A RESEARCH NOTE ON RHEOLOGICAL BEHAVIOR OF SOME PROCESSED PRODUCTS FROM CACTUS PEAR (*OPUNTIA FICUS-INDICA* [L.] MILL.)

P. CEREZAL[1,3], E. CASTRO[2] and G. DUARTE[1]

[1]*Departamento de Alimentos*
Facultad de Recursos del Mar
Universidad de Antofagasta. Avda.
Universidad de Antofagasta # 02800, Campus Coloso
Casilla 170, Antofagasta, Chile

[2]*Departamento de Ingeniería en Alimentos*
Facultad de Ciencias Químicas y Farmacéuticas
Universidad de Chile. Avda.
Vicuña McKenna # 20, Santiago, Chile

ABSTRACT

A study of some textural and rheological properties of cactus pear products was carried out in this research. The parameters of brittleness and hardness in peeled cactus pears in syrup (PCPS), and the compression stress in the sweetened pulp from cactus pears with partial addition of their skins (SPCPS) and in the marmalade from cactus pear skins (MCPS) were evaluated. Flow behavior was only evaluated in SPCPS and MCPS. A significant variation of the maximum force between different samples of PCPS in the range of 544.3–975.3 N was determined, while the deformation was 17.1–21.2 mm. In the SPCPS and MCPS products, the maximum stresses were low, 0.254 and 0.366 N/mm^2, although the deformation was of 28.8 and 29.07 mm, respectively. SPCPS was a pseudoplastic fluid, with a better fit to the general model of Herschel–Buckley. Meanwhile, MCPS was represented by a polynomial model of third order.

PRACTICAL APPLICATIONS

The results of this study indicate that there is a relatively wide interval in the degree of ripeness of the fruits, detected by instrumental evaluation, which

[3] Corresponding author. TEL: 56 (55) 637456; FAX: 56 (55) 637265; EMAIL: pcerezal@uantof.cl

can be used for the development of the peeled cactus pears in syrup product despite the previous selection of the fruits carried out in the elaboration process and are accepted by trained testers without being a sensorial defect. On the other hand, the partial or total incorporation of the skins in sweetened pulp from cactus pears with partial addition of their skins and in marmalade from cactus pear skins semisolid products, respectively, allows a greater use of the fruit, while the mixture of pectin and mucilage in the skins contributes to increasing the consistency without making necessary the addition as external additives.

KEYWORDS

Herschel–Buckley model, non-Newtonian flow, peeled cactus pears, semisolid products from cactus pears, textural and reologhical properties

INTRODUCTION

Textural and rheological properties of the processed fruits integrate a set of physical parameters that have direct influence in the processing, packaging and selection of the type of package. In addition, they define the quality of product for commercialization. Texture is an important aspect of food quality, sometimes even more important than flavor and color. It significantly influences people's image of food. It is the most important in bland foods and foods that are crunchy or crispy. Texture refers to those qualities of food that can be felt either with fingers, tongue, palate or the teeth. The range of textures in foods is very wide, and a deviation from an expected texture is considered a quality defect (Ragaee 2003).

The viscosity of fluid foods is an important transport property, and within the food industry, the study of the rheological behavior of products is very useful for (1) designing of food processes and processing equipment; (2) calculations of engineering processes such as selection of pumps, determination of pressure drops in pipes, unit operations like sterilization and evaporation, among others; (3) determination of the characteristics of an ingredient and its behavior within a product; (4) prediction of storage and stability measurements; (5) study of shelf life; and (6) quality evaluation and control of food products to evaluate the texture of the food and to correlate it with the sensorial evaluation (Holdsworth 1993; Krokida *et al.* 2001; Medina *et al.* 2003).

Theoretical and semiempirical models do not adequately predict the viscosity of foods because of the complex chemical and physical structure. Therefore, empirical models are necessary to characterize fluid food experimental measurements (Krokida *et al.* 2001).

The materials under investigation can range from low-viscosity fluids to semisolids and gels, to hard, solid-like food products (Medina *et al.* 2003).

Newtonian fluids have a straight line relationship between the shear stress (σ) and the shear rate (γ) with a zero intercept. The behavior of non-Newtonian fluids can be described by the Herschel–Buckley model $\sigma = \sigma_o + K\gamma^n$, where K is the consistency coefficient, n is the flow behavior index and σ_o is the yield stress. Many fluid foods are governed by this model which is applicable to shear thinning (pseudoplastic) when $0 < n < 1$, or shear thickening (dilatent) when $1 < n < \infty$, while Bingham plastic behavior may be considered as a special case. For the Newtonian and Bingham plastic models, K is commonly called the viscosity (μ) and plastic viscosity (μ_p), respectively (Steffe 1996; Schramm 2000).

Cactus pear (*Opuntia ficus indica* [L.] Miller) is a fruit that has had an excellent development during the last two decades, specifically as related to the industrial exploitation and characterization of each of its parts: skins, seeds and pulp (Forni *et al.* 1994; El Kossori *et al.* 1998; Sáenz 2000; Majdoub *et al.* 2001; Ramadan and Mörsel 2003a,b; Cerezal and Duarte 2004, 2005a,b; Habibi *et al.* 2004a,b, 2005; Ennouri *et al.* 2005).

Only a few studies have dealt with the rheological properties of extracted products from cactus pears. Some examples such as rheological and mechanical properties of the mucilage gum (*Opuntia ficus indica*) and of gels formed by mixtures with carrageenans (Medina *et al.* 2000, 2003), rheological behavior of prickly pear seed oils (Ennouri *et al.* 2005), characterization of pectin from prickly pear (*Opuntia ficus indica*) peel (Forni *et al.* 1994), isolation and structural characterization of protopection from the skin of *Opuntia ficus-indica* prickly pear fruits (Habibi *et al.* 2005) and structural features of pectic polysaccharides from the skin of *Opuntia ficus-indica* prickly pear fruits (Habibi *et al.* 2004a) have been studied.

The objective of the present research was a preliminary study of the textural and rheological properties of three products from cactus pears. The parameters, brittleness and hardness in peeled cactus pears in syrup (PCPS), and the compressive stress in both semisolid products, sweetened pulp of cactus pear with partial addition of skins (SPCPS) and marmalade from cactus pear skins (MCPS), were evaluated. Flow behavior was also determined for the last two semisolid products.

MATERIALS AND METHODS

The processing of products was carried out on a pilot scale using cactus pears (*Opuntia ficus-indica* [L.] Miller) of green coloration, corresponding to the winter harvest, in their fresh condition with a time $\leq$48 h of harvest and selected based on firm ripeness, and being free of diseases. The origin of place of harvest was the community of Caspana, located at an altitude of 3,260 m over the sea level to the Northeast of the Calama City (Anonymous, 2004), belonging to the Andean High Plain of II Region of Chile.

For the characterization of texture and rheological behavior, packages were selected at random, after 2 weeks of storage at room temperature ($T = 20 \pm 5$C), for three products made from cactus pears. PCPS, SPCPS and MCPS were prepared and packaged as described by Cerezal and Duarte (2004, 2005b). Calcium chloride was only added to peeled cactus pears in syrup (PCPS) to influence its firmness. The consistency acquired in both semisolid products, SPCPS and MCPS, was by the addition of sucrose, as well as by the inherent contents of pectin and calcium.

Description of Products and Their Characteristics

PCPS. Whole cactus pears without skins were prepared in syrup with the addition of sucrose, and a phosphoric and citric acid mixture (50% v/v) to obtain in the final product, pH = 4.0–4.2, water activity (A_w) = 0.96 (≈20°Brix). The calculation of sucrose concentration in the syrup to obtain the A_w in equilibrium was performed using Ross equation that is expressed as $A_{w\ equilibrium} = (A_{w\ fruit})\ (A_{w\ syrup})$, where $A_{w\ fruit}$ and $A_{w\ syrup}$ are the initial water activities of the fruit and the sucrose syrup, respectively. Water activity ≈ 0.99 for fresh cactus pears was assumed, but the value $A_{w\ syrup} = 0.97$ was calculated. The sucrose syrup concentration was determined by Norrish equation according to $A_{w\ sucrose\ syrup} = X_1 \exp\left(-KX_2^2\right)$, where X_1 and X_2 are water and sucrose molar fractions, respectively, and K value is a constant whose value is 6.47 for sucrose (Alzamora 1997; Welti and Vergara 1997; Barbosa-Canovas *et al.* 2003).

PCPS was also prepared using added potassium sorbate, ascorbic acid and calcium chloride in concentrations of 1,000, 500 and 120 ppm, respectively. The relation of weight drained to syrup was considered in 60:40 (w/w), and it was poured and sealed in glass jars of 440 mL of capacity with twist-off lids (Cerezal and Duarte 2004).

SPCPS. Ground cactus pear fruits, without skins and seeds, with skins ground incorporated (in pulp–skins relation of 3:1), sucrose addition to obtain in the final product, A_w = 0.94 (≈40°Brix); pH = 3.2–3.4 was obtained by the addition of phosphoric acid solution, 50%, as well as potassium sorbate, ascorbic acid and sodium bisulphate in concentrations of 1,000, 500 and 100 ppm, respectively. They were poured and thermally sealed in polyethylene bags (Cerezal and Duarte 2005b).

TABLE 1.
COMPOSITION DATA OF THE PROCESSED PRODUCTS FROM CACTUS PEAR

Determinations*		Peeled cactus pears in syrup	Sweetened pulp from cactus pears with partial addition of their skins	Marmalade from cactus pear skins
Soluble solids	(°Brix)	19.8	40.0	63.5
Moisture content	(%)	75.48	50.41	37.03
Total solids	(%)	24.25	49.59	62.97
pH		4.20	3.34	3.94
Acidity	(% citric acid)	0.10	0.33	0.25
Crude fiber	(%)	2.30	0.19	–
Pectin	(%)	0.08	0.28	–

* Average of five observations.

MCPS. Ground cactus pear skins were mixed with sucrose, citric acid and potassium sorbate to obtain in the final product a soluble solid concentration of 63°Brix, pH = 4.0 and 250 ppm, respectively. They were poured and sealed in glass jars of 440 mL capacity with twist-off lids (Cerezal and Duarte 2005b).

The main physical and chemical characteristics of the three products are shown in Table 1. The sensorial evaluation conducted by trained tasters, directed only to the texture characteristic (PCPS) and consistency (SPCPS and MCPS), produced the results that are shown in Table 2. In these cases, the technique of characterization by means of scale by attributes was used. All characteristics were evaluated on a 10-cm linear scale, with verbal anchors on the extremes. However, each attribute had a maximum value of 5 points (Cerezal and Duarte 2004, 2005b).

Ten students aged between 18 and 25 were selected for sensory evaluation based on a prescreening questionnaire. The training of the panel included theoretical and practical sessions on the assessment attributes belonging to two categories (firmness and consistency). Analysis of variance (ANOVA) was performed in order to determine which tasters had slight deviations of the average values in comparison with the rest. The evaluation of each product was performed in a room with a controlled temperature inside of the standard sensory cabinet with illumination. In each session, the products were presented in their containers.

Instrumental Evaluation. Compressive Stress. Five glass jars of PCPS were opened, and the syrup was separated from solids by means of draining on a stainless steel sieve, and four fruits were chosen at random out of a sample of about 40. A Universal Machine (Materials Testing Machine,

TABLE 2.
SENSORY EVALUATION OF CACTUS PEAR PRODUCTS FOR THE TEXTURE CHARACTERISTIC

Products	Attributes*	Values			Category of qualification
		Obtained		Maximum	
		$\bar{X}$	S		
Peeled cactus pears in syrup	Firmness	3.58	0.70	5.00	Acceptable
Sweetened pulp from cactus pears with partial addition of their skins	Consistency	4.56	0.57	5.00	Excellent
Marmalade from cactus pear skins	Consistency	4.00	0.74	5.00	Good

* Attributes measured on a 10-cm scale with a maximum score of 5.
$\bar{X}$ and S are the mean and SD of 10 panelists.

model LR-5K Plus series, Lloyd Instruments, Fareham, U.K.) interfaced with a microcomputer for data acquisition was used. The test sample was placed in the Kramer shear compression cell, and the crosshead speed was set at 100 mm/min. From the force–deformation curve (Fig. 1), different texture parameters, brittleness and hardness (Borges and Peleg 1997; Costell *et al.* 1997; Corradini and Peleg 2006) were calculated for PCPS product using the following relationships:

$$\frac{\text{Brittleness}}{d_1} = \text{N/mm} \tag{1}$$

$$\frac{\text{Hardness}}{d_2} = \text{N/mm} \tag{2}$$

where the term on the left-hand side of Eq. (1) is known as the first maximum peak in the force–deformation curve (N), and the term on the left-hand side of Eq. (2) is known as the second maximum peak in the force–deformation curve (N) (Fig. 1).

For the products, SPCPS and MCPS, from the three packages, were taken and placed in the Universal Machine with a 100-N load cell. The runs were carried out with a 12.5-mm diameter cylindrical probe at a compression rate of 100 mm/min, registering the values through a microcomputer provided with the DAPMAT 3.05 software (DAPMAT 40-0465, v. 3.05), giving the value of force, the maximum force applied, the deformation to the maximum load

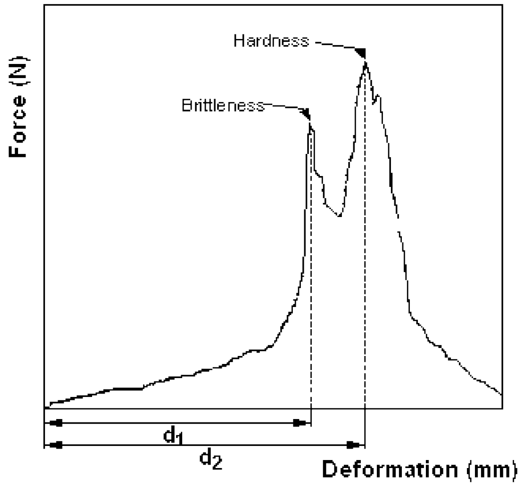

FIG. 1. A TYPICAL FORCE–DEFORMATION CURVE FOR CACTUS PEARS
Where d_1 and d_2 are the distances in millimeter until the first and second maximum peak of the curve, respectively.

(mm) and the differential stress when the maximum weight is applied (%). All tests were performed at $T = 20 \pm 5$C in triplicate samples.

Instrumental Evaluation. Rheological Behavior. In addition, these last products were measured using a controlled stress rheometer (Haake RS 100, Haake-Mess-Technik GmbH, Karlsruhe, Germany) with a 35/4° cone-plate system. The measurements were conducted in a controlled-shear rate mode, and the resulting shear stress was measured. All tests were performed at $T = 20 \pm 5$C in triplicates. The shear rate ranged from 0 to 650/s. Finally, the rheological behavior was evaluated for fit with different traditional models such as Bingham, Power Law as two-parameter models and Herschel–Buckley as a three-parameter model.

Statistical Evaluation. All the results of the determinations are in the form of average values ($\bar{\mathbf{X}}$) and their corresponding standard deviations (S). Adjustments between dependent and independent variables were made, choosing the mathematical models whose coefficients of determination (R^2) were the highest (Gutiérrez and de la Vara 2003). In all the analyses, 95% confidence was used. The values of linear correlation coefficients were calculated, and their significance was examined by Student's t-test or ANOVA test at $P < 0{:}05$. The statistical calculations were made with the support of software Statgraphics 5.1 for Windows.

RESULTS AND DISCUSSION

Texture Behavior

PCPS. Figure 2 shows the compression curves made to different samples of whole PCPS. As it can be seen in Table 3, there is a significant variation between the values of the maximum force applied in brittleness of samples 1 and 4 (<758 N) with samples 2 and 3 (>962 N); nevertheless, the deformation has a wide range, 17.1–18.8 mm, if it is compared with the other narrow range of 17.1–17.7 mm, respectively. The same does not happen for hardness, because samples 1 and 2 (>764 N) have a deformation range slightly higher, 20.7–21.2 mm, in comparison with the group formed by samples 3 and 4 (<605 N) whose deformations range from 19.7 to 19.8 mm. These results agree with the values calculated for the relation brittleness/d_1 and hardness/d_2.

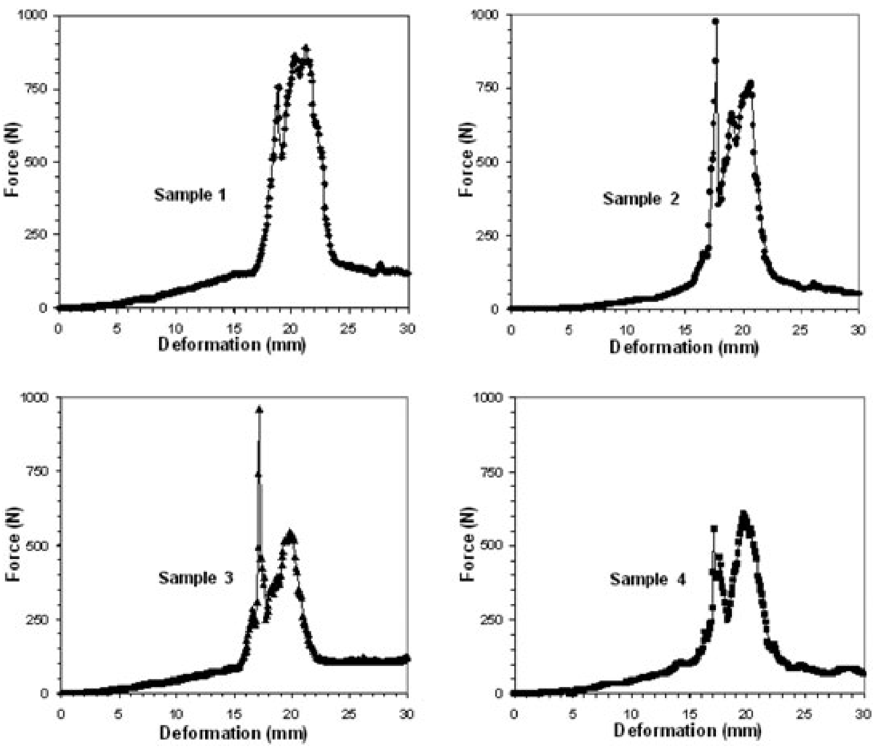

FIG. 2. TYPICAL FORCE–DEFORMATION CURVE FOR PEELED CACTUS PEARS IN SYRUP (SAMPLES 1–4)

TABLE 3.
SOME PARAMETERS OF THE TEXTURE PROFILE OF PEELED CACTUS PEARS IN SYRUP

Samples*	Brittleness (1st peak)	Hardness (2nd peak)	Deformation (distance)		Brittleness	Hardness
					d_1	d_2
	Force (N)	Force (N)	d_1 (mm)	d_2 (mm)	N/mm	N/mm
1	757.3	890.4	18.8	21.2	40.3	42.0
2	975.3	764.0	17.7	20.7	55.1	36.9
3	963.7	544.3	17.1	19.8	56.4	27.5
4	555.3	604.4	17.1	19.7	32.5	30.7

* Average of three replications.
d_1 and d_2, the distances in millimeter until the first and second maximum peak of the curve, respectively.

The force–deformation curves for PCPS were very heterogeneous under the same processing conditions. Vincent (2004) has suggested that heterogeneity is probably, therefore, an important adjunct to brittleness in generating a crisp texture.

These results of the measurement of the instrumental texture indicated that all the fruits did not have a similar state of ripeness, in spite of the previous selection of the fruits made by the staff in the PCPS elaboration process based on the "visual" inspection and "firmness" determination to the touch. In other words, those fruits that displayed greater brittleness were not necessarily those of greater hardness or those with a greater deformation (samples 2 and 3).

On the other hand, sample 1 reached average values of brittleness and maximum values of hardness, and was the one that presented the highest values for the deformation, in the first and second peaks, respectively. Sample 3, in spite of being one of the two samples with greater brittleness, was the one with greater relation of brittleness/d_1 = 56.4 N/mm, and at the same time was less hard, because it presented a hardness/d_2 = 27.5 N/mm. Although firmness was evaluated by a group of tasters trained with the category of "acceptable" for this product (Table 2), there are relatively wide intervals in the degrees of ripening of fruits that are detected by the instrumental evaluation. Some studies of other fruits, such as banana and mango, have obtained similar results (Banjongsinsiri *et al.* 2004; Hofsetz and Lopes 2005).

These results of the measurement of the instrumental texture indicated that not all the fruits had a similar state of ripeness, despite the prior selection of the fruits carried out by the staff in the process of elaboration. Another effect in relation to the few definitions of the force–deformation curves is the content of seeds in the cactus pear fruits, resulting in alterations in the measurement of texture, creating small zones that move from the average values.

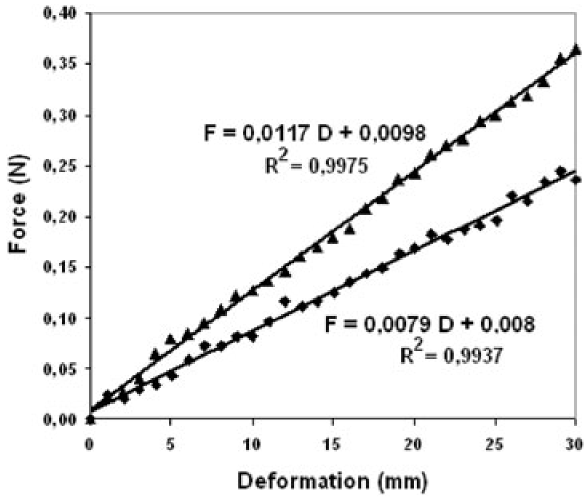

FIG. 3. TYPICAL FORCE–DEFORMATION CURVES FOR THE SWEETENED PULP FROM CACTUS PEARS WITH PARTIAL ADDITION OF THEIR SKINS AND MARMALADE FROM CACTUS PEAR SKINS

SPCPS and MCPS. In Fig. 3, the compression curves for SPCPS and MCPS are presented, to which the linear regression model was applied, obtaining high coefficients of determination, $R^2 = 0.9937$ and $R^2 = 0.9975$, respectively. The slope of the straight line for MCPS reached values of 1.5 times higher than that of SPCPS; both products needed a low initial penetration force, represented by the intercept of the straight lines with the axis *y*. The ANOVA showed the calculated *F* value (force versus deformation) for SPCPS ($F_{\text{calc.}} = 4404.83$) and MCPS ($F_{\text{cal}} = 11750{,}48$) is much greater than 10 times the tabulated *F* value ($F_{0.95;1;28}$) = 4.20. Besides, the *P* value is less than 0.05, demonstrating that there is statistically a significant relation between force and deformation for the level of confidence established.

These values are a result, first, of the concentration of total solids of the products (MCPS = 62.97% and SPCPS = 49.59%), and second, by the partial incorporation of skins in SPCPS and total skins in the MCPS. It must be remembered that the skins have high contents of mucilage and pectin, which contribute to greater stiffness in semisolids foods (Forni *et al.* 1994; Medina *et al.* 2000, 2003; Majdoub *et al.* 2001; Habibi *et al.* 2004a,b, 2005).

Table 4 shows a summary of the maximum values of stress and deformation applied, as well as the differential of stress in the tests of compression for both products.

TABLE 4.
MAXIMUM STRESS APPLIED DURING COMPRESSION IN THE SWEETENED PULP FROM CACTUS PEARS WITH PARTIAL ADDITION OF THEIR SKINS (SPCPS) AND MARMALADE FROM CACTUS PEAR SKINS (MCPS)

Rheogram parameters	SPCPS		MCPS	
	$\bar{X}$	S	$\bar{X}$	S
Maximum stress applied (N/mm^2)	0.254	0.004	0.366	0.017
Deformation to the maximum load (mm)	28.80	0.43	29.07	0.70
Differential of stress when the maximum load was applied (%)	72.00	1.07	72.67	1.74

$\bar{X}$ and S are the mean and SD of six replications.

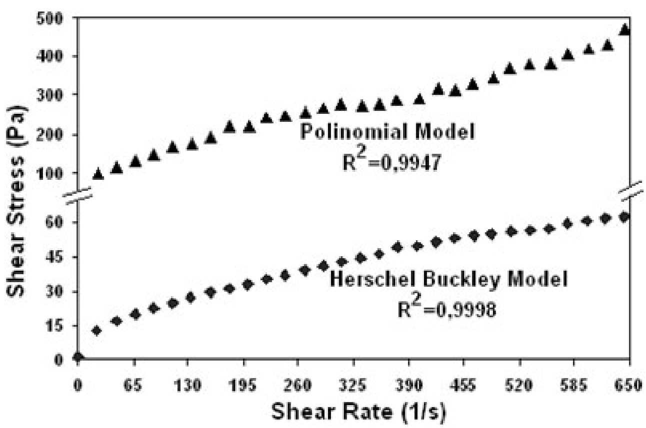

FIG. 4. FLOW CURVES OF THE SWEETENED PULP FROM CACTUS PEARS WITH PARTIAL ADDITION OF THEIR SKINS AND THE MARMALADE FROM CACTUS PEAR SKINS

Rheological Behavior

Figures 4 and 5 show the flow curves of the SPCPS and MCPS, and in both cases, a non-Newtonian behavior is seen. A behavior of pseudoplastic fluid for the SPCPS at $T = 20 \pm 5$C is observed (Table 5) and the Power Law and Herschel-Buckley models are those that better adjustments have, being the latter the one with greater R^2 (Fig.4). Similar results were obtained for banana puree with soluble solids of 22.2°Brix, 0.9% of starch and 2.4% total fiber, and the values of the coefficients were $\sigma_o = 81.04–40.94$ Pa, $K = 4.67–0.03$ Pa $\cdot$ s^n and $n = 0.442–0.966$ for an interval of temperatures of 30–120C, respectively (Ditchfield *et al.* 2004). The rheological behavior of Brazilian orange juice

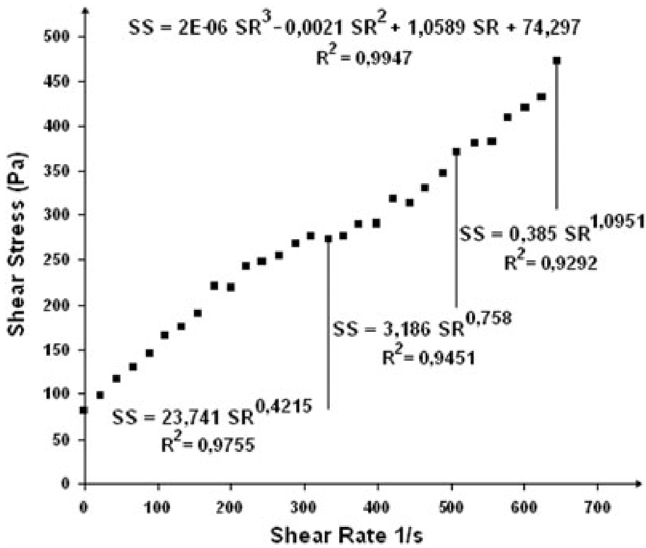

FIG. 5. SPECIFIC BEHAVIOR OF DIFFERENT ZONES OF THE FLOW CURVE FOR MARMALADE FROM CACTUS PEAR SKINS

TABLE 5.
MODELS OF REGRESSION OF NON-NEWTONIAN FLUID APPLIED FOR SWEETENED PULP FROM CACTUS PEARS WITH PARTIAL ADDITION OF THEIR SKINS (SPCPS) AND MARMALADE FROM CACTUS PEAR SKINS (MCPS)

Products	Bingham			Power Law			Herschel-Buckley			
	σ_o (Pa)	K (Pa · s)	R^2	K (Pa · s^n)	n	R^2	σ_o (Pa)	K (Pa · s^n)	n	R^2
SPCPS	14.76	0.0815	0.955	2.4	0.500	0.997	13.86	2.4	0.500	0.999
MCPS	91.20	0.5508	0.958	14.5	0.517	0.994	82.55	18.6	0.473	0.968

σ_o, yield stress; K, consistent coefficient; n, flow behavior index; R^2, determination coefficient.

with different water content (0.34–0.73 w/w) was studied at a wide range of temperatures (0.5–62C) using a concentric cylinder viscometer. The results indicated that the juices behave as pseudoplastic fluids with yield stress, being represented by the Herschel–Bulkley model (Telis *et al.* 1999).

With regard to the MCPS, it was observed in Table 5 that the value of R^2 increased as it passed from Bingham model to Power Law model, and decreased again in the Herschel–Buckley model; however, when a more complex model was adjusted, such as the polynomial of third order, the value of coefficient of determination increased significantly to $R^2 = 0.995$ (Fig. 4), and the general equation of the model is shown in the Fig. 5. This model is an indicator that the fluid had a pseudoplastic behavior up to 334.4/s; next, there was a period of evident transition, given by the increase in 1.8 times of the n value, and the decrease in seven times from the K values, but the pseudoplasticity was still seen up to 508.5/s, and from here to 644.3/s, the shear rate values were much higher that those of shear stress, being $n > 1$ and diminishing the K values over eight times, which demonstrates the beginning of a dilating fluid behavior.

Apparently, the mixture of pectin and mucilage contents in the skins, reported by Medina *et al.* (2000, 2003) and Habibi *et al.* (2004a,b, 2005), contributes to this complexity in the fluid. Steffe (1996) indicated that the pseudoplastic fluids are characterized by specific values of K and n constants, and they must be in the interval specified for this type of fluid ($K > 0$ and $0 < n < 1$). In the high values of shear rate ($\dot{\gamma} > 508$/s) begins the dilatent behavior ($n > 1$). The equations of these three models with the values of K and n are shown in Fig. 5.

Figure 6 shows the rheological behaviors of SPCPS and MCPS in a log η versus log γ graph, obtaining linear equations of the functions, whose slopes are negative and have a parallelism among them, approximately decrease

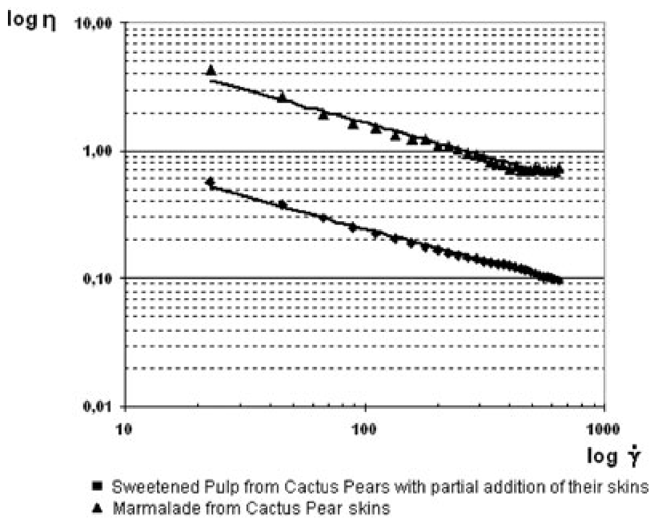

FIG. 6. APPARENT VISCOSITIES OF THE SWEETENED PULP FROM CACTUS PEARS WITH PARTIAL ADDITION OF THEIR SKINS AND THE MARMALADE FROM CACTUS PEAR SKINS

η, apparent viscosity in Pa · s; $\dot{\gamma}$, shear rate in 1/s.

values in 12% between SPCPS and MCPS. In both cases, for low values of shear rate (<250/s), meaningful variations in the apparent viscosity were observed, occurring so that in the values of shear rate (>300/s), the values of apparent viscosity slightly decreased and only small deviations were found in the model fit.

CONCLUSIONS

From this preliminary research that consisted of a study about different products made from the cactus pears, in the product PCPS a range of brittleness and hardness were observed because of different degrees of ripeness that were detected by the instrumental methods. On the other hand, the SPCPS and MCPS semisolid products with partial and total addition of skins, respectively, were non-Newtonian fluids. For high values of shear rate, the MCPS showed a dilatent fluid behavior (these fluids are also called shear thickening). Further study should be undertaken to analyze the variation of shear stress versus shear rate with time and also in a wide range of temperature, with the objective of determining the exact flow behavior of these products.

NOMENCLATURE

K	consistent coefficient ($Pa \cdot s^n$)
n	flow behavior index (dimensionless)
T	Temperature (C)
t	seconds (s)
F	force (N)
D	deformation (mm)
γ	shear rate (1/s)
η	apparent viscosity ($Pa \cdot s$)
σ	shear stress (Pa)
σ_o	yield stress (Pa)

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