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Steady and dynamic shear rheological properties of açai pulp (*Euterpe oleraceae* Mart.)

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ABSTRACT

The aim of this work was to study the rheological behavior of açai pulp at different temperatures (10, 25, 40, 55 and 70 °C) under steady and dynamic shear conditions. The measurements were carried out using a stress-controlled rheometer with stainless steel plate-plate geometry and smooth or grooved surfaces. In the tests performed with smooth surface geometry, the slip effect was shown at low shear rates under steady and dynamic shear conditions, while the use of grooved surface geometry minimized this effect. At any given temperature, the açai pulp showed shear thinning behavior with yield stress in the steady shear measurements. However, an increase in temperature led to an increase in thixotropy and a decrease in yield stress. In addition, the açai pulp viscosity at 100 s^{-1} showed Arrhenius type temperature dependence, with activation energies of 4.18 and 6.21 kJ/mol for smooth and grooved surfaces, respectively. With respect to the dynamic shear measurements, the açai pulp showed weak gel-like behavior, and the storage and loss moduli decreased with increasing temperatures.

1. Introduction

Açai is the fruit of the *Euterpe oleracea* Mart. Palm and native to the Amazonian region. In recent years, this berry has been recognized not only for its high energy potential but also for its functional properties for use in food and nutraceutical products, especially due to its high antioxidant activity, related to its high anthocyanin and phenolic contents (Coisson et al., 2005; Schauss et al., 2006). However, açai is a highly perishable fruit and must be pulped within 24 h after harvest. The pulp, even when stored under refrigeration, has a maximum shelf life of 12 h. Thus most industries producing açai-based products, located far from the harvest area, are obliged to store and use the frozen pulp and process the thawed material. The methods used to process the berries can affect their overall quality, including their antioxidant activity. In addition, the application of heat over extended periods of time coupled with continuous shearing during pumping can cause irreversible structural breakdown of the products and make them unappealing to consumers (Nindo et al., 2007). Thus, knowledge of the rheological properties of açai pulp under different conditions of temperature and shear is essential for product development, quality control, consumer acceptability and the design of processing equipment. There are several approaches to carry out rheological characterizations, and the technique selected depends on the specific product and the functional characteristics that need to be analyzed (Tabilo-Munizaga and Barbosa-Cánovas, 2005).

Fruit purees and pulps are generally characterized as non-Newtonian fluids, as a result of complex interactions amongst their components. These kinds of fluid are generally described by empirical rheological models that represent the most convenient rheogram fit. The most widely used models are the Power Law (Dak et al., 2007; Haminiuk et al., 2006) and the Herschel–Bulkley model (H–B) (Ahmed and Ramaswamy, 2004; Dutta et al., 2006). Several factors affect the rheological behavior of fruit purees and pulps, including temperature (Bhattacharya, 1999; Ditchfield et al., 2004; Guerrero and Alzamora, 1998), concentration (Cepeda et al., 2002; Hernández et al., 1995; Nindo et al., 2007) and particle size (Genovese and Lozano, 2000).

Fruit suspensions consist of disintegrated cells and cell wall material dispersed in a serum composed of soluble components such as sugars, salts and acids (Bayod et al., 2007). However, açai presents a particular chemical composition, rich in proteins, fibers and lipids (Rogez, 2000), which can provide a different type of rheological behavior as compared to other fruit pulps. In addition, its high lipid content associated with a great number of insoluble particles can lead to a slip between the sample and the surface of the rheology measuring device, which can give rise to experimental artifacts. Apparent wall slip is a phenomenon often observed during the rheological measurements of particulate systems such as foams, emulsions, gels or suspensions, such as fruit pulps (Bertola et al., 2003).

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Nomenclature

$\dot{\gamma}$	shear rate (s^{-1})	k	consistency index ($Pa \cdot s^n$)
σ	shear stress (Pa)	R	universal gas constant (8.316 kJ/mol · K)
σ_o	yield stress (Pa)	η	viscosity (Pa · s)
A	Arrhenius model fitting parameter		
E_a	activation energy (kJ/mol)		

Wall slip effects in dispersion flows have been associated with a depletion of particles in the wall and several techniques have been developed to eliminate the slip effects and extract the bulk rheology. In particular, suspensions show particle migration towards regions of less intense shear rates, and particle depletion near the fluid solid interface at rest (Bertola et al., 2003). Thus, in practice, a homogeneous flow does not occur for particulate systems below a critical shear rate, as only a small region of the material actually moves (Moller et al., 2006). Despite being commonly observed in steady shear measurements, the effects of wall slip are less observed in dynamic oscillatory shear measurements. However, they must be eliminated before the measurements, since they can lead to a non-linear stress response, making it difficult to distinguish between the slip effects and fluid rheology (Yoshimura and Prud'Homme, 1988). The use of geometries with grooved or rough surfaces can prevent the formation of a separated phase layer, since the serrations, when they dip into the bulk fluid, provide a good grip on the sample, thus eliminating or decreasing the occurrence of slip effects (Pal, 2000). Another suggestion to prevent slip effects is the use of geometries with hydrophobic or hydrophilic surfaces, depending on the material polarity. For a hydrophobic material, for example, the use of a hydrophobic surface significantly reduces slip effects, due to the interaction between the sample and the plates, while a hydrophilic surface would repel the samples, causing very little change in wall slip (Walls et al., 2003).

The aim of this work was to evaluate the rheological properties of açai pulp under steady and dynamic shear at different process temperatures. In addition, different shear conditions were used (measurement devices with smooth and grooved surfaces) in order to evaluate the presence and the causes of slip effects in açai pulp.

2. Material and methods

2.1. Material

Frozen açai pulp was purchased from CAMTA (Belém, PA, Brazil) and stored at −18 °C. Samples were thawed according to the amount required and carefully homogenized with a magnetic stirrer before performing the rheological measurements. The composition of açai pulp is shown in Table 1.

Table 1
Composition of açai (*Euterpe oleraceae* Mart.) pulp.

Analyzed item	Mean value	Analysis method
Moisture (%)^{wb}	86.01 ± 0.31	A.O.A.C. (1990)
Proteins (%)db	10.69 ± 0.66	A.O.A.C. (1990)
Lipids (%)^{db}	48.24 ± 0.12	Bligh and Dyer (1959)
Fibers (%)^{db}	31.67 ± 2.06	A.O.A.C. (1990)
Total sugars (%)^{db}	3.55 ± 0.20	A.O.A.C. (1990)
Ash (%)^{db}	3.04 ± 0.24	A.O.A.C. (1990)
pH	5.2 ± 0.1	pH meter

^{wb}, wet basis, ^{db}, dry basis.

2.2. Methods

Rheological measurements were carried out using a stress-controlled rheometer Carri-Med CSL^2 500 (TA Instruments, Crawley, England), with stainless steel plate-plate geometries and smooth surface (SS) or grooved surface (GS) with a diameter of 40 mm. The grooved geometry used was a system with a 45° serration angle and 0.5 mm depth. Firstly, steady shear measurements were performed to determine an adequate gap using smooth surface geometry. The açai particle size can reach 1 mm in diameter, which can affect the rheological measurements if the gap is not large (or small) enough. All the rheological tests (steady and dynamic shear) were performed at temperatures of 10, 25, 40, 55 and 70 °C. At the higher temperatures, a thin silicone oil layer was spread around the sample in order to minimize water evaporation from the product.

2.2.1. Steady shear rheology

Three flow ramps (up, down and up-cycles) were performed in a range of shear stresses corresponding to shear rates from approximately 0 to 300 s^{-1}, in order to evaluate and eliminate thixotropy. All measurements were performed in triplicate, using a new sample for each repetition. The third flow curve data were fitted to rheological models using the software Statistica 5.0 (Statsoft, Tulsa, EUA). The Power Law, Herschel–Bulkley and Bingham models were tested and the determination coefficient (R^2) was used as a parameter to verify the goodness-of-fit. Significant differences between the results obtained with different temperatures or geometries were analyzed by the Analysis of Variance (ANOVA) and Tukey Test, both using the software Statistica 5.0 (Statsoft, Tulsa, EUA).

2.2.2. Dynamic shear rheology

Oscillatory stress sweeps between 0.1 and 5.0 Pa were performed to determine the linear viscoelastic range at a frequency of 0.1 Hz. Frequency sweep measurements were carried out to determine the mechanical spectra of the açai pulp at a fixed shear stress value within the linear viscoelastic range. The frequencies ranged from 0.01 to 10 Hz, but in most cases some results had to be discarded due to equipment limitation at low frequencies or resonance effects at high frequencies. The tests were performed in triplicate.

3. Results and discussion

3.1. Gap determination

A typical flow curve showing clear dependence between the shear stress values and the gap used can be observed in Fig. 1. During the measurements performed with gaps of 1.7 and 2.1 mm, separation of the serum from the pulp was clearly visible within the product, which probably led to a higher value for the viscosity reading. Conversely the curve obtained with a 2.8 mm gap showed shear stress values slightly inferior to the others, probably due to a non-homogeneous shear rate throughout the sample at higher