

## Effect of temperature and concentration on rheological properties pomelo juice concentrates

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**Abstract:** Rheology is the science of deformation and flow behavior of fluid. Knowledge of rheological properties of fluid foods and their variation with temperature and concentration have been globally important for industrialization of food technology for quality, understanding the texture, process engineering application, correlation with sensory evaluation, designing of transport system, equipment design (heat exchanger and evaporator), deciding pump capacity and power requirement for mixing. The aim of this study was to determine the rheological behavior of pomelo juice at different concentrations (20-60.4%) and temperatures (23-60°C) by using a rotational rotational Haake Rheostress 600 rheometer. Pomelo juice was found to exhibit both Newtonian and Non-Newtonian behavior. For lower concentration the Newtonian behavior is observed while at higher concentration Non-Newtonian behavior was observed. Standard error (SE) method was selected on the basis to carry out the error analysis due to the best fit model. For the four models the values of SE show that the Herschel-Bulkley and Power Law models perform better than the Bingham and Casson models but Herschel-Bulkley model is true at higher concentration. The rheological model of pomelo juice, incorporating the effects of concentration and temperature was developed. The master-curve was investigated for comparing data from different products at a reference temperature of 40°C. Multiple regression analysis indicated Master-Curve presents good agreement for pomelo juice at all concentrations studied with  $R^2 > 0.8$ .

**Keywords:** Rheology, shear rate, apparent viscosity, flow behavior index, consistency coefficient, Bingham, power law; Herschel-Bulkley, Casson, standard error (SE), Arrhenius model, master-curve

### Introduction

Drying is considered as final process for the manufacturing the industrial products such as dairy powders, dried grains and fruits. Fruit juices are valuable semi finished products for use in the production of fruit juice beverages and fruit juice powders. The conventional mode in which fruits are processed and preserved is the form of fruit juices/pulps (purees). However, preservation of juices is not economical, since the water content of fruit juices is very high, i.e. 75 to 90% (Ramteke *et al.*, 1993).

Concentration of fruit juices not only provides microbiological stability, but also leads to economical packaging, transportation and distribution of the final products. However, it is due to reduction in bulk of the products (Belibagli and Dalgic, 2007). In concentration processes, the solids content is increased up to 65 to 75% so that the final product is still in liquid form (Ramteke *et al.*, 1993).

The rheological behavior of juices is studied due

to application of its knowledge for equipment design and its relation to juice acceptance by consumers. The properties of fruit juices are depend on shear time as well as on shear rate (Cepeda *et al.*, 2002). And also the viscosity of fruit juice influences the selection of evaporators which are used for concentrating juices (Belibagli and Dalgic, 2007). In industrial operations a product is submitted to a range of shear rates and it is important to know how the viscosity will change with temperature at these shear rates to adequately design the equipment for these operations (Alvarez *et al.*, 2006).

The rheological behavior of fluids depends on their molecular structures. The flow behavior of fruit juices can be described by different rheological models (e.g. Newtonian, Power Law, Bingham, Herschel-Bulkley and Casson equations (1), (2), (3), (4), (5) respectively), depending on the nature of the juices (Belibagli and Dalgic, 2007; Altan and Maskan, 2005).

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$$\tau = \mu \cdot \dot{\gamma} \quad (1)$$

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

$$\tau = \tau_0 + k' \cdot \dot{\gamma} \quad (3)$$

$$\tau = \tau_0 + \kappa \cdot \dot{\gamma}^n \quad (4)$$

$$\tau^{0.5} = \kappa_0^{0.5} + \kappa_1 \cdot \dot{\gamma}^{0.5} \quad (5)$$

Where,  $\mu$  is the Newtonian viscosity,  $\tau$  is the shear stress,  $\tau_0$  is the yield stress,  $\dot{\gamma}$  is the shear rate,  $\kappa$  is the consistency coefficients,  $n$  is the flow behavior index,  $\kappa_0^{0.5}$  Casson yield ( $\text{Pa}^{0.5}$ ),  $\kappa_1$  is the Casson constant ( $\text{Pa s}^{0.5}$ ), and  $k'$  is the plastic viscosity (Holdsworth, 1993; Ahmed *et al.*, 2007).

Most fluid foods do not have the simple Newtonian rheological model; in other words, their viscosities are independent of shear rate or shear stress and not constant with temperature. Therefore, it is necessary to develop more complex models to describe their behavior (Alvarez *et al.*, 2006). Juices such as orange, grape, pineapple and apple are well established. Now, attention is drawn to the tropical juices and juice products (Anon., 2004; Kortbech-Olesen, 1990). Pomelo, Pommelo, Shaddock or limua bali [*Citrus grandis*(L) Osbeck] is giant citrus fruit native to southern Asia and Malaysia. It is thought to be the ancestor of the grape fruit. The pomelo is also called Shaddock after an English sea captain who introduced the fruit to the West Indies from the Malay Archipelago. In New Zealand and North American region, the fruit is still known as shaddock, but the name pomelo is also well known (Herbst, 1995).

Pomelo is commonly consumed as fresh fruit. They are also good for salad, jams, jellies, marmalades and syrups. Pomelo is used in religious ceremony, especially during Chinese New Year and the moon or autumn festival. Pomelo is used as a symbol of good luck and prosperity in Chinese New Year celebrations. The skins and the leaves could be boiled to prepare a ceremonial bath to ritually cleans a person and repel evil. The word for Pomelo in Chinese is pronounced the same as the word for blessing, or protection, thus its widespread presence in many buddhist shrines (Turk, 2002).

Many studies have been published on the characteristics and physical properties of pomelo, there are limited data demonstrating the rheological behavior of pomelo juice. The rheological data of pomelo juice at different concentrations and temperatures are used in producing the pomelo juice with various changes in their viscoelastic behavior. The pomelo juice is used for various purposes.

Concentrated pomelo juice is normally used for making health drink while thin pomelo juice is use for coloring or favoring (Chuah *et al.*, 2008). There are many publications on flow properties of juice concentrates and effects of temperature and concentration which most of them are based on viscometry data (Ahmed *et al.*, 2007). Table 1.1 gives some reported studies concerning the rheological behavior. The aim of this work was to investigate the rheological behavior of pomelo juice prepared from fresh pomelo. The various models such as Newtonian, Power Law, Bingham, Herschel-Bulkley and Casson were fitted to the experimental data to describe the flow behavior of the pomelo juice in different temperature and pomelo juice concentration.

**Table 1.** Reological models of some fruit juice

Researcher	Sample	Researcher	Sample
Constenia <i>et al.</i> (1989)	Apple Juice	Dak <i>et al.</i> (2007)	Mango juice
		Cepeda <i>et al.</i> (2002)	Blueberry cloudy juice
Ibarz <i>et al.</i> (1987)	Apple Juice	Kaya and Belibagli (2002)	Solid Gaziantep Pekmez (grape juice)
Saravacos (1970)	Apple Juice & Grape Juice	Assis <i>et al.</i> (2006)	yellow ombin
Juszczak and Fortuna (2003)	Strawberry Juice	Cabral <i>et al.</i> (2007)	blackberry juice
Kaya, and S <sup>o</sup> zer, (2005)	Sour pomegranate Juice	Arslan <i>et al.</i> (2005)	sesame paste / concentrated grape juice
Giner <i>et al.</i> (1996)	clarified cherry juices	Ibarz (1996)	Sloe ( <i>Prunus Spinosa</i> ) Fruit Juices
Belibagli and Dalgic (2007)	Sour-cherry Juice	Arslan <i>et al.</i> (2005)	sesame paste / concentrated grape juice
Altan and Maskan (2005)	Pomegranate Juice	Cabral <i>et al.</i> (2007)	blackberry juice
Singh and Eipeson (2000)	Clarified Mango Juice	Assis <i>et al.</i> (2006)	yellow ombin
Akbulut <i>et al.</i> (2008)	Juniperus drupacea Fruit Juice (pekmez) Concentrated		

## Materials and Methods

### Preparation of samples

Fruits were purchased from a local market and washed with water to remove any adhering substances, sliced and hand peeled. Juice was extracted from the fruits by homogenizing in waging blender at 8000 rpm for 3 min and centrifugation at 9000 rpm for 10 min. In order to select the variables which are likely to be important in preparing the juice concentrates, response surface methodology (RSM) is used. It is usually called a screening experiment. The objective of factor screening is to reduce the list of candidate variables to a relatively few so that subsequent experiments will be more efficient and require fewer runs or tests. The purpose of this phase is the identification of the important independent variables. This is done and reported in our previous study (Keshani *et al.*, 2010). The relate from the RSM can be used to prepare different concentration of pomelo juice under different process conditions. The pomelo

juice was prepared at different concentrates 20, 30.4, 40.4, 53.4 and 60.4°Brix by a small scale laboratory vacuum evaporation (HEIDOLPH, Germany) at 60 rpm and 50°C. The extracted juice was stored at 4°C until used. The concentrate is a stable liquid in cold storage (Cepeda *et al.*, 2002).

#### Rheological measurements

The rheological measurements were performed in a controlled-stress rheometer (RheoStress 600, Haake, Karlsruhe, Germany). Shear stress/shear rate rotation ramp tests (mechanical spectra) were performed using a cone sensor (C35/2° Ti; 222–1632; 35 mm diameter, 2° angle), with 0.105 mm gap and a measuring plate cover (MPC 35; 222–1549). The sample compartment was controlled at a temperature of 25°C using a water bath/circulator Haake DC-30 and a Haake Universal Temperature Controller System (UTC) (Haake, Karlsruhe, Germany). Ascending and descending flow curves of shear stress versus shear rate were carried out in the range of 0–1000 s<sup>-1</sup> during 15 s. The sample was not reused after heating due to the change in rheological properties (Izidoro *et al.*, 2008).

Experimental data were evaluated and fitted according to the rheological models of Newtonian (Eq. (1)), Power Law (Eq. (2)), Bingham (Eq. (3)), Herschel-Bulkley (Eq. (4)) and Casson (Eq. (5)). The flow behaviors of pomelo juice were analyzed at different temperature of 23, 30, 40, 50, and 60°C and different concentrates of 20, 30.4, 40.4, 53.4 and 60.4°Brix. Three replications were made with a fresh sample used for each run. The equipment was driven through the Haake software, Rheowin Job Manager Version 3.30. The data of the rheological measurements were analyzed using the Rheowin Data Manager software Version 3.30. The best fit model was selected on the basis of standard error, which is defined as

$$SE = \left( \sum (Y_m - Y_c)^2 / n - 1 \right)^{1/2} \quad (6)$$

Where  $Y_m$  is the measured value,  $Y_c$  is calculated value for each data point, and  $n$  is the number of observations (Nindo *et al.*, 2007).

## Results and Discussion

### Rheological behavior

The rheological behavior of the pomelo juices at different concentrates (20, 30.4, 40.4, 53.4 and 60.4°Brix) and temperatures within the range 23 to 60°C. The flow behaviors of pomelo juice

were analyzed at 23, 30, 40, 50, and 60°C. Three replications were made with a fresh sample used for each run. The measurement of viscosity is not necessarily a simple matter, and many factors have to be taken into consideration. The shear stress versus the shear rate data at each experimental condition were tested for various models (Newtonian, Power Law, Bingham, Herschel-Bulkley and Casson). Data derived from each model were fitted by least-squared regression method. The line will have  $R^2=1$  if the model matches the data perfectly. A significant deviation from these values can be used as an indication of the inadequacy of the model. Thus, the best model is determined by analyzing standard errors (SE) value which determined from equation. The effects of concentration and temperature on the models were also evaluated. Subsequently, general models describing the rheological behavior of pomelo juice at different temperature were developed. The rheology properties on the effect of concentration of pomelo juice were conferred.

In general, the plot of shear stress versus shear rate is known as rheogram. Rheograms of pomelo juice at 23°C room temperature at 60°C at concentration range from 20°Brix to 60.4°Brix were illustrated in Figures 1(a-e). The higher concentration (60.4°Brix) demonstrates at higher stress compare to the rheogram at lower concentration (20°Brix).

### Flow models

#### Newtonian model

A wide range of dilute solutions of food products show simple Newtonian behavior (Chuah *et al.*, 2007). Products showing Newtonian behavior have a linear relationship between shear stress,  $\tau$  (N/m<sup>2</sup>), and a shear rate,  $\dot{\gamma}$  (s<sup>-1</sup>) in equation (1). The viscosity values estimated from the Newtonian model for all experimental conditions studied are given in Table 2. The results show a reasonable fitting of these data on Newtonian model with good regression (0.92~0.99). It can be observed that flow behavior show the Non-Newtonian while at lower concentration pomelo juice tends to Newtonian behavior.

#### Non-Newtonian model

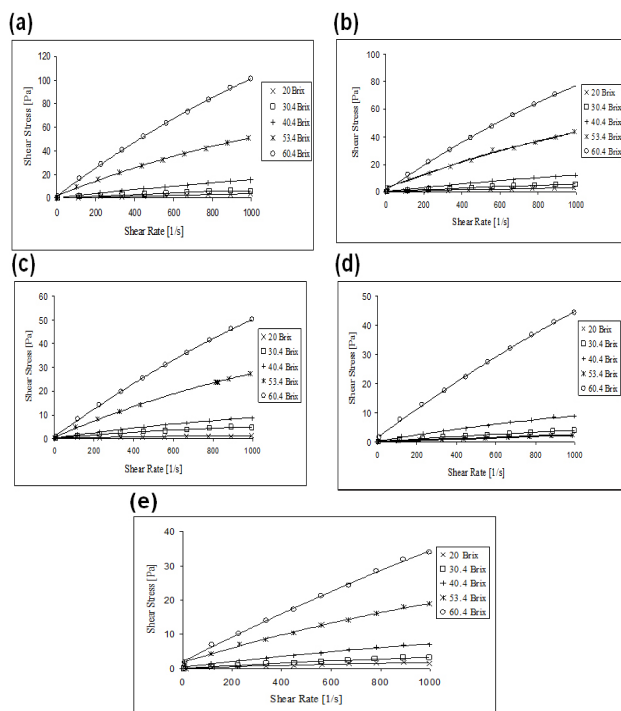
In spite of high coefficient of determination ( $R^2$ ) for fitting Newtonian model (e.g, at 23°C and concentration, 20°Brix,  $R^2$  is 0.996) it can be observed from Figures 1(a-e) the fluids show Non-Newtonian behavior. It can also be observed that as the values of shear rate increase linear form of the curves (specially, at higher concentration) tends toward non-linear form. This can be attributed to the wide range of



**Table 2.** Parameters of Newtonian Model

T (°C)		20 (°Brix)	30.4 (°Brix)	40.4 (°Brix)	53.4 (°Brix)	60.4 (°Brix)
23	R	0.003862 0.9960	0.006713 0.9828	0.01611 0.9935	0.05553 0.9845	0.1069 0.9936
30	R	0.003249 0.9934	0.006083 0.9876	0.01292 0.9915	0.04694 0.9776	0.08100 0.9918
40	R	0.001146 0.9268	0.005283 0.9727	0.009379 0.9863	0.02899 0.9920	0.05279 0.9948
50	R	0.002437 0.9967	0.004188 0.9901	0.009601 0.9863	0.02556 0.9834	0.04692 0.9936
60	R	0.001995 0.9494	0.003581 0.9814	0.007774 0.9788	0.02079 0.9646	0.03612 0.9883

shear rate 0-1000 s<sup>-1</sup>, used in flow curves of shear rate versus shear stress. Thus Non-Newtonian models are investigated to evaluate the Non-Newtonian behavior of pomelo juice.



**Figure 1.** Rheogram of pomelo juice at concentration of 20, 30.4, 40.4, 53.4, 60.4°Brix at (a)23°C (b)30°C (c)40°C (d)50°C (e)60°C

### Bingham plastic model

Many types of foodstuff, although showing Newtonian linear relationship of  $\tau / \dot{\gamma}$ , exhibit a yield stress, and are said to show plastic or viscoplastic behavior (Chuah *et al.*, 2008). The yield stress represents the minimum amount of stress that is needed to initiate flow (Kyereme *et al.*, 1999). Equation (2) indicates that the Bingham plastic model will show a straight line in terms of shear rate and stress. This model required to parameters, *i.e.*  $k'$  is the Bingham plastic viscosity and  $\tau_0$  is the yield stress and both parameters can be acquired from the linear graph, where  $k'$  obtained from the intercept. The graph shows a reasonable fitting of these data on Bingham model with good regression (0.95~0.99). The values of parameters were summarized in Table 3. It can be observed that the yield stress and Bingham plastic

viscosities increases as the concentrate increase. This implies that pomelo juice have tendency to remains rigid when the magnitude of shear stress is smaller than yield stress  $\tau_0$ .

### Power law model

One of the most widely spread models is the so-called power law for approximation of viscosity data. Power law model contains only two parameters (K and n) that can describe shear stress-shear rate data, thus it is used extensively to characterize fluid foods. The main reason for the power law being so popular is that the shearing rheological behavior of a fluid is represented simply by a straight line (Steffe, 1996). In order to quantify power law model, values of shear stress and shear rate were fitted into equation (7) shown below, by means of a statistical program Rheowin Data Manager Software Version 3.30 to plot performance graph that uses the power law algorithm.

$$\log \eta = \log k + n \cdot \log \dot{\gamma} \quad (7)$$

The results showed reasonably good fitting to power law model with R<sup>2</sup> 0.96 to 0.99. It is seen that the flow behavior index of the samples is between 0.65 and 0.89. As indicated by (Holdsworth, 1993) these samples are exhibiting the nature of pseudoplastic. Thus, shear thinning flow behavior is found at all samples where  $n < 1$ . The majority of fruit juice products are found to be pseudoplastic ( $0 < n < 1$ ), a situation that may be regarded as an indication of breakdown of structural units in a food due to the hydrodynamic forces generated during shear (Rao, 1999a; Arslan *et al.*, 2005; Chuah *et al.*, 2008; Nindo *et al.*, 2007). Most non-Newtonian food exhibit shear thinning behavior, including citrus e.g. orange juice, pomelo juice, grape juice (Hernandez *et al.*, 1995; Rao, 1999b; Kaya and Belibagli, 2002; Chuah *et al.*, 2008). Hence the overall average value of n is 0.762 for all samples. The flow behavior index practically did not change with the temperature (Table2). These results agree with those obtained in Kesar mango juice (Dak *et al.*, 2007), Totapuri mango juice (Dak *et al.*, 2006), yellow mombin (Assis *et al.*, 2006), and pineapple juice (Dak *et al.*, 2008).

### Herschel-Bulkley model

The rheological model that has been generally used for non-Newtonian fluids is the Herschel-Bulkley model. This model is appropriate for many fluid foods (Ahmed, 2004). The Herschel-Bulkley Model describes materials which combine power law and Bingham behavior. Above the yield stress

**Table 3.** Parameters for Power-law Model, Bingham Plastic Model, Casson Model, Herschel-Bulkley Model, Regression Analysis and Standard Error

Power-law Model					
T (°C)	C (°Brix)	K (Pa.s <sup>n</sup> )	n	R <sup>2</sup>	SE
23	20	0.008555	0.8799	0.9993	0.047
	30.4	0.02490	0.8019	0.9933	0.24
	40.4	0.04635	0.8403	0.9997	0.12
	53.4	0.2680	0.7614	0.9999	0.23
	60.4	0.3075	0.8404	0.9999	0.38
30	20	0.008957	0.8466	0.9990	0.045
	30.4	0.02040	0.8171	0.9963	0.16
	40.4	0.04181	0.8225	0.9996	0.11
	53.4	0.2386	0.7540	0.9958	1.24
	60.4	0.2553	0.8265	0.9994	0.88
40	20	0.01126	0.6539	0.9660	0.19
	30.4	0.03150	0.7299	0.9952	0.35
	40.4	0.03860	0.7861	0.9988	0.085
	53.4	0.1099	0.8006	0.9999	2.5
	60.4	0.1379	0.8549	0.9999	0.44
50	20	0.004875	0.8951	0.9991	0.032
	30.4	0.01371	0.8207	0.9981	0.078
	40.4	0.03858	0.7898	0.9982	0.17
	53.4	0.1221	0.7635	0.9995	14.37
	60.4	0.1319	0.8438	0.9996	0.39
60	20	0.01115	0.7398	0.9704	0.15
	30.4	0.01538	0.7796	0.9948	0.11
	40.4	0.04060	0.7501	0.9975	0.16
	53.4	0.1595	0.6917	0.9965	0.48
	60.4	0.1294	0.8072	0.9982	0.65
Bingham Plastic Model					
T (°C)	C (°Brix)	$\tau_0$ (pa)	k' (pa s)	R <sup>2</sup>	SE
23	20	0.1436	0.003658	0.9982	0.073
	30.4	0.3707	0.006187	0.9877	0.32
	40.4	0.7665	0.01502	0.9972	0.27
	53.4	4.014	0.04975	0.9941	1.81
	60.4	5.180	0.09955	0.9974	2.39
30	20	0.1479	0.003037	0.9969	0.080
	30.4	0.3142	0.005637	0.9920	0.24
	40.4	0.6964	0.01193	0.9963	0.34
	53.4	4.501	0.04053	0.9950	1.34
	60.4	4.098	0.07517	0.9960	2.27
40	20	0.1422	0.0009437	0.9582	0.23
	30.4	0.4305	0.004671	0.9846	0.37
	40.4	0.5825	0.008551	0.9929	2.82
	53.4	1.683	0.02679	0.9968	2.82
	60.4	2.385	0.04939	0.9981	1.43
50	20	0.01623	0.002320	0.9985	0.085
	30.4	0.2316	0.003858	0.9952	0.12
	40.4	0.6248	0.008713	0.9935	0.33
	53.4	2.175	0.02247	0.9966	14.35
	60.4	2.607	0.04321	0.9987	0.72
60	20	0.1564	0.001773	0.9600	0.17
	30.4	0.2366	0.003244	0.9889	0.16
	40.4	0.7390	0.006726	0.9959	0.20
	53.4	2.557	0.01716	0.9962	0.5
	60.4	2.760	0.03220	0.9987	0.55
Casson Model					
T (°C)	C (°Brix)	$\tau_{oc}$	$k_c$	R <sup>2</sup>	SE
23	20	0.02630	0.003152	0.9993	2.20
	30.4	0.1038	0.004902	0.9915	3.83
	40.4	0.1800	0.01237	0.9993	9.43
	53.4	1.367	0.03686	0.9991	32.32
	60.4	1.195	0.08208	0.9994	64.30
30	20	0.03382	0.002516	0.9987	1.79
	30.4	0.08211	0.004541	0.9950	3.47
	40.4	0.1737	0.009646	0.9988	7.50
	53.4	1.414	0.02975	0.9968	27.80
	60.4	1.041	0.06091	0.9986	48.46
40	20	0.05988	0.0006131	0.9662	1.28
	30.4	0.1541	0.003379	0.9918	3.13
	40.4	0.1755	0.006613	0.9971	3.08
	53.4	0.4920	0.02109	0.9995	19.4
	60.4	0.4890	0.04155	0.9996	36.53
50	20	0.01326	0.002032	0.9993	1.35
	30.4	0.05973	0.003090	0.9978	2.33
	40.4	0.1820	0.006753	0.9973	5.50
	53.4	0.6814	0.01671	0.9997	0.62
	60.4	0.5582	0.03570	0.9998	27.98
60	20	0.05313	0.001305	0.9664	1.052
	30.4	0.07159	0.002492	0.9933	1.95
	40.4	0.2352	0.004927	0.9983	4.34
	53.4	1.004	0.01145	0.9993	11.93
	60.4	0.7105	0.02522	0.9994	21.37
Herschel-Bulkley Model					
T (°C)	C (°Brix)	$\tau_0$	K	n	R <sup>2</sup>
23	20	0.01044	0.008235	0.8850	0.9993
	30.4	0	0.03621	0.7518	0.9937
	40.4	0	0.05001	0.8300	0.9997
	53.4	0	0.2701	0.7604	0.9999
	60.4	0	0.3254	0.8327	0.9999
30	20	0	0.009176	0.8433	0.9990
	30.4	0	0.02725	0.7783	0.9965
	40.4	0	0.04718	0.8062	0.9966
	53.4	2.342	0.1217	0.8449	0.9971
	60.4	0	0.2906	0.8091	0.9994
40	20	0.02524	0.008672	0.6888	0.9662
	30.4	0	0.04715	0.6765	0.9959
	40.4	0	0.05014	0.7511	0.9990
	53.4	0	0.1154	0.7941	0.9999
	60.4	0	0.1424	0.8505	0.9999
50	20	0.01853	0.004360	0.9103	0.9991
	30.4	0	0.01409	0.8170	0.9982
	40.4	0	0.04387	0.7725	0.9983
	53.4	0.6907	0.08620	0.8103	0.9999
	60.4	0.9526	0.09916	0.8826	0.9998
60	20	0	0.01893	0.6698	0.9713
	30.4	0	0.01981	0.7457	0.9950
	40.4	0.3291	0.02307	0.8263	0.9984
	53.4	1.463	0.06391	0.8144	0.9994
	60.4	1.749	0.06450	0.9017	0.9994

the rheogram is non-linear and may display either shear thinning or shear thickening. The model can be expressed mathematically as into equation (4). This relationship reduces (i) to the power law when there is no yield stress and  $\tau_c = 0$  and (ii) to the Bingham model when  $n=1$  (Smith, 2003). Herschel-Bulkley parameter is presented in Table 3. The  $n$  values ranged between 0.66 and 0.90 which indicate shear-thinning nature of pomelo juice. However, according to (Ahmed *et al.*, 2007), there is no trend for  $n$  values with temperature.

#### Casson model

The Casson model had been reported to applied widely in the range of food products such as molten chocolate, fruit purees, gums, fruit juice concentrate and tomato products (Rao, 1999b; Holdsworth, 1993). Casson model are closely related with the effect of particle size distribution on the flow behavior of pigment –oil suspensions (Chuah *et al.*, 2007; Chuah *et al.*, 2008). There are two parameter determined from Casson model, namely Casson yield,  $\tau_{cc}^{0.5}$  and the Casson Plastic viscosity,  $\eta_{ca}$ . Casson model parameters are tabulated in Table 3. The  $R^2$  value obtained is in the range of 0.96 to 0.99. The pomelo juice samples exhibited definite yield stress due to significant particle-particle interactions and crowding. Throughout the range of concentration studied, it is observed that the yield stress,  $\tau_{cc}$  obtained for pomelo juice at 60.4°Brix is higher than yield stress of the juice at 20°Brix. Proximate analysis result shows at least sugar content 0.113%, protein 6.26%, fiber 0.113%, Fat 0.30% and Moisture content 91% in the juice. Since sugar content plays a major role in the magnitude of the viscosity by (Hernandez *et al.*, 1995), thus higher sugar content will results in higher viscosity. Also, other particles (e.g. sugar content, fiber, pectin (according to Baker, (1997) pomelo contains 3.30-4.50% pectin) present in the high concentrated sample which cause the crowding occurs.

#### Apparent viscosity

Apparent viscosity,  $\eta$  is plotted as a function of shear rate. It is observed that the apparent viscosity decreased with the increasing shear rates for pomelo juice with different concentration that can be explained by the structural breakdown of the blend due to the hydrodynamic forces generated and the increased alignment of the constituent molecules e.g. sugar, oil and protein (Arslan *et al.*, 2005; Rao, 1999). Shearing caused progressive deformation and disruption of oil droplets, resulting in less resistance to flow (Arslan *et al.*, 2005). Also, at constant shear

rate, that the temperatures have a direct effect on the apparent viscosity as the results shows the apparent viscosity decrease with increasing temperature.

If given sufficient time to equilibrate, *i.e.* conduct the experiment with a larger data range of shear rate (e.g. 0-1000  $s^{-1}$ ), the apparent viscosity is expected to decrease until they reach a constant value, *i.e.* Newtonian viscosity,  $\mu$ . When shear force, associated with temperature, was applied, the particles would under go rearrangement among themselves in a direction parallel to the shear force. The particle-particle interaction is disrupted as particles tend to undergo vibration at higher temperature. Also, at higher temperature, many big particles would break into smaller particles. For example, the globular protein molecule may unfold or break into chain segments if render in a high temperature environment. This would results the particles in the fluid to flow easily due to the reduced particle-particle interaction and less restriction in flow. Thus the viscosity of the fluids decreases (Haminiuk *et al.*, 2006).

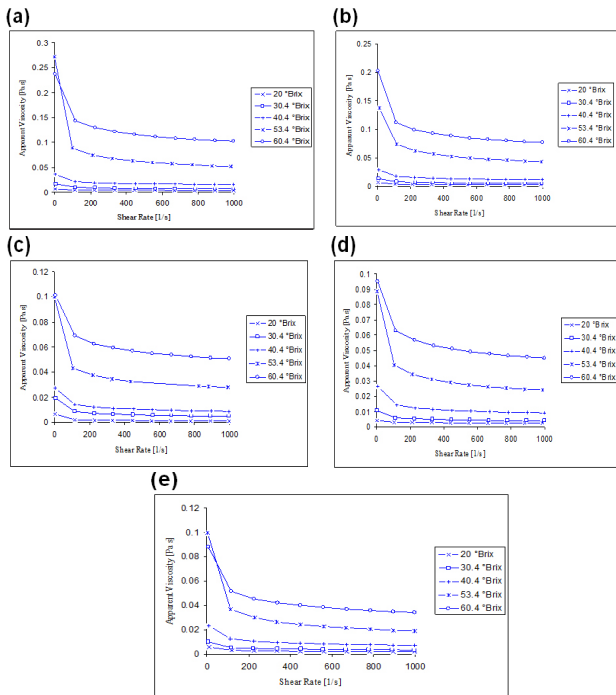
In Figure2(a-e), It is also observed that apparent viscosity decreases at a faster rate for higher concentration than at low concentration (Dak *et al.*, 2007) which suggested the pseudoplastic or shear-thinning nature of juice. The shear-thinning behavior indicates that the high water content of pomelo juice contribute to the lubricating effect between the particles. Thus, flow is relatively unhindered (Steffe, 1996; Chuah *et al.*, 2007; Chuah *et al.*, 2008). The data computed for apparent viscosity using equation (8).

$$\eta = k \dot{\gamma}^{n-1} \quad (8)$$

#### Comparison of selected rheological models

Comparing between coefficient of determination  $R^2$  and SE(equation 6), it can be inferred that the coefficient of determination for the five models are all close to one. Pomelo juice shows different behaviors at different temperatures and concentrations. It can be observed from Figures 2(a-e) that when the concentration is low, the juice tends to Newtonian behavior while with increase of concentration the juice presents Non-Newtonian behavior. In other words, It is observed that variation of viscosity on versus shear rate in high concentration is more than low concentration, e.g. at 30°C when shear rate varies from 4 to 1000  $s^{-1}$  the apparent viscosity varies from 0.00394 and 0.13(Pa.s) for 20 and 60°Brix, respectively.

As shown in Table 3, it can be observed that the values of standard error for Non-Newtonian models Power Law and Herschel-Bulkley models are less



**Figure 2.** Variation in Apparent viscosity of pomelo juice with shear rate for (a)23°C, (b) 30°C, (c) 40°C, (d) 50°C and (e) 60°C Cat different concentrate

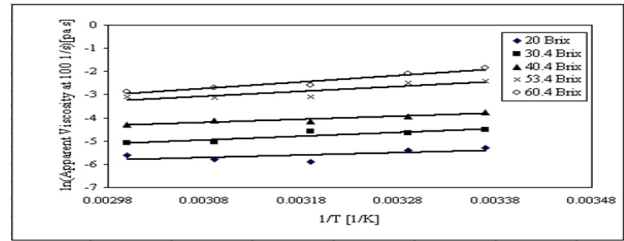
than Bingham and Casson models. On the other hand, Herschel-Bulkley model shows two different trends that observed in pomelo juice at different temperature and Brix. There is no yield stress observation in low temperature for all range concentrations however at higher temperature (50-60°C) the values of yield stress are between 0.3 and 1.7. Thus, it can be concluded that Herschel-Bulkley model fits well at higher concentration.

*Effect of temperature*

Temperature has an important role on rheological characteristics of any food products. The effects of temperature on the viscosity of pomelo juice at a specified shear rate were determined. The applicability of the Arrhenius model to describe the effect of temperature on the apparent viscosity (equation 10) at a constant shear rate of 100 s<sup>-1</sup> was investigated.

$$\eta = \eta_0 \cdot \exp\left(\frac{E_a}{RT}\right) \tag{9}$$

As expected, the temperature effects on apparent viscosity of pomelo juice at different concentration that the apparent viscosity was reduced with increasing temperature and shear rate. The applicability of the Arrhenius model to the apparent viscosity versus temperature data on a pomelo juice sample at different concentration are shown in Figure 3.



**Figure 3.** Applicability of the Arrhenius model to the apparent viscosity,  $\eta$  versus temperature data on pomelo juice at different concentration

In general, from Figure 3 it is observed that apparent viscosity decrease with increased temperature. These results applicability of the Arrhenius model to the apparent viscosity versus temperature data agree with this obtained in tamarind juice concentrates (Ahmed *et al.*, 2007). The parameters obtained from Arrhenius model by curve fitting is tabulated in Table 4.

**Table 4.** Arrhenius model parameters for the apparent viscosity,  $\eta$  at 100 s<sup>-1</sup> and the consistency index, K of different concentration pomelo juice samples

Sample (°Brix)	$\eta_0$	$E_a$ (J/mol)	R <sup>2</sup>
20	1.13x10 <sup>-4</sup>	9103.83	0.4079
30.4	4.74x10 <sup>-5</sup>	13492.80	0.788
40.4	2.60x10 <sup>-4</sup>	10974.48	0.8982
53.4	5.60x10 <sup>-5</sup>	18134.50	0.7907
60.4	1.13x10 <sup>-5</sup>	23391.43	0.9354

In this study, the magnitudes of activation energy of the samples for the entire temperature range were determined by linear regression analysis. It seems that sample at lower concentration (R<sup>2</sup>=0.4079) and higher concentration (R<sup>2</sup>=0.9354), using the apparent viscosity at 100 s<sup>-1</sup> for the Arrhenius model, gives a good fit from the least-square technique. These values indicated apparent viscosity at specific shear rate follow Arrhenius model adequately. From Table 3, the magnitude of activation energy increase with the concentration. Except for the 40.4°Brix that value then dropped suddenly. As noted by (Guerrero and Alzamora 1997; Nindo *et al.*, 2007), there are probably some interactions promoted by high temperatures that could affect the temperature dependence of the consistency index. This is owing to the important role of sugar content on the activation energy of the pomelo juice. A significant increase in activation energy relating to apparent viscosity with concentration is observed. This is because activation energy value indicates the sensitivity of the apparent viscosity to the temperature changes. Higher activation energy means that the apparent viscosity is relatively more sensitive to temperature change (Kaya, and Sö zer, 2005; Haminiuk *et al.*, 2006).

*Effect of concentration*

At all temperatures, the consistency index



of pomelo juice increased with increasing concentrations as shown in Figures 4 and 5. The effect of concentration, C of pomelo juice on either apparent viscosity,  $\eta$  or the consistency index, K of the power law model can be described by either exponential or power law relationship. In this study, exponential relationships as shown below, at five level of temperature (23°C, 30°C, 40°C, 50°C and 60°C) were satisfactory in analyzing this effect.

$$K = A \exp(BC) \quad (11)$$

$$\eta = A' \exp(B'C) \quad (12)$$

Equations 11 and 12 were linearised to ease the data analysis by linear-square regression. In these equations, the terms other than viscosity, consistency index, apparent viscosity at constant shear rate and concentration were calculated at different temperatures by regression analysis.

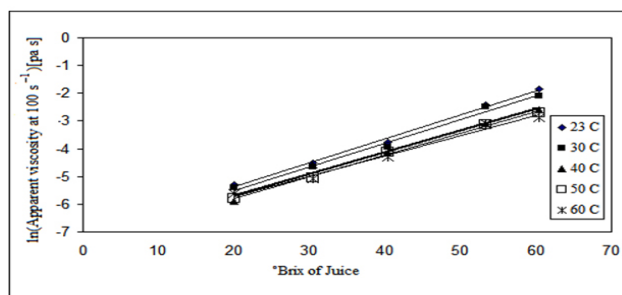


Figure 4. The influence of concentration on Apparent Viscosity at shear rate  $100 \text{ s}^{-1}$  of pomelo juice at different temperature

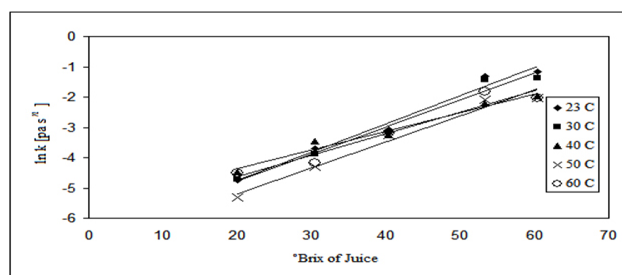


Figure 5. The influence of concentration on consistency index of pomelo juice at different temperature

The influence of concentration on  $\eta$  (at shear rate of  $100 \text{ S}^{-1}$ ) and on K, is shown in Figures 4 and 5, respectively. It is observed that juice at higher concentration will have higher viscosity. This is probably due to the presence of more low and high molecular weight solutes such as salts, acids, pectin and other particles in pomelo juice in the higher concentration. It is important to note that the consistency index and apparent viscosity increased as concentration increased. This agreed with the findings by Bayindirli, 1993 and Ibarz *et al.*, 1994 who showed that the apparent viscosity decreased as the concentration of the grape juice and orange juice

decreased, respectively. In this study, the regression shows a very good agreement for all application for consistency index and apparent viscosity in exponential relationship.

#### Master curve

Master –curve can be very useful in comparing data from different products such as concentrated orange juice made from different varieties of oranges (Steffe, 1996). A reference temperature of 40°C will be used in developing a master-curve of the experimental data. Developing a master-curve requires a horizontal shifting of the data at 23°C, 30°C, 50°C and 60°C to the 40°C curve. A dimensionless shift factor ( $a_T$ ) is numerically found to account for the movement of each curve. Shear stress curves shear rate divided by the shift factor ( $\frac{\tau}{a_T}$ ) are plotted to produce master curve. The shear stress, of pomelo juice with different concentrate of 100 Pa will be used as the basis for determining  $a_T$ . The master curves were produce from the aforementioned data is illustrated at 60.4°Brix in Figure 6 and can be given as:

$$\text{for } 20^\circ\text{Brix} \quad Y=0.0001x + 0.4595 \quad R=0.8752 \quad (19)$$

$$\text{for } 30.4^\circ\text{Brix} \quad Y=0.0028x + 0.6231 \quad R=0.8938 \quad (20)$$

$$\text{for } 40.4^\circ\text{Brix} \quad Y=0.0064x + 1.364 \quad R=0.9588 \quad (21)$$

$$\text{for } 53.4^\circ\text{Brix} \quad Y=0.026x + 1.117 \quad R=0.8371 \quad (22)$$

$$\text{for } 60.4^\circ\text{Brix} \quad Y=0.0453x + 4.8487 \quad R=0.9922 \quad (23)$$

Horizontal shifting causes the data to overlap on a trend line obtain from linear regression. Multiple regression analysis indicated Master-Curve presents good agreement for pomelo juice at all concentrations studied with *R-squared*,  $R^2 > 0.8$ .

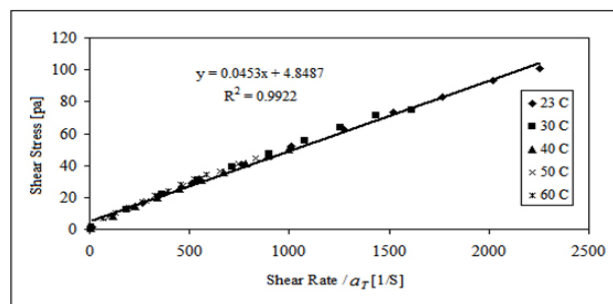


Figure 6. Plot of shear stress versus ( $\frac{\tau}{a_T}$ ) providing a master-curve of pomelo juice at 60.4°Brix having a reference temperature of 40°C

#### Conclusions

Pomelo juice show different trends that observed at different temperature and concentration. It can be observed that the concentration increases, the flow behavior show the Non- Newtonian while at lower concentration pomelo juice tends to Newtonian behavior. Herschel-Bulkley model exists at higher concentration of pomelo juice in as much as there is no



yield stress observation in low temperature for almost range concentrations. Power Law model indicates that pomelo juice presents pseudoplastic behavior for all concentration and temperature studied, as the flow behavior index obtained is less than 1. The analysis of apparent viscosity curve for pomelo juice shows typical shear thinning behavior. This is due to the high water content of pomelo juice contributing to the lubricating effect between the particles. The effect of temperature was described by an Arrhenius model, on apparent viscosity at a constant shear rate. From results, the magnitude of activation energy increase with the concentration. Master- curve obtained from the experimental data of pomelo juice presents good agreement at all concentrations studied. It can be very useful in comparing data from different products.

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