Study of rheological properties of açai berry pulp: an analysis of its time-dependent behavior and the effect of temperature

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Abstract The industry of açai-based products has been growing in the last few years. Knowledge about the physical properties of açai pulp, including its rheology, is essential to the optimization of industrial processes. This work presents the rheological behavior of açai berry pulp in relation to the effects of shear rate, temperature, and time of shearing. The entire study was carried out in the temperature range of 10–70 °C. Açai pulp showed a non-Newtonian, pseudoplastic, and time-dependent behavior. Four upward and backward shear rate cycles were evaluated, resulting in complex hysteresis loops, in which thixotropy and anti-thixotropy zones were observed. Downward flow curves could be satisfactorily represented by the Power-Law rheological model. The stress profiles as a function of shear rate obtained in the first upward curves suggest a breakdown of the initial fluid structure at low shear rates. Tests were also carried out at a constant shear rate of 20 s⁻¹ and, in this case, the Weltman model of thixotropy satisfactorily fit the experimental data. The activation energy, which was calculated by the Arrhenius equation, was 29.0 kJ/mol. The achievements of this work may be useful to further studies about açai pulp rheology and may contribute to process design in the açai industry.

Keywords Açai · Fruit pulp rheology · Food thixotropy · Food anti-thixotropy · Non-Newtonian

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Nomenclature

d_p	Particle diameter
$d_{63.2}$	Rosin–Rammler–Bennet model parameter
m	Rosin–Rammler–Bennet model parameter
D_S	Sauter mean diameter
$\Gamma(p)$	Gamma function
σ	Shear stress
σ_e	Shear stress at steady state
σ_0	Yield stress
γ	Shear rate
k	Consistency index
n	Flow index
A_1	Weltman model linear coefficient of thixotropy
B_1	Weltman model angular coefficient of thixotropy
A_2	Hahn model linear coefficient of thixotropy
B_2	Hahn model angular coefficient of thixotropy
t	Time of shearing
η	Apparent viscosity
η_{eq}	Apparent viscosity at steady state
η^o	Pre-exponential constant of Arrhenius equation
E_a	Activation energy for the effect of temperature on açai pulp rheology
R	Ideal gases constant
T	Absolute temperature
$RSD_{mean} (\%)$	Relative standard deviation mean
SD	Standard deviation of an experimental point
M	Mean value of an experimental point
N	Number of experimental points

1 Introduction

Açai berry is a fruit native to the Amazon that has been receiving significant attention in recent decades due to scientific studies that have pointed out the potential association of açai consumption with many health benefits, including antioxidant, anti-inflammatory, anti-proliferative, and cardio-protective properties [1–3]. Açai is commercially valuable due to its widespread use in beverage and energy drink production. A wide variety of açai-based products have been commercialized, including smoothies, juices, energetic and isotonic drinks, dried pulp, and oils destined for the pharmaceutical industry [4].

Although different species of açai exist, only two of them are economically important: the most common, *Euterpe oleracea*, which is known as “açai-do-pará”, and *Euterpe precatoria*, known as “açai-do-amazonas” [4].

According to official Brazilian government data [5], Brazil produced approximately 1.1 million tons of açai berry fruit in 2016, which corresponded to a commercial value of 3.9 billion reais (Brazilian currency—approximately US\$ 1.2 billion). The major production comes from the state of Para, which produced 98.9% of the total Brazilian açai in 2016.

Açai pulp is extracted mechanically, usually in horizontal turning rods, which have a central mixer and a sieve at the bottom. Fruit is dropped into the top of the pulping machine, and water

is added to facilitate pulp extraction. An emulsion is then formed, which flows through the sieve and is subsequently frozen, resulting in the final açai pulp [6]. In some factories, açai pulp is pasteurized before freezing in order to inactivate undesirable enzymes and microorganisms. The manner in which the fruit is processed affects the overall quality of the final product, such as its color, antioxidant capacity, flavor, and rheology. The latter is highly influenced by the amount of water added for pulp extraction and continuous shearing during processing, which can cause unwanted structural breakdown of the fruit, making it undesirable to consumers [7, 8].

Although there are several studies about açai, most of them are related to its chemical characteristics and its nutritional and pharmaceutical properties [3, 9, 10]. Literature about its physical properties is scarce. Understanding more about these properties is important for new product development, quality control, industrial processing, etc. Among these physical properties, knowing about the rheological behavior of açai-based products may be helpful, for instance, in designing equipment and pumping systems. The best report on açai pulp rheology available in the literature is by Tonon et al. [7]. In this study, the authors characterized the dynamic and steady-state shear rheological properties of the pulp. Concerning the importance and growth of the açai industry, other studies about açai rheology and other physical properties are warranted.

Numerous aspects of food science and technology depend on characterizing and understanding the rheological aspects of a food material, which is essential, for instance, for standardized characterization of raw materials and innovative products or for prediction of heat and mass transfer coefficients [11]. Knowledge of rheological parameters is also necessary for process simulations that involve fluid motion [12, 13]. In summary, determination of rheological properties allows the optimization of unit operations, which helps the food industry to reduce economic resource waste [14].

Food fluids, such as açai pulp, are one of the most challenging systems to study rheology. They have a multi-compound structure, forming a dispersed phase in which fibers, proteins, lipids, and other insoluble biological compounds can be found. These compounds interact with each other and with the continuous phase (serum), which is formed by an aqueous solution of sugars, polysaccharides, salts, organic acids, and small peptides. The rheological properties of food fluids are defined by these interactions, which in most cases are affected by shearing stresses, which cause food fluids in general to have a variable viscosity at a given temperature [11]. In this case, viscosity changes with the applied shear rate; therefore, the fluid is considered as non-Newtonian. Low solid-concentrated liquid foods, such as juices and isotonic drinks, for example, are usually Newtonian fluids, that is, they have a constant viscosity at a certain temperature independently of the shearing applied. The rheological behavior of fluids may also be dependent on shearing time. In this case, the fluid may be thixotropic or anti-thixotropic (also known as rheopectic). In the former, flow resistance decreases with time, whereas in the latter, the opposite occurs [15].

Although little information about açai pulp flow behavior is available in the literature, there are plenty of studies on the rheological properties of other fruits and vegetables, as can be found in a review by Diamante and Umemoto [14]. Fruit pulp presents a shear thinning behavior, which means that the fluid viscosity decreases under shear strain. This characteristic flow was found, for instance, in siriguela (*Spondias purpurea* L.) [16], jaboticaba (*Myrciaria jaboticaba*) [17], blueberry [18], blackberry [19], and mango [20] pulps. Tonon et al. [7] also observed this flow behavior in açai pulp. Moreover, these authors noted that açai showed thixotropy, which is common in many other food materials [17, 18, 21].

Considering the importance of characterizing the rheology of a food material and the growth of the açai industry in these past few years, this work proposes an evaluation of the effects of temperature, shear rate, and shearing time on açai pulp rheological behavior.

2 Materials and methods

2.1 Materials

Açai berry pulp was procured from a local company. It was properly frozen and packed in polyethylene bags in 1-kg quantities. According to the manufacturer's information on the product label, the açai pulp was type C, according to Brazilian legislation, which classifies açai pulp in three classes depending on the solid content: A—more than 14%, B—more than 11 up to 14%, and C—from 8 up to 11% [22]. The label also indicated that the product was made entirely of water and açai pulp; hence, no other ingredient had been added.

The pulp contained in the manufacturer's package (1 kg) was thawed at 5 °C, poured into a 2-l plastic container, and divided into 12-ml portions in small polyethylene bags in order that a freshly thawed sample would be used in each analysis. Before portioning, the açai was smoothly homogenized with a glass rod. After portioning, the pulp was frozen again and kept at −18 °C.

2.2 Methods

2.2.1 Physical-chemical analysis

Firstly, the main chemical constituents of the pulp were determined. Moisture was quantified in a drying oven at 105 °C and the ash content, in a muffle furnace at 600 °C. Protein content was measured by using the Kjeldahl method [23].

Fiber content was measured by gravimetry according to the AOAC [23]. This method is based on treating a sample with acid and alkaline solutions at boiling temperature and taking the remaining insoluble matter after digestion as crude fiber.

Previously dried samples of açai pulp (obtained from moisture content analysis) were packed and sealed in filter bags (model F57 - Ankon Technology™), which consist of an inert and heat-resistant material properly developed for fiber determination. An empty bag was used as a blank. Firstly, the fat of the açai was extracted by manually soaking the bags in petroleum ether for 10 min. After the excess solvent was removed, the bags were placed in layers in a pile of stainless-steel plates, which was mechanically soaked for 30 min in H₂SO₄ solution (1.25% w/v) at the boiling temperature (~97 °C) using a fiber analysis vessel (Tecnal TE-149). Afterwards, the acid solution was dumped and the samples washed by soaking the bags for 5 min in boiling distilled water. The washing procedure was performed four times, dumping the previously used water and filling the bath with fresh water. After washing, the samples were soaked in boiling NaOH solution (1.25% w/v) for 30 min (~97 °C). Washing was repeated, and the bags were subsequently soaked manually in acetone for 5 min. Afterwards, the acetone was evaporated and the bags dried in an oven at 105 °C for approximately 4 h. Finally, the bags with the samples were burned in a muffle furnace at 600 °C in order to subtract the weight of the ashes.

Lipid content was determined according to Bligh and Dyer [24]. Digestible carbohydrates, which include mainly simple sugars and starch, and other compounds were calculated by subtracting all other components from the total mass to complete the analysis of açai composition. Analysis of each component was carried out in triplicate.

2.2.2 Particle size distribution (PSD)

PSD analysis of açai pulp was carried out by applying the technique of laser-ray diffraction using a Mastersizer 2000 (Malvern™ Instruments) diffractometer set to the diffraction index of Fraunhofer.

Before the runs, 20 ml of Calgon ((Na(PO₃)_n) at 25 g/l was diluted into 480 ml of distilled water, and this solution was used to calibrate the instrument's optical device, generating a blank reference. Then, approximately 20 ml of the açai pulp was poured into this solution, which was sonicated for 30 s in order to avoid particle coalescence. The displacement of the tip of the ultrasonic probe was set to 13.0 µm. The sample was kept agitating by a mixer at 2200 rpm, coupled to the Mastersizer. Subsequently, the laser-ray diffraction of the sample was measured, and the particle size distribution (PSD) was given by the instrument.

Results were expressed in terms of frequency and cumulative particle distribution. In the latter, the Rosin–Rammler–Bennet (RRB) model (Eq. 1) was fit to the experimental data. Moreover, the Sauter mean diameter (D_s), also known as surface-weighted mean-diameter, was calculated considering the model presented by Eq. 2a and 2b, which is used for PSD represented by RRB with $m > 1$. D_s is widely used in PSD characterization of food materials [25–27].

$$X(d) = \left(1 - \exp \left[- \left(\frac{d_p}{d_{63.2}} \right)^m \right] \right) \cdot 100\% \quad (1)$$

$$D_s = \frac{d_{63.2}}{\Gamma \left(1 - \frac{1}{m} \right)} \quad (2.a)$$

$$\Gamma(p) = \int_0^\infty \exp(-x) \cdot x^{p-1} dx \quad (2.b)$$

2.2.3 Rheological analysis

Rheological measurements were carried out with the Brookfield™ viscometer, model LVDV2T (low-viscosity digital viscometer) with a specific cylindrical compartment to contain the fluid (Small Sample Adapter from Brookfield™) and a thermostatic bath to control the temperature. Tests were performed with the aid of Rheocalc T software. The effects of shear rate, temperature, and shearing time were evaluated. Triplicate runs were performed at 10, 20, 30, 40, 50, 60, and 70 °C. The cone-plate Brookfield™ spindle SC4-21 was used in the tests performed with changing the shear rates, while the cone-plate spindle SC4-34 was used in the analyses carried out at a constant shear rate.

Before each experimental run, samples of açai pulp were thawed at room temperature (25 °C) 30 min before each rheological test. Subsequently, the thawed açai pulp was poured into beakers (50 ml volume) and smoothly homogenized with a lab spatula. Açai pulp was then poured into the viscometer compartment. New samples were used in each run.

Rheological measurements on a sample may be affected by the procedures applied before the fluid is loaded into the viscometer compartment [28]. Therefore, in order to standardize all the açai samples, a pre-shearing at 40 s^{-1} for 30 s followed by a resting time of 3 min was applied to the fluid before rheological data were taken. This procedure was sufficient to 233 eliminate earlier shear effects. Then, rheological analyses were carried out according to the sections.

Shear rate and temperature effects Shear stress and viscosity data recording was begun after the sample was maintained at a shear rate of 1.5 s^{-1} for 20 s. After the first data point was collected, the shear rate was raised by 0.5 s^{-1} every 5 s until it reached 5 s^{-1} and was subsequently increased by 1.0 s^{-1} every 5 s up to 40 s^{-1} . This step of shearing increase was named “UP.” Once the UP step was concluded, the backward shearing step (named “DOWN”) started by decreasing the shear rate from 40 s^{-1} to 1.5 s^{-1} following the same path as for UP but in reverse. These UP and DOWN loops were repeated four times on the same sample and were respectively named 1st UP, 1st DOWN, 2nd UP, 2nd DOWN, 3rd UP, 3rd DOWN, 4th UP, and 4th DOWN. The experimental design of these analyses is shown in Fig. 1. A 5th cycle was performed at 10, 40, and 70 °C; however, there was no considerable difference between this and the 4th cycle (data not shown). Therefore, four cycles were sufficient to consider a steady flow behavior in all the experimental runs.

Such UP and DOWN loops are widely used to check the occurrence of thixotropy in fluids [7, 21, 29]. When shear stress data are plotted against the shear rate, a thixotropic fluid will present a hysteresis loop, the area of which may be calculated in order to estimate the degree of thixotropy [28].

The Herschel–Bulkley (Eq. 3) and Power-Law (Eq. 4) rheological models were fit to the experimental results of shear stress (σ) vs. shear rate ($\dot{\gamma}$) over the entire shear rate range ($1.5\text{--}40 \text{ s}^{-1}$) by non-linear curve fitting applying the method of Levenberg–Marquardt using the software Statistica™ 7.0. The parameters σ_0 , k , and n were

Fig. 1 Experimental design of tests to study the effect of shear rate on the rheological behavior of açai pulp

estimated considering a relative error of 10^{-6} as the convergence criterion. Models were assumed to be statistically significant when the value of probability (p value) calculated for each parameter was less than or equal to 5%.

$$\sigma = \sigma_0 + k\gamma^n \quad (3)$$

$$\sigma = k\gamma^n \quad (4)$$

Hysteresis loops between each UP and DOWN curve in each cycle were calculated from their graphical area. Additionally, the area between the first upward (1st UP) and the last downward curves (4th DOWN) was determined. Each area was calculated numerically by applying the trapezoidal rule.

Shearing time and temperature effects In order to evaluate the time-dependent rheological behavior of açai pulp, viscosity and shear stress measurements were performed, maintaining a constant shear rate of 20 s^{-1} for 30 min followed by 30 min of rest, and then a new shearing step at 20 s^{-1} for an additional 10 min.

The Weltman (Eq. 5) and Hahn (Eq. 6) models, which have been used to represent thixotropic behavior in food fluids [20, 25], were fit to the experimental data achieved in the first 10 min of shearing. The equilibrium shear stresses (σ_e) were determined from the average of the last 5 σ values obtained in the first shearing step.

$$\sigma = A_1 - B_1 \cdot \ln t \quad (5)$$

$$\ln(\sigma - \sigma_e) = A_2 - B_2 \cdot t \quad (6)$$

Activation energy for the effect of temperature on apparent pulp viscosity was calculated by the Arrhenius model (Eq. 7), considering the steady-state viscosities reached after the first 30 min of shearing:

$$\eta_{eq} = \eta^0 \exp\left(\frac{E_a}{RT}\right) \quad (7)$$

3 Results and discussion

3.1 Açai composition

The composition of açai pulp is shown in Table 1. It was noted that almost half of the dried matter was composed of lipids (47.59%), which makes the açai pulp a food of high energetic value. The results of protein, lipid, and ash determination agree with the findings reported by Tonon et al. [7] and Carvalho et al. [30]. However, the amounts of digestible carbohydrates and fiber are discordant between our study and the previous two. This may be related to differences in açai growing and processing conditions.

Table 1 Chemical composition of açai berry pulp

Nutrient	%		
	Present work	Tonon et al. [7]	Carvalho et al. [30]**
Moisture ^(wm)	88.34 ± 0.13	86.01	—
Proteins ^(dm)	9.47 ± 0.23	10.69	8.56
Lipids ^(dm)	47.59 ± 0.10	48.24	52.43
Fiber ^(dm)	19.80 ± 0.34	31.67	7.87
Digestible carbohydrates and other compounds* ^(dm)	18.73 ± 0.29	3.55	26.44
Ash ^(dm)	4.41 ± 0.01	3.04	4.71

Results represent the mean value of triplicate runs with their respective standard deviation

wm wet matter, dm dry matter

*Calculated by subtracting all other components from the entire dry matter

**Mean values between three commercial açai pulps.

3.2 Particle size distribution

In a fluid, solid particles interact with each other and with the continuous phase (serum). The intensity of these interactions depends on many factors, including the solid concentration, size, and shape. Because these parameters have a tremendous effect on the rheological properties of a fluid, PSD is often analyzed in studies that involve rheological measurements [25–27].

Cumulative and frequency PSD of açai pulp are shown in Fig. 2, which indicates that açai pulp presented a monomodal PSD. The particle diameters were in the approximate range of 5–250 µm. The RRB model was a good fit to the experimental data ($R^2 = 0.998$), with parameter values of $d_{63.2} = 67.16$ µm and $m = 1.37$. The former parameter means that 63.2% of the total solids have an equivalent diameter equal or smaller than 67.16 µm. The Sauter mean diameter (D_s) was 19.93 µm. To the best of our knowledge, no other report about açai pulp PSD exists in the literature.

The PSD band observed for açai pulp was smaller and narrower than that of jaboticaba pulp [17], tomato [25], citrus [31], and mango [27] juices. The Sauter mean diameter of the açai pulp was approximately equal to that of mango juice ($D_s = 17.55$ µm) [27] and much smaller than that of tomato juice ($D_s \sim 180$ µm) [25].

Sato and Cunha [17] studied the effect of particle size distribution on the rheological properties of jaboticaba pulp. In this study, the flow properties of the initial pulp were compared with those of reconstituted pulps, and the two were found to have different PSDs. The authors observed that these parameters greatly affected the apparent viscosity, thixotropy, and pseudoplasticity of the pulp. It was noted that the apparent viscosity of the pulp increased linearly as the particle size increased from 64 to 550 µm. However, for larger particles, shearing forces promoted instability of the internal network, leading to sedimentation, which caused an abrupt decay of yield stress and the consistency index. When compared with the initial pulp, the reconstituted pulp had a narrower PSD band, a similar particle mean diameter, and a much higher viscosity.

Augusto et al. [25] studied the effect of high pressure homogenization on the rheological properties of tomato juice, as this process affects PSD. The apparent viscosity of the juice increased as the particle size decreased. This same behavior was observed by Zhou et al. [27]

Fig. 2 Cumulative (a) and frequency (b) particle size distribution of açai berry pulp

with mango pulp. According to these authors, smaller particles have higher surface areas, which creates a stronger interaction between the network that composes the system. Hence, foods are more likely to have higher viscosities when the particle size of the dispersed phase is smaller. However, there are many other factors that affect the rheological properties of a food, such as its solid content, composition, and shape [32].

3.3 Rheological behavior

Figures 3 and 4 show, respectively, the results of shear stress and apparent viscosity as a function of shear rate. Relative standard deviation means (Eq. 8) were 7.4, 3.1, 3.8, 6.1, 3.7, 6.5, and 6.1% for σ (or η) vs. $\dot{\gamma}$ curves at 10, 20, 30, 40, 50, 60, and 70 °C, respectively.

Fig. 3 Açai pulp shear stresses as a function of shear rate at different temperatures for upward and downward shear rate cycles

Fig. 4 Açai pulp apparent viscosities as a function of shear rate at different temperatures for upward and downward shear rate cycles

$$RSD_{mean}(\%) = \frac{\sum_{i=1}^N \left(\frac{SD_i}{M_i} \right)}{N} \times 100\% \quad (8)$$

Fig. 5 Example of açai pulp rheological behavior, showing thixotropy and anti-thixotropy for a hysteresis loop. These data represent the mean of triplicate runs for the second loop at 20 °C

Concerning the two models used for experimental data fitting, it was noted that the p values of the estimated parameters of the Herschel–Bulkley equation were greater than 5% for many of the σ vs. γ curves, and in many of the fittings, the estimated value of yield stress (σ_0) was negative, and hence, without physical meaning (data not shown). In the case of Power-Law fittings, p values were less than 1% for all curves. Nevertheless, this model fit better to the downward data points ($R^2 \geq 0.97$) than to the upward points ($R^2 \geq 0.93$, except the first curve) (Table 2). This result shows that even though açai pulp exhibited pseudoplastic behavior ($n < 1$), some deviations from this behavior occurred in the UP curves, especially at low temperatures. These flow deviations can be better observed by the sharp stress slope shown in Fig. 3 and the viscosity peaks in Fig. 4. While DOWN curves had a smooth σ vs. γ profile over all shear ranges, UP curves showed an increase in the slope stress curve for shear rates up to approximately 10 s^{-1} at temperatures from 10 to 30 °C and up to approximately 20 s^{-1} for temperatures from 40 to 70 °C. This deviation of flow behavior may likely be associated with breakdown of the initial structure of the fluid at the beginning of shearing, which results in a stress overshoot. As shearing continues, fibers and other dispersed solids are aligned in the same direction as the shear, which reduces the resistance of flow, and as a result, the stress slope decreases [15]. Although this effect is more evident on the first upward curve, it also occurs on the subsequent UP curves. This demonstrates that the pulp partially recovers its structure when subjected again to low stresses at subsequent downward shear rates. It is also noted that higher temperatures shift the stress overshoot to higher rates.

Tonon et al. [7] also observed a shear thinning behavior for açai pulp. However, the flow of the pulp studied by these authors was better represented by the model of Herschel–Bulkley, with yield stresses (σ_0), consistency (k), and flow indexes (n) ranging from 2.29 to 5.48 Pa, 0.16 to 1.18 $\text{Pa}\cdot\text{s}^n$, and 0.39 to 0.83, respectively, in the temperature range from 10 to 70 °C. These author's tests were carried out by applying three flow ramps (1st UP, DOWN, and 2nd UP) with shear rates ranging from 0 to 300 s^{-1} . The apparent viscosities of the pulp studied by Tonon et al. [7] were higher than those observed in the present work, and shear stresses were in the range of up to 10 Pa for shear rates between 0 and 40 s^{-1} . As in the present work, Tonon et al. [7] observed a sharp decrease in stresses after the 1st UP curve. Nevertheless, the differences between these reports and the findings of the present work may be related to the

Fig. 6 Hysteresis areas and the ratio between thixotropy and anti-thixotropy in each upward and downward shear rate loop.

Fig. 7 Apparent viscosity curves at a constant shear rate of 20 s^{-1} . Vertical bars represent standard deviation

differences in chemical composition of the açai pulp and the characteristics of the dispersed phase, which in turn may vary due several issues related to açai growing conditions, such as soil and climate, level of fruit ripeness, and how the açai was processed.

Growing conditions will impact directly on açai composition, such as the amount of fiber, protein, and lipid. These compounds create a network in the dispersed phase, which also interacts with the continuous phase. The strength of these interactions will govern the flow behavior of the fluid [11].

Moisture content is one of the most important factors affecting the rheology of fluid foods [8, 32, 33]. Since water is added during pulp extraction, this processing step will strongly affect the rheological properties of the pulp. Moreover, the characteristics of suspended solids immersed in the serum will also depend on how pulp extraction is carried out, and variables such as the speed of the mixer, amount of fruit loaded, design of the equipment, and mesh size of the sieve will impact on the quality of the dispersed phase, which includes particle sizes and PSD, affecting the rheology of the pulp. Additionally, shearing forces during fluid transportation may breakdown its structure irreversibly, causing significant changes in its rheological properties [8].

According to the literature, shear thinning behavior is very common in different fruit pulps. The Power-Law model was used to describe the flow behavior of other fruit pulps and purees, such as capuaçu (*Theobroma grandiflorum*) [34], guava [35], and blackberry [19]. However, in contrast to the present work, most studies point out that fruit pulps present a yield stress, as in the case of mango [20], blueberry [18], siriguela (*Spondias purpurea* L.) [16], and umbu (*Spondias tuberosa*) [36] pulps.

Usually, the consistency index (k) of non-Newtonian fluids decreases with temperature [14, 37]. Although this tendency was observed over the temperature range between 10 and 40°C , k tended to increase slightly at temperatures greater than 40°C . The flow indexes (n) ranged from 0.353 to 0.647, which were lower than the values found by Tonon et al. [7]. This means that the rheology of açai pulp in the present work was more sensitive to shear forces. Diamante and Umemoto [14] reported flow indexes for most berry fruits in the range of 0.25 to 0.60, which agrees with the findings of the present work.

It was noted that the stresses on the upward curves were different from those on the downward curves, which resulted in hysteresis (Fig. 3). This indicates that the fluid showed

Table 3 Parameters of the thixotropic models at a constant shear rate of 20 s^{-1}

Model	Parameter	Temperature						
		10 °C	20 °C	30 °C	40 °C	50 °C	60 °C	70 °C
Weltman (Eq. 3)	A_1	8.61	6.18	4.75	3.54	2.33	1.77	1.31
	B_1	0.79	0.59	0.47	0.37	0.25	0.19	0.14
	R^2	0.99	0.99	0.99	0.98	0.98	0.98	0.98
Hahn (Eq. 4)	σ_e	3.18	2.34	1.69	1.10	0.71	0.54	0.40
	A_2	1.03	0.69	0.45	0.05	0.39	0.66	0.96
	B_2	3.55×10^{-3}	4.85×10^{-3}	4.66×10^{-3}	4.09×10^{-3}	4.18×10^{-3}	4.19×10^{-3}	4.19×10^{-3}
	R^2	0.98	0.99	0.97	0.92	0.92	0.92	0.92

Table 4 Temperature effect on the Weltman parameters A_1 and B_1

Equation	a	k	R^2
$A_1 = a_{A1} \cdot \exp(-k_{A1} \cdot T)$	11.92	3.17×10^{-2}	0.99
$B_1 = a_{B1} \cdot \exp(-k_{B1} \cdot T)$	1.08	2.89×10^{-2}	0.99

time-dependent rheological behavior. In addition, stresses tended to decrease as the number of cycles increased, which indicates that açai pulp has thixotropy [28]. Nevertheless, this time-dependent behavior was not well established, since there are regions, mainly at low shear rates, where the stresses achieved on the UP curves were lower than those on the DOWN curves, and this in turn indicates anti-thixotropy (Fig. 5) [28]. As discussed previously, the breakdown of the initial structure of the fluid may be related to this behavior, as it is noted that anti-thixotropy occurs in the shear rate range until the stress overshoot is achieved. Hysteresis in tests performed with upward and downward shear rates were also reported in mango [20], jaboticaba [17], blueberry [18], and in the açai pulp studied by Tonon et al. [18].

According to Mewis and Wagner [28], low shear rates in up and down cycles, such as those applied in the present study, may induce structure formation in the fluid, which gives an anti-thixotropic behavior over a certain low shear range. The σ vs. γ profiles of starch dispersions reported by Wang et al. [38] showed similar flow behavior, with anti-thixotropic zones at low shear rates and thixotropic zones at higher rates. Wang et al. [38] assigned such behavior to the increase in intermolecular forces among the dispersed phase at low shear rates, which caused an increase in flow resistance. This flow profile has been reported in many studies on the rheology of starch dispersions [21, 39–41]. Although this behavior appeared in açai pulp, it is not suitable to predict that a similar mechanism of interaction within the network of the pulp may be occurring, especially because it is known that açai is poor in starch. To the best of our knowledge, this anti- and thixotropic behavior has not been reported for other fruit pulps.

Figure 6 shows the hysteresis areas and the ratio between thixotropy and anti-thixotropy for each loop and for the first and last curves (1st UP and 4th DOWN), indicated as “Global.” These areas are directly related to the degree of thixotropy or anti-thixotropy. A decreasing trend in hysteresis as the number of cycles increased was noted, indicating that the fluid approached a steady state. It was also observed that the effect of shearing time diminished as the temperature increased. This result is more evident when the “Global” hysteresis area is compared for each temperature. Considering the ratio between thixotropy and anti-thixotropy from a general point of view, higher anti-thixotropy was noted in the temperature range between 10 and 40 °C, while it decreased considerably from 50 °C upward.

Results of the tests performed at a constant shear rate of 20 s^{-1} showed a completely thixotropic behavior for all evaluated temperatures (Fig. 7). As expected, apparent viscosity decreased as temperature increased, and a steady state could be reached after 30 min of shearing. Açai pulp was unable to recover its structure, even after resting for 30 min after its first shearing.

The activation energy was 29.0 kJ/mol, which is considerably higher than that found for açai pulp by Tonon et al. [7], who reported an activation energy of 4.18 kJ/mol. This result may be related to the differences in açai particle size distribution and composition, mainly its fiber content. According to Diamante and Umemoto [14], fluid foods with lower fiber contents have higher activation energies. Indeed, the fiber content of açai pulp found in the present work was considerably lower than the amount of açai fiber measured by Tonon et al. [7]. Also,

the activation energy in the present work was higher than those reported for blackberry (18.27 kJ/mol) [19], blueberry (9.36 kJ/mol) [18], and siriguela (17.51 kJ/mol) [16] pulps.

Parameters of the thixotropic models (Eqs. 3 and 4) calculated by linear fittings are shown in Table 3. The equation that best represented açai thixotropy was the Weltman model. It was noted that A_1 and B_1 decreased exponentially with temperature (Table 4). Other studies about time-dependent rheological behavior of different food materials [42–44] also pointed out that A_1 and B_1 decreased as temperature increased. In contrast to the observations of the present work, Ramaswamy and Basak [44] observed a linear dependency of these parameters on temperature. This linearity was also present in the results of Dolores and Canet [43].

Considering that parameter B_1 is directly related to the rate of structure breakdown by the shear stress [20], these results point out that the steady state is reached faster as the temperature increases. In other words, thixotropy tends to decrease as the temperature increases. This agrees with the results reported for the tests performed with the UP and DOWN cycles (Figs. 3, 4, and 7).

Parameter A_1 , which is directly related to the initial value of shear stress [20], decreased as temperature increased. This was expected because temperature has an inverse effect on açai pulp viscosity.

Although the parameters A_2 and B_2 (Eq. 6) are analogous to the parameters A_1 and B_1 (Eq. 5), the Hahn model did not fit well over the entire temperature range, especially from 40 to 70 °C. As expected, the equilibrium stress (σ_e) decayed as temperature increased. This occurs because temperature has a negative effect on fluid viscosity. However, A_2 and B_2 did not show a clear tendency.

4 Conclusions

The effects of shear rate, temperature, and shearing time on the rheological behavior of açai berry pulp were evaluated. Tests performed with upward and downward shear rate cycles showed that açai pulp, like other fruit pulps, presents a shear thinning flow. Although the Herschel–Bulkley model has been widely used to describe the flow behavior of other fruit pulps, açai pulp rheology could not be described by this model. The downward σ vs. $\dot{\gamma}$ curves were better described by the Power-Law model, which means that no yield stress (σ_0) was significant in the flow of the pulp. The upward curves presented unusual behavior, with a stress overshoot between 10 and 20 s⁻¹, which likely indicates that a significant breakdown of the fluid structure occurs at these shear rates.

The differences between the upward and downward flow curves resulted in hysteresis, with anti-thixotropic-like loops at low shear rates and thixotropic-like loops at higher rates (above 10–20 s⁻¹, approximately). This behavior was unusual and was not reported in other studies about fruit pulp rheology.

When the açai pulp was subjected to a constant shear rate of 20 s⁻¹ for 30 min, it showed an entirely thixotropic behavior, which was best represented by the Weltman model. In these tests, açai was unable to recover its initial structure, even after resting for 30 min. This indicates that care must be taken during açai pulp processing in order to avoid a serious breakdown of its network.

The activation energy of the pulp was 29.0 kJ/mol, considerably higher than that of other fruit pulps, which indicates that the apparent viscosity of açai is more sensitive to temperature changes.

Concerning the importance of the emerging industry of açai-based products and the scarce information about açai pulp rheology available in the literature, the results obtained in the present work may be useful for the development and optimization of processing methods for this fruit. The rheological parameters found in this study may also be used in computational simulations that involve açai flow. Additionally, the achievements presented herein may contribute to further research about açai and the rheology of other fruit pulps.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflicts of interest.

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