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SHORT COMMUNICATION

Modelling of rheological behaviour of guava, pomelo and soursop juice concentrates via shear rate-temperature-concentration superpositioning

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Abstract The steady-state flow test was conducted on pink-fleshed guava, pink-fleshed pomelo and soursop juice concentrates using a rheometer to understand its rheological behaviour. The power law model was used and a master-curve was created using the shear rate-temperature-concentration superposition technique to predict rheological properties from a wide range of temperatures and concentrations. All three juice concentrates undergo a double horizontal shift whilst the pink-fleshed guava required an additional vertical shift. The final equations show shear-thinning behaviour of pink-fleshed guava, pink-fleshed pomelo and soursop with flow behaviour index of 0.2217, 0.7507 and 0.6347, respectively. The final master-curve predicts shear stress at wide range of shear rates, i.e. between 10^{-2} and 10^{6} s^{-1} for the pink-fleshed guava, 10^{0} and 10^{6} s^{-1} for the pink-fleshed pomelo and 10^{0} and 10^{7} s^{-1} for the soursop. The results provide useful information and effective technique to predict fruit juice concentrates behaviour affected by heat changes during processing.

Keywords Superposition · Master-curve · Vertical shift · Guava · Pomelo · Soursop

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Introduction

The understanding of rheological properties of fruit juice is important for the design and optimisation of its processing and its product stability and quality. As fruit juice concentrates behave differently at different shear, it is important to have a rheology test performed to determine how the juice concentrates behave when sheared over a period of time. The juice concentrates start to shear as it travels through the middle of the pipe and as it moves in relation to the pipe walls. Rheological data coupled with information on the flow rate, pipe and pump dimensions, and pump rotational speed can help one to select the correct pump and sizing leading to a proper design of unit operations which helps in optimisation of processing system, prevention the over-dimensioned of facilities and reduction of wasteful use of economic resources (Falguera et al. 2010). Fruit juice that flow easily in a pipe also leaves less fouling.

The power law model (Eq. 1) is the most widely used empirical model for non-Newtonian fluids to describe the flow properties of fluids theoretically and practically in engineering applications. It gives good agreement with experimental data of "Totapuri" mango juice (Dak et al. 2006), pomelo juice concentrates (Chin et al. 2009), reconstituted tomato concentrates (Barbana and El-Omri 2012) and soursop juice concentrates (Quek et al. 2013). The non-Newtonian fluids change in viscosity depending on the shear rate. The fluid is considered as the shear-thinning fluid if the n value is between 0 and 1 ($0 < n < 1$), and the shear-thickening fluid if n value is more than 1 ($1 < n < \infty$). The viscosity of Newtonion ($n = 1$) fluids is retained regardless of the shear rate.

$$\sigma = K\dot{\gamma}^{n} \tag{1}$$

where σ is the shear stress, K is the consistency coefficient, $\dot{\gamma}$ is the shear rate, and n is the flow behaviour index.

The data from flow tests performed at various temperatures can be superimposed to a reference temperature using time–temperature superposition technique to develop a master-curve. The time–temperature superposition technique is the most useful extrapolation technique with a wide range of applications and it has been applied to viscoelastic properties of polymers (Gupta et al. 2012; Luo et al. 2012; Chang et al. 2013). The superposition gives a master-curve with a much longer time interval at a specified temperature with assumption that the polymer will behave similarly at higher temperatures and longer time. Some other studies found that the measured data could not be superimposed smoothly by only horizontal shifting, and a proper addition of vertical shifting was needed to make a smooth master curve (Tajvidi et al. 2005; Cai et al. 2013; Chang et al. 2013). Although vertical shifting has been studied on polymers (Guedes 2011; Nakada et al. 2011; Chang et al. 2013), there is no literature on vertical shift of rheological data of fruit juice. Literature on horizontal shift of rheological data of fruit juice concentrates includes those by Chin et al. (2009) and Quek et al. (2013).

This study aimed to model rheological behaviour of thermosonic-extracted juice concentrates by applying the shear rate-temperature-concentration superposition technique using the power law model.

Materials and methods

Fruit juice concentrates preparation

The pink-fleshed guava (*Psidium guajava* L.), pink-fleshed pomelo (*Citrus maxima* M.) and soursop (*Annona muricata* L.) fruits used in this study were purchased from local markets and prepared for thermosonication extraction. For pink-fleshed guava, floral remnants at the apex and the tip ends of the ripened and washed fruits were removed and cut. Then, it was diced into small pieces. For pink-fleshed pomelo, its thick spongy rind of yellow rinds was cut into four sections as close as possible to the flesh of the pomelo prior to pull away the peel from the fruit. The fruit was then sliced and broken into half. The seeds and bitter remaining pith were completely removed. For the soursop, ripen fruits were washed, cut into half and cored. Their skin was hand-peeled and seeds were removed from the pulp manually.

In order to get a homogenised fruits pulp mash, 2.5 kg of pulp of each fruit was crushed into a mash using a commercial food blender (XB409 1300 W, Ceado, Italy) with pulse duration of 60 s at 28,000 rpm, then 30 s off and 60 s at 22,000 rpm. Fruit juices of guava, pomelo and soursop were extracted at optimum conditions of thermosonic-assisted extraction methods (Abdullah 2015). Guava and pomelo juices were extracted using indirect thermosonication technique with power, time and temperature settings of 1 kW, 30 min, 55 °C, and 2.5 kW, 23 min, 54 °C, respectively. Soursop juice was extracted using direct thermosonication at ultrasonic amplitude of 10% for 10 min at 55 °C. For indirect thermosonic-assisted extraction, a glass bottle of the pulp sample mixture was partially immersed in an ultrasonic water bath (Chin et al. 2013), which contains distilled water as a medium to spread waves at optimum power. The direct thermosonic-assisted extraction was carried out using a 400 W digital ultrasonic processor (S-450D, Branson, USA) with its probe tip immersed to a depth of 25 mm into a 150-mL beaker containing pulp mixture samples. The pulp mixture samples were mixtures of 50 g of blended pulp with distilled water at a ratio of 1:1. The specified temperature was checked using temperature probe and maintained manually by adding cold water into water container which places the beaker for direct thermosonication or applying continuous flow of water in the bath for indirect thermosonication.

The treated pulp was separated from the juice by centrifugation at 4000 rpm and 4 °C for 20 min using a refrigerated centrifuge (Mikro 22R, Hettich Zentrifugen, Germany). The supernatant was collected and freeze-dried. 100 mL of each juice was poured in a round plastic container and frozen in a freezer at −20 °C prior to freeze-drying. 1 L of each juice was concentrated in a freeze-dryer (FreeZone, Labconco, USA) for 72 h. Guava juice concentrate at 47.8 ± 0.7 °Brix, pomelo juice concentrate at 73.1 ± 0.4 °Brix and soursop juice concentrate at 73.8 ± 0.7 °Brix were produced and they were reconstituted by dilution with distilled water to get juices of different concentrations of 5, 15, 30, 45 and 70 °Brix for flow evaluation with exception of 70 °Brix for guava. The concentration was determined in terms of total soluble solids content using a digital refractometer (PAL-Alpha, Atago, USA).

Rheological measurements and master-curve construction

Steady-state flow test was conducted on different combinations of 5 juice concentrates from 5 to 70 °Brix and 7 temperatures from 0, 4, 10, 25, 40, 60 to 80 °C which produced 35 individual shear stress-shear rate curves using a rheometer (ARG2, TA Instruments, USA). The temperature of samples was controlled and maintained by a peltier plate. The 2° cone plate with diameter of 40 mm was used and shear rate ranging from 0 to 400 s^{-1} was applied for each sample. The experiment was conducted using a same batch of fruits and duplicated entirely.

Table 1 Comparison of rheological parameters at various reference temperatures after the first shift of superposition

Reference temperature, T_{ref} (°C)	Guava		Pomelo		Soursop	
	K' (Pa s^n)	n'	K' (Pa s^n)	n'	K' (Pa s^n)	n'
5 °Brix						
0	0.1112	0.7526	0.0021	0.9952	0.0045	0.9042
4	0.0983	0.7526	0.0019	0.9952	0.0038	0.9042
10	0.0892	0.7526	0.0015	0.9952	0.0032	0.9042
25	0.0562	0.7526	0.0011	0.9952	0.0024	0.9042
40	0.0367	0.7526	0.0009	0.9952	0.0019	0.9042
60	0.0244	0.7526	0.0006	0.9952	0.0011	0.9042
80	0.0126	0.7526	0.0004	0.9952	0.0007	0.9042
15 °Brix						
0	7.7569	0.4170	0.0043	0.9750	0.0182	0.8461
4	7.0926	0.4170	0.0037	0.9750	0.0145	0.8461
10	6.4238	0.4170	0.0030	0.9750	0.0125	0.8461
25	5.0279	0.4170	0.0021	0.9750	0.0097	0.8461
40	3.6417	0.4170	0.0015	0.9750	0.0058	0.8461
60	2.7276	0.4170	0.0011	0.9750	0.0045	0.8462
80	2.1524	0.4170	0.0009	0.9750	0.0035	0.8461
30 °Brix						
0	95.4044	0.2553	0.0203	0.8864	0.0844	0.7663
4	85.7008	0.2553	0.0167	0.8864	0.0633	0.7663
10	74.3332	0.2553	0.0141	0.8864	0.0479	0.7663
25	56.5479	0.2553	0.0089	0.8864	0.0333	0.7663
40	46.2091	0.2553	0.0064	0.8864	0.0257	0.7663
60	44.4012	0.2553	0.0049	0.8864	0.0206	0.7663
80	41.9947	0.2553	0.0046	0.8864	0.0172	0.7663
45 °Brix						
0	317.9775	0.2131	0.0784	0.8772	0.3256	0.7360
4	293.5147	0.2131	0.0654	0.8772	0.2607	0.7360
10	264.8533	0.2131	0.0522	0.8772	0.2268	0.7360
25	220.8170	0.2131	0.0300	0.8772	0.1579	0.7360
40	192.1179	0.2131	0.0203	0.8772	0.1147	0.7360
60	175.8173	0.2131	0.0152	0.8772	0.0782	0.7360
80	162.2264	0.2131	0.0144	0.8772	0.0691	0.7360
70 °Brix						
0	NA	NA	12.2722	0.7491	40.2469	0.6329
4	NA	NA	9.7152	0.7491	32.3371	0.6329
10	NA	NA	5.9860	0.7491	24.2473	0.6329
25	NA	NA	3.0361	0.7491	14.8254	0.6329
40	NA	NA	1.5820	0.7491	7.0383	0.6329
60	NA	NA	0.8399	0.7491	3.0276	0.6329
80	NA	NA	0.6197	0.7491	3.0995	0.6329

NA means data are not available

A single master-curve was developed from all individual curves by the shifting method in two steps using reference temperatures and concentrations to see the variations of K, n and R^2. All data were shifted horizontally along $\dot{\gamma}$-axis to a reference temperature for the first shift and a reference concentration for the second shift. With exception for the guava, the second shifting needed both the horizontal and vertical (along σ-axis) shifting because of the inadequacy of a single horizontal shifting to produce a smooth superimpose (Nakada et al. 2011). The horizontal shift amount was defined by Eqs. 2 and 3, while the vertical shift was determined by Eq. 4. At reference temperature and concentration, the values of a_T, a_C and b_C are equal to 1.

$$a_T = \dot{\gamma}_T / \dot{\gamma}_{T_{ref}} \quad (2)$$

$$a_{C(T)} = \dot{\gamma}_{C(T)} \Big/ \dot{\gamma}_{C(T)_{ref}} \quad (3)$$

$$b_C = \sigma_C / \sigma_{C_{ref}} \quad (4)$$

where a_T is the shear rate-temperature shift factor, $\dot{\gamma}_T$ is the shear rate at temperature T, $\dot{\gamma}_{T_{ref}}$ is the shear rate at reference temperature, $a_{C(T)}$ is the shear rate-temperature-concentration shift factor, $\dot{\gamma}_{C(T)}$ is the shear rate at concentration C, $\dot{\gamma}_{C(T)_{ref}}$ is the shear rate at reference concentration, b_C is the shear stress-concentration shift factor, σ_C is the shear stress at concentration C, $\sigma_{C_{ref}}$ is the shear stress at reference concentration.

Equation 5 was then fitted to the all concentrations master-curves prior to shift for the second time. The final master-curve was then obtained after the second step of shifting and fitted to Eq. 6 for pomelo and soursop, while Eq. 7 is for guava in order to get a single equation of rheological behaviour of each guava, pomelo and soursop.

$$\sigma = K' (\dot{\gamma}/a_T)^{n'} \quad (5)$$

$$\sigma = K'' \left(\dot{\gamma}/a_T \cdot a_{C(T)}\right)^{n''} \quad (6)$$

$$\sigma / b_C = K'' \left(\dot{\gamma}/a_T \cdot a_{C(T)}\right)^{n''} \quad (7)$$

Results and discussion

The tropical fruit juice concentrates can be categorised as either simple or structured fluids from the power law's constant parameters of K and n whilst being affected by other factors of temperature and concentration which arise due to processing requirements. The tropical fruit juice concentrates used in this study exhibited both shear-thinning and Newtonian behaviour depending on the type of fruit, temperature and concentration. The shear-thinning behaviour shows a large decrease in viscosity and increase of shear stress when the shear rate increased (Jalil and Asghar 2013). The viscosity drops upon shearing causes the juices to pour and flow easily. Newtonion behaviour occurs when the viscosity is not influenced by the shear rate. In general, higher concentration juices require high shear stress to flow due to its relatively thick texture, which is related to the increment of solids content. In this study of juice concentrates, the power law model has fitted well (graph not shown) to all the individual shear stress-shear rate curves with $R^2 > 0.9892$. All juice concentrates show shear-thinning, except for the pomelo and soursop at low concentration of 5 °Brix at 80 °C which has Newtonian behaviour. Pomelo needs less force to flow as it has the lowest K values, while guava requires high force to make it flow as it possesses high K values. The K values represent the level of viscosity, where thicker juice has high K values and watery juice has low K values. The thick, creamy, and fleshy guava pulp results in difficulty in cell wall disruption of the fruit tissue. Thus, only a small amount of juice could be pressed or squeezed out.

Table 1 shows the difference of K and n when using different reference temperature to develop a master-curve by the shear rate-temperature method. K values changed accordingly when different reference temperature was used. When the reference temperature is high, the value of K became lower. However, values of n and R^2 were similar when using any reference temperature. No change of n value indicates that the shapes of all curve lines are maintained, while the curve lines move from their original positions due to the changes of K value. A higher K value at higher concentration indicates higher juice concentrate viscosity. Juice concentrates show more shear-thinning behaviour at higher concentration with corresponding lower n values. A lower n value means the juice concentrate is further from the Newtonian behaviour and more difficult to flow. The guava owns the highest K and the lowest n, and vice versa for the pomelo. The reference temperature was chosen arbitrarily as there was no shape change when using any temperature as a reference curve. This result is consistent with Steffe (1996) who found that

Table 2 Comparison of rheological parameters at reference temperature of 25 °C and various reference concentrations after the second shift of superposition

Reference concentration, C_{ref} (°Brix)	Guava		Pomelo		Soursop	
	K'' (Pa s^n)	n''	K'' (Pa s^n)	n''	K'' (Pa s^n)	n''
5	15.3062	0.2377	0.0156	0.7507	0.0578	0.6347
15	23.5189	0.2377	0.0212	0.7507	0.0975	0.6347
30	54.8622	0.2377	0.0336	0.7507	0.1312	0.6347
45	191.0759	0.2377	0.0877	0.7507	0.3823	0.6347
70	NA	NA	3.0012	0.7507	14.6436	0.6347

NA means data are not available

any value of the considered temperature range could be chosen as the reference temperature. In this study, 25 °C was selected randomly as a reference temperature for the first shift and its rheological parameters were fitted to Eq. 5. The obtained $R^2 > 0.9$ indicates that the measured data are close to the fitted line and it generates precise prediction of K and n values.

Table 2 illustrates the variations of K and n when different reference concentration for the second shift of a master-curve construction was used. Changes were observed only in K values and not with the n values despite varying the reference concentration. This observation suggested that the data moved to the reference data without changing its curve shape. A higher value of reference concentration contributed to a higher K value. For the second shift of the data, 30 °Brix was chosen as a reference concentration and the values of K and n at this reference concentration was fitted to Eq. 6 for the pomelo and soursop, and Eq. 7 for the guava (Table 2). The obtained $R^2 > 0.9$ explains that all the variability of the K and n values are close to their means. These master-curves confirm that all guava, pomelo and soursop juices are having shear-thinning behaviour with n values within 0 and 1, where n are 0.2217, 0.7507 and 0.6347, respectively. The guava and soursop are more pulpy than the pomelo. Therefore, the values of n for guava and soursop are lower

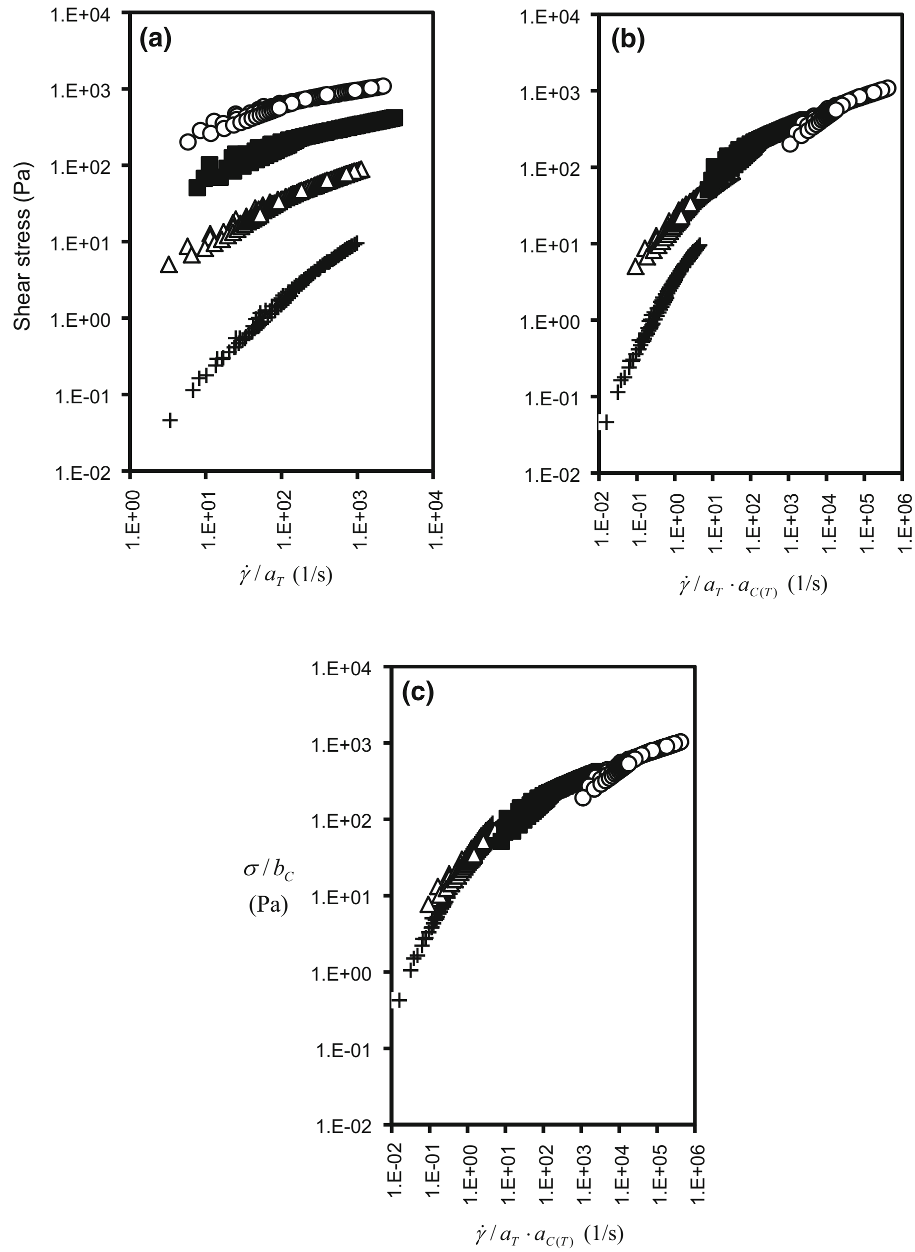

Fig. 1 **a** Shear rate-temperature superposition horizontally, **b** shear rate-temperature-concentration superposition with second horizontal shift and **c** shear rate-temperature-concentration plus addition of shear stress-concentration shift for guava. + 5 °Brix; 15 °Brix; 30 °Brix; 45 °Brix

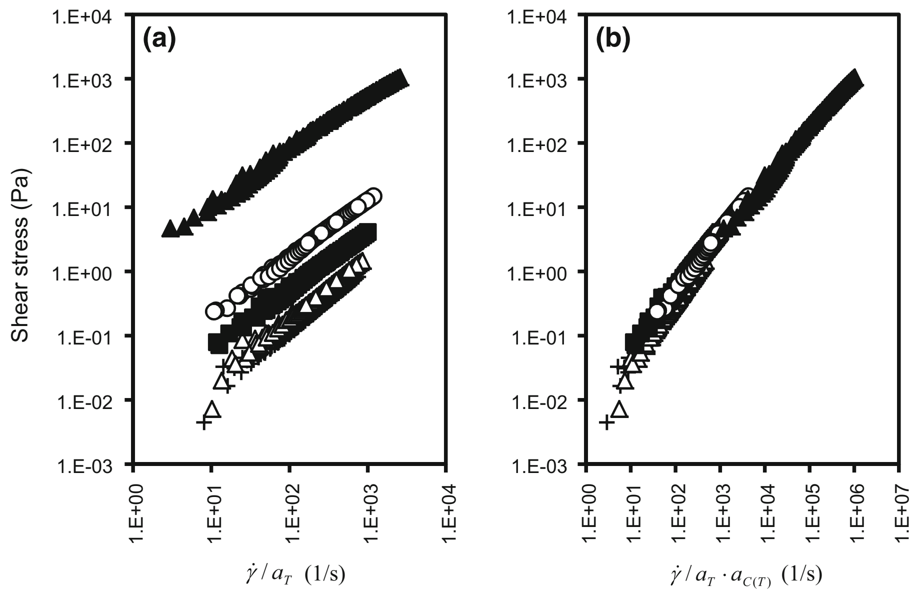

Fig. 2 **a** Shear rate-temperature superposition and **b** shear rate-temperature-concentration superposition with second horizontal shift for pomelo. + 5 °Brix; 15 °Brix; 30 °Brix; 45 °Brix; 70 °Brix

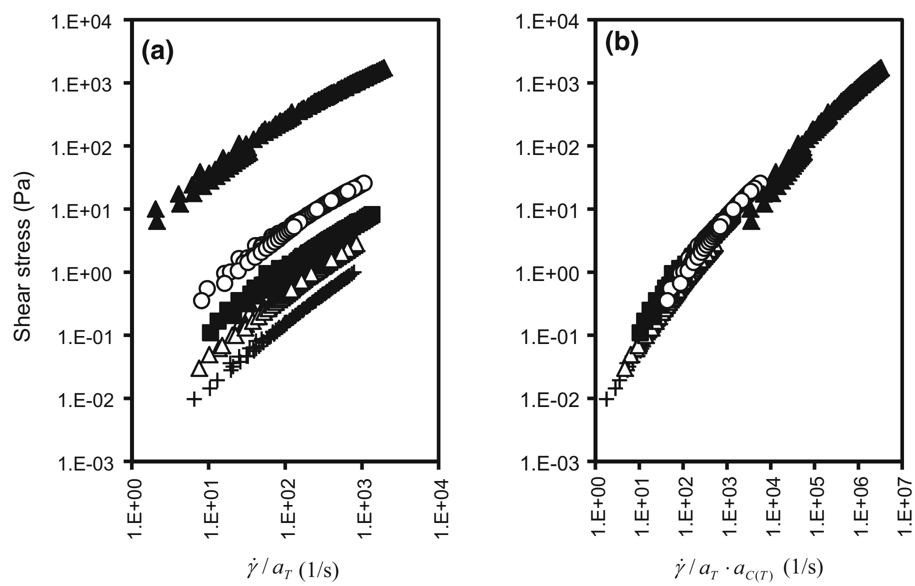

Fig. 3 **a** Shear rate-temperature superposition and **b** shear rate-temperature-concentration superposition with second horizontal shift for soursop. + 5 °Brix; 15 °Brix; 30 °Brix; 45 °Brix; 70 °Brix

than the *n* value for pomelo. These results are consistent with Krokida et al. (2001) who concluded that the pulpy products have *n* values of close to 0.5, while clear juices have *n* values of close to 1.

Figures 1, 2 and 3 illustrate the graphical results of master-curves for all three fruit juice concentrates after a double horizontal shifting and a vertical shifting for the guava juice. The guava juice had to be shifted in vertical direction at its third shift, encompassing a shear rate-temperature-concentration and shear stress-concentration superposition to get a smooth line. The 5 °Brix line for guava of Fig. 1b moves up along the shear stress axis to the reference concentration of 30 °Brix to obtain a smooth master-curve as Fig. 1c. The master-curve was necessary to achieve high shear rates of 10^6 s^{-1}, that of a typical shear rate range in food processing, i.e., pouring from a bottle (10^1–10^2 s^{-1}), swallowing (10^1–10^2 s^{-1}), mixing and stirring (10^1–10^3 s^{-1}), pipe flow (10^0–10^3 s^{-1}) and spraying (10^3–10^5 s^{-1}) (Steffe 1996). Figures 1c, 2b and 3b show that the rheological properties of guava cover the shear rate range of 10^{-2}–$10^6 s^{-1}$, while pomelo and soursop cover the shear rate range of 10^0–$10^6 s^{-1}$, and 10^0–$10^7 s^{-1}$, respectively. As these shear rate range encompasses the shear rate requirement in juice processing, the shear stress would be referred from these generated master-curves.

Conclusion

The rheological behaviour of guava, pomelo, and soursop were satisfactorily explained by the shear-thinning behaviour. For modelling of rheological properties of these juice concentrates using master-curve, the reference temperature and concentration could be arbitrarily chosen because the shape of curves remained unchanged during shifting. The rheological data was superimposed by a one dimensional double horizontal shifting or a two dimensional shifting involving a double horizontal and an additional vertical shifting to get a smooth master-curve. The master-curve gives information on flow behaviour over a broad range of shear rate. The rheological properties of fruit juice concentrates are very essential when heat transfer occur during unit operations like chilling, concentration, pasteurisation, pumping and agitation.

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