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Rheological evaluation of *Prunus mume* pulp

Avaliação do comportamento reológico da polpa de Prunus mume

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Summary

The rheological behaviour of mume pulp at 6, 7, 8 and 9 °Brix was investigated using a rotational viscometer at temperatures ranging from 15 to 75 °C. The rheological models of Herschel-Bulkley and Ostwald-Waele (Power Law) were fitted to obtain the rheological parameters of the mume pulp. The product was described as time non-dependent and presented a viscosity of 1.9 Pa.s at 15 °C and 1.1°Pa.s at 65 and 75 °C for the 9 °Brix pulp. The pulp showed non-Newtonian behaviour and the Herschel-Bulkley model was used to describe this behaviour. The activation energy ranged from 6.6-10.6 kJ.mol^{-1} and the consistency index from 18.0-22.9 Pa.s^{n} for the 9 °Brix pulp and 8.3-12.2 Pa.s^{n} for the 8 °Brix pulp at temperatures varying from 15 to 75 °C. The models presented high correlation values for all the rheological data obtained in the present work.

Key words: *Mume pulp; Rheological model; Consistency; Non-Newtonian; Concentration.*

Resumo

O comportamento reológico de polpa de umê, com 6, 7, 8 e 9 °Brix, foi avaliado utilizando-se um viscosímetro rotacional nas temperaturas de 15 a 75 °C. Modelos reológicos de Herschel-Bulkley e Ostwald-Waele (Lei da Potência) foram ajustados para se obterem os parâmetros reológicos para a polpa de umê. A viscosidade da polpa de umê foi descrita como independente com o tempo e apresentou valores de 1,9 Pa.s a 15 °C e 1,1 Pa.s a 65 e 75 °C, para polpas com 9 °Brix. O produto apresentou comportamento não-newtoniano e o modelo reológico de Herschel-Bulkley descreveu o seu comportamento. A energia de ativação variou de 6,6 a 10,6 kJ.mol^{-1}. O índice de consistência foi mais influenciado pelo teor de sólidos solúveis totais, comparado com a temperatura, com valores de 18,0 a 22,9 Pa.s^{n}, para polpa com 9 °Brix, e de 8,3 a 12,2 Pa.s^{n}, para polpa com 8 °Brix, para temperaturas entre 15 e 75 °C. Os modelos reológicos apresentaram elevado índice de correlação para os dados obtidos no presente trabalho.

Palavras-chave: *Polpa de umê; Modelo reológico; Consistência; Não-Newtoniano; Concentração.*

1 Introduction

Prunus mume Sieb. et Zucc (commonly called ume, mume or mei) belongs to the *Rosaceae* family and has been widely cultivated in Asia for over 3,000 years. There are about 200 mume cultivars. Initially the plants were cultivated because of their flowers, but in the past 2,000 years interest has been focused on the medicinal properties of the fruit (SHI and MOY, 2005; SHI et al., 2009; TSUBAKI et al., 2010). Mume fruit is consumed mainly in Korea, China and Japan, and the tree is cultivated in about 2,700 ha in Korea, 9,000 ha in China and 17,000 ha in Japan, with an average yield of 6.7 ton/ha (JUN and CHUNG, 2008; TOPP et al., 2007).

Prunus mume Sieb. et Zucc. has been studied as rootstock for peach (*P. persica*), showing promising characteristics such as rusticity, high resistance to noxious weeds and diseases, adaptation and the capacity to reduce the size of peach and nectarine trees (MAYER et al., 2005; 2008). Several processed mume products are consumed as health foods and for the treatment of several diseases, since they are rich in bioactive compounds such as anti-cancer and antioxidant substances (SHI and MOY, 2005; SHI et al., 2009; TSUBAKI et al., 2010).

Studies on the effects of substances present in mume juice and fruit on human health showed that mumefral, a citric acid derivative, improves blood flow, helping prevent cardiovascular diseases (UTSUNOMIYA et al., 2002). In Brazil, the consumption of mume is still almost completely restricted to Asian descendants. Mume fruits can be used to make jam by mixing with peaches and plums (CAMPO DALL'ORTO et.al., 1995-1998).

On an industrial scale, it is better to use the fruit pulp instead of the fresh fruits themselves, and therefore it is important to understand the physical-chemical and rheological properties, which are mainly influenced by the composition of the juice or pulp and will depend on the type of fruit and the treatments to which it was subjected during the manufacturing process (VANDRESEN et al., 2009).

Knowledge of the rheology of fruit juices is useful in quality control, sensory evaluation and in engineering applications when industrial plants are being designed. The effect of temperature and concentration on the flow properties must be known to calculate and choose the right kind and size of equipment to ensure adequate heat and mass transfer operations (ALTAN and MASKAN, 2005; CHIN et al., 2009; FALGUERA and IBARZ, 2010; NINDO et al., 2005). The aim of this work was to evaluate the rheological behaviour of mume pulp at different concentrations and temperatures and fit the data obtained to well-known rheological models.

2 Material and methods

2.1 Raw Material – mume pulp

The mume fruits were picked in São Paulo State, Brazil, latitude: 23° 30' 43" S; longitude: 48° 16' 38" W; elevation: 737 m. The fruits were green-to-yellow in colour, and soon after this natural fruit dropping of the fruit occurs, approximately 88 days after flowering (DAF). They were held for 6 days at 26 °C until complete maturation, which can be noted by a significant change in colour to yellow and the development of a strong peach-like perfume with floral notes. Overripe fruits present a brownish colour and wrinkled skin.

Batches of about 5 kg ripe fruits were heat blanched by immersion in boiling water for 120 seconds, and then immediately pulped in a pilot-scale brush pulper with round 2.2 mm perforations. This equipment was set for maximum speed rotation to maximize pulp extraction. The pulp was poured into a jacketed vat equipped with vacuum, and deaerated at –0.7 bar for 30 minutes at 50-60 °C. Since the pH of the product is below 4.5 (pH 2.6), the pulp was submitted to pasteurization at 80 °C for 10 minutes in a tank with a mechanical mixer.

The pasteurized pulp was hot-filled into glass jars, closed with glazed metal covers, deaerated for 1 minute and placed upside down for 10 minutes. After cooling, the hermetically closed glass jars were stored at room temperature until used.

The total soluble solids (TSS) content was obtained using a Reichert AR200 digital refractometer expressed in Brix. The original mume pulp showed a value of 9 °Brix. For values of 8, 7 and 6 °Brix, potable water was used for dilution.

The total solids (TS) content and total titration acidity (TTA) were determined according to The Instituto Adolfo Lutz (2008) and were carried out in duplicate.

The pectin content was determined by mixing 4 g of freeze dried mume pulp with 1:50 50 mM nitric acid at 80 °C for 25 minutes. After filtration and cooling to 4 °C, the acid extract was mixed with 1:2 96 °GL ethanol at 4 °C and allowed to stand for 30 minutes. The mixture was then filtered, placed inside permeable bags, and maintained overnight under agitation with 70% ethanol. s After washing once more with 95% ethanol and drying at 40 °C, the pectin content was determined in triplicate.

2.2 Rheological measurements

A programmable Brookfield DVII+Pro viscometer equipped with concentric cylinders was used to measure the viscosity of the mume samples. A set of small sample adapters (SC4-27 spindle and a SC4-45Y assembly) was used with a Brookfield TC-501thermostatic bath. The equipment set presented a viscosity accuracy: ±1% on the full-range scale, repeatability: 0.2% full-range scale and temperature accuracy: ±0.1 °C. The measurements were carried out at 15, 25, 45, 65 and 75 °C for each concentration level (9, 8, 7 and 6 °Brix). Before each test, the samples were placed in a water bath for 10 minutes at the same temperature programmed for the rheological tests. DVLOADER software was used to produce the program to control the viscometer and the data were exported using REOCALC32 software and a computer attached to a RS-232 port viscometer.

To verify the dependence of the rheological properties on time, the changes in apparent viscosity were determined for 180 s at a constant shear rate of 40.8 s^{-1}. The flow-curves were determined using ascending and descending flow curves of shear stress versus shear rate for 180 seconds in the range from 0 to 70 s^{-1}, for pulp concentrations of 6 to 9 °Brix and temperatures ranging from 15 to 75 °C. A shear rate of 0-70 s^{-1} was chosen because it approximates the shear rates of tumbling, pouring and transport conditions (CHIN et al., 2009). The experiments were carried out in duplicate and a new sample was used for each measurement.

The Arrhenius equation is commonly used for rheological studies reported in the literature, and the effect of temperature on the viscosity can be determined in juices and pulps (ARSLAN et al., 2005; BELIBAGLI and DALGIC, 2007; FALGUERA and IBARZ, 2010; VANDRESEN et al., 2009):

$$\eta_a = \eta_0 \exp \frac{E_a}{RT} \tag{1}$$

where η_a is the apparent viscosity (Pa.s), η_0 is an empirical constant (Pa.s), E_a is the flow activation energy (J.mol^{-1}), the R value is 8.314 J.mol^{-1}K^{-1} and represents the ideal gas constant and T is the absolute temperature (K).

The Arrhenius equation can be linearized as shown:

$$\ln(\eta_a) = \ln(\eta_0) + \frac{E_a}{R} \times T^{-1} \tag{2}$$

Applying Equation (2), the values for η_0 and E_a are obtained from the linear and angular coefficients of the plot of ln(η_a) as a function of (1/T):

$\ln(\eta_0)$ = linear coefficient

E_a/R = angular coefficient

After determining the behaviour of the apparent viscosity, the Ostwald-Waele (Power law) model was used to determine the rheological parameters:

$$\tau = k(\dot{\gamma})^n \tag{3}$$

where: τ is the shear stress (Pa), *k* the consistency index (Pa.s^n), $\dot{\gamma}$ is the shear rate (s^{-1}) and n is the fluid behaviour index (dimensionless).

The Herschel-Bulkley model is commonly used to describe non-Newtonian fluids and fruit pulps:

$$\tau = \tau_0 + k(\dot{\gamma})^n \tag{4}$$

where: τ_0 is the yield stress (Pa).

2.3 Statistical analysis

The rheological models were fitted to the experimental data using the Microsoft Office Excel™ Software to calculate the activation energy, consistency index, fluid behaviour index and yield stress values. In all models The coefficient of correlation (R^2) and chi-square (χ^2) were determined for all the models.

The differences were analyzed for significance by Tukey ($p<0.05$) using the Statistica™ version 8 software.

3 Results and discussion

Figure 1 shows the results for apparent viscosity and its variation with time, using different concentrations of mume pulp. The data were obtained maintaining the shear rate constant at 40.8 s^{-1}.

The apparent viscosity was practically constant at temperatures ranging from 15 to 75 °C and concentrations from 6 to 9 °Brix for a period of 3 minutes. No significant difference was observed in the apparent viscosity with time for most samples (Tukey, $p<0.05$). However, samples with 9 °Brix at temperatures of 15 and 25 °C presented significant differences in apparent viscosity after 160 s, when compared with the initial values. At higher concentrations of mume pulp and lower temperatures, there was a slight dependence of the apparent viscosity on time, probably due to the higher pectin and soluble solids contents. The mume pulp exhibited typical juice behaviour, where the apparent viscosity decreased with increase in temperature.

Figure 1 also showed that the effect of temperature on apparent viscosity was more noticeable at lower temperatures. An increase in temperature from 15 to 25 °C lowered the apparent viscosity values by 21% from 1.9 to 1.5 Pa.s. Raising the temperature by 20 °C, from 45 to 65 °C lowered the apparent viscosity by 12% from 1.25 to 1.1 Pa.s, but no difference was observed in the apparent viscosity of the mume pulp with 9 °Brix at 65 and 75 °C.

As can be noted in Figure 1, the present study showed that a decrease of 1 °Brix resulted in a significant reduction in the apparent viscosity. This same behaviour was observed at higher temperatures. Thus the total soluble solids content should be strictly controlled in an industrial application such as heat treatment, where the flow curves can greatly influence the quality of the final product. For all the cases ($d\eta_a/dt$) the slopes were very close to zero, ranging from –0.001 to 0.0005, except for samples with 9 °Brix at 65 °C, where the values ranged from –0.002 to 0.001 probably due to the presence of insoluble fibres in these samples. Figure 2 shows the variation in Shear Stress (Pa) versus Shear Rate (s^{-1}) for ascending flow curves, since the ascending and descending flow curves showed coincident data. The measurements were carried out at temperatures ranging from 15 to 75 °C and different pulp concentrations. In Figure 2, the lines connecting the experimental points were only inserted to assist visualization of the experimental data. These lines do not represent a mathematical fit. The rheological models were fitted to obtain the rheological parameters for mume pulp and will be shown in Tables 1 to 4.

All the samples (6, 7, 8 and 9 °Brix) evaluated at temperatures from 15 to 75 °C, showed a non-linear relation between shear stress and shear rate, indicating non-Newtonian behaviour, similar to that reported by several authors (CHIN et al., 2009; FALGUERA and IBARZ, 2010; IGUAL et al., 2010; TIZIANI and VODOVOTZ, 2005) for fruit juices. Preliminary experiments showed no significant dependence or slight thixotropy for mume pulp (BELIBAGLI and DALGIC, 2007).

Table 1 presents the calculated values for η_0, E_a and the regression coefficient (R^2) obtained from linearization of the Arrhenius equation for the flow of mume pulp at different shear rates. The Chi-square (χ^2) values were below 0.0054, indicating a good fit between the experimental data and the mathematical fit.

E_a indicates the dependence of the viscosity on temperature variations. High E_a values indicate that the viscosity varies a lot with changes in temperature. As can be seen in Table 1, R^2 exceeded 94% in all cases, indicating an adequate fit. The flow activation energy value increased slightly with the increase in shear rate. Thus heat transfer processes can be helped by agitation at different stages of the process (FALGUERA and IBARZ, 2010). Considering the highest shear rate (68 s^{-1}), the E_a value for 9 °Brix was 10.64 $kJ.mol^{-1}$. This result is similar to that obtained by Altan and Maskan (2005), (E_a = 8.6 $kJ.mol^{-1}$) for pomegranate juice at 35.2 °Brix and by Gratão et al. (2007), (E_a = 11.59 $kJ.mol^{-1}$) for soursop juice at 9.3 °Brix. The values for E_a showed no clear trend regarding changes in the concentration. Similar behaviour was observed by Gratão et al. (2007) for soursop juice and by Chin et al. (2009) for grapefruit juice.

Table 2 presents the data obtained for the fit of the mume pulp at different concentrations and temperatures according to the Power Law model.

From the results shown in Table 2, it can be seen that the n values were all less than one, indicating non-Newtonian behaviour at all concentration and temperature levels. The n values decreased with increase

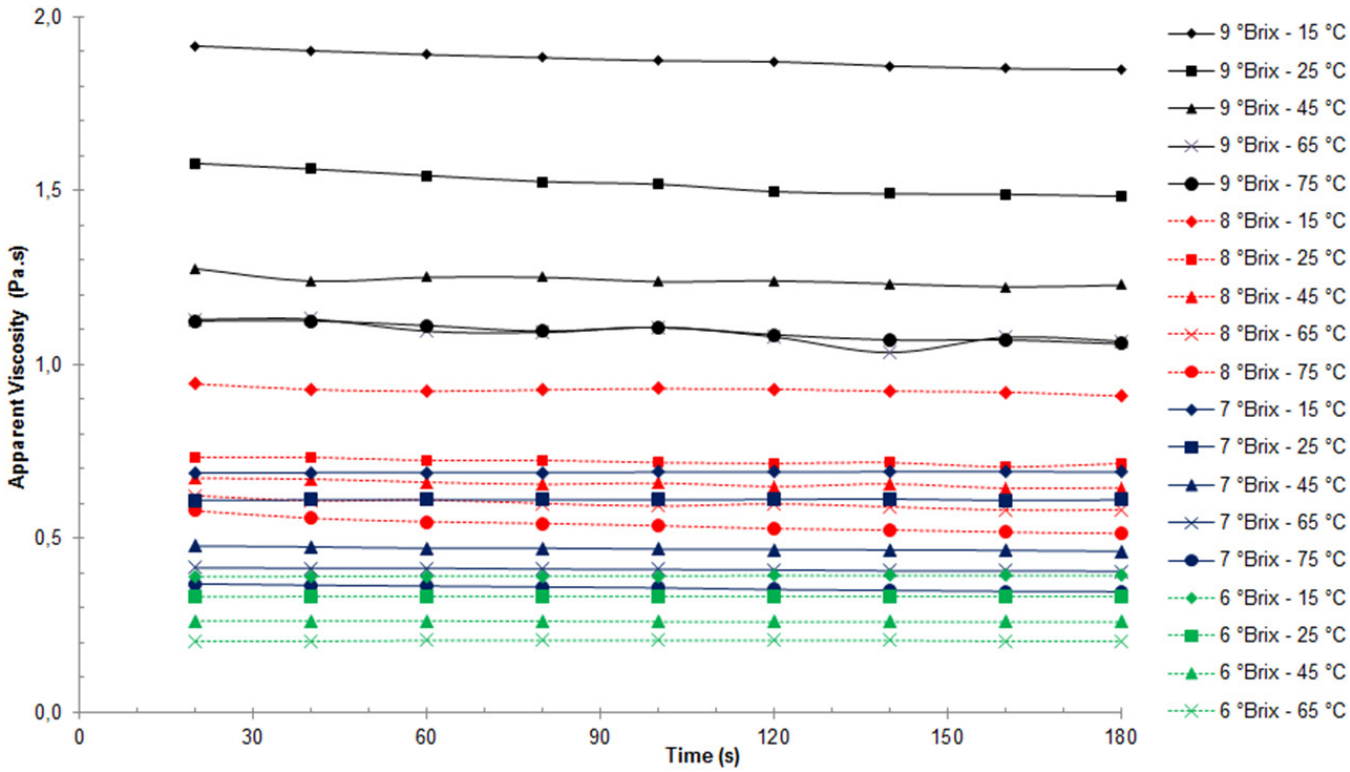

Figure 1. Variation in apparent viscosity with time at temperatures of 15 to 75 °C for different concentrations of mume pulp.

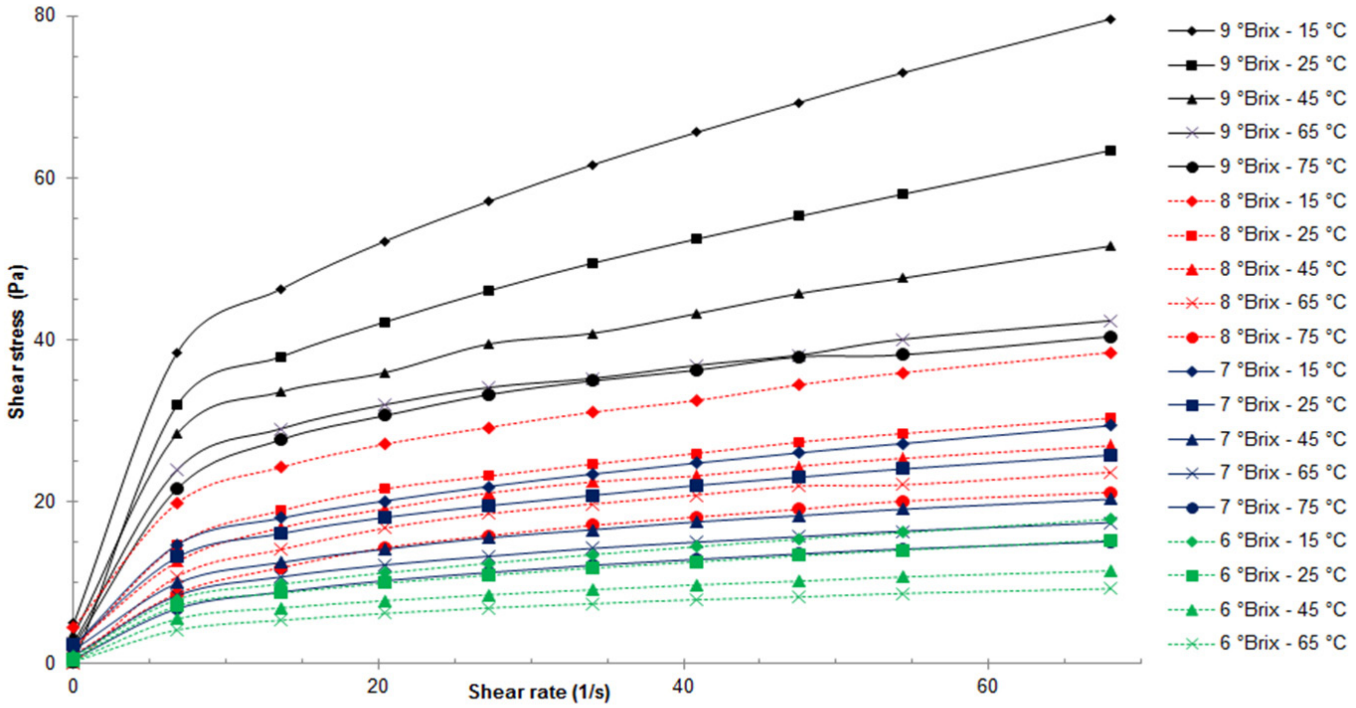

Figure 2. Shear Stress versus shear rate at temperatures from 15 to 75 °C with different concentrations of mume pulp.

Table 1. Model parameters obtained for the Arrhenius equation.

Shear rate:	20 s^{-1}			41 s^{-1}			68 s^{-1}		
Samples (°Brix)	η_0 (Pa.s)	Ea (kJ.mol⁻¹)	R^2	η_0 (Pa.s)	Ea (kJ.mol⁻¹)	R^2	η_0 (Pa.s)	Ea (kJ.mol⁻¹)	R^2
6	0.0153	8.4739	0.9934	0.0055	9.9076	0.9969	0.003	10.6463	0.9991
7	0.0342	8.0023	0.9958	0.0179	8.4095	0.9959	0.0108	8.819	0.9958
8	0.0819	6.6012	0.9472	0.0484	6.6037	0.9429	0.0255	7.2672	0.9492
9	0.142	6.9361	0.9406	0.0573	7.9126	0.9466	0.0237	9.2167	0.9798

Table 2. Power law parameters for mume pulp at different concentrations and temperatures.

Samples	Parameters	Temperature (°C)				
(°Brix)		15	25	45	65	75
6	n	0.405	0.384	0.3489	0.2903	-
	k (Pa.s^n)	3.1756	2.9711	2.5834	2.6686	-
	R^2	0.999	0.9987	0.9968	0.9962	-
7	n	0.3288	0.3104	0.2913	0.2829	0.2752
	k (Pa.s^n)	7.2623	6.8795	5.8905	5.2258	4.7333
	R^2	0.9994	0.9991	0.9983	0.9989	0.9997
8	n	0.2702	0.2497	0.2275	0.2243	0.2249
	k (Pa.s^n)	12.1609	10.487	10.2519	9.1811	8.3157
	R^2	0.9978	0.9981	0.9961	0.9963	0.9897
9	n	0.2937	0.2757	0.2465	0.1789	0.1519
	k (Pa.s^n)	22.9439	19.5389	18.0164	19.7227	21.4914
	R^2	0.9996	0.9976	0.9937	0.9538	0.9706

Table 3. Herschel-Bulkley model parameters for mume pulp at 6 and 7 °Brix.

Temperature	6 °Brix				7 °Brix			
(°C)	τ_0	*k*	n	R^2	τ_0	*k*	n	R^2
15	4.236	4.236	0.587	1.000	7.203	3.011	0.471	1.000
25	4.054	4.054	0.583	1.000	7.570	2.341	0.484	1.000
45	4.408	4.408	0.678	1.000	7.720	1.300	0.537	1.000
65	4.133	4.133	0.636	1.000	5.929	1.516	0.478	1.000
75	-	-	-	-	1.979	3.333	0.325	1.000

Table 4. Herschel-Bulkley model parameters for mume pulp at 8 and 9 °Brix.

Temperature	8 °Brix				9 °Brix			
(°C)	τ_0	*k*	n	R^2	τ_0	*k*	n	R^2
15	15.360	2.593	0.516	1.000	19.335	10.464	0.414	1.000
25	7.947	4.862	0.360	0.998	25.633	3.900	0.535	1.000
45	12.881	1.645	0.508	0.998	26.734	1.715	0.632	0.998
65	1.272	8.235	0.237	1.000	19.606	4.689	0.371	0.956
75	2.531	10.496	0.196	0.991	4.884	17.371	0.172	0.968

in concentration. Similar behaviour was observed by Arslan et al. (2005) for sesame paste/concentrated grape juice blends and by Gratão et al. (2007) for soursop juice.

Newtonian fluids show a flow behaviour index equal to unity (n = 1). For fruit juices, this index usually increases slightly with rise in temperature. As expected, the flow behaviour index slightly decreased with increase in temperature for several concentrations. Since these variations are quite small, some authors consider n to be constant (GRATÃO et al., 2007). The fluid behaviour of mume pulp at 8 and 9 °Brix was similar to that found for prune puree from 20 to 40 °C as studied by Maceiras et al. (2007).

The determinations of the *n* and *k* values are important in juice processing. For example, an increase in the consistency index can decrease the flow rate of the juice in the pipes due to the greater resistance to flow. Also, an increase in the consistency index can change the flow and temperature profile and modify the holding time of the product in the flow lines during pasteurization (CHIN et al., 2009).

It can also be seen in Table 2 that the consistency index of the mume pulp changed more significantly with the change in concentration than with the variation in temperature. Thus a small difference in the pulp concentration of different products from the same production batch can result in a different consistency index, and this effect can be more significant than a rise in temperature. The consistency index increased exponentially with the increase in concentration. Tables 3 and 4 show the Herschel-Bulkley model parameters for mume pulp at temperatures from 15 to 65 °C and total soluble solids concentrations varying from 6 to 9 °Brix.

Non-linear regression of the Herschel-Bulkley model showed that the yield stress (τ_0) did not show consistent behaviour with regards to the variation in temperature between 15 and 75 °C for mume pulp from 6 to 9 °Brix. This situation was reported by Falguera and Ibarz (2010) for concentrated orange juice (65.3 °C with 6% pulp) at temperatures from –12 to 30 °C. However, it can be seen that the values for yield stress increased with an increase in concentration from 6 to 9 °Brix. Similar behaviour was observed for the consistency index (*k*). Taken as an example, mume pulp with 8 °Brix at temperatures from 15 to 25 °C showed a consistency index of 3.73 Pas^n. In the same temperature range, carrot juice at 8.04 °Brix showed a consistency index of 0.03067 Pas^n (VANDRESEN et al., 2009).This indicates that mume pulp, in addition to its soluble solids, has other factors which increase its consistency, such as pectin and low pH values, and that an increase in pectin results in an increase in the consistency index. The values for the consistency index of mume pulp were similar to those obtained for concentrated orange juice (FALGUERA and IBARZ, 2010). Tables 3 and 4 show that the values for the fluid behaviour index (n) were higher than those determined by the power law (Table 2). However, all the values remained below one, confirming a Herschel-Bulkley behaviour.

4 Conclusions

The experimental data for mume pulp described a non-Newtonian behaviour and could be fitted using the Herschel-Bulkley model. This pseudoplastic behaviour is typical of dilute solutions in the presence of solids, similar to other fruit juices. The consistency of mume pulp depends strongly on its concentration and to a lesser degree on the temperature.

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