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RHEOLOGICAL AND FUNCTIONAL PROPERTIES OF ROSELLE (*HIBISCUS SABDARIFFA*) LEAVES PUREE

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ABSTRACT

Pureed form of leaves (*Hibiscus sabdariffa* L. (Roselle)) was taken for physicochemical and rheological analysis at temperatures and TSS range of 278 K – 318 K and 3 – 5 °Brix respectively. The steady-state rheological analysis was performed with a shear rate of 1 – 100 $s^{-1.}$ Different rheological models are tried; Power-law was best fitted with the experimental data ($R^2 \geq 0.98$). Temperature dependence of viscosity was found out using an Arrhenius-type relationship at a shear rate of 10, 50, 100 s^{-1} IR analysis was done to know the influence of functional groups on rheological properties of purees. Consistency index (K) of puree increases with increase in TSS content but at a fixed TSS, there is a decrease in K with an increase in temperatures but the opposite was observed for flow behavior index (n). Puree showed a shear thinning behavior with an increment in temperature level and puree having 5 °Brix (8.37) has higher activation energy ($kJ.mol^{-1}$) than 3 °Brix (6.32).

Keywords: Roselle leaves; puree; TSS; temperature

INTRODUCTION

Roselle (*Hibiscus sabdariffa*) locally known as gongura, belongs to the *Malvaceae* family and are cultivated in large quantities in the tropical regions of Asia, Africa and Central and South America. It is an annual or perennial herb with leaves is 8 – 15 cm long and profoundly 3 to 5 lobed and arranged alternately on the stem (Figure 1). These leaves are most commonly used by the people as curries, potherbs and mixed green salad **(Adegunloye et al., 1996)**. Roselle consumed throughout the southern part of India, is one of the unique dishes in many eateries, hotels, restaurants and food joints, and often called as the king of all Andhra food. The leaves are used for making pickles and chutney and are often named as gongura pickle, ambadi, and chutney. It is a very good source of folic acid and iron **(Sutton, 2004)**. The high iron content imparts the leave sourness and bitterness. It is a rich source of minerals, organic acids, vitamins, and antioxidants, which have distinctive medical benefits such as antiscorbutic, diuretic impacts, emollient, narcotic and many more uses **(Ramakrishna et al., 2008)**.

Fresh Roselle leaves are perishable in nature and the quality of the leaves deteriorates due to microbial and physiological activities during the period of storage and transportation and hence requires immediate processing and preservation **(Singh et al., 2014)**. Minimally processed state in form of puree has been very attractive in processing, handling, storing and selling. These are easily consumable and hence, there is a need for methods to preserve and maintain the quality of purees within fresh form until consumed by the customer. As puree is a mixture of soft particles in serum or viscous gel and finds uses in as topping, seasoning and as recipes for the fast-food industry **(Colin-Henrion et al., 2007)**.

Flow properties of purees product are of extensive interest for the development of new product items for manufacturing, quality control, and engineering applications **(Bayod, 2008; Alvarado and Romero, 1989; Rao et al., 1981)**. Also, there is a need to characterize the effect of rheological properties during food handling operations and storage period. With increasing demand, and its commercial importance of puree in our day to day activity, the present research was conducted: a) to assess the physicochemical properties of the puree, b) to assess the change in the rheological behavior of the purees and try to decide a best rheological model to the concerned flow curves obtained during rheological analysis, c) study the effect of various parameters such as TSS (3 – 5 °Brix), pH (1.3 – 5.3), and temperature (278 K – 318 K) on the rheological behavior of puree concentrate.

Scientific hypothesis

Temperatures, TSS and pH are the important parameters, which affect the rheological properties of Roselle leaves puree.

MATERIAL AND METHODOLOGY

Preparation of blanched puree

Roselle leaves were purchased from the local market of Raipur (Chhattisgarh, India), washed thoroughly with running water, and then destemmed. Leaves were blanched at 363 K for 5 min. Efficacy and duration of blanching were determined by peroxidase inactivity test. Chilled leaves after removing excess amount of water were grounded using a mixer grounder (Bajaj, India) at 298 K for 15 min with the addition of distilled water. The grounded puree was filtered through filter paper (Whatman, 125 mm). Uniform consistency of the puree was maintained by passing through a 12-mesh screen. The lab size rotary evaporator (Cole-Parmer, India) prepared the puree concentrates of different TSS at 323 K and 160 mm Hg pressure. Samples of the desired range of TSS (3, 4.35, 5 °Brix) were obtained by diluting with appropriate amounts of distilled water.

Physico-chemical analysis

Moisture content was determined as per **Garcia-Segovia et al. (2010)** at 333 K on a wet basis. Ash content was calculated as per the standard method of **AOAC (1984)**. Chlorophyll content was analyzed using the method described by **Arnon (1949)**. The ascorbic acid content of the puree was found out by titration method **(Ranganna, 1986)**. Titratable acidity was calculated in terms of citric acid equivalent as per the process proposed by **Wang et al. (1995)**. TSS (°Brix) and pH was measured using digital refractometer (Atago, Japan) and pH meter (Handy) respectively. The acidic pH (1.3 – 5.3) of purees was maintained by using a buffer for physicochemical and rheological analysis.

FTIR analysis of Roselle puree

The spectra of puree are used for analysis by FTIR instrument (Bruker, Germany) in the frequency ranging from 500 cm^{-1} to 4000 cm^{-1}. The functional groups present in the puree are identified using the spectral data obtained with the reference.

Rheological measurement

The rheological test was conducted in a modular compact Rheometer MCR 102 (Anton Paar, Germany), mounted with ST22-4V-40 (four-bladed vane geometry). The temperature of the cup was varied from 278 K – 318 K, for various experimental runs. The experiment was performed within shear rate (1 – 100 s^{-1}). The rheological estimations were investigated utilizing the Rheoplus software package of Anton Paar.

Rheological models

Numerous factors influence the selection of a rheological model to describe the flow behavior of a particular fluid. In this work, the experimental values of shear rate and shear stress were fitted to the various non-Newtonian models as listed in Eq. (1 – 6).

Power Law: $$\tau = K(\gamma)^n \quad (1)$$

Herschel-Buckley: $$\tau^{0.5} = \tau_0 + K(\gamma)^n \quad (2)$$

Casson : $$\tau^{1/2} = \tau_0^{1/2} + K\gamma^{1/2} \quad (3)$$

Heinz-Casson: $$\tau^n = \tau_0 + K\gamma^n \quad (4)$$

Vocadlo: $$\tau^{1/n} = \tau_0^{1/n} + K\gamma \quad (5)$$

Mizrahi- Berk: $$\tau^{1/2} = \tau_0^{1/2} + K\gamma^n \quad (6)$$

where, τ , γ , K, n, τ_0 is shear Stress (Pa), shear rate (s^{-1}), consistency index ($Pa.s^{-1}$), behavior index (dimensionless), yield stress (Pa), respectively.

Statisic analysis

The selection of most suitable model among these, for prediction of the flow behavior of puree, was investigated taking account of the statistical parameters, root means square error (RMSE) (Eq. (7)) and coefficient of determination (R^2) (Eq. (8)). The fitted data, which showed the lowest values for RMSE and highest values for the R^2, was considered as the best-fit model. ANOVA and t-test were used to determine the mean differences. The significant difference was defined at p <0.05 with 95%

Figure 1 Photo of Hibiscus Sabdariffa leaves attached with stem part.

level of confidence.

The average value of all the experimental data was analyzed using statistical parameters including Root mean square error (RMSE) and coefficient of determination (R^2) were determined and were defined as,

$$RMSE = \sqrt{\frac{\sum_{i=1}^{N}(EV - MV)^2}{N}} \qquad (7)$$

$$R^2 = 1 - \frac{\sum_{i=1}^{N}(MV - EV)^2}{\sum_{i=1}^{N}(EV - EV_{avg})^2} \qquad (8)$$

Where EV is the experimental value, MV is the modeled value EV_{avg} is the average of the experimental value and N is the number of observations.

Effect of temperature on apparent viscosity

Temperature dependency on its viscosity was determined using Arrhenius model (Eq. (9)) **(Sengül et al. 2005)**.

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

Where, η_0 is the proportionality constant, E_a the activation energy (kJ.mol^{-1}), R the universal law gas constant (J.mol^{-1} K^{-1}), and T the absolute temperature (K).

Effect of TSS on activation energy

Two models, namely, the power law Eq. (10) and exponential model Eq. (11) were used to describe the variation of the activation energy with the TSS, as given below.

$$E_a = a(C)^{b} \qquad (10)$$

$$E_a = a \exp(bC) \qquad (11)$$

Where, a (Empirical constant (kJ.mol^{-1})), C (TSS (°Brix)) and b (Constant).

The combined effect of temperature and TSS content on apparent viscosity

The combined effect of temperature and TSS on apparent viscosity was evaluated by Power law type Eq. (12), first-order exponential model Eq. (13) and second-order exponential model Eq. (14) **(Juszczak and Fortuna 2004; Manjunatha et al. 2012)**. The estimates and fits of the different parameters were found at 5% significant level ($p < 0.05$). The suitability of the model was choose based on RMSE values and correlation coefficient (r).

$$\eta = a(C)^{C} Exp(\frac{E_a}{RT}) \qquad (12)$$

$$\eta = aExp(\frac{E_a}{RT} + cC) \qquad (13)$$

$$\eta = aExp(\frac{E_a}{RT} + cC + dC^2) \qquad (14)$$

Where, η, E_a, R, C, T is apparent viscosity (Pa.s^{-1}) at shear rates (10, 50 and 100 s^{-1}), flow activation energy (kJ.mol^{-1}), gas constant, TSS (°Brix), absolute temperature (K), respectively, a is pre-exponential constant (mPa.s^{-1}), b=Ea/R, c is constant (°Brix^1) and d is constant (°Brix^2).

Effect of temperature on K and n

The effect of temperature on flow behavior index (n) and consistency coefficient (K) was evaluated using a modified Turian approach through a regression analysis **(Turian, 1964)** (Eqs. (15) and (16)):

$$\log k = \log k_0 - A_1 T \qquad (15)$$

$$n = n_0 + A_2 T \qquad (16)$$

Where A_1 and A_2 are the slopes for Turian models. Higher A_1 and A_2 represent more dependency of n and k to the temperature.

Effect of pH on viscosity of puree

Changes in viscosities of puree with TSS (3 °Brix) having different pH value were assessed. The pH range was varied from 1.3 to 5.3 (increment of 2 was adjusted using 0.2 M Na_2HPO_4 and 0.1 M citric acid) at shear rates (10, 50 and 100 s^{-1}) and fixed temperature of 303 K.

RESULTS AND DISCUSSION

Physico-chemical measurements

The physicochemical characteristics of purees are shown in Table 1. The purees having 3 °Brix shows the high moisture content, due to the presence of higher concentration of water. Purees having higher TSS showed the highest value of acidity, compared to that of lower TSS value, probably due to the presence of the highest amount of total organic acid (like ascorbic acid) in the leaf (57.2 mg.100g^{-1}).

The amounts of ascorbic acid reported in the present

Table 1 Physicochemical composition of Roselle leaves puree concentrates used for experiments.

Parameter		Quantity		
		3 °Brix	4.35 °Brix	5 °Brix
Moisture (%)		84.25 ±1.81	81.19 ±1.11	77.55 ±0.81
pH		1.4 ±0.011	2.3 ±0.014	5.8 ±0.023
Density		1.052 ±0.04	1.066 ±0.05	1.069 ±0.082
Ash content (%)		4.52 ±0.11	4.64 ±0.19	4.72 ±0.17
Dry matter content (%)		38.29 ±0.032	39.5 ±0.092	40.1 ±0.019
Chlorophyll (mg.mL^{-1})	a	2.19 ±0.13	2.2 ±0.16	2.30 ±0.18
	b	0.31 ±0.03	0.25 ±0.08	0.36 ±0.02
	Total	2.50 ±0.16	2.54 ±0.24	2.66 ±0.20
Ascorbic acid (mg.100g^{-1})		54 ±0.18	55.6 ±0.34	57.2 ±0.21
Titrable acidity (%)		0.62 ±0.012	0.65 ±0.022	0.69 ±0.015

Note: The results were expressed as mean values ±SD (n = 2).

work were found higher than the ones previously reported in the literature **(Nnam and Onyeke, 2003)**.

The differences observed might be due to different varieties, harvest conditions, genetics, environment, and ecology. The different TSS of purees showed different values of chlorophyll content due to the difference in the soluble solid present in the puree of all TSS. Ash content and density of puree increase slightly with an increase in TSS value, as TSS value represent its solid content.

The FTIR spectra confirmed the presence of –OH stretching (have a similarity to 3672 – 29,345 cm^{-1}), –CH stretching (have a similarity to 2856 cm^{-1}), bending aromatic compounds overtone (have a close similarity to 1792 and 1734.8), C=N stretching imine/oxime or C=O stretching (have a similarity to 1659.1 cm^{-1}), conjugated ketone or alkenes (have a similarity to 1635.1 cm^{-1}), and OH bending phenol (have a similarity to 1368 cm^{-1}).The frequency of absorbance at 2856 cm^{-1} confirmed the presence of –CH stretching due to the formation of H-bond. At 1659.1, broad-shaped bands with medium intensities were observed which indicates the C=O stretching of COOH and Acetyl groups in the sample **(Femenia et al. 2003)**. It was noted that with the increase in TSS, the intensity of the peaks also increased, this might be due to the reason that during the concentration process there is a conversion in functional groups. The presence of the biologically active compound is indicated by the presence of acetyl group, which helps to develop cross-molecular barrier in the cell **(Chang et al. 2011)**. In the case of Aloe Vera powder, **Kim et al. (2009)** reported similar band formation. The previous studies of strong bands on Aloe Vera depict the presence of monosaccharide units in the branched regions such as glucan and galactose in the range of 1156 – 1230 cm^{-1} **(Gentilini et al.2014; Ray and Aswatha, 2013)**.

Rheological analysis of Roselle puree

Figure (1-3) exhibits the effect of temperature on steady shear properties of the purees with a TSS of 3, 4.35 and 5 °Brix.

The obtained data were fitted to six non-Newtonian models (Eqs. (1-6)). The best numerical models for characterizing the flow characteristics of purees was chosen based on the coefficient of determination (R^2), RMSEs, the presence of yield stress and overall bias factors acquired during the fitting (Table 3 – 8). Although all models fitted well with the test data, at 5 °Brix esteem, Herschel-Buckley, Heinz-Casson, Vocadlo, and Mizrahi-Berk fitted model demonstrated negative values, which are having no physical importance. Hence RMSE values were not calculated for the rest of these unfitted models. Power law and Casson model fit satisfactorily at all temperature ranges examined and the R^2 value was found to be greater than 0.97 and hence were more appropriate to describe the rheological flow behavior of the puree.

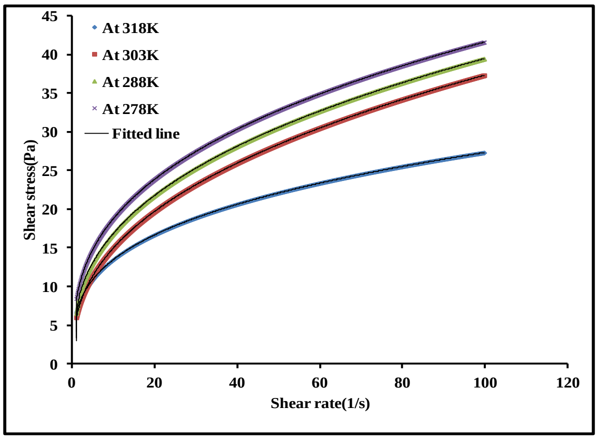


Figure 1 Shear rate vs. shear stress for the 3 °Brix of Roselle leaves puree at various temperatures (T=278 K – 318 K).

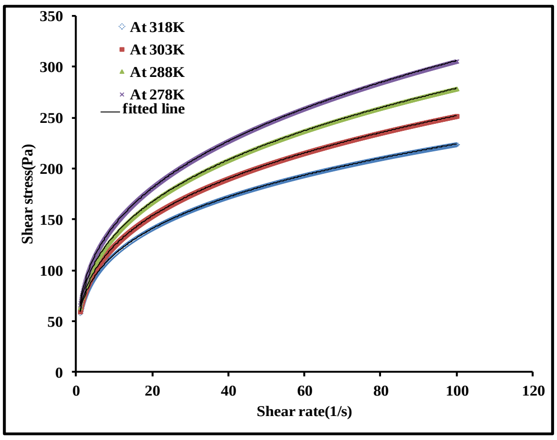


Figure 2 Shear rate vs. shear stress for the 4.35 °Brix of Roselle leaves puree at various temperatures (T = 278 K – 318 K).

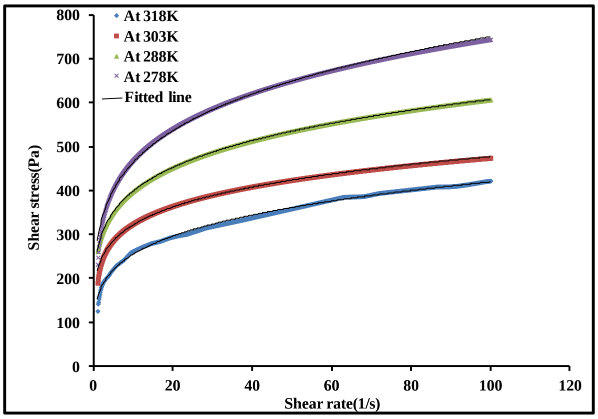


Figure 3 Shear rate vs. shear stress for the 5 °Brix of Roselle leaves puree at various temperatures (T = 278 K – 318 K).

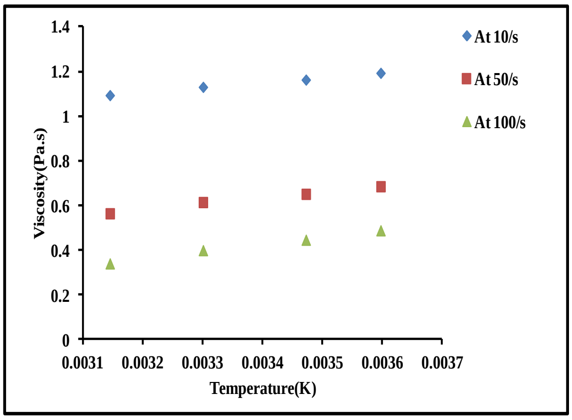


Figure 4 Shear rate vs. viscosity for the 3 °Brix of Roselle leaves puree at various temperatures (T = 278 K– 318 K).

Table 2 Functional groups present in Roselle leaves puree of 5 °Brix by FTIR analysis.

Functional groups	**Wave number**
Unknown	3750.3
Unknown	3708
Free alcohol O-H Stretching	3672
Intermolecular bonded alcohol O-H Stretching	3408
Intermolecular bonded alcohol O-H Stretching	2934.5
C-H stretching alkane	2856
C-H bending aromatic compounds overtone	1792 1734.8
C = N stretching imines/ oxime or C = O stretching	1659.1
Conjugated ketone or alkenes	1635.1
O-H bending phenol	1368
C-N stretching amine	1230 1156

Table 3 Parameters of the Ostwald-de-Waelle (power law) model fitted to the data of Roselle leaves puree.

TSS	**T (K)**	***n***	**K**	**R^2**	η_{50}	**RMSE**
3	278	0.396	8.477	1.00	0.642	3.360
	288	0.345	7.854	0.98	0.592	3.394
	303	0.343	6.010	1.00	0.543	4.125
	318	0.307	6.645	0.98	0.449	2.412
4.35	278	0.323	68.915	0.98	4.850	4.105
	288	0.310	65.340	0.99	4.485	4.013
	303	0.304	61.810	1.00	4.120	3.394
	318	0.285	60.170	0.97	3.670	2.351
5	278	0.193	304.940	0.98	12.600	4.324
	288	0.182	261.738	0.98	10.280	4.054
	303	0.158	350.280	1.00	9.250	3.387
	318	0.068	228.240	0.98	7.960	3.312

Table 4 Parameters of the Heschel-Buckley model fitted to the data of Roselle leaves puree.

TSS	**T (K)**	τ_0	***n***	**K**	**R^2**	**RMSE**
3	278	6.319	0.442	4.640	0.97	2.541
	288	6.673	0.461	3.816	0.97	3.145
	303	6.491	0.471	3.277	0.98	4.051
	318	5.699	0.440	2.896	0.89	3.360
4.35	278	59.199	0.431	33.945	0.97	3.394
	288	57.799	0.430	30.658	0.95	4.125
	303	56.399	0.428	27.301	0.94	2.111
	318	49.214	0.396	28.364	0.97	1.854
5	278	401.122	0.533	30.064	0.98	1.594
	288	365.004	0.507	23.418	0.96	1.141
	303	389.834	4.865	20.100	0.94	1.845
	318	-25528.300	0.001	25.240	0.84	1.098

Effect of K and n on the temperature at different TSS

The variation of K value with temperature (278 – 318 K) of puree is given in Table 3 for power law fitted model. K varied from 6.01 to 350.28 Pa.s^{-1} in puree with different TSS range. The lower values for K were observed at a higher temperature (318 K). The variation was more noticeable on puree having lower TSS value (3 °Brix). Higher TSS purees were found to have higher K value in magnitude. The increments in K were found with an increase in TSS value at a fixed temperature probably due to increment in the particle-to-particle contact **(Chin et al., 2009)**. A similar result was obtained for another vegetable **(Ahmed et al., 2013; Karababa and Develi Isikli, 2005)**. The impact of factors was discovered significant ($p < 0.05$) for all TSS (Table 3). The dependence of K on the temperature variation was also performed using Turian model (Eq. (15)) for Puree. Highest flow behavior dependency was found for 5 °Brix at varying temperatures

(Table 9). Increments in the temperature from 278 K to 318 K significantly affect the consistency index.

The *n* value of the puree ranged from 0.068 to 0.396 (Table 3) for various TSS studied, signifying gently non-Newtonian pseudoplastic shear thinning behavior of puree **(Muller 1974, Steffe, 1996)**. Turian models (Eq. (16)) were used to monitor the dependence of *n* on its temperature change and the model parameters are shown in Table 9. The variation of *n* value with temperature was lower (0.307 – 0.396) in lower TSS value as compared with the higher TSS value (0.068 – 0.193) in all temperature range. This may be because of the increase in soluble solid content. The decrement in n value of puree showed the steady loss of pseudo-elasticity **(Sikora et al., 2007)**. The sample having different temperature demonstrated different flow behavior values for different TSS sample. When the temperature varied from 278 K to 318 K, the n values decrease with increment in temperature. Comparative patterns for the n were accounted by **Rao et al. (1981)** for tomato juice concentrate. Besides TSS, the impact of every single other variable was found significant (p <0.05) (Table 3).

Effect of temperature on viscosity at a different shear rate

Effect of temperature on viscosity is important in determining the rates of heat exchange, energy utilization, and flow rates so it is feasible to assure persistent product flow **(Nindo, 2005)**. **Karwoski (2013)** reported that TSS and temperature influence physical properties, such as viscosity, specific heat, refractive index, density and boiling point, of the fruit product. The plot of the log of viscosity against 1/T (K^{-1}) at various shear rates shown in Figure 5 and the constants of Eq. (9) were determined. It was observed that viscosity of puree concentrates diminish with increment in temperature over the range of shear rates. Figure 6 shows that viscosity decreases with a shear rate, which may be because of increased versatility of macromolecules, because of temperature rise, causing less resistance to flow. The change of the magnitude of viscosity was more at shear (10 s^{-1}) contrasted with the little distinction in a shear rate of 50 and 100 s^{-1}. Interaction impact of temperature and shear rate was additionally assessed and was significant for puree at all Brix value (p <0.05).

Activation energy is minimum energy required for particles to get away from the impact of its neighboring particles during the characterizing the viscous flow. The extent of the vitality of initiation for flow increased with increment in the TSS of the puree, it demonstrates that more energy was required to defeat potential vitality hindrance at higher TSS. Both the power law (Eq. (10)) and exponential model (Eq. (11)) were tried to determine the effect of flow activation energy on TSS of Puree. A nonlinear trend of increment in activation energy ($p \leq 0.05$) of puree with the increase in solids content was observed. The exponential model (r >0.75) was more effective in explaining the impact of the TSS on the flow activation energy of the puree than the power law model (r <0.75) as shown in Table (10). A similar type of results was recorded in the case of pomegranate **(Kaya and Sözer, 2005)**. The activation energy ranged between 3.544 – 8.610 kJ.mol^{-1} for puree having different TSS range (3 – 5 °Brix) (Table 11). Higher values of activation energy may be associated with the higher temperature and TSS of the puree **(Ahmed et al., 2007)**. In pseudoplastic vegetable product, the activation energy was proportional to the n i.e., the more pseudoplastic the product, the less the impact of temperature on its apparent viscosity **(Sharoba et al., 2005)**. In this study, it was found that the activation energy was discovered lower for the puree having lower TSS value at a fixed shear rate (Table 11). Similar types of trends were observed by **Hernandez et al. (1995)** and **Vitali and Rao (1984)**. However, it was also observed that for all TSS Value, the activation energy of puree concentrates increase with an increase in shear rate. Higher activation energy values show a more impact of temperature on its viscosity, i.e. faster change in viscosity with temperature **(Sanchez et al., 2009)**. The outcomes demonstrated that temperature and TSS had significantly affected the thickness of puree concentrates. The viscosity value increases altogether with increment insoluble solid substance. Similar results were obtained by **Alpaslan and Hayta (2002); Akbulut et al. (2012); Arslan et al. (2005)**.

Combined Effect of Temperature and TSS on apparent viscosity

From the food process designing perspective, it is necessary to get a single equation, which represents the effect of both temperature and TSS dependency on the viscosity of puree. Few authors have utilized different types of equation to depict the joined impact of temperature and TSS on viscosity of the liquids **(Juszczak et al., 2010; Kaya and Sozer, 2005; Ibarz et al., 2009; Nindo et al., 2005; Nindo et al., 2007; Altan and Maskan, 2005; Juszczak and Fortuna, 2004)**.

Table 12 demonstrates the parameters for the various models for depicting the combined impact of temperature and TSS on the viscosity of puree. The correlation coefficients values of 0.992, 0.998 and 0.999 and % RMSE values of 10.259, 10.324 and 6.887 were obtained for the first order, exponential first order, and exponential second-order models, respectively. The second order exponential condition was ideal, because of high correlation coefficient and low percent RMSE values. The Estimated parameter values of Eqs. (12), (13) and (14) are given in Table 12 and model equation may be represented as (Eqs 17 – 19).

$$\ln\eta = \text{-}8.13685 + (\frac{602.275}{T}) + 6.045833 \qquad (17)$$

$$\ln\eta = \text{-}6.1791975 + (\frac{602.275}{T}) + 1.56987034C) \qquad (18)$$

$$\ln\eta = \text{-}4.900 + (\frac{602.275}{T}) + 0.89C + 0.085C^2) \qquad (19)$$

The viscosity of puree was significantly (p <0.05) influenced by temperature and TSS of puree. The surface plot for the joined impact of temperature and TSS on the consistency of puree at various shear rates is shown in Figure 7 (a – c).

Table 5 Parameters of the Casson model fitted to the data of Roselle leaves puree.

TSS	T (K)	τ_0	K	R^2	RMSE
3	278	35.121	1.077	1.00	1.234
	288	31.509	1.056	0.97	2.012
	303	29.604	0.898	0.98	1.058
	318	25.431	0.660	0.98	4.211
4.35	278	274.146	7.641	0.97	4.121
	288	256.604	6.811	0.98	3.124
	303	238.888	5.988	0.98	2.458
	318	221.455	5.159	0.97	4.354
5	278	967.060	11.756	0.97	4.001
	288	848.185	7.950	0.98	2.055
	303	693.148	5.589	0.98	4.517
	318	746.176	5.744	0.97	3.845

Table 6 Parameters of the Heinz-Casson model fitted to the data of Roselle leaves puree.

TSS	T (K)	τ_0	*n*	K	R^2
3	278	20.788	0.648	15.190	0.95
	288	23.532	0.755	3.460	0.940
	303	22.098	0.761	2.702	0.96
	318	18.129	0.785	1.446	0.95
4.35	278	909.601	0.598	838.955	0.99
	288	923.254	0.594	788.168	0.94
	303	942.032	0.588	734.583	0.91
	318	1047.212	0.569	898.567	0.99
5	278	21310.460	0.601	2439.761	0.92
	288	19364.434	1.017	2215.253	0.92
	303	22154.221	0.351	1984.214	0.90
	318	19238.830	0.561	3617.393	0.91

Table 7 Parameters of the Vocadlo model fitted to the data of Roselle leaves puree.

TSS	T (K)	τ_0	*n*	K	R^2
3	278	9824.763	-23.653	-6.372	89.000
	288	6335.603	20.053	5.296	92.000
	303	8773.116	24.345	5.470	88.254
	318	10552.290	28.807	4.753	0.910
4.35	278	332.122	1.556	2.973	0.990
	288	249.187	1.393	2.373	0.920
	303	181.312	1.232	1.844	92.000
	318	143.135	1.136	1.466	88.254
5	278	592.742	1.107	3.254	0.910
	288	200.640	-0.687	-1.367	0.925
	303	173.914	-0.708	-0.989	0.971
	318	139.636	-0.611	-0.878	0.891

At lower temperatures the magnitude of viscosity increases quickly with a fixed TSS of Puree and increases insignificantly at higher temperatures, this was because of increment in thermal energy of the particles, which increase its intermolecular spacing. A comparable sort of results was accounted for other liquid food **(Nindo et al., 2005; Ibarz et al., 1989)**. Size, shape, nature of solute and its condition of hydration and the type of solute and solvent influences the viscosity determination, thus might account for deviation in various cases. In the case of Roselle leaves, the solids present in the puree may be mainly fiber, diverse sugars (sucrose, fructose), vitamins, minerals, amino acids, proteins, hormones etc. The condition of hydration was distinctive for various TSS, the magnitude of viscosity relies on upon the sort of solids parts present in the puree **(Nindo et al., 2005; Telis et al., 2007)**.

Effect of pH on apparent viscosity

The influence of pH on the apparent viscosity of puree is shown in Figure 8. As the initial puree prepared from leaves is having pH of 1.3 ±0.26, so the puree is an anionic polysaccharide containing a great number of negative charge-bearing groups such as carboxyl groups, which may undergo different degrees of ionization with changing pH value, leading to changes in viscosity and rheological properties **(Lapasin and Pricl, 1995)**. At shear rates less than 50 s^{-1}, the highest viscosity was obtained over the pH range of 1.3 – 5.3.

Table 8 Parameters of the Mirazi-Berk model fitted to the data of Roselle leaves puree.

TSS	T (K)	τ_0	*n*	K	R^2
3	278	31.905	0.480	7.471	0.960
	288	43.776	0.625	3.300	0.950
	303	41.774	0.612	3.008	0.990
	318	31.615	0.527	3.518	92.000
4.35	278	401.213	0.582	28.740	88.254
	288	378.931	0.574	26.671	0.910
	303	358.100	0.582	22.778	0.990
	318	328.848	0.552	22.787	92.000
5	278	2537.131	-157.274	-146.966	88.254
	288	1719.754	1.001	3.736	0.960
	303	1668.560	-3163.220	2.962	0.950
	318	1782.440	-101.650	8.893	0.910

Table 9 The modified Turian parameters for Roselle puree at different concentrations.

TSS (°Brix)	$\log k = \log k_0 - A_1 T$		$n = n_0 + A_2 T$	
	$\log k_0$	A₁	n_0	A₂
3	2.0013	-0.0039	0.9202	-0.0019
4.35	2.1839	-0.0013	0.5829	-0.0009
5	3.8133	-0.0046	1.0489	-0.0030

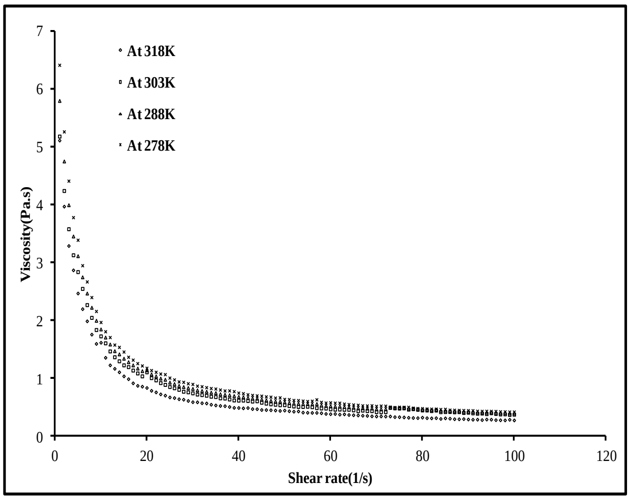


Figure 5 Rheogram of viscosity vs. temperature for the 4.35 °Brix of Roselle leaves puree at various shear rates.

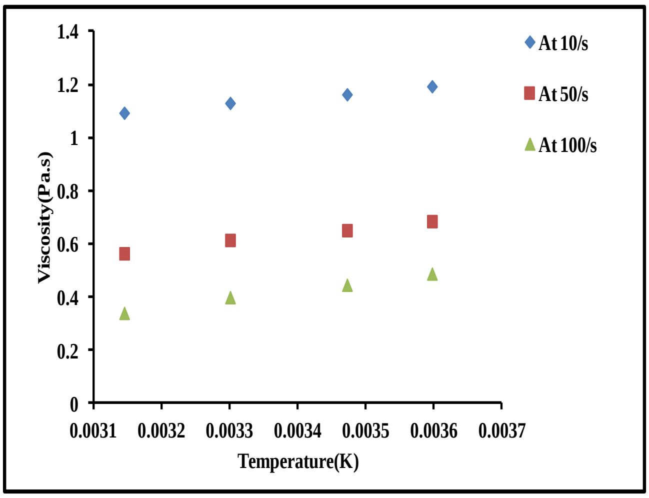


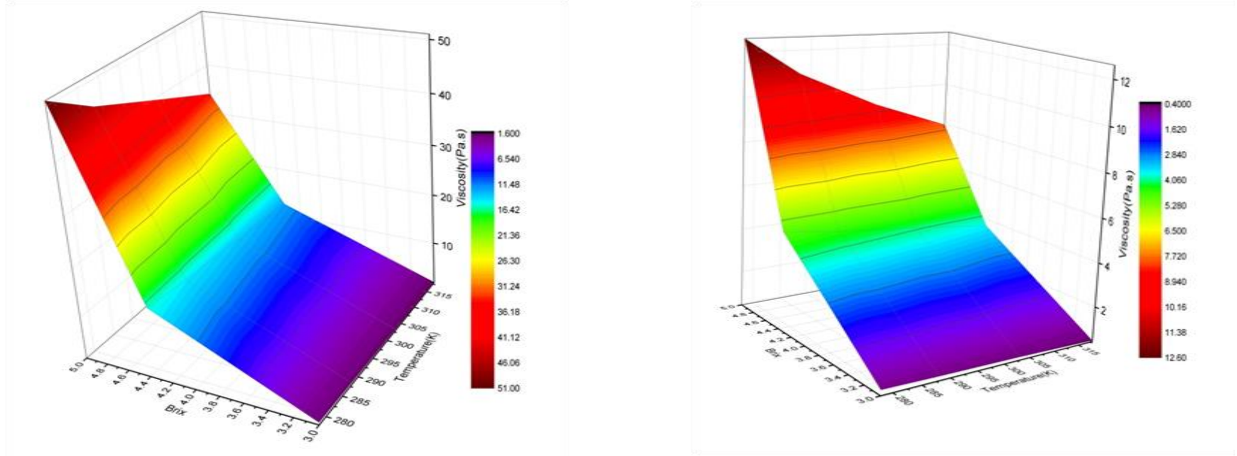
Figure 6 Shear rate vs. viscosity for the 3 ^{0}Brix of Roselle leaves puree at various temperatures (T = 278 K – 318 K).

Table 10 Effect on TSS (°Brix) on activation energy (E$_a$) values of Roselle leave puree.

Model	*a* (KJ.mol^{-1})	*b* (%)	r
$E_a = a(C)^b$	0.8741	1.2166	0.7041
$E_a = a \exp(bC)$	1.248	0.3261	0.755

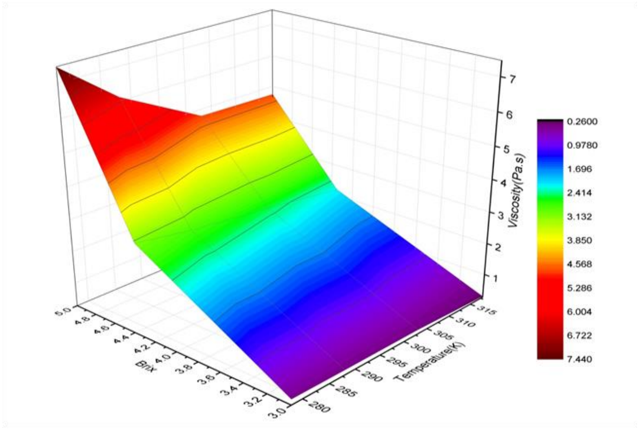
Table 11 Activation energy values at different concentrations and shear rates.

TSS (°Brix)	Shear rate (s^{-1})	η_0	Activation Energy(E$_a$) (KJ.mol^{-1})	r
3	10	-0.8594	3.5444	0.9977
	50	-3.1631	6.3210	0.9590
	100	-3.9252	7.1869	0.8364
4.35	10	0.9528	4.1428	0.9977
	50	-0.5848	5.0047	0.9934
	100	-1.5313	6.1411	0.9956
5	10	0.7569	7.3348	0.9980
	50	-1.0983	8.3772	0.9986
	100	-1.7639	8.6100	0.8988



(a) At a shear rate of 10 s^{-1}

(b) At a shear rate of 50 s^{-1}



(c) At a shear rate of 50 s^{-1}

Figure 7 Surface plots representing the combined effect of temperature and concentration on the viscosity of Roselle leaf puree at different shear rates: (a) 10 s^{-1}; (b) 50 s^{-1}; (c) 100 s^{-1}.

Table 12 Parameters of different models relating the combined effect of temperature and total soluble solid content of Roselle leaves puree.

Model	a (mPa.s^{-1})	b=Ea/R (K)	c (°Brix^1)	c (°Brix^2)	r	RMSE
Power Law	0.000293	602.2754	6.045833	---	0.992	10.25937
First order exponential	0.002072	602.27667	1.56987034	---	0.998	10.324
Second order exponential	0.007444	602.27667	0.89111167	0.08590097	0.999	6.887765

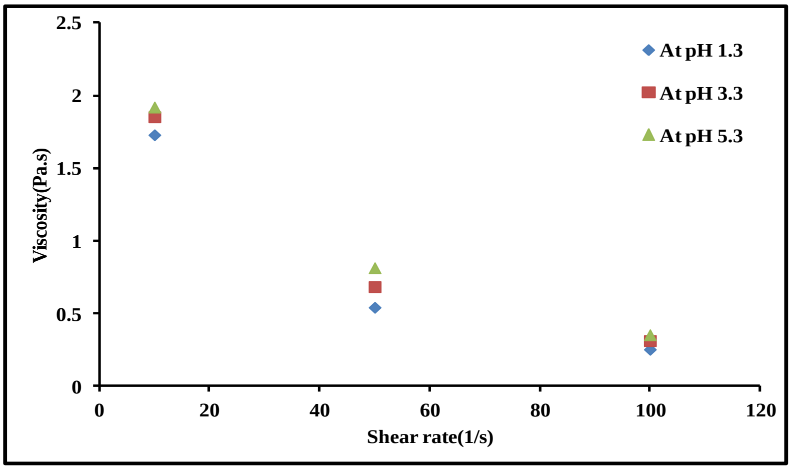


Figure 8 Rheogram of viscosity vs. shear rate for the 3 °Brix of Roselle leaves puree with varying pH (1.3 – 5.3).

There is an increment in viscosity is due to the ionization of carboxyl groups. As per **Feng et al. (2007)**, the viscosity will be at a most extreme when its atomic chains present in puree are entangling with each other. Past research work by the researcher on the impact of pH on viscosity of sweet potato puree and quince puree demonstrated that the consistency of puree increase with pH value **(Ice et al., 1980)**. On the other hand, in the more basic region, the solution consistency dropped at a fixed TSS. As the pH is raised, the functional group present induces electrostatic repulsion that tends to keep the atoms in a developed shape, in this manner producing an exceedingly thick solution **(Onweluzo et al., 1994; Launay et al., 1986)**. Results likewise demonstrated that at higher shear rates (more than 50 s^{-1}); changes in pH had no extensive impact on the viscosity.

In addition, the pH-dependence of viscosity may be due to change in puree confirmation. At low pH, polysaccharide chains tend to appear in coil state with acid groups in free acid form. With increasing pH, acid groups of coils are gradually ionized and the coils are expanded due to increase in electrostatic repulsion between functional groups, leading to more intermolecular interactions among the coils and consequent higher viscosity of the puree **(Feng et al., 2007)**.

The maximum viscosity was obtained around the pH 5.3 for Puree, where the shape of chains may be close to rod conformational state **(Achi and Okolo, 2004)**. This condition usually appears at where acid groups are ionized and electrostatic repulsion reaches a maximum and consequently, tends to keep the molecules in an extended form, leading to a high viscous solution and higher K values **(Medina-Torres, 2000; Coupland, 2013)**. The decrease of viscosity from pH 1.3 to 5.3 may be explained by the neutralization effect of added alkali on the negative charges of the puree, which reduces the hydrodynamic volume of the puree and consequent viscosity **(Chen et al., 2001; Porto et al., 2015)** and puree depolymerization under alkali condition, proposed by **Achi and Okolo, (2004)**.

CONCLUSION

Physicochemical properties of the Roselle leaf puree was found to increase with an increase in TSS value. FTIR spectra confirmed the presence of free alcohol, alkanes, alkene, intermolecular bonded alcohol, aromatic compounds, ketone, amine, phenol, imine, and oxime stretching in the puree. The observation of bands in the IR region made it evident resulting in the chlorophyll molecule masking the other molecules. The rheological analysis also plays a vital role to know the effect of the presence of different compounds on its flow behavior. In the present study, there was a 4 to 5% variation in chlorophyll contents with an increase in TSS contents of puree.

Rheological models were tried to fit with the experimental data of Puree and *best model was selected*. The power-law model display well-fitted data and was considered as a favored model for depicting the rheological properties of puree as impacts of temperatures, TSS and pHs. Puree samples exhibited pseudoplastic behavior (n <1) with the flow behavior index (n) between 0.28 and 0.82. The values of K were found to increase with TSS at a fixed temperature. The Arrhenius model describes satisfactorily, the temperature dependence of viscosity of puree. The activation energy of puree concentrates increase with an increase in TSS and shear rate. The model equation was developed and evaluated by the combined effect of TSS and temperature on the viscosity of puree.

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