °C increases the product's viscosity and that, in general, concentrated tucupi behaves as a pseudoplastic fluid. Additionally, hysteresis were observed between the up-ramp and down-ramp flow curves at temperature from 40 °C, showing that concentrated tucupi is characterized as a rheopectic fluid. The Ostwald-de Waele model predicted with excellent accuracy the flow curves of the analyzed product and the dependence of apparent viscosity on temperature was well described by an Arrhenius-like equation. The activation energy values calculated for concentrated tucupi ranged from 16.86 kJ/mol to 25.23 kJ/mol.

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