PHYSICAL PROPERTIES OF THE AÇAÍ PULP IN FUNCTION OF TEMPERATURE

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Abstract. *Açaí pulp has recently been the focus of international interest as a functional ingredient in fruit juices, beverage blends, dietary supplements, and dairy products due to potential health benefits associated with its polyphenolic composition. The knowledge of the rheological behavior of food fluids in function of the temperature is essential for the quality control and also it is very important for the processing industry to design agitation and filtration equipments, and to ecomomize energy. Considering the consumer demand for processed foods with high quality, there is a need to define changes in rheological properties of foods in processing operations that may affect their overall acceptability. The aim of the present paper was to investigate the rheological behavior of açaí (*Euterpe oleracea *Mart.) pulp brands available commercially in the city of São Carlos (SP). To verify the influence of total solid content on rheology, açaí pulps diluted in distillated water were also analysed. The rheological measurements of the açaí pulps were carried out at 15ºC, 25ºC and 35ºC using a Brookfield rotational rheometer DVIII+ equipped with stainless steel cone-plate and concentric cylinders of smooth surface. The experimental data were evaluated and fitted according to the rheological models of Power Law and Herschel-Bulkley, in the 2nd up-cycle, and the determination coefficient (R^2) was used as a parameter to verify the goodness-of-fit. At any given temperature, the samples showed tixotropic behavior with both geometries, decreasing the apparent viscosity with shearing time. After three shear cycles, the thixotropic behavior was not observed and the samples showed shear thinning behaviour with yield stress. The determination coefficient (R^2) for both models was higher than 0.94 (except brand 2 at 15°C), indicating that both models showed a good fit to the experimental data for pulps. However, the Herschel-Bulkley model is more suitable to describe its rheological behavior once the power law model does not take account the yield stress.*

Keywords*: açai pulp, rheological behavior, physical properties, cross-flow microfiltration*

1. INTRODUCTION

Açai (*Euterpe oleracea* Mart.) is a fruit that grows on multi-stems of palm trees widely distributed in the Amazon estuary floodplains. Due to its highly perishable nature, the consumption and commercialization of açai have long been restricted to a regional level in Brazil. However, açai pulp has recently been the focus of international interest as a functional ingredient in fruit juices, beverage blends, dietary supplements, and dairy products due to potential health benefits associated with its polyphenolic composition (Gallori et al., 2004; Lichtenthäler et al., 2005; Pacheco-Palencia et al., 2007; Pozo-Insfran et al., 2004; Rogez, 2000). In order to minimize the loss of physical and chemical quality attributes of juices made from açai fruits, careful design of equipment for processing and handling is necessary.

The knowledge of the rheological behavior of food fluids is important to identify some of the food process engineering operations such as pipeline transport and mechanical separations, heat transfer operations, mass transfer processes and physical changes during processing in which flow and viscoelastic properties play an outstanding role (Krokida, 2001; Vélez-Ruíz, 2002). Rheological measurements have also been considered as an analytical tool in understanding changes in the structure and macromolecular conformation of food during processing (Ahmed and Ramaswamy, 2004).

Fluids are generally characterized by graphical analyses of the shear stress in function of the shear rate. Depending on their behavior under imposed shearing forces, materials are classified as Newtonian or non-Newtonian. Complex fluids, like fruit pulps, are in general non-Newtonian; the viscosity is not constant, but it is dependent on the shear rate. Non-Newtonian fluids are frequently found in different industrial processes, such as fruit juice clarification and concentration using microfiltration and ultrafiltration. Viscosity can be measured with rheometers which show different data of shear stress as a function of shear rate, characterizing the behavior of a fluid. The pseudoplastic behavior is common in most non-Newtonians fluids, mainly in fruit pulp or juices that contain high level of solids (Zeman and Zydney, 1996; Carrère, 2000; Queiroz and Fontes, 2008).

Fruit pulps are considered concentrated suspensions where solid particles (pulp) are dispersed into an aqueous medium (Bertola et al., 2003; Sato and Cunha, 2009). Rheology of food suspensions can be affected by several factors such as the total solid content, particle shape, particle size and particle size distribution, wetting properties of the suspended particles in the continuous phase, temperature and pH (Servais et al., 2002; Ahmed and Ramaswamy, 2004).

It has been reported that fruit pulp behave as non-Newtonian fluid as a result of complex interactions among soluble sugars, pectic substances, and suspended solids and therefore rheological parameters of various fruit pulps are needed for process control and engineering applications as design and optimization of processing units (Ahmed and Ramaswamy, 2004).

Data in the literature concerning physical properties such as rheological behavior of açai pulp and juice are scarce. Recently, Tonon et al. (2009) studied the rheological behavior of açai pulp at different temperatures under steady and dynamic shear conditions. In this work, it was observed a decrease in apparent viscosity with shear application time indicating thixotropic behavior, probably due to a complex interaction amongst the product components in açai pulp. The authors also verified that the açai pulp apparent viscosity presented Arrhenius dependence on the temperature, but the activation energy was lower than for other fruit derivatives, indicating a less pronounced effect of temperature on the apparent viscosity of açai pulp probably due to the presence of high fiber or solids contents.

Considering the consumer demand for processed foods with high quality, there is a need to define changes in rheological properties of foods in processing operations that may affect their overall acceptability. Taking into account these considerations, the aim of the present paper was to investigate the rheological behavior of different açai (*Euterpe oleracea* Mart.) pulp brands available commercially in the city of São Carlos (SP). To verify the influence of total solid content on rheology, açaí pulps diluted in distillated water were also analyzed.

2. MATERIAL AND METHODS

2.1. Raw material

In this work, two commercial frozen açai pulps manufactured in Belém city (Pará, Brazil) were purchased from distributers in São Carlos city (São Paulo, Brazil) and stored at -18°C. Samples were thawed at 4°C (refrigeration) according to the amount required and homogenized with a magnetic stirrer before performing the rheological measurements.

2.2. Rheological measurements

Rheological measurements of the açai pulps were carried out using a rotational rheometer DVIII+ (Brookfield Engineering Laboratory, Massachusetts, USA) equipped with stainless steel cone-plate and concentric cylinders of smooth surface. The geometries, concentric cylinder and cone-plate, were studied with spindles SC4-18 and CP-42, respectively. The measurements were made at 15ºC, 25ºC and 35ºC, and the temperature was controlled by a thermostatic bath coupled to the equipment, with a precision of 0.5ºC.

The measurements were performed in this temperature range considering which 10ºC-15ºC is the usual thawed pulp temperature, 25ºC is the temperature of processing of the pulp (microfiltration process), and 35ºC is the temperature of enzymatic activity utilized in the fruit juice industry.

For the cone and plate geometry was determined the narrow gap between the two surfaces ranging from 0.650mm to 1.300mm at 25ºC.

To evaluate and eliminate the thixotropic phenomenon, three flow ramps were performed in a range of shear stresses corresponding to shear rates from 0 to 200s^{-1}. All readings were taken in triplicate, utilizing a new sample for each repetition.

To verify the influence of total solid content on rheology, açaí pulps diluted in distillated water were also analyzed using the cone plate geometry at 15ºC, 25ºC and 35ºC.

2.3. Calculation of rheological parameters

The Power Law and Herschel-Bulkley models were fitted to the rheological data obtained for pulps, and the determination coefficient (R^2) was used as a parameter to verify the goodness-of-fit. The softwares Origin 7.5 and Statistica 8.0 were utilized to analyze the results obtained.

In the Power Law Model, two rheological constants *K* and *n* are required to characterize the flow behavior (eq. 1).

$$\tau = K(\dot{\gamma})^n \tag{1}$$

where τ is the shear stress, $\dot{\gamma}$ the shear rate. *K* is the Consistency Index and *n* is the Flow Behavior Index. The *K* value corresponds to the viscosity of Newtonian fluids (Krokida, 2001).

The Herschel–Bulkley model is one of a number of relations that are commonly used to describe the behavior of certain fluids, including various food products. This model relates the shear stress τ to the strain rate $\dot{\gamma}$ by the formula (Holdsworth, 1993):

$$\tau = \tau_0 + k(\dot{\gamma})^n \quad (2)$$

where τ_0 is the yield stress of the material, K is the Consistency Index, $\dot{\gamma}$ is the shear rate, and n is the Flow Behavior Index.

3. RESULTS AND DISCUSSION

3.1. Gap determination

The wall slip effect occurs in the flow of two-phase (or multi-phase) liquids in rheometers because of the displacement of the disperse phase(s) away from solid boundaries, leaving a lower-viscosity, depleted layer of liquid. In that case it appears that there is discontinuity in shear rate near the wall: close to the wall there is a thin fluid layer in which the shear amplitude is much larger than in the rest of the material. The liquids that give large slip effects in the appropriate geometries are concentrated solutions of high molecular weight polymers; suspensions of large or flocculated particles and emulsions of large droplet size (Barnes, 1995; Bertola et al., 2003).

To prevent this phenomenon using the cone-plate geometry, tests were performed with gaps between the two surfaces ranging from 0.650mm to 1.300mm. The dependence between the shear stress values and the gap between the cone and plate used to the steady state is showed in Figure 1. The curve obtained with 0.650mm, 0.715mm and 0.780mm gap showed the highest shear stress values, probably because the açai particle size is larger than the gap. On the other hand, the curve obtained with 1.040mm and 1.300mm gap exhibited smaller shear stress values, probably due to a non-homogeneous shear stress throughout the sample (Tonon et al, 2009). Large gaps can lead to apparent slip effect. According to the equipment stability and the difference on the rheological behavior of the brands (1 and 2), gap of 0.910mm were considered to analyze the brand 1, and gap of 1.300mm for brand 2.

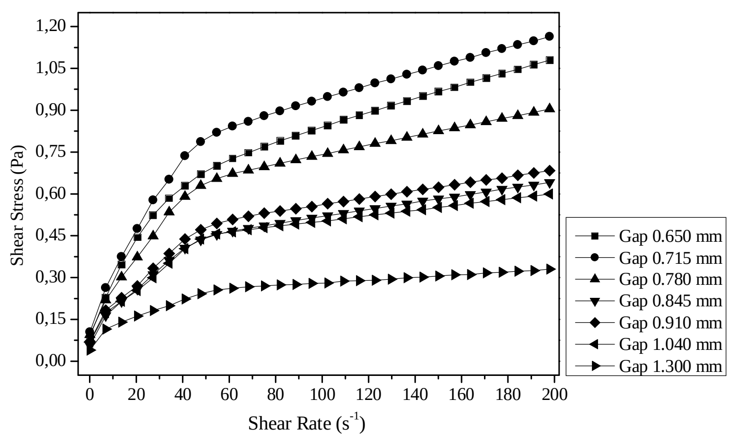

Figure 1. Flow curves of the açai pulp at 25ºC using cone-plate geometry for different gaps.

3.2. Rheological behavior

Both açai pulps showed thixotropic behavior at all temperatures (15ºC, 25ºC and 35ºC), with cone-plate and concentric cylinder geometries, decreasing the apparent viscosity with shearing time. This thixotropic behavior for both brands can be observed in Figs. 2 and 3, with cone-plate and concentric cylinder geometries, respectively. Similar results were also found in other products, such as jaboticaba pulp (Sato and Cunha, 2009), gilaboru juice (Altan et al., 2005), and mustard (Juszczak et al., 2004). Sato and Cunha (2009) verified which enhance of particle size promoted increase of thixotropy, viscosity, pseudoplasticity and yield stress of reconstituted jaboticaba pulps. In Figs. 2a and 2b, in the second up-cycle, higher values of stress or apparent viscosity were observed at low shear rates, as compared to the down-cycle. After three shear cycles, the thixotropic behavior was not observed. This effect can be attributed to breakdown of the inner structure of fluid which is formed through physical interaction between molecules (Moller et al. 2006).

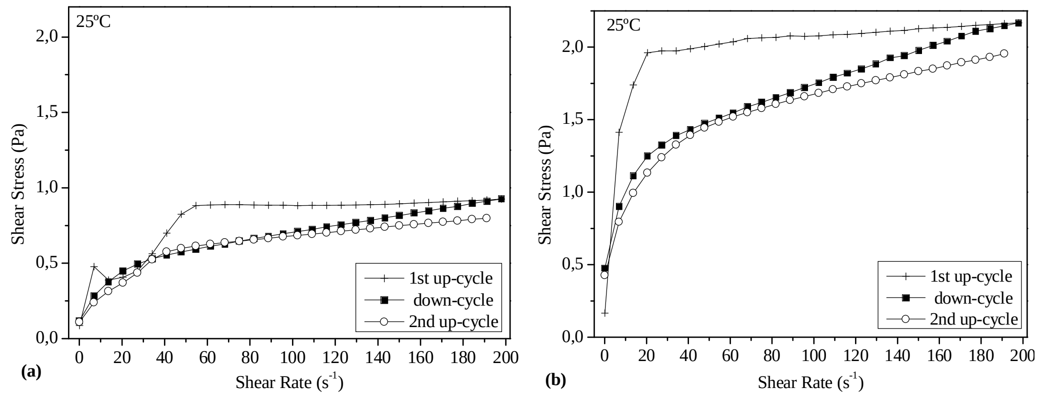

Figure 2. Flow curves of the açai pulps at 25ºC, using cone-plate geometry. Brand 1 (a) and brand 2 (b).

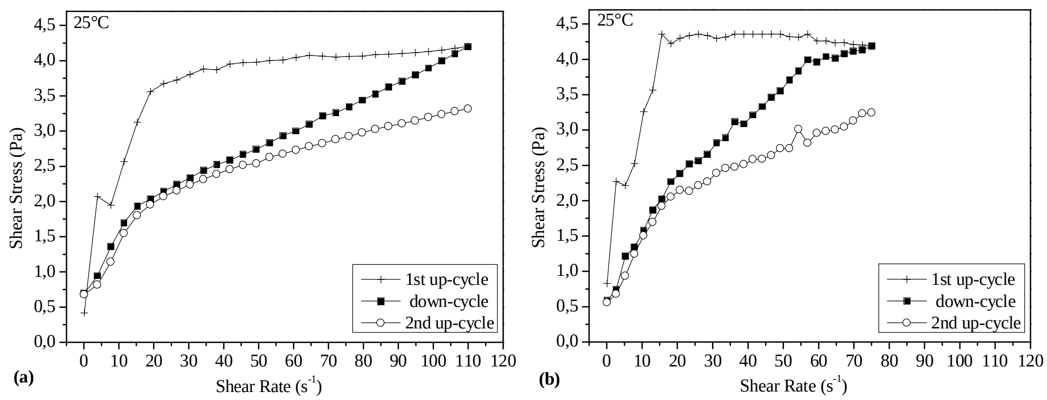

Figure 3. Flow curves of the açai pulps at 25ºC, using concentric cylinder geometry. Brand 1 (a) and brand 2 (b).

The experimental data were evaluated and fitted according to the rheological models of Power Law and Herschel-Bulkley, in the 2nd up-cycle, i. e., steady state. The rheological parameters for both models within temperatures of 15-35°C can be observed in Tables 1 and 2, for cone-plate and concentric cylinder geometries, respectively.

The determination coefficient (R^2) for both models was higher than 0.94 (except brand 2 at 15°C), indicating that both models showed a good fit to the experimental data (Tables 1 and 2) for pulps.

The rheological behavior of açai pulps using both cone-plate and concentric cylinder geometries was classified as non-Newtonian fluid for all temperature ranges and models studied. The non-Newtonian fluid is characterized by non-linear relationship between the applied shear stress (τ) and the shear rate ($\dot{\gamma}$). Conversely, the apparent viscosity is not constant and is a function of $\dot{\gamma}$ (Chhabra and Richardson, 1999).

The values of the flow behavior index (n) for the açai pulps ranged between 0.21 and 0.45 for Herschel-Bulkley model and ranged between 0.30 and 0.38 for Power Law model, considering the both rheometer geometries. It can be observed the flow behavior index was smaller than 1 for all cases, indicating pseudoplastic characteristics for the açai pulps analyzed. However, according to the flow curves of açai pulp which showed yield stress, the Herschel-Bulkley model is more suitable to describe its rheological behavior once the power law model does not take account this rheological parameter that is very important in industrial applications (Juszczak et al., 2004; Nindo et al., 2007).

The consistence index (K) decreased with increasing temperature for both açai pulp brands using the concentric cylinder geometry (Table 2), and for brand 2 using cone-plate geometry (Table 1). However, the constant K decrease with increasing temperature for Power Law model using the cone-plate geometry was observed only from 25 to 35°C for both brands (Table 1). This same finding was verified for brand 1 with Herschel-Bulkley model using the cone-plate geometry.

Similar results had been reported by other authors (Silva et al., 2005; Pereira et al., 2008; Vandresen et al., 2009). The rheological behavior of pasteurized carrot juice carried out using a rotational viscosimeter at temperatures between 8 and 85°C showed a pseudoplastic behavior and was well fitted to Herschel-Bulkley model (Vandresen et al., 2009). In the study conducted by Pereira et al. (2008) on rheological behavior of umbu pulp, the values for flow behavior index, n, were less than 1, which was indicative of the pseudoplastic behavior. The Herschel-Bulkley equation was found to be an adequate model to describe the flow behavior of the sample in this study, because values of R^2 greater than 0.98 were found. The rheological behavior of the industrialized acerola juice was studied at different soluble solids concentrations 4, 7, 10, 13 and 16°Brix and temperatures from 5 to 85°C, using a concentric cylinder rheometer. The authors observed that the best results were obtained using the Herschel-Bulkley model and the low values presented by the behavior index (0.338-0.759) confirmed the pseudoplastic behavior of the acerola juice (Silva et al., 2005).

Gehrke (1996) measured the apparent viscosity of the concentrated juice of cashew apple, orange, lemon and passion fruit with the aim of comparing the results obtained in the measurement systems and parallel plate and concentric cylinders. In this work differences in the value of apparent viscosity for the two rheograms was observed for the same temperature and concentration.

Table 1. Rheological parameters of Power-law and Herschel-Bulkley models at 15ºC, 25º and 35ºC, using cone-plate geometry for both brands.

Brand 1							
T (°C)	Power Law Model			Herschel-Bulkley Model			
	K (Pa.s)	n	R^2	τ_0 (Pa)	K (Pa.s)	n	R^2
15	0.176 ± 0.009	0.29 ± 0.02	0.99	0.06 ± 0.01	0.132 ± 0.008	0.335 ± 0.009	0.99
25	0.18 ± 0.01	0.29 ± 0.02	0.96	0.01 ± 0.05	0.18 ± 0.04	0.29 ± 0.03	0.97
35	0.16 ± 0.02	0.32 ± 0.02	0.94	-0.01 ± 0.07	0.16 ± 0.05	0.32 ± 0.05	0.94
Brand 2							
T (°C)	Power Law Model			Herschel-Bulkley Model			
	K (Pa.s)	n	R^2	τ_0 (Pa)	K (Pa.s)	n	R^2
15	0.57 ± 0.01	0.222 ± 0.05	0.99	0.167 ± 0.05	0.43 ± 0.04	0.26 ± 0.01	0.99
25	0.58 ± 0.02	0.217 ± 0.008	0.99	0.23 ± 0.05	0.39 ± 0.04	0.28 ± 0.02	0.99
35	0.30 ± 0.01	0.217 ± 0.008	0.98	0.13 ± 0.04	0.19 ± 0.03	0.28 ± 0.02	0.98

τ_0 - Yield Stress; K - Consistency Index; n - Flow Behavior Index; R^2 - Determination Coefficient.

Table 2. Rheological parameters of Power-law and Herschel-Bulkley models at 15ºC, 25º and 35ºC, using concentric cylinder geometry.

Brand 1							
T (°C)	Power Law Model			Herschel-Bulkley Model			
	K (Pa.s)	n	R^2	τ_0 (Pa)	K (Pa.s)	n	R^2
15	0.93 ± 0.05	0.33 ± 001	0.97	0.2 ± 0.2	0.8 ± 0.2	0.35 ± 0.04	0.98
25	0.72 ± 0.04	0.33 ± 0.01	0.98	0.3 ± 0.1	0.51 ± 0.09	0.38 ± 0.03	0.98
35	0.33 ± 0.01	0.35 ± 0.01	0.99	0.27 ± 0.03	0.15 ± 0.02	0.45 ± 0.02	0.99
Brand 2							
T (°C)	Power Law Model			Herschel-Bulkley Model			
	K (Pa.s)	n	R^2	τ_0 (Pa)	K (Pa.s)	n	R^2
15	1.4 ± 0.4	0.30 ± 0.03	0.86	-1 ± 1	3 ± 1	0.21 ± 0.08	0.88
25	0.63 ± 0.04	0.38 ± 0.02	0.98	0.2 ± 0.2	0.5 ± 0.3	0.41 ± 0.04	0.98
35	0.61 ± 0.03	0.35 ± 0.01	0.98	0.2 ± 0.1	0.5 ± 0.1	0.38 ± 0.04	0.98

τ_0 - Yield Stress; K - Consistency Index; n - Flow Behavior Index; R^2 - Determination Coefficient.

In Figures 4c and 4d for brands 1and 2, using cone-plate geometry, was not observed significant difference between flow curves at three temperatures, nevertheless, the decrease in apparent viscosity in function with increasing temperature was observed in Figs. 5c and 5d when concentric cylinder was utilized. The apparent viscosity decrease with increasing temperature was not observed for brand 2 at temperatures of 15 and 25°C using cone-plate geometry (Fig. 4d) and at 25 and 35°C using concentric cylinder geometry (Fig. 5d). This apparent viscosity decrease can be attributed to the increase in intermolecular distances, because of the thermal expansion caused by the increase in temperature (Constenla et al., 1989; Haminiuk et al., 2006).

Activation energy is necessary for moving of a molecule, and as the temperature increases, the liquid flows more easily due to higher activation energy in high temperatures (Haminiuk et al, 2006). Besides, Rha (1975) noted that the decrease in apparent viscosity with increasing shear rate is related to the increasing alignment of constituent molecules.

According to Hassan and Hobani (1998), the increase in the temperature and consequently in the thermal energy leads to an increase in the molecular distances due to a reduction in the intermolecular forces. Also, with a temperature increase, the shear stress is enhanced, causing a rearrangement of the particles in parallel directions and their breaking into smaller particles. These particles can flow more easily due to the decrease in the particle–particle interactions, resulting in an apparent viscosity decrease.

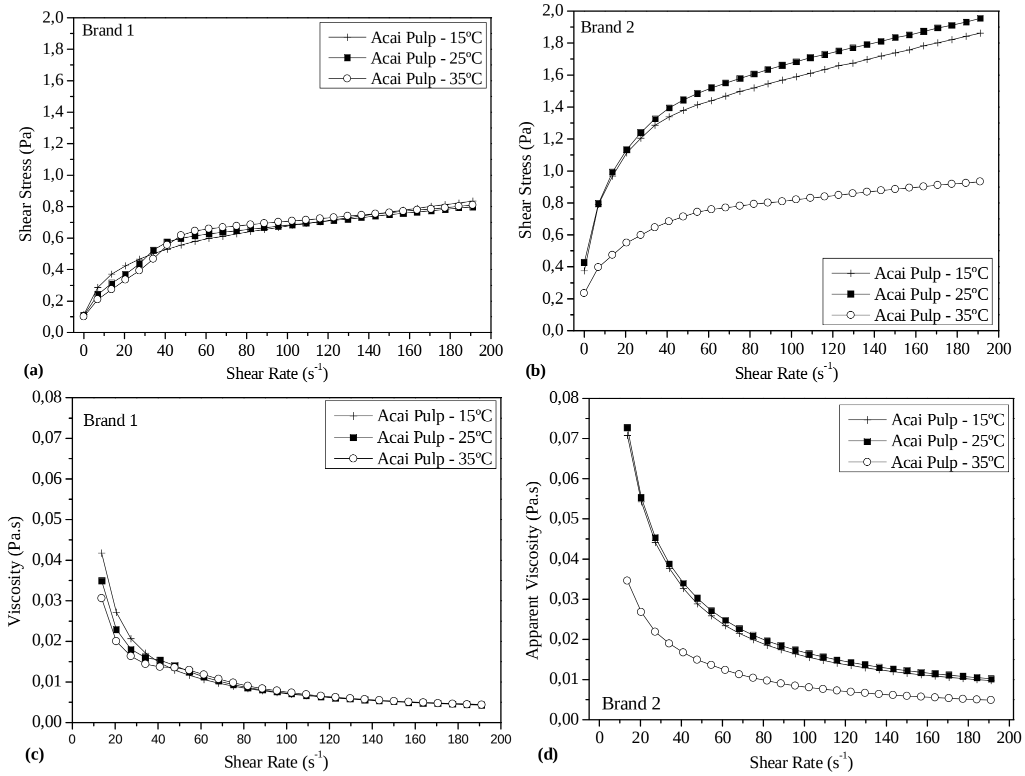

Figure 4. Flow curves of shear stress in function of shear rate (a – brand 1and b – brand 2), and apparent viscosity versus shear rate (c – brand 1 and d – brand 2) for açai pulps using cone-plate geometry.

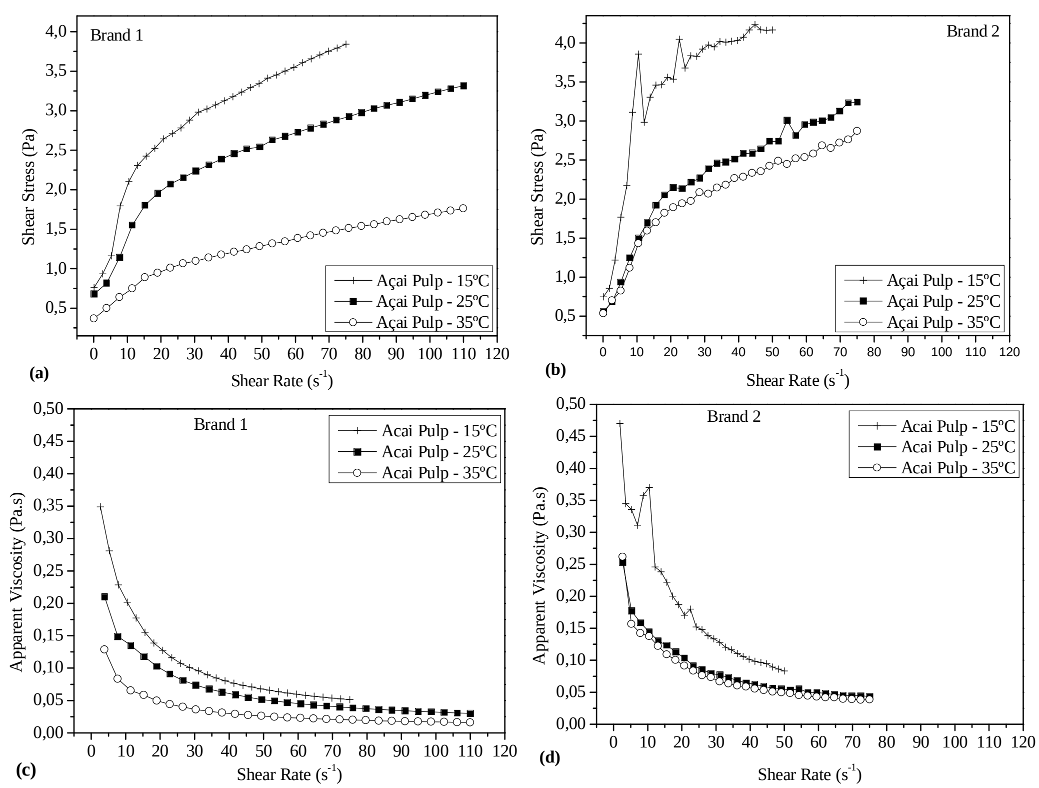

Figure 5. Flow curves of shear stress in function of shear rate (a – brand 1and b – brand 2), and apparent viscosity versus shear rate (c – brand 1 and d – brand 2) for açai pulps using concentric cylinder geometry.

Diluted açai pulp showed thixotropic behavior at all temperatures (15ºC, 25ºC and 35ºC), with cone-plate geometry and after three shear cycles, the thixotropic behavior was not observed. This thixotropic behavior for brand 1 can be observed in Figs. 6.

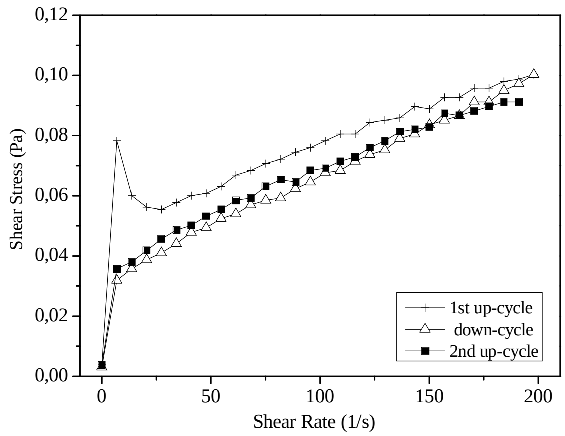

Figure 6. Flow curves of the diluted açai pulp at 25ºC, with cone-plate geometry.

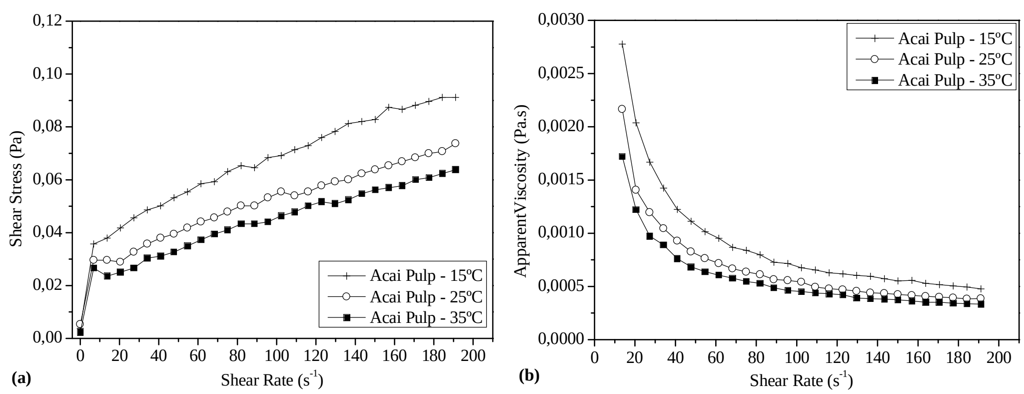

Figure 7. Flow curves of shear stress in function of shear rate (a), and apparent viscosity versus shear rate (b) for diluted açai pulp (brand 1).

In Figures 7.a and 7.b are presented the flow curves of shear stress in function of shear rate and apparent viscosity versus shear rate for diluted açai pulp (brand 1) using cone-plate geometry. The slope of the curves decreased with increasing shear rate and the apparent viscosity decreased with the increase of the shear rate. This behavior is typical from shear-thinning fluids. The majority food fluids derived from fruits shows pseudoplastic behavior, where viscosity apparent decreases with increase of the shear rate (Holdsworth, 1971).

The diluted açai pulp presented smaller apparent viscosity than the whole açai pulp probably due to the higher solid content in the latter and a decrease in the viscosity with the increase of the temperature.

Usually, the flow behavior of fruit derivates is strongly influenced by the amount of suspended particles, which imparts a shear-thinning characteristic to the pulps (Saravacos, 1970; Sato and Cunha, 2009).

Açai pulp and its products are becoming more attractive to consumers due to their high levels of antioxidants that promote human health. However, the process calculations, modelling, design, and optimization of the açai processing are related to the knowledge of its physical properties. When such data, even approximate, cannot be found, it becomes necessary to have them measured.

Nowadays, few studies have been conducted that relate to rheological properties to the optimal design and better control of the açai processing. Each day the international and national interest by this fruit is increasing, and then, the açai rheological data are been required.

4. CONCLUSIONS

The rheological behavior of açai pulp was investigated using cone-plate and concentric cylinder geometries. To prevent the wall slip effect and high shear stress values using the cone-plate geometry, gaps of 910mm and 1300mm were considered to analyze the different brands.

Açai pulps showed shear thinning behavior with yield stress, and data were best fitted to the Herschel-Bulkley model at all temperatures. The samples presented thixotropic behavior in function of the temperature, and after three shear cycles this effect was eliminated.

Açai pulps presented differences in rheological behavior using both cone-plate and concentric cylinder geometries. However, the pulps were classified as non-Newtonian fluid for all temperature ranges and models studied. Pseudoplastic characteristics (flow behavior index smaller than 1) for the açai pulps analyzed were observed.

The influence of the total solid content reduction on rheology of açai pulp was verified with a decrease in the viscosity with the increase of the temperature.

The results obtained in this study have been used as parameters in cross-flow microfiltration processes of açai pulp in development by this research group. The technology of separation by membranes can be an alternative to the removal of particulates (clarifying) of the fruit juices as a clean technology that neither produces residues, nor utilizes chemical preservatives and thermal energy.

5. ACKNOWLEDGEMENTS

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