RHEOLOGICAL BEHAVIOR OF MIXED DRINK OF ANNONA AND MILK

MARIA E. M. DUARTE[1], DEYZI S. GOUVEIA[2], MARIO E. R. M. C. MATA[3], ALEXANDRE J. DE M. QUEIROZ[4]

ABSTRACT: In this study the rheological behavior in different temperatures (0; 6; 18 e 24 °C) and physicochemical parameters of integral annona (Annona squamosa) pulp and the annona pulp with milk in different percentages pulp/milk (75g of annona pulp/25g of milk, 50g of annona pulp/50g of milk, 25g of annona pulp/75g of milk) have been availed, in order to verify the effect of temperature and pulp concentration in the rheological behavior of these beverages. To obtain the rheological parameters a concentric cylinder rheometer has been used and the rheograms were analyzed using the Ostwald-de-Wael (power Law) and Herschel-Bulkley models. The physicochemical parameters (sugars, pH, ash, acidity and soluble solids) were determined in order to establish correlations with the rheological behavior. Finally, the best results had been obtained using the Herschel-Bulkley model; the low values for the behavior index (n <1) obtained confirm the pseudoplastic behavior of all samples.

KEYWORDS: fruit, rheology, agro- industry, processing.

COMPORTAMENTO REOLÓGICO DE BEBIDA MISTA DE PINHA E LEITE

RESUMO: Estudou-se o comportamento reológico em diferentes temperaturas (0; 6; 18 e 24 °C) e os parâmetros físico-químicos da polpa de pinha (*Annona squamosa*) integral e também da polpa de pinha com diferentes percentuais de leite (75g de polpa de pinha/25g de leite; 50g de polpa de pinha/50g de leite; 25g de polpa de pinha/75g de leite), com o intuito de verificar o efeito da temperatura e da concentração de polpa no comportamento reológico desses blends. Para a obtenção dos parâmetros reológicos, foi utilizado um reômetro de cilindros concêntricos. Os parâmetros físico-químicos das formulações pH, acidez total titulável, sólidos solúveis totais, sólidos totais e cinzas foram determinados com o intuito de correlacioná-los com o comportamento reológico obtido. Os reogramas foram analisados utilizando os modelos de Ostwald-de-Wael (Lei da Potência) e Herschel-Bulkley. Os melhores resultados foram obtidos usando o modelo de Herschel-Bulkley, e os baixos valores obtidos para o índice de comportamento (n<1) confirmam o comportamento pseudoplástico de todas as amostras.

PALAVRAS-CHAVE: fruta, reologia, agroindústria, processamento.

INTRODUCTION

There are many Brazilian fruits processed in the form of pulps and juices (SANTANA et al., 2008) and others, although well accepted for its taste, are not yet industrialized, among these is the sugar apple (*Annona squamosa*). The family *Annonacea* is composed of approximately 120 genera distributed in tropical and subtropical regions around the world, in which *Annona* is the most important genus within this family, with about 50 species. In the domestic market, lower prices are charged for this fruit in the months of January to mid-March, with a significant increase from April to August (ACAGA & HAL, 2006).

[1] Doutor, Profa. Associado III, Departamento de Engenharia Agrícola, CTRN/UFCG Campina Grande - PB, Fone: 83-2101.1552, melitamd@gmail.com.
[2] Mestre, Profa. Assistente, Depto. de Engenharia Agrícola, CTRN/UFCG, Campina Grande - PB, deyzigouveia@yahoo.com.br.
[3] Prof. Associado III, Departamento de Engenharia Agrícola, CTRN/UFCG, Campina Grande - PB, mmata@deag.ufcg.edu.br.
[4] Prof. Associado III, Departamento de Engenharia Agrícola ,CTRN/UFCG, Campina Grande - PB, alex@deag.ufcg.edu.br.
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There is much interest from major industries in the knowledge of the physical properties of fruit pulps, in view of the rapid growth in national production of juices (SANTANA et al., 2008) and also nectars, ice creams, desserts, gelled, yogurt among others, where the fruit is generally used as raw material in the form of concentrated pulp.

To take advantage of this emerging market, by providing lower loss than the export of fresh fruits, it can guarantee higher profits for exporters; certain domestic industries begin to study the physical characteristics and behavior of certain products to designing new ways of processing. This knowledge is essential to the specific projects of industrial plants, piping, sizing of pumps and stirring and mixing systems, which precede the arrival of the juices on the shelves of supermarkets (TORALLES et al., 2006, VIDAL et al., 2006; SATO & CUNHA, 2007).

The viscosity is the resistance of fluids to flow and it can be considered synonymous with internal friction. Otherwise it can be said that the viscosity corresponds to the internal friction in the fluid due primarily to intermolecular interactions, it is often a function of temperature. The force per unit area required to produce motion is called the shear tension, τ, is proportional to the velocity gradient or strain rate, γ. The proportionality constant between the two is called the consistency index, k (VIDAL et al., 2006).

According to FERNANDES et al. (2009), the fluids that obey Newton postulate are called Newtonian fluids. The flow occurs as soon as voltage is applied, and the coefficient of viscosity is independent of strain rate and shear tension, and the behavior exponent, n, equal to 1. The consistency coefficient (k), this case is referred to as viscosity and is represented by μ. The rheological behavior of fruit pulp in general cannot be described by a Newtonian equation. The non-Newtonian fluids are characterized by apparent viscosity η at a strain-specific rate. The words used to describe the rheological behavior of fluids that do not change their behavior over time are Equation (1) Herschel-Bulkley (OLIVEIRA et al., 2009, OLIVEIRA et al., 2011).

$$\tau - \tau_{OH} = K_H \dot{\gamma}^{nH} \quad (1)$$

In which,

τ- shear tension, Pa;

τ_{OH} - initial tension, Pa;

n_H - fluid behavior index, dimensionless;

K_H - consistency factor (Pa.s), and

$\dot{\gamma}$ - strain rate, s^{-1}.

The food fluids generally is a special case of this expression, in which the threshold voltage of flow ($\tau0$) is null or is assumed to be zero, so that eq.(1) becomes eq.(2), known as Ostwald-de-Waelle model or Power Law (FERNANDES et al., 2009):

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

In which,

τ- shear tension, Pa;
$\dot{\gamma}$ - strain rate, s^{-1}, and
K – consistency index (Pa.s).

The objective of this study was to evaluate the effect of temperature and the proportion of pulp in the rheological behavior of drinks made with sugar-apple pulp and milk.

MATERIAL AND METHODS

The sugar-apples (*Annona squamosa L.*) were obtained from farmers in the state of Paraiba. It was chosen the best looking fruit, opting for firmer fruits, without apparent damage in the skin and in mature stage. The fruit pulp was manually extracted, proceeding the removal of the seeds, and then carrying out the homogenization of the pulp in a blender. In addition to the whole pulp three formulations were prepared of milk sugar-apple pulp, using the following proportions (pulp weight / weight of milk): 75/25, 50/50 and 25/75. The formulations were prepared in a blender to ensure perfect homogeneity.

Rheological measurements were performed on a Brookfield viscometer, model LV - DVII. The study of the rheological behavior was carried out at temperatures of 0; 6; 12; 18 and 24 °C and measurements were made by varying the strain rate from 0.22 to 44 s^{-1} and 0.34 to 68 s^{-1}. The experiments were performed in triplicate using, for each repetition, a new sample equal to the previous one to avoid possible effects of time. The speeds 1; 5; 10; 15; 30; 60; 90; 120; 150; 180 and 200 rpm were used. The measurements were performed with the use of the spindles 25 and 31 and the torque readings always performed after the first 30 seconds of shear. The experimental data obtained for each sample at different temperatures were correlated by two rheological models: Ostwald-de-Waelle (Power Law) and Herschel-Bulkley. The parameters of each model were determined using the software Statistica, version 8.0, using the Quase-Newton method.

The physico-chemical parameters, pH, soluble solids, total solids, ash and titratable acidity were determined according to the rules of the Adolfo Lutz Institute, IAL (2008). The pH was obtained by using a digital pH meter, Model ET-902 brand Digimed accurately 0.01 pH units, previously calibrated with buffer solution pH 4.0 and 7.0. The content of soluble solids (° Brix) was determined using a bench type Abbe refractometer, brand Quimis model Q-109B. The solids were determined by weighing 20g of the pulp at room temperature, followed by drying in an oven at 70 °C to constant weight. The method used to determine ash is the amount of inorganic substances obtained through the ash or the ash sample, based on the residue obtained by burning the sample in an oven at 525 °C. The total acidity of the product was determined by titrating the sample with a solution of 0.1 of N sodium hydroxide, expressing the final result as a percentage of citric acid.

Data analyses were treated according to completely randomized design with three replicates for each sample, using the program Assistat (SILVA & AZEVEDO, 2006).

RESULTS AND DISCUSSION

Physico-chemical characterization

In Table 1 it is found the average values of physico-chemical whole pulp and with the addition of milk in different proportions.

TABLE 1. Values of physico-chemical parameters, with standard deviation, for whole annona pulp with different percentages of milk.

Sample		pH	ATT (%citric acid)	SST (ºBrix)	ST (%)	Ashes (%)	SST/ATT
Pulp	Milk						
100%	0%	5.4 ± 0.053	0.417 ± 0.065	28.467 ± 0.503	29.108 ± 0.467	0.801 ± 0.082	68.266
75%	25%	5.6 ± 0.035	0.357 ± 0.042	23.667 ± 0.416	24.012 ± 0.224	0.795 ± 0.030	66.294
50%	50%	6.0 ± 0.055	0.344 ± 0.046	18.067 ± 0.808	20.005 ± 0.250	0.784 ± 0.023	52.520
25%	75%	6.4 ± 0.053	0.270 ± 0.014	15.467 ± 0.306	15.727 ± 0.765	0.720 ± 0.241	57.285

ATT- Total titrable acidity, TSS- Total soluble solids, ST- total solids

It is observed from Table 1 that the pH and total acidity of the whole pulp are within the range of values determined by SALGADO et al. (1999) for the same product which was 5.49 units of pH and acidity of 0.45. The variation in physico-chemical composition of fruit juices is due to many factors such as variety, maturation stage, planting location, time of planting and harvesting, among others (SILVA et al., 2005).

It appears that the addition of milk to sugar-apple pulp brought to a higher pH, corresponding to an increase in pH of the sample with a higher proportion of milk to 17.26%. This is due to the pH of the milk being greater than that of the fruit pulp. The pH of the fruit in general is around 3.5. This value expresses the strength of the acids present in the juice which is correlated with a sensation of taste (VICENZI, 2006).

With respect to the total acidity of the integral sugar-apple pulp it was found that there was a decrease of 35.72% in the sample with 75% milk. This behavior was expected because the milk has low acidity which contributes to the dilution of organic acids existing in the pulp. Just as occurred with the total acidity, soluble solids also decreased with increasing proportion of milk in the samples. The total soluble solids (º Brix) of the sugar-apple pulp are in the same range of values determined by ALVES et al. (2000), UGULINO et al. (2005) for this product. The maximum reduction of soluble solids (º Brix) was 45.66%, occurred in the sample with the highest percentage of milk.

The values of total solids of the whole pulp is greater than the value determined by UGULINO et al. (2005), decreases with increasing concentration of milk. There was a reduction of 49.4% of total solids in the sample with 75% milk in the fruit pulp. The ash content of the whole pulp fruit is 10.11% less than the sample with 75% milk.

The relationship between soluble solids and titratable acidity (TSS / TA) was higher in the whole pulp, followed by the sample with 25% milk and 75% of the fruit pulp. It is noted that the lower relation in relation to TSS / TA was observed in the composition of the sample with 50% milk and 50% pine pulp. SALGADO et al. (1999) found for the integral sugar-apple pulp values for TSS / TTA rate in order of 32.06, and the total soluble solids, TSS, were equal to 14.43 ° Brix and total titratable acidity, TTA, 0.45 %. This ratio is well below that found in this study (68.266). This fact can be explained due to the high amount of soluble solids in the order of 28.4 ° Brix.

Rheological behavior

Effect of pulp temperature and concentration of the apparent viscosity

Table 2 shows the parameter values of the Ostwald-Waelle (Power Law) and Herschel-Bulkley models obtained through the adjustment to experimental data for the whole sugar-apple pulp and pulp with different percentages of milk at temperatures of 0; 6; 12; 18 and 24 °C.

Analyzing the results in Table 2 for the Ostwald-Waele (Power Law) model, it appears that the parameter k (consistency index) of different samples at the same temperature increases with increasing pulp concentration, except for the whole pulp. Several factors affect the rheological behavior of fruit pulp, including temperature and concentration of soluble solids. Higher concentrations of pulp used in the formulations provide the concentration of soluble solids, and thus the amount of free water in the mixture decreases, thereby increasing the consistency and enhancing the stabilizing effect.

In this study it was found that the milk when added in small proportion (up to 25%) causes a certain arrangement which increases the consistency of these samples. This fact was observed for samples of sugar-apple pulp with 25% of the whole milk at all temperatures. However, when the percentage is greater than 25%, as expected, there is less consistency at all temperatures. Perhaps the high water content of the milk with the fat can occasion dilution, with consequent loss of consistency. Food materials are complex rheological structure consisting of a mixture of solids, liquids and structural components and any change in the content of these constituents may lead to changes sometimes unpredictable in its structure (OLIVEIRA et al, 2009; PEREIRA et al., 2008).

TABLE 2. Adjustment parameters of Ostwald-de-Waelle (Power Law) and Herschel-Bulkley models, for whole annona pulp and annona pulp with different percentages of milk, as a function of temperature.

			Ostwald-de-Waele (Power Law)			Herschel-Bulkley			
Concentration Pulp	Milk	Temp (ºC)	K(Pas^n)	n	R^2	K_H (Pa.s)	n_H	τ_{OH}(Pa)	R^2
100%	0%	0	224.417	0.370	99.161	124.185	0.495	117.798	99.463
		6	209.313	0.375	99.180	115.600	0.502	110.801	99.481
		12	170.796	0.414	98.543	47.707	0.708	160.378	99.638
		18	158.888	0.420	98.647	48.942	0.691	142.900	99.544
		24	158.745	0.402	98.690	58.287	0.627	125.658	99.395
75%	25%	0	309.578	0.285	99.843	249.439	0.324	63.601	99.875
		6	265.801	0.318	99.517	197.677	0.376	73.959	99.578
		12	246.571	0.322	98.919	156.387	0.413	98.502	99.046
		18	189.335	0.370	99.030	82.482	0.552	125.948	99.481
		24	162.994	0.390	98.640	44.913	0.684	148.010	99.679
50%	50%	0	81.669	0.309	99.301	145.348	0.214	66.599	99.536
		6	66.987	0.351	99.773	54.859	0.391	13.519	99.804
		12	63.294	0.359	99.505	38.865	0.461	27.864	99.679
		18	52.733	0.398	99.536	42.791	0.441	11.624	99.570
		24	44.821	0.432	99.356	33.545	0.495	13.824	99.421
25%	75%	0	14.849	0.311	99.038	23.208	0.239	-9.140	99.616
		6	10.721	0.351	99.587	10.607	0.352	0.133	99.587
		12	10.430	0.354	99.657	11.695	0.333	-1.474	99.664
		18	11.190	0.315	99.423	16.708	0.249	-6.065	99.502
		24	9.109	0.357	99.546	8.406	0.372	0.836	99.550

For the Herschel-Bulkley model, the results obtained for consistency index (K_H) showed oscillations and without tendency defined in relation to concentration and temperature.

The values for the fluid behavior index (n and n_H), for Ostwald-de-Waelle and Hershel and Bulkley models, respectively, are below 1.0, characterizing the behavior of fluid mixtures as non-Newtonian and pseudoplastic, as it was expected, since such behavior is common in pulp and fruit juice, as reported by numerous authors as PEREIRA et al. (2008) in a study of the rheological behavior of umbu. BEZERRA et al. (2009) to analyze the model parameters for Hershel Bulkley rheological behavior of strawberry pulp found that all the n_H values were also below 1.0, with values of at most 0.24.

Pictures 1-2 are curves representing the shear tension as a function of strain rate, as described by the model of Ostwald-de-Waelle and Hershel Bulkley, respectively, obtained at different temperatures and concentrations. Based on statistical parameters analyzed (R^2), the adjustments of the two models to the experimental data are considered satisfactory.

It can be seen by analysis of the Figures 1 and 2 that with the increase of pulp concentration in the samples, the higher shear stress is required. This behavior is characteristic of a pseudoplastic fluid that follows the model of power (Power Law).With the increase of shear tension increases in the strain rate at all concentrations. It is also noted that inasmuch as the increase in the temperature, a decrease of the shear tension. OLIVEIRA et al. (2011) working with strawberry pulp and Pereira et al. (2008), working with umbu observed the same behavior.

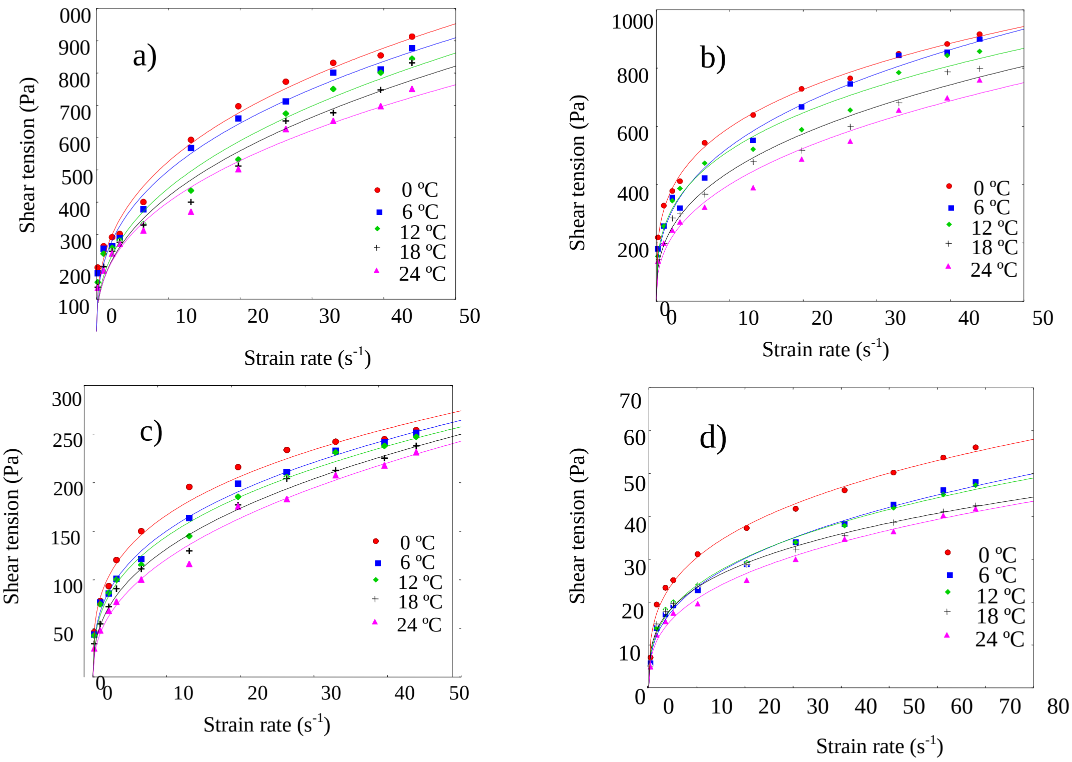
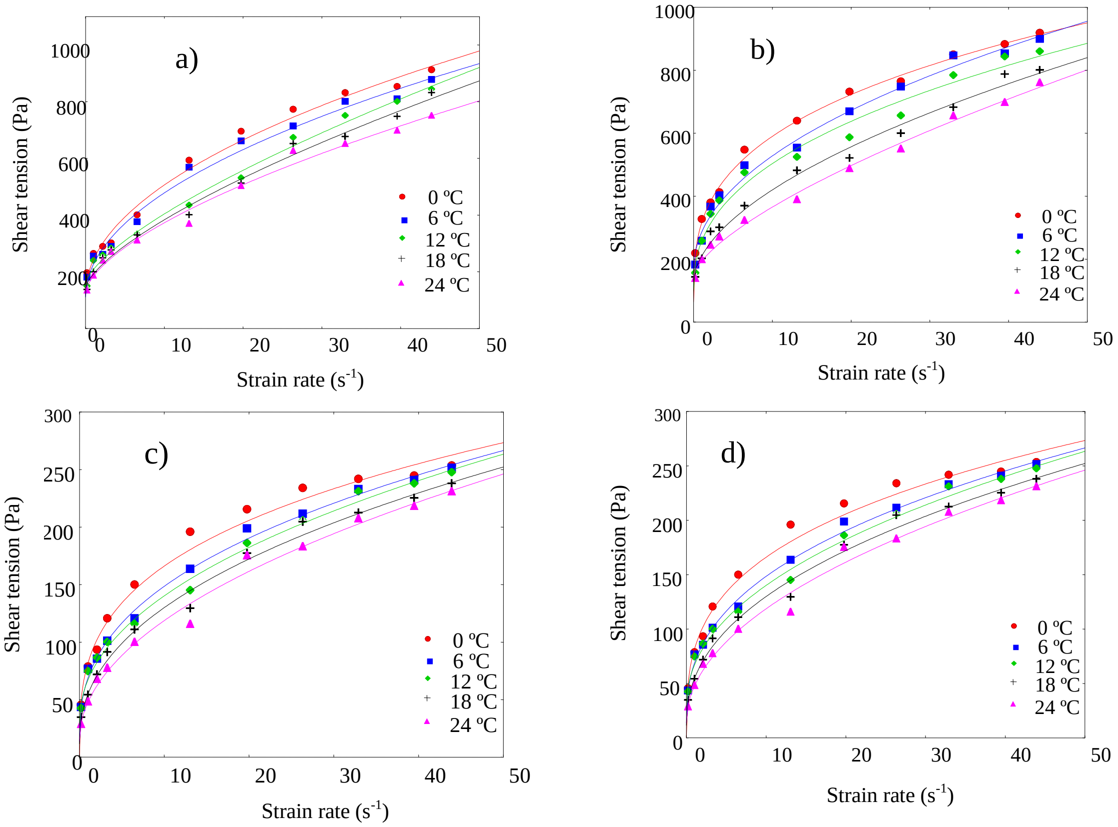


FIGURE 1. Relationship between shear tension and strain rate for samples with a concentration of annona pulp/milk as follows: a) 100% pulp and 0% milk; b) 75% pulp and 25% milk; c) 50% pulp and 50% milk; d) 25% pulp and 75% milk, described by the Ostwald-de-Waelle model (Power Law) at different temperatures (0; 6; 12; 18 and 24 °C).



PICTURE 2. Relationship between shear tension and strain rate for samples with a concentration of annona pulp/milk as follows: a) 100% pulp and 0% milk; b) 75% pulp and 25% milk; c) 50% pulp and 50% milk; d) 25% pulp and 75% milk, described by the HershelBulkley model at different temperatures (0; 6; 12; 18 and 24 °C).

It can be observed in Pictures 1 and 2 that for strain rates fixed, the shearing stress decreases as the temperature increases, indicating reduction of the apparent viscosity during the heating process.

Table 3 shows the values of apparent viscosity of the different mixtures annona / milk obtained for a rotation of 200 rpm and a strain rate of 40 s^{-1} setting.

TABLE 3. Apparent viscosity of the annona pulp with different percentages of milk as a function of temperature.

Concentration		0 ºC	6 ºC	12 ºC	18 ºC	24 ºC
Pulp (%)	Milk(%)					
100	0	2097.409	2041.682	1948.682	1888.409	1724.320
75%	25%	2068.727	1990.591	1915.409	1817.727	1702.045
50%	50%	575.909	571.136	560.909	539.772	524.318
25%	75%	82.353	70.588	69.558	62.647	61.470

It is noted from Table 3, a decrease in apparent viscosity with increasing temperature, at all concentrations and an increase in the apparent viscosity with increasing pulp concentration in the sample. The same behavior was observed by Oliveira et al. (2011) to study the rheological behavior of strawberry pulp; PEREIRA et al. (2008), working with umbu.

When the pulp concentration in the sample decreases the viscosity decreases intensely, indicating that the viscosity has a strong dependence on the solids content, a phenomenon also observed by GUEDES et al. (2010) studying the effects of temperature and concentration in the physical properties of the watermelon pulp.

The value of the apparent viscosity decreases with increasing temperature and increases with increasing concentration of solids. Many authors have observed this type of behavior such as GRANGEIRO et al. (2007), studying the viscosities of concentrated pulps of India-fig; BRANCO and GASPARETTO (2005) studied the rheology of mango pulp with different proportions of orange juice and acerola fruit; VIDAL et al. (2006) studied the rheological properties of the centrifuged mango pulp.

CONCLUSIONS

The rheological parameters initial tension and the viscosity increase with the addition of annona pulp, i.e. the increase in viscosity occurs with the increase in soluble solids content.

The apparent viscosity obtained by the Ostwald-Waelle (Power Law) and Hershel Bulkley model, for all formulations, decreased with the increasing of the strain rate and with the temperature increase.

The flow behavior index (n), determined by the rheological model of Ostwald-de-Waelle (power law) and Hershel Bulkley, had values lower than the unity under all conditions, which defines the sugar-apple pulp to different percentage of milk as a non-Newtonian fluid with time-independent behavior and fitting into the pseudoplastic category (BEZERRA et al., 2009).

The models of Herschel-Bulkley and Ostwald-de-Waelle, based on statistical parameters, provided a good adjustment to experimental data. The flow behavior index determined through these models showed lower values than the unit, characterizing the whole annona pulp and the pulp with milk in the proportions pulp weight / milk weight of 75/25 50/50 and 25/75 as a non-Newtonian fluid with time-independent behavior and fitting into the pseudoplastic category.

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