EFFECT OF XANTHAN GUM ADDITION ON THE RHEOLOGICAL PROPERTIES OF UMBU FRUIT PULP

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In this study, the rheological properties of an umbu pulp suspension containing a food hydrocolloid (xanthan gum) at many concentrations (0.25, 0.50, 0.75 and 1.00%) and temperatures (10, 20, 30, 40 and 50ºC), were evaluated by means of shear flow, and at 30ºC for the oscillatory shear tests. The experiments were conducted in a Haake RS 600 rheometer. The tests for steady shear were conducted in the shear rate range between 0.1 – 1000 s^{-1}. In the dynamic shear tests, frequency sweeps between 0.01 and 100 Hz were used to characterize the viscoelastic behavior of the material. The storage moduli (G'), loss moduli (G") and tangents (tan δ) were determined. Suspensions containing the biopolymer xanthan exhibited shear-thinning behavior, characterized by yield stress, and hence the rheograms were fitted to the Herschel-Bulkley model. An exponential model was used to evaluate the effect of concentration on the apparent viscosity and the Arrhenius model was used to describe the temperature effect. The umbu pulp suspension behaved like a weak gel, with the storage greater than loss moduli. Both elastic (G') and viscous (G") moduli increased with increase in frequency (ω).

KEY-WORDS: UMBU PULP; XANTHAN GUM; RHEOLOGY; DYNAMIC AND STEADY-SHEAR.

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1 INTRODUCTION

Umbu (*Spondias tuberosa* Arruda Câmara) is a native fruit from the semi-arid area of northeastern Brazil. The umbu tree produces succulent fruits, which are rich in mineral salts and vitamins, contributing considerably to the diet of the local population (CAZÉ FILHO et al., 2005). Due to its pleasant flavor, the fruit shows good potential for consumption, similar to that of other native species from other areas (CAVALCANTI, RESENDE & BRITO, 2000).

The fruit is consumed "*in nature*" and its pulp can be manufactured into many different products such as juice, nectars, ice cream and jelly by means of unit operations such as pumping, agitation, heat exchange and separations. The technical and economic feasibility of these industrial processes depends greatly on the knowledge of the physical-chemical properties, and of these properties, the rheological behavior is one of the most important (IBARZ, GONÇALVEZ & ESPLUGAS, 1996). The rheological properties play a role in the process design, evaluation and modeling. These properties are sometimes measured as an indicator of product quality (e.g. indication of total solids or change in molecular size). Rheological data are required for the calculations of any process involving fluid flow (e.g. pump sizing, extraction, filtration, extrusion, purification) and play an important role in the analyses of flow conditions in food processes such as pasteurization, evaporation, drying and aseptic processing (MARCOTTE, HOSHAHILI & RAMASWAMY, 2001).

The rheological behavior of hydrocolloids is of special importance when they are used to modify textural attributes. Gums are used in food primarily as thickeners and gelling agents as a result of their ability to alter the rheological properties of the solvent in which they are dissolved. The changes in viscosity occur as a result of the right molecular weight polymeric nature of the gums that are dissolved or dispersed. These properties have been exploited for their functionality in food systems, including textural attributes and mouthfeel (YASEEN et al., 2005; KOKSOY & KILIC, 2004; SIKORA et al., 2007).

Xanthan gum is a microbial hetero-polysaccharide produced by the aerobic fermentation of *Xanthomonas campestris* (BRESOLIN et al., 1998). It has a cellulose backbone substituted on every second residue, by a side chain consisting of two mannose units separated by a glucuronic acid residue. The mannose residue attached to the cellulosic backbone is variably acetylated, and the terminal mannose can contain a pyruvate group (SCHORSCH, GAMINIER & DOUBLIER, 1997). The gum is soluble either in hot or cold water, has a high viscosity at low concentrations (ALEXANDRE, 1999) and shows excellent stability in hot and acid systems (CASAS, SANTOSA & GARCIA-OCHOA, 2000).

Several benefits of controlling the texture and sensorial properties by adding gums have recently been reported (GIBINSKI et al., 2006; KOSKSOY & KILIC, 2004; SAHIN & OZDEMIR, 2004; SIKORA et al.; 2003).

The objective of this paper was to investigate the effect of different concentrations of the hydrocolloid xanthan on the rheological properties of umbu pulp on the steady and dynamic shear at different temperatures.

2 MATERIAL AND METHODS

2.1 MATERIAL

The Umbu fruits (*Spondias tuberosa* Arruda Câmara) were supplied by EMBRAPA/CPATSA, Petrolina, PE, Brazil.

The xanthan gum was donated by CP KELCO BRASIL S/A, Limeira, SP, Brazil.

2.2 SAMPLE PREPARATION

The fruits were washed and processed through a pulping machine. Samples with 10ºBrix (soluble solids content) were obtained.

Four concentrations of xanthan were employed (0.25, 0.50, 0.75 and 1.00%) in the range frequently used in food systems. They were prepared by mixing the desired amount of dry xanthan with the umbu pulp at room temperature, with continuous mild agitation provided by a magnetic stirrer.

2.3 RAW MATERIAL CHARACTERIZATION

Total soluble solids (ºBrix) and pH were determined using refractometer (WY1A, ABBE, USA) and pH-meter with glass electrode (model 710 A, Orion Research, Boston, USA) at 20ºC, respectively. Acidity and pectin were measured according to standard method of AOAC (1990).

2.4 RHEOLOGICAL MEASUREMENTS

Rheological evaluations were performed using a controlled stress Rheometer (Haake RheoStress 100, Karlsruhe, Germany) with temperature control. A cone and plate C60/2ºTi (6 cm in diameter, cone angle 2º) geometry was used. Paraffin oil was used to cover the sample to avoid evaporation of the water during measurement.

Two types of measurements were performed: steady state and oscillatory experiments. In the steady state experiments, the shear stress versus shear rate was calculated in the range from 0.01 – 1000 s^{-1}, and for each measurement 2 replicates were used.

The experimental data were fitted to the Herchel-Bulkley model ($\tau = K(\dot{\gamma})^n + \tau_o$) where τ is the shear stress, $\dot{\gamma}$ is the shear rate, K is the consistency coefficient, n is the flow behavior index, and τ_o is the yield stress.

The temperature dependency of the viscosity at each concentration was evaluated by applying the Arrhenius model, $\eta = A \exp^{(Ea/RT)}$, where η is the apparent viscosity, A is the frequency factor, *Ea* is the activation energy, T is the temperature and R is the gas constant.

The variation in viscosity with concentration can be described by various expressions, though they are generally of the exponential type $\eta = \eta_1 \exp(a_1 C)$, where η is the apparent viscosity, η_1 and a_1 are constants, and C is the concentration.

Viscoelastic materials exhibit solid and liquid characteristics simultaneously, and the storage (G') and loss (G") moduli refer to the elastic and viscous character of a given material, respectively, it being possible to quantify the predominance of the solid or liquid character of a sample from dynamic measurements. Thus the determinations of the moduli G' and G" were recorded versus frequency. Before performing these frequency spectra, the linear viscoelastic region was determined and an appropriate strain was selected. Frequency sweeps between 0.01 and 100 Hz were used to characterize the viscoelastic behavior of the material. Each run was performed in duplicate with different samples.

The rheological properties of fluids are associated with their internal structure and the Cox-Merz rule provides information on simple polymeric solutions (RAO & COOLEY, 1992). This empirical relationship states that the complex dynamic viscosity $|\eta^*(\omega)|$ and steady-shear viscosity $\eta(\dot{\gamma})$ are equivalent when the angular frequency ω is equal to the steady-shear rate $\dot{\gamma}$: $|\eta^*(\omega)| = \eta(\dot{\gamma} = \omega)$.

3 RESULTS AND DISCUSSION

3.1 PULP CHARACTERIZATION

The physical and chemical characteristics of umbu pulp are given in Table1.

TABLE 1 - THE PHYSICAL-CHEMICAL CHARACTERISTICS OF UMBU PULP

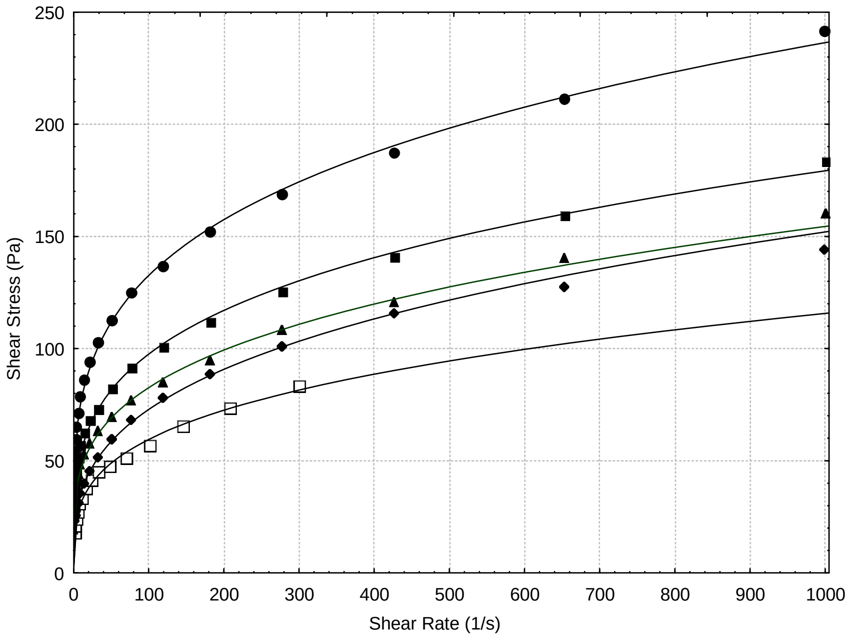
	Content
Acidity (g/100g)	1.32
Pectin (g/100 g)	0.536
pH	2.25
Soluble solids (ºBrix)	10

The experimental values are according to ALMEIDA (1999) and FERREIRA (2000).

3.2 STEADY SHEAR PROPERTIES

The shear stress versus shear rate data for the umbu pulp dispersions with different concentrations of xanthan gum at 30ºC, are shown in Figure 1. The experimental results indicate the non-Newtonian behavior.

FIGURE 1 - SHEAR STRESS-SHEAR RATE PLOTS FOR UMBU PULP SUSPENSIONS CONTAINING XANTHAN GUM - 0.00% (□), 0.25% (♦), 0.50% (▲), 0.75% (■) AND 1.00% (●), AT 30ºC



All the samples showed highly shear-thinning behavior with values for the flow behavior index (*n*) as low as 0.26 – 0.35 (Table 2). The *n* values did not change much with increase of concentration. The magnitudes of the apparent viscosity (η_{100}) and consistency index (*K*) obtained from the Herchel-Bulkley model increased with increase in the gum concentration. All the dispersions also showed low magnitudes for yield stress (τ_o) in the range from 0.03 – 23.07 Pa. Similar results were observed by FREITAS (2002) for passion fruit pulp-xanthan gum systems.

TABLE 2 - THE EFFECT OF CONCENTRATION ON THE STEADY SHEAR RHEOLOGICAL PROPERTIES OF UMBU PULP SUSPENSIONS AT DIFFERENT TEMPERATURES, AS DETERMINED BY THE HERSCHEL-BULKLEY EQUATION

C, % / T, ºC	*K*, Pa.s^n	*n*	τ_o, Pa	R^2
0.00				
10	14.67	0.33	4.70	0.994
20	14.00	0.31	3.18	0.997
30	14.12	0.30	3.17	0.994
40	12.65	0.29	0.75	0.997
50	11.29	0.30	0.75	0.996
0.25				
10	16.50	0.33	8.17	0.999
20	15.18	0.34	3.35	0.999
30	15.25	0.33	3.22	0.999
40	13.77	0.33	2.76	0.999
50	11.61	0.35	2.39	0.999
0.50				
10	18.10	0.31	13.48	0.999
20	19.79	0.29	11.17	0.999
30	18.57	0.30	9.60	0.996
40	19.73	0.29	0.26	0.998
50	18.49	0.29	0.16	0.999
0.75				
10	29.65	0.27	21.32	0.999
20	29.88	0.27	6.00	0.998
30	26.68	0.27	4.00	0.998
40	28.21	0.27	3.37	0.996
50	28.48	0.27	0.09	0.995
1.00				
10	38.07	0.27	23.07	0.999
20	37.02	0.27	16.41	0.999
30	36.80	0.26	8.43	0.998
40	34.58	0.27	7.04	0.998
50	35.57	0.26	4.66	0.988

C = concentration, T = temperature, *K* = consistency coefficient, *n* = flow behavior index, τ_o = yield stress.

3.2.1 Effect of temperature on apparent viscosity

The apparent viscosity values decreased with increase in temperature from 10 to 50ºC (Table 3). Reasonably good agreement was obtained when these results were compared with those reported in studies with fluid food products (SINGH & EIPESON, 2000; TELIS-ROMERO et al., 2001). The observed decreases in the viscosity values can be attributed to the increase in intermolecular distances as a result of thermal expansion with the increasing temperature (CONSTELA, LOZANNO & CRAPISTE, 1989). The effect of temperature on the apparent viscosity at a specified shear rate can be described by the Arrhenius relationship, in which the apparent viscosity decreases as an exponential function with temperature. The calculated values for Ea and the constant *A* were in the range from 1.03 – 1.08 Kcal/gmol and 0.25 – 0.11 Pa.s, respectively, with high determination coefficients (Table 4). The observed E_a values did not change much with the increase in concentration, indicating that there was no appreciable effect of the concentration on E_a. At the concentrations studied, the low values meant that their rheological behavior properties were less temperature-dependent, as also indicated by IBARZ, PAGÁN & MIGUELSANZ (1992), IBARZ, GONÇALVEZ & ESPLUGAS (1994) for clarified blackcurrant and orange juices, respectively.

TABLE 3 - APPARENT VISCOSITY OF UMBU PULP AT DIFFERENT GUM CONCENTRATIONS AND TEMPERATURES

C, %	T, °C				
	10	20	30	40	50
0.00	0.75	0.70	0.67	0.63	0.59
0.25	0.80	0.78	0.75	0.70	0.61
0.50	0.90	0.87	0.84	0.77	0.71
0.75	1.25	1.13	1.10	1.05	0.99
1.00	1.57	1.46	1.38	1.30	1.25

C = concentration, T = temperature.

TABLE 4 - ACTIVATION ENERGIES OF UMBU PULP AT DIFFERENT GUM CONCENTRATIONS

C, %	A, Pa.s	E_a, Kcal/g.mol	R^2
0.00	0.11	1.08	0.999
0.25	0.12	1.06	0.861
0.50	0.14	1.04	0.950
0.75	0.20	1.03	0.929
1.00	0.25	1.03	0.996

C = concentration, A = frequency factor, E_a = activation energy.

3.2.2 Effect of concentration on the apparent viscosity

The viscosity of fluid foods increases with concentration. Table 5 summarizes the regression model parameters a_1 and a_2 for the apparent viscosity at 100 s^{-1}. The coefficients of determination obtained were high, indicating that the models adequately described the associated variability. This model was applied to several hydrocolloids (MARCOTTE, HOSHAHILI & RAMASWAMY, 2001), Korean rice flour dispersions (CHUN & YOO, 2004), and acerola juice (SILVA, GUIMARÃES & GASPARETTO, 2005).

TABLE 5 - PARAMETERS OF THE EXPONENTIAL MODEL

T, °C	η_1	a_1	R^2
10	0.66	0.85	0.96
20	0.63	0.80	0.97
30	0.61	0.78	0.96
40	0.57	0.80	0.96
50	0.51	0.88	0.96

η_1 and a_1 = constants, C = concentration.

3.3 OSCILLATORY EXPERIMENTS

Figures 2, 3, 4 and 5 shows the changes in the storage (G') and loss (G") moduli as a function of frequency (ω) for the sample dispersions at 30ºC. The elastic modulus was higher than the viscous modulus (G'>G") over a wide range of ω, demonstrating the weak gel-like behavior of all the samples (pure pulp and pulp with gum). The addition of xanthan increased a little G' and G". The ratio of G"/G' (tan δ) was smaller than 1, meaning that the samples were not true gels. ALEXANDRE (2002) and FREITAS (2002) characterized a similar behavior for assai pulp and for the biopolymer xanthan in passion fruit pulp, respectively.

FIGURE 2 - MECHANICAL SPECTRA OF THE SAMPLES CONTAINING 0.25% XANTHAN: VALUES OF G' (■) AND G" (□)

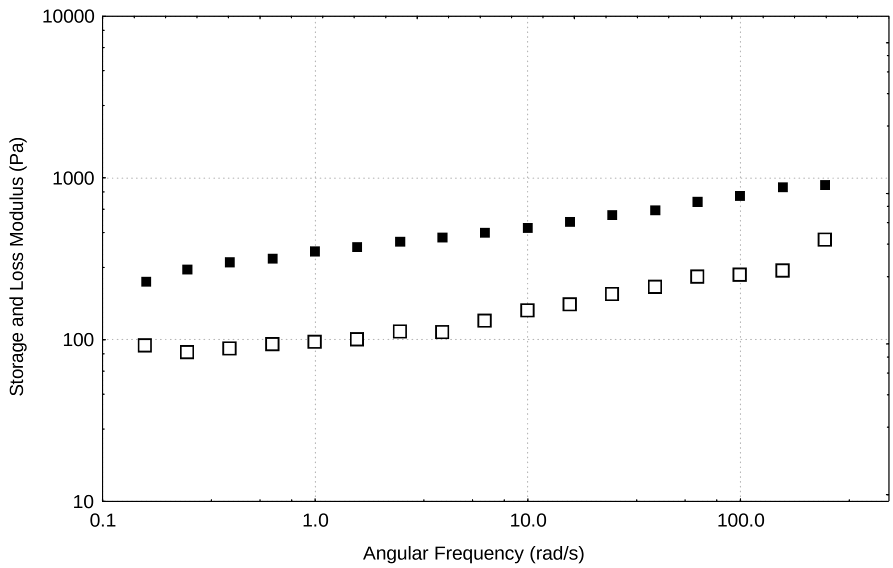


FIGURE 3 - MECHANICAL SPECTRA OF THE SAMPLES CONTAINING 0.50% XANTHAN: VALUES OF G' (▲) AND G" (△)

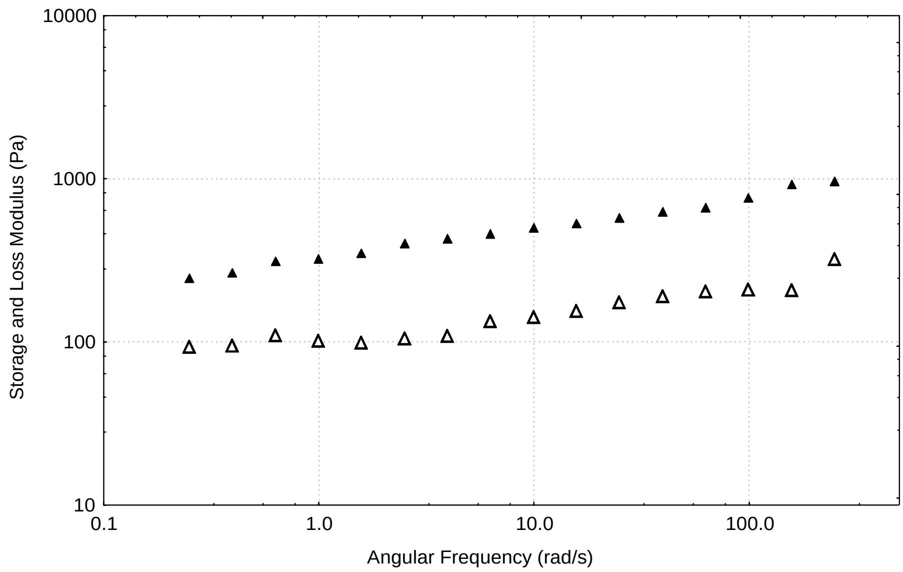


FIGURE 4 - MECHANICAL SPECTRA OF THE SAMPLES CONTAINING 0.75% XANTHAN: VALUES OF G' (◆) AND G" (◇)

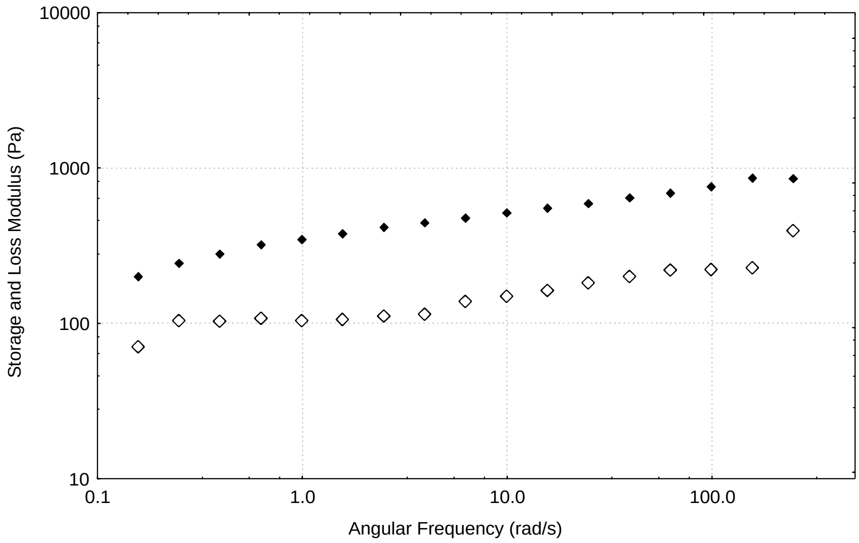
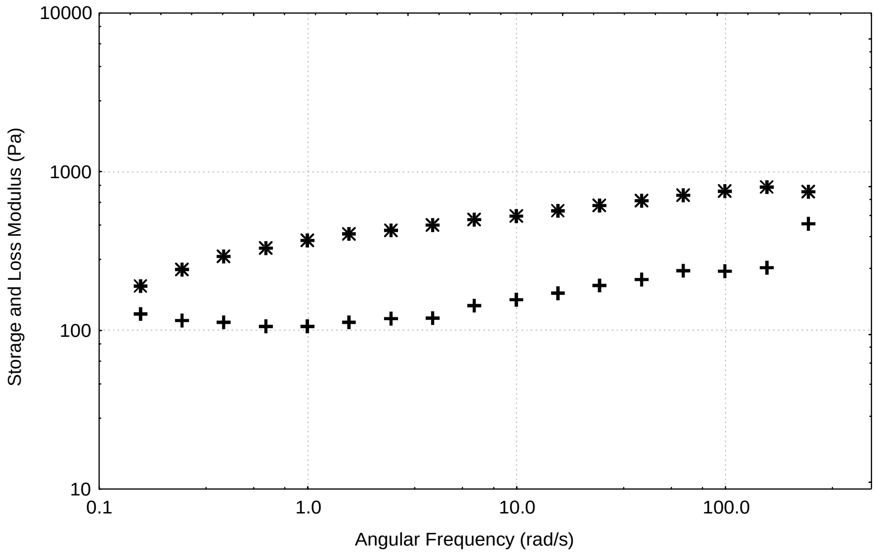


FIGURE 5 - MECHANICAL SPECTRA OF THE SAMPLES CONTAINING 1.00% XANTHAN: VALUES OF G' (✳) AND G" (+)



3.3.1 Cox-Merz rule

The Cox-Merz correlation has been studied for a great number of polymer solutions and complex food systems. In order to examine the applicability of the rule, the apparent viscosity and complex viscosity of samples were plotted against shear rate and frequency, respectively. It was observed that the magnitudes of η^* were lower than those of η, indicating that the Cox-Merz rule was not applicable to these dispersions. Many products such as tomato paste concentrates (RAO & COLOLEY, 1992), waxy maize starch dispersions (CHAMBERLAIN & RAO, 1999), and pectin dispersions (LOPES DA SILVA, GONÇALVES & RAO, 1993), do not obey the Cox–Merz rule.

4 CONCLUSION

Umbu pulp containing different concentrations of xanthan (0.25 - 1.00%) exhibited high shear-thinning behavior with yield stress values (τ_o).The effect of temperature on the apparent viscosity (η_{100}) was clearly depicted by the Arrhenius relationship. Both the power law and the exponential type modes were found to be satisfactory in describing the relationship between concentration and apparent viscosity. The consistency index and apparent viscosity increased with the addition of hydrocolloid and decreased with temperature. Based on the dynamic rheological data of the storage (G') and loss (G") moduli as a function of frequency (ω), the samples displayed a rheological behavior similar to that of weak gel-like macromolecular dispersions, with G' greater than G" for all the values of ω applied. The Cox-Merz rule was not applicable.

RESUMO

EFEITO DA ADIÇÃO DE GOMA XANTANA NAS PROPRIEDADES REOLÓGICAS DE POLPA DE UMBU

Avaliaram-se as propriedades reológicas de suspensões de polpa de umbu contendo o hidrocolóide xantana em várias concentrações (0,25, 0,50, 0,75 e 1,00%) e temperaturas (10, 20, 30, 40 e 50ºC) em regime estacionário e a 30ºC em regime oscilatório. Os experimentos foram conduzidos em reometro Haake RS 600. Os testes em estado estacionário foram conduzidos com taxa de deformação de 0,1 – 1000 s^{-1}. No estado dinâmico, os testes foram conduzidos na freqüência de 0,01 e 100 Hz para caracterizar o comportamento viscoelástico do material. Os módulos de estocagem (G'), perda (G") e tangente (tan δ) foram determinados. Suspensões contendo o biopolímero xantana exibiram comportamento pseudoplástico e apresentaram tensão inicial. Os dados foram modelados com o auxílio da equação de Herschel-Bulkley, sendo o modelo exponencial usado para avaliar o efeito da viscosidade aparente e o modelo de Arrhenius para descrever o efeito da temperatura. As suspensões com a polpa de umbu apresentaram comportamento de gel fraco, no qual os valores dos módulos de estocagem são maiores do que os do módulo de perda. Os módulos elásticos (G') e viscosos (G") aumentaram com a elevação da freqüência (ω).

PALAVRAS-CHAVE: POLPA DE UMBU; GOMA XANTANA; REOLOGIA; ESTADO ESTACIONÁRIO E DINÂMICO.

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