

Rheometry of Roselle (*Hibiscus sabdariffa* L.) Seed Oil

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Received: 13.04.2017 | Revised: 22.04.2017 | Accepted: 23.04.2017

ABSTRACT

The viscosity of Roselle seed oil was studied to investigate the flow consistency index (K) and flow behavior index (n) while subjecting the oil to a shear rate of 10-10,000 per second for 4 minutes modeled with SAS¹⁵ software based on Ostwald-de Waele power law equation. The outcome of the analysis showed index properties of K and n as 0.029 and 2.068 respectively. The value of n (2.068) > 1; predicts a dilatancy a shear thickening behavior evident in the increased viscosity and decreased fluidity with increased shear rate. Dilatancy is a rare behavior in food material typical in sausage slurry, homogenized peanut butter, some types of honey and 40 % raw corn starch solution. The linoleic fatty was the major essential fatty acid found in the Roselle seed oil. These finding shows that Roselle seed oil possesses a huge industrial novel application.

Key words: Roselle seed oil, Ostwald-de Waele, Dilatancy

INTRODUCTION

Roselle (*Hibiscus sabdariffa* L.) is a plant found predominantly in the tropic belonging to the family malvaceae widely cultivated for its jute like fibre in India and the red calyces used for local beverage globally. Roselle calyces and petals of the flower are extensively used to improve herbal drinks, cold and warm beverages, as well as making jams and jellies^{14,20}. The Roselle drink has been shown to be a good source of ascorbic acid (Vitamin C)¹¹ while the seeds of this crops from which edible oil could be extracted are being wasted in its production areas after the farmers might

have taken the quantity needed for the next planting season for calyces production^{3,7}. In some parts of Africa the seeds are reported to be used for its oil. The seeds are reported to be rich in proteins, dietary fiber, carbohydrate and oil^{2,6,9,14}. It contain substantial amount of oil that resembles that of cotton seed¹⁰ and a rich source of carotenoids¹³. A lot of preliminary investigations have been conducted on the physical and nutritional qualities of different parts of the Roselle plant; however, the nature and behavior of the oil have not been studied extensively.

Cite this article: Karma, B.R., Chavan, U.D., Nimbalkar, C.A. and Kahar, S.P., Rheometry of Roselle (*Hibiscus sabdariffa* L.) Seed Oil, *Int. J. Pure App. Biosci.* 5(2): 987-993 (2017). doi: <http://dx.doi.org/10.18782/2320-7051.2846>

The nature of food raw materials is a critical constituent that affects its handling the choice of processing techniques to be adopted and its effects as additives in the conversion to finish product. Viscosity is one of such attributes that defines the material science of food. Rheology is the study of the flow of matter primarily in a liquid state but also as soft solids¹⁷. Different food raw materials exhibit unique rheological properties depending on their natural consistency and thus affecting its functionality in food product development, food product quality and shelf life.

The unique behavior of food materials can be simulated using the *power law* which expressed a functional relationship between two quantities on which the study of viscosity hinges on; a study on the rate of change of shear stress (units in Pascal) on shear rate (units per seconds). One of such power law model is the *Ostwald-de Waele* relationship. The power law fluids can be subdivided into three different types based on the value of their flow behavior index (n): Pseudoplastic (n<1) a shear thinning fluid, Newtonian fluid (n=1) and Dilatancy (n>1) a shear thickening fluid, less common¹⁸.

An exhaustive literature search suggest little has been done to study the rheology of Roselle seeds oil, thus the objective of this study was to evaluate the numerical rheological behavior of Roselle seed oil in relation to its flow consistency index (K) and flow behavior index (n).

MATERIAL AND METHODS

Materials

Seeds of Roselle (*Hibiscus sabdariffa L*) were obtained from the vegetable markets in Ahmednagar District, Maharashtra State, India.

Sample preparation and Equipment

Roselle seeds were cleaned by washing to separate poor quality seeds, adhering dust particles, stones, plant debris and were dried carefully at ambient conditions to preserve its nutritive value, packed in a high density polyethylene bag and stored in a cool dry place

until used. The laboratory scale hammer mill used in milling the seed samples and the Rheometer used for the study the viscosity behavior of the Roselle seed oil extract were available in the Department of Food Science and Technology and the instrumentation laboratory in Dr. Annasaheb Shinde College of Agricultural Engineering Department of Agricultural Process Engineering all in Mahatma Phule Agricultural University, Rahuri, Maharashtra State, India.

Methods

Oil Extraction

A modified cold extraction method was adopted. 200g of the cleaned Roselle seeds flour was transferred into a 500 mL Erlenmeyer beaker to which petroleum ether was poured in up to the 400 mL mark and swirled using a glass rod to mix, the beaker was tightly sealed using a polythene film to minimize loss of ether. The set up was kept overnight in a cool dry place. The ether-oil supernatant extract was then carefully decanted and the ether was recovered using a soxhlet set up at 20 °C) to obtain the clear oil extract.

Rheometry Determination

Viscosity of Roselle seed oil was estimated using a rheometer (Brookfield, Mode: R/S SST coaxial rheometer) mounted with CC3-40 spindle and coaxial cylinder with a separation of 1mm at shear rate 10 to 1000 sec⁻¹. The results obtained from the studies were modeled with SAS¹⁵ (2002-2010) software based on Ostwald-de Waele Power law (Eq. 1).

$$\eta = K(\dot{\gamma})^{(n-1)}$$

Fatty acid profile analysis

Fatty acid profile analysis of Roselle seeds oil extracts were analyzed by gas-liquid chromatography using flame ionization detector. The GC was outsourced.

Statistical Analysis

Statistical analysis were conducted using excel 2010 software¹², and modeled with SAS¹⁵ software with significance level taken at p<0.05.

RESULTS AND DISCUSSION

The data obtained from rheometry studies was modeled with SAS¹⁵ software based on Ostwald-de Waele Power law (Eq. 1) to ascertain the flow behavior index (n) and flow consistency index (K) of the Roselle seed oil. The equation represents an apparent or effective viscosity as a function of shear rate (S.I unit Pa.s). Where η is the viscosity, K is a material based and $\delta u / \delta y$ is the applied shear rate⁵. The law was adopted owing to its simplicity for describing the behavior of the Roselle oil across a range of shear rates to which the coefficients were fitted. However, there are a number of models that better describes the entire flow behavior of shear dependent fluids, but they do so at the expense of simplicity. The result obtained described the Roselle seed oil behavior, permit mathematical predictions and correlate the experimental data. The values for K and n were estimated as 0.029 and 2.068 respectively suggesting a dilatants behavior as the n-value (flow behavior index) is greater than one. Dilatancy is rarely encountered behavior¹⁸ making Roselle seed oil a potential raw material for novel industrial application. This behavior could possibly have occurred because the Roselle seed oil had crystallize under stress and behaved more like a solid than a solution like a colloidal suspension transition from a stable state to a state of flocculation⁵. This was evident from the results (Table 1) where viscosity increased and fluidity decreased respectively with increased shear rate.

The trend function in excel 2010 was also used to confirm the relationship between the shear stress (Pa) and shear rate (per seconds) outcomes of the rheological study on

Roselle seed oil (Table 1). Trend is a powerful statistical tool in excel used to calculate the relationship between two variables and defines if they are linear or non-linear²¹. It is called a multiple cell ray function which returns a number in a linear trend; it returns all predicted value of the dependent variable (Shear stress) and states how far they are predicted off. The difference between observed and predicted values gives the residual values. The plot of the standardized residual values against shear rates (per second) proved as shown in a defined parabolic graph (Figure 1) confirmed a non-linear relationship between Shear stress (Pa) and shear rate (per second). The direct plot of shear stress versus shear rate gave a polynomial relationship in a fourth order with coefficient of correlation ($R^2=0.999$; Figure 2). There was a significant difference ($P<0.05$) in the treatments showing an increasing trend in viscosity with increasing shear rate (per second) (Figure 3).

The fatty acid profile of oil extracts of Roselle seed is given in Table 2, which showed substantial and significant ($p<0.05$) lipid contents. The ratio of saturated to unsaturated fatty acids in whole Roselle seed was approximately 1:3. The major fatty acids found for raw seed oil were linoleic acid (41.18) followed by oleic acid (31.22%), palmitic acid (21.45) and stearic acid (3.94%). The results obtained in these research findings are in close harmony with El-Adawy and Khalil⁶; Emmy *et al*⁷., and Cissouma *et al*⁴. The Roselle seed oil can be classified as belonging to linoleic-oleic category as the most abundant unsaturated fatty acids present, suggesting it beneficial use in lowering blood pressure and serum cholesterol^{4,8,16,19}.

Table 1: Effect of shear rate (sec⁻¹) and time (sec.) on the viscosity of Roselle seed oil

| Time(sec) | Shear rate (sec ⁻¹) | Shear stress (Pa) | TREND | Residual value(Pa) | Standardized Residual value (Pa) | Apparent Viscosity (mPa.s) | Fluidity (mPa.s) ⁻¹ |
|-----------|---------------------------------|-------------------|--------|--------------------|----------------------------------|----------------------------|--------------------------------|
| 2 | 10.010 | 0.000 | -0.516 | 0.516 | 3.209 | 0.000 | |
| 6 | 36.910 | 0.666 | 0.686 | -0.021 | -0.128 | 18.030 | 0.0555 |
| 10 | 63.800 | 1.838 | 1.887 | -0.049 | -0.307 | 28.809 | 0.0347 |
| 14 | 90.700 | 2.975 | 3.089 | -0.114 | -0.710 | 32.800 | 0.0305 |
| 18 | 117.600 | 4.138 | 4.291 | -0.153 | -0.954 | 35.183 | 0.0284 |
| 22 | 144.500 | 5.358 | 5.492 | -0.134 | -0.836 | 37.080 | 0.0270 |
| 26 | 171.400 | 6.584 | 6.694 | -0.110 | -0.685 | 38.413 | 0.0260 |
| 30 | 198.300 | 7.791 | 7.896 | -0.105 | -0.655 | 39.286 | 0.0255 |
| 34 | 225.200 | 9.015 | 9.097 | -0.082 | -0.513 | 40.031 | 0.0250 |
| 38 | 252.100 | 10.245 | 10.299 | -0.054 | -0.337 | 40.639 | 0.0246 |
| 42 | 279.000 | 11.470 | 11.501 | -0.031 | -0.192 | 41.111 | 0.0243 |
| 46 | 305.900 | 12.720 | 12.702 | 0.018 | 0.109 | 41.582 | 0.0240 |
| 50 | 332.800 | 13.955 | 13.904 | 0.050 | 0.314 | 41.931 | 0.0238 |
| 54 | 359.700 | 15.160 | 15.106 | 0.054 | 0.337 | 42.146 | 0.0237 |
| 58 | 386.600 | 16.395 | 16.307 | 0.088 | 0.545 | 42.408 | 0.0236 |
| 60 | 400.000 | 17.035 | 16.906 | 0.129 | 0.803 | 42.588 | 0.0235 |
| SE(±) | 0.13 | 0.03 | | | | | |
| CD at 5% | 0.38 | 0.08 | | | | | |
| CV (%) | 0.24 | 1.28 | | | | | |

*Each value is an average of three determinations

Table 2: Fatty acid profile of Roselle seed oil

| Fatty Acids (g/100g) | Roselle Seed Oil | |
|--------------------------------------|------------------|-------------------|
| | Raw Seed Oil | Sprouted Seed Oil |
| Lauric acid (C12:0) | 0.01 | 0.01 |
| Myristic acid (C14:0) | 0.20 | 0.20 |
| Palmitic acid (C16:0) | 21.45 | 21.41 |
| Stearic acid (C18:0) | 3.94 | 3.87 |
| Arachidic acid (C20:0) | 0.59 | 0.56 |
| Eicosenoic acid (C20:0) | 0.01 | 0.10 |
| Behenic acid (C22:0) | 0.25 | 0.25 |
| Lignoceric acid (C24:0) | 0.14 | 0.15 |
| Total Saturated Fatty Acid | 26.2 | 26.55 |
| Palmitoleic acid (C16:1) | 0.44 | 0.43 |
| Oleic acid (C18:2n6c) | 31.22 | 31.99 |
| Linoleic acid (C18:2n6c) | 41.18 | 40.07 |
| Gamma linolenic acid (C18:2n6) | 0.01 | 0.01 |
| Alpha linolenic acid (C18:3n3) | 0.27 | 0.52 |
| Cis-11,14-eicosadienoic acid (C20:2) | 0.01 | 0.01 |
| Total Unsaturated Fatty Acid | 73.13 | 73.03 |
| Lipid Dietary Profile Ratio | 1: 2.79 | 1: 2.75 |

*Results of triplicate determinations

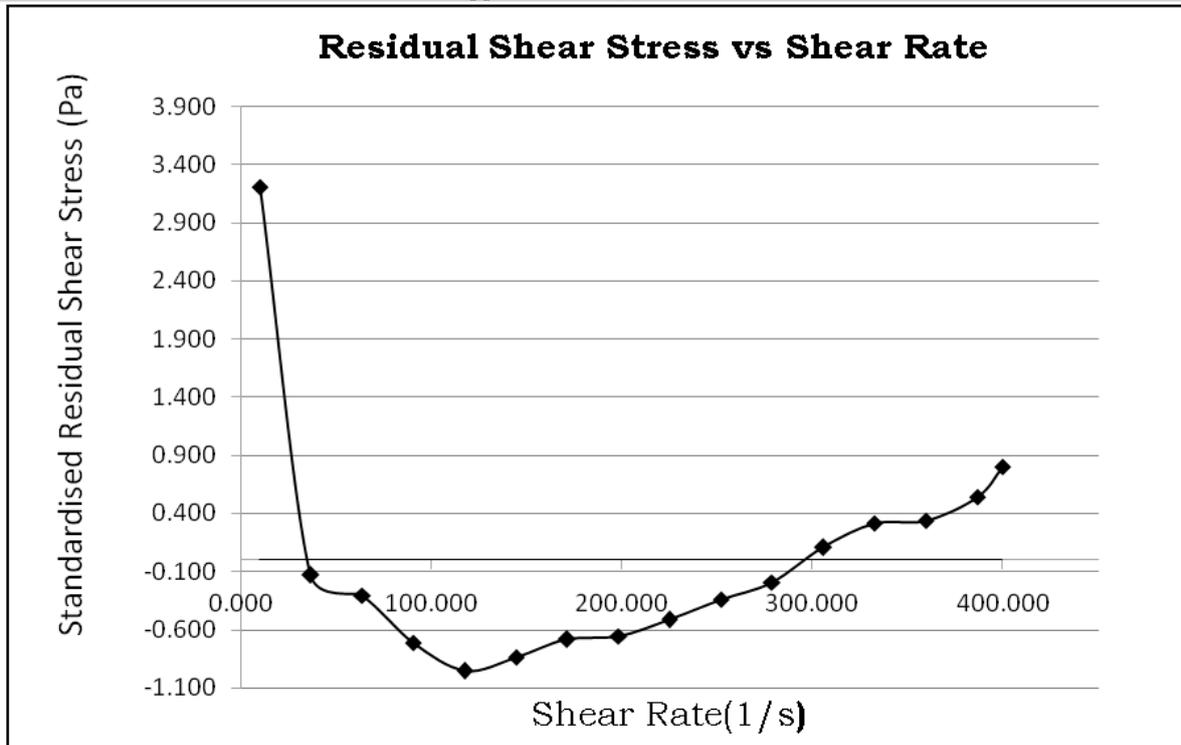


Fig. 1: Residual profile analyses of Roselle seed oil

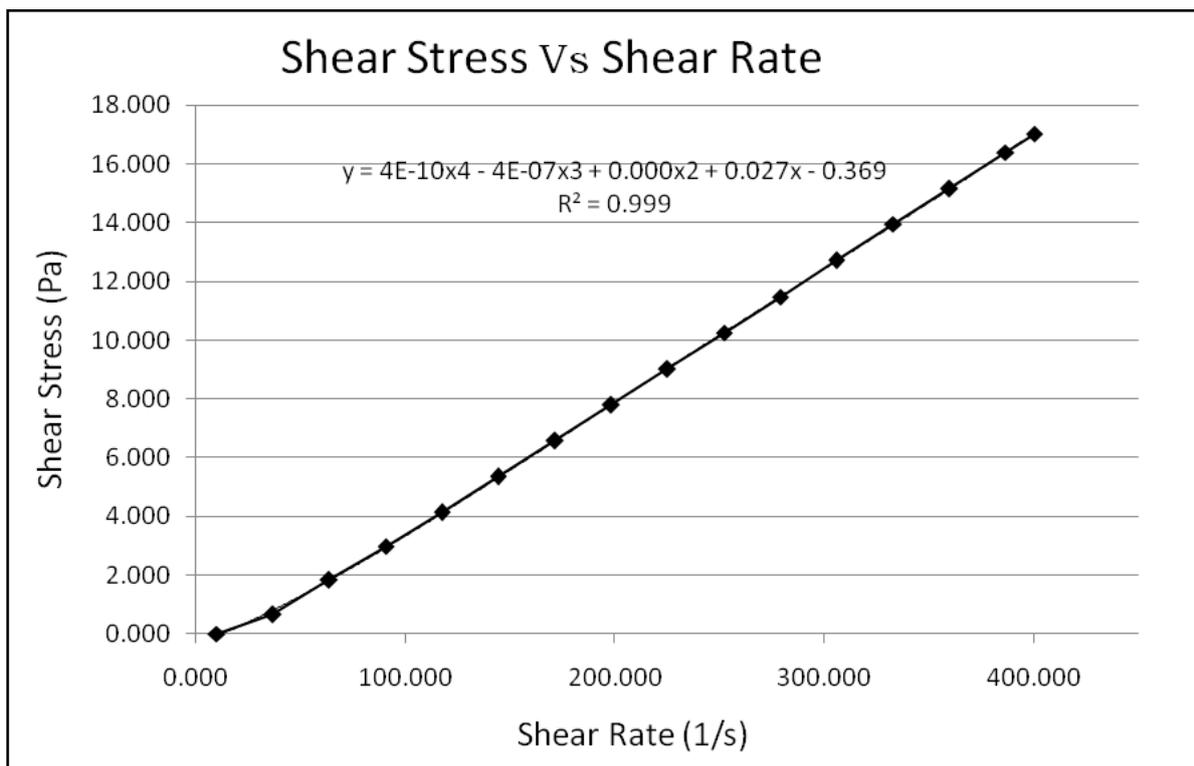


Fig. 2: Viscosity profile of Roselle seed oil

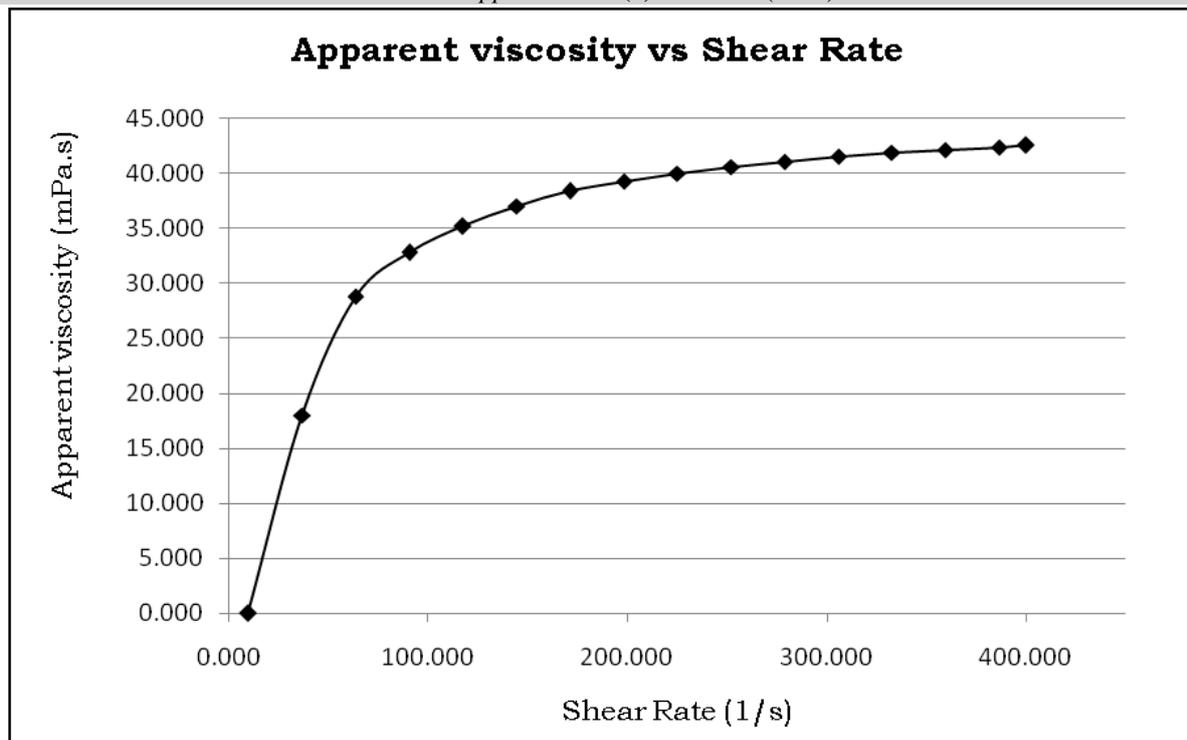


Fig. 3: Apparent viscosity profiles of Roselle seed oil

CONCLUSION

From the outcome of the rheometry studies, Roselle seed oil showed a shear thickening behavior, a case of *dilatancy* as shown by the upward trending curve a rare behavior among food ingredients. The fatty acid composition of Roselle seed oil showed very good essential oil profile. These findings suggest that Roselle seed oil could have a huge industrial application with enormous potential as a natural thickener in food processing, in the formulation of new products and as a lubricant also in medicine as a potential source of possible therapy for deficiency treatments of synovial fluids defects.

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