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Influence of concentration on the steady and oscillatory shear behavior of umbu pulp[1]

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ABSTRACT

In this experimental work the rheological behavior of umbu pulp has been studied by shear flow (pseudoplasticity, apparent viscosity) and in oscillatory mode (dynamic modules) in the linear domain of viscoelasticity. The studies were carried out with the use of a controlled stress Rheometer Haake RS 100, at different soluble solid concentrations (10, 15, 20 and 25 °Brix), measured at 30 °C. Tests in steady shear were conducted over a shear rate range of 0.1 – 300 s^{-1} and oscillatory measurements over a frequency range of 0.01 – 100 Hz. The results indicated that umbu pulp behaves as a non-Newtonian fluid, with pseudoplastic characteristics and yield stress appearance and exhibits tixotropic properties. Rheograms were fitted to the Herschel-Bulkey model. From the dynamic test the umbu pulp showed storage modulus (G′) values that were always higher than loss modulus (G″), indicating weak gel-like behavior. Storage and loss modulus increased with increase in the concentration.

Key words: viscosity, viscoelasticity, soluble solid content (°Brix)

Influência da concentração no comportamento reológico nos estados estacionário e oscilatório de polpa de umbu

RESUMO

Neste trabalho experimental o comportamento reológico da polpa de umbu foi analisado nos estados estacionário (pseudoplaticidade, viscosidade aparente) e oscilatório (módulos dinâmicos, viscosidade complexa) determinado na região de viscoelasticidade linear. Os estudos foram conduzidos com o uso do reômetro de tensão controlada Haake RS 100, em diferentes concentrações de sólidos solúveis (10, 15, 20 e 25 °Brix), medidos na temperatura de 30 °C. Testes em estado estacionário foram conduzidos sobre taxa de cisalhamento de 0,1 – 300 s^{-1} e as medidas em regime oscilatório em uma taxa de freqüência de 0,01–100 Hz. Os resultados indicam que a polpa de umbu se comporta como um fluido não-Newtoniano, com características pseudoplásticas, apresenta tensão inicial e exibe propriedades tixotrópicas. Os reogramas foram ajustados através do modelo de Herschel-Bulkey. Nos testes em estado dinâmico a polpa de umbu apresentou os valores do módulo de estocagem (G′) maiores que os do módulo de perda (G″), indicando comportamento de gel fraco, enfim os módulos de estocagem e perda aumentam com a concentração.

Palavras-chave: viscosidade, viscoelasticidade, sólidos solúveis (°Brix)

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INTRODUCTION

The umbu fruit (*Spondias tuberosa* Arruda Câmara) is of great economic importance for the semi-arid region of the Brazilian Northeast, because many of its derived products are sold in the markets, especially pulp, that can be converted to jam, nectar, sweets, jellies, juices and concentrates (Cazé Filho et al., 2005).

Rheological measurements have been considered as an analytical tool to provide a fundamental insight into the structural organization of food. Various factors affecting the rheological behavior of fruit pulp include temperature (Giner et al., 1996) and total soluble solids/concentration (Ahmed et al., 2000). Foods, however, are structurally and rheologically complex materials and, in many cases, they consist of mixture of solids as fluid structural components (Finney, 1972).

Rheological properties have been reported and summarized in many publications of various foods (Rao & Steffe, 1992; Steffe, 1996) and fluid fruit and vegetable puree products (Krokida et al., 2001).

The viscosity of fluid foods is an important transport property, which is useful in many applications of Food Science and Technology, such as design of food process and processing equipment, quality evaluation and control of food products, and understanding the structure of food materials (Barbosa-Cánovas et al*.*, 1996; Krokida et al., 2001).

In oscillatory instruments, samples are subjected to harmonically varying stresses or strains. This testing procedure is the most common dynamic method for studying the viscoelastic behavior of food. Results are very sensitive to chemical composition and physical structure and so are useful in a variety of applications including: gel strength evaluation, monitoring starch gelatinization, studying the glass transition phenomenon, observing protein coagulation or desnaturation, evaluating curd formation in dairy products, cheese melting, texture development in bakery and meat products, shelf-life testing, and correlation of rheological properties to human sensory perception. Food scientists have found oscillatory testing instruments to be particularly valuable tools for product development work (Steffe, 1996).

According to Trifiró et al. (1987) apud Queiroz (1998), juices and pulps are considered as pseudoplastic fluids, and the Newtonian behavior is determined by the pulp content of the product – juices without pulp, or with little pulp, behave as Newtonians fluids. Increasing pulp content increases the pseudoplastic character. Ahmed et al. (2004) report that fruit pulp behaves as a non-Newtonian fluid as a result of complex interactions among soluble sugars, pectic substances and suspended solids.

The concentration process results in the removal of the water and a consequent reduction in the transport costs and storage space. The apparent viscosity of the products varies widely during the process (Silva et al., 2005).

The objective of the present research was to determine the influence of soluble solids concentration on the rheological behavior of umbu pulp.

MATERIAL AND METHODS

Material

The fruits of umbu (*Spondias tuberosa* Arruda Câmara) were supplied by EMBRAPA/CPATSA – Petrolina, Brazil. The fruits were washed and processed through a pulping machine, followed by concentration by evaporation. Samples with soluble solids contents of 10 (in nature), 15, 20 and 25 °Brix were obtained.

Analytical methods

The total content of soluble solid was measured in °Brix using a digital refractometer (Model RDA 8600, ACATEC). The pH was read using a pH meter (pH 300 M, ANALYSER). The standard method (AOAC, 1984) was used to determine the content of moisture on triplicate samples for the umbu pulp.

Rheological measurements

The studies were carried out using a controlled stress Rheometer Haake RS 100. The sensor used was the Z40 DIN (Gap 4.2 mm) coaxial cylinder system with a cylindrical rotor to steady shear behavior and a cone and plate device C60/2ºTi (6 cm diameter, cone angle 2°, Gap 0.105 mm), to study oscillatory shear behavior. The temperature of the sample was adjusted to 30 °C and kept constant with a thermostatic bath and Peltier effect temperature regulation for different systems.

Tests in steady shear were conducted over a shear rate range of 0.1 – 300 s^{-1}. The Herchel-Bulkley model were then fitted to the shear rate and shear stress data using the non-linear estimation module of Statistica software (Release 5.0, StatSoft Inc., Tulsa, OK, USA). The closeness of fit to the model was based on correlation coefficient (R^2).

The Dynamic rheological behavior was characterized. Initially, stress sweeps were performed to establish the linear viscoelastic range of the material. Frequency sweeps between 0.01 and 100 Hz was used to characterize the viscoelastic behavior of the material. Storage modulus (G’), loss modulus (G”) and tangents (Tan δ) were determined. Each run was performed in triplicate with different samples.

RESULTS AND DISCUSSION

Physicochemical properties

The umbu pulp had a total content of soluble solid of 10 °Brix. The pH of pulp was 2.25. The moisture was 87%. Similar results have been reported by Ferreira (2000) and Almeida (1999).

Steady shear properties

The relations fitted the Hershel-Bulkley equation ($t = K(\dot{\gamma})^n + \tau_o$), (Table 1), where τ is shear stress, $\dot{\gamma}$ is shear rate, K is the consistency coefficient ($Pa.s^n$), n is the flow behavior index (dimensionless), and τ_o is yield stress (Pa). The umbu pulp has no straight line relationship between the shear stress and the shear rate, thus showing itself to be a non-Newtonian fluid with yield stress. Figure 1 shows the shear stress versus shear rate for umbu pulp at 30 °C.

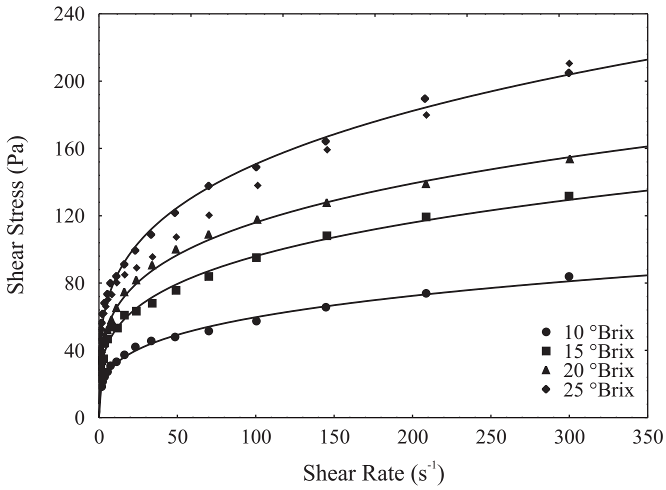

Figure 1. Rheogram of umbu pulp: shear stress (τ) versus shear rate ($\dot{\gamma}$). Effect of the concentration

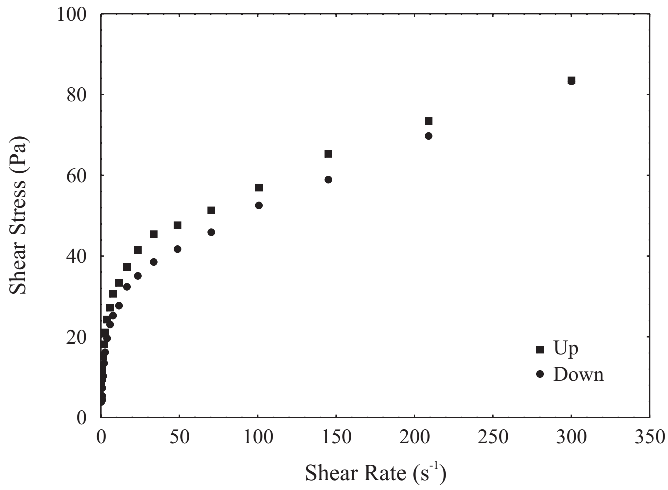

Figure 2. Thixotropic loop for umbu pulp (10 °Brix), temperature of measurement is 30 °C

The values for flow behavior index, *n*, were less than 1, which was indicative of the pseudoplastic behavior (shear thinning). 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. Similar results had been reported for clarified cherry juices (Giner et al., 1996), clarified blackcurrant juice (Ibarz et al., 1992) and acerola juice (Silva et al., 2005). Results of the relationship between viscosity, at a shear rate of 100 s^{-1}, and soluble solids contents of umbu pulp (C) are shown. Various factors affecting the rheological behavior of fruit pulp include total soluble solids/concentration (Hernandez et al., 1995).

Table 1. Flow parameters of the umbu pulp determined by Herschel-Bulkley equation

C	η	K_H	n_H	τ_0	R^2
10	0.5694	14.1216	0.3004	3.1752	0.9935
15	0.9459	23.6173	0.2934	4.4899	0.9941
20	1.1752	30.5599	0.2801	4.6961	0.9939
25	1.4700	37.7443	0.2887	8.0640	0.9961

Table 2 shows the values of the parameters obtained by the exponential ($\eta = a_1 . \exp(a_2 . C)$) and power-law ($\eta = a_1 . C^{a_2}$) relations, where C is the soluble solids concentration (°Brix), a_1; a_2 are constants (dimensionless). According to the values of the regression coefficient obtained, the power model seems to describe better the effect of the soluble solids on the viscosity of umbu.

A hysteresis loop, typical of thixotropic behavior, was obtained (Figure 2) for umbu pulp (10 °Brix). The area enclosed by the hysteresis loop indicates the degree of structural breakdown due to shearing. The curve for increasing rate was developed first and was at a higher position than the decreasing rate curve, showing that umbu pulp exhibited a thinning behavior with time. Thixotropy indicates continuous breakdown or rearrangement of structure with shearing time (Rha, 1978 apud Bhattacharya, 1999).

Table 2. Parameters of exponential and power-law model

Model	a_1	a_2	R^2
Exponential	5.9512	1.3966	0.9883
Power-law	16.7751	1.0195	0.9912

Dynamic shear properties

Figures 3A and 3B, respectively, show changes in storage modulus (G') and loss modulus (G") as functions of frequency

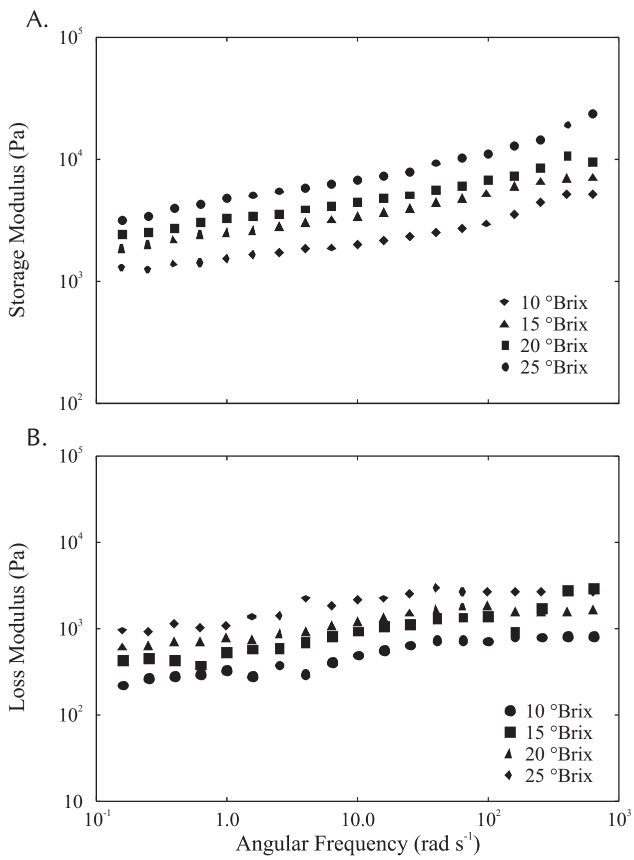

Figure 3. Mechanical spectra of umbu pulp at different concentrations, showing the values of storage modulus (A) and loss modulus (B) versus angular frequency

(ω) for umbu pulp at 30 °C. The magnitudes of G' and G" increased with increasing ω with a high frequency dependence. According to dynamic experiments, G' values are greater than G" values through the whole range of frequencies considered, corresponding to a solid state. As was expected, a higher soluble solid concentration showed higher G'and G" values than ones with a lower concentration. The ratio of G" to G' (Tan δ) is greater than 0.1 (Figure 4), meaning that the sample is not a true gel, but could be characterized as a weak gel. This type of behavior has been reported by other authors for mango pulp (Iagher et al., 2002), rice flour dispersions (Chun & Yoo, 2004), assai pulp (Alexandre, 2002) and jaboticaba pulp (Sato, 2005).

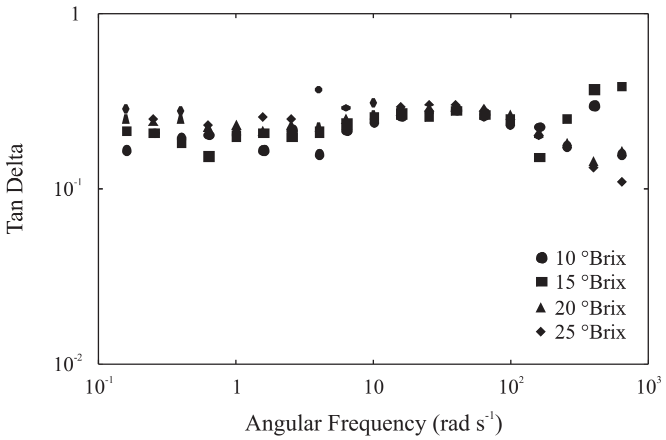

Figure 4. Tan (δ) as a functions of angular frequency at 30 °C for umbu pulp at different concentrations

Conclusions

1. This study demonstrated that the umbu pulp exhibited a high shear-thinning behavior with yield stress value.

2. The Herschel-Bulkley equation clearly depicted the behavior with correlation (R^2).

3. Based on dynamic rheological data on storage (G') and loss modulus (G") as functions of angular frequency (ω), the pulp in different concentrations displayed the rheological behavior similar to weak gel-like macromolecular dispersions with G' much greater than G" at all values of ω applied.

4. Storage and loss modulus increased with increase in the concentration.

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