The Effect of Temperature Modulation on the Viscoelastic Properties of Jujube (*Ziziphusjujuba* L.) Puree

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RESEARCH

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ABSTRACT

The impact of temperature on the functionality of fruits purees is critical. Hence the objective of this research is to investigate the effect of temperature on the rheological properties of three Li, SW, and TT of Jujube varieties. The raw Jujubes fruits were peeled, pureed, and pasteurized. The samples were subjected to rheological tests using a controlled shear stress-strain rheometer at 5, 25, and 50°C. The results revealed that the Jujube purees exhibit an elastic behavior with G' > G". Except for the Li as the frequency increases beyond 1 Hz the purees losses its structural integrity, hence transformed into a viscous fluid as shown by the crossover point at 10 Hz for SW and 4 Hz for TT. The crossover occurs when the full elastic structure is broken and the puree becomes more viscous in the region where G” > G’. The modulus of each variety significantly ($p < 0.05$) varied at different temperatures. The steady shear viscometry (0.1 to 1000 s-1), generally showed a more pronounced pseudo-plastic ($n < 1$) behavior and the presence of yield stresses. Herschel-Bulkley model was found to fit adequately (R2 ≥ 0.90) over the entire shear rate range.

Key words: Jujube-purees, elastic modulus, viscous modulus, steady shear, and flow behavior index.

Abbreviations

f	-	Frequency
η*	-	Complex viscosity
η’	-	Dynamic Viscosity
n	-	Behavioral Index
τ0	-	Yield Stress
τ	-	Shear Stress (Pa)
ω	-	Angular Frequency
γ	-	Shear rate (1/s)
Ca	-	Calcium
Cu	-	Cupper
DHR	-	Discovery Hybrid Rheometer
Fe	-	Iron
G'	-	Storage Modulus
G"	-	Loss Modulus
GI	-	Gastrointestinal Tract
HB	-	Herschel-Bulkey
Li	-	Li
LNP	-	Loaded Nanoparticles
K	-	Consistency Index
Ln µ	-	Natural Log of viscosity
LVR	-	Linear Viscoelastic Range
Mg	-	Magnesium

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NIFA - National Institute of Food and Agriculture

SW - Shuttle Worth-Wallace

Tan δ - Phase Angle

TT - Tigertooth

USDA - United States Department of Agriculture

INTRODUCTION

Jujube (Ziziphusjujuba L.) is a deciduous tree with thorny branches and consists of over 45 genera and 550 species and it belongs to the Rhamnaceae family [1]. Jujube is known as one of the most important fruits grown in the tropical and subtropical climates, it is popular with consumers because of the associated nutritional and health benefits. They are rich sources of vitamins (A, C, B-complex, etc.) and minerals (Ca, Cu, Fe, Mg, etc) [2, 3, 4]. Amongst the vast varieties of jujube fruits, the Li, Tigertooth, and Shuttle Worth-Wallace varieties are the most common.

Traditionally, Jujube fruits are mostly consumed fresh, hence postharvest storage possesses significant challenges to extend availability beyond the harvest season. Hence, value-added processing into a puree will minimize the postharvest losses and add the economic viability of the fruit into its application as a food ingredient. Therefore, understanding the physiochemical and structural attributes of jujube puree is important to enhance processing and the design of processing and handling equipment [5]. Also, the structural organization of food fruit puree has a significant effect on thermal processing, hence the flow characteristics during heat and mass transfer will significantly influence its quality [6].

Furthermore, blending, pumping, constant agitation, heating, and cooling during the transport of the purees in a production line is a common unit process. Thus, continuous shearing and structural transformation are endued during this stage of the process, hence with time, it might ultimately impact the sensory quality [5]. The extent of the structural transformation, either reversible or irreversible is vital to the development of fruit purees, and based on literature search such information is not available on Jujube. The information on the physical and rheological properties of jujube puree will provide insights into the structural and rheological behavior. Perona [7] reported the structural breakdown of apple purees during flow pattern changes from laminar to turbulent while flowing in pipes. Saenz & Costell [8] reported the composition and the added ingredients in the food product significantly influence the functionality of the end product. In the serum or continuous medium of a typical plant, food dispersion contains sugar, organic acids, salts, and pectic substances. This composition varies depending on the environmental factors such as a particular commodity, the cultivar, and the extent of ripening. The differences in the composition of plant food dispersions can lead to significant interparticular forces compared to viscous forces and thus some of these physical phenomena are described by their viscoelastic and rheological behaviors [9]. Sharma, Kristo, Corredig, & Duizer [10] observed an elastic nature of the carrot puree when treated with hydrocolloids.

In a complex food system particles are usually exposed and subjected to intermolecular attractive forces (e.g. Hydrogen bonding) [11, 12] and the strength of these forces influence the degree of energy loss compared to the energy gained in a cyclic deformation describe by physical parameters such as the tan delta (tan δ). Tan $\delta < 1$ suggests a high intra-particle interactive force, hence materials behave more like an elastic, and otherwise the materials trends towards a viscous fluid ("Understanding Rheology of Structured Fluids, n.d.). Hass Avocados exhibit elastic behavior during ripening under refrigerated conditions hence Ortiz-Viedma et al. [13] represent the rheological behavior with tan δ < l. Most fruit juices and purees exhibit a non-Newtonian pseudoplastic/shear-thinning behavior [12, 14, 15, 16, 17] hence characterized based on flow behavioral index (n) [18]. Therefore, the objective of this study was to determine the rheological and structural properties of Jujube purees by oscillatory and steady-shear measurements as a function of temperature.

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MATERIALS AND METHODS

2.1. Sample preparation

The three varieties of the jujube fruits Li (Li), Shuttle Worth-Wallace (SW), and Tigertooth (TT) were collected from the Winfred Thomas Agricultural Research Station, Alabama A & M University, Normal, Alabama. The fruits collected were washed properly with portable water and dried, and the seeds were subsequently removed with a sharp knife and them crushed in a mechanical blender (Hamilton Beach 10 speed blender, Model: 50123, Wisconsin, USA) until a homogenous smooth puree was obtained. The purees were filled in a glass jar and were subsequently pasteurized using a water bath (Model: 2872, ThermoScientific, Norcross, GA, USA) at 90°C for 60 min. After pasteurization, the sealed samples and cooled rapidly to 4°C, and then stored at room temperatures (25±5 °C) till needed.

2.2 Rheological measurements

2.2.1. Steady Shear Test

The steady shear flow measurement was conducted using the Hybrid Rheometer (DHR-2, TA Instruments Inc, New Castle, DE) and it was programmed to equilibrate at a predetermined temperature for 1 min, followed by a shear ram ranging from 0.1 to 1000 s -1. Rheological parameters (shear stress vs. shear rate) were obtained via the software (TA-Advantage). Various rheological flow models (Newtonian, Bingham, Casson, power low, and Herschel Bulkley) were tested and the best fit model was selected based on the R2 values.

2.2.2. Viscoelastic Frequency Sweep Test

Rheological measurements were carried out using a Hybrid Rheometer (DHR-2, TA Instruments Inc, New Castle, DE) controlled with commercial computer software (TA-Advantage Data Analysis Software v.4.1.2, TA Instruments Inc, New Castle, DE). The experiments were carried out using a 40 mm cone (2°) and plate geometry with a 1000 µm gap. The temperature of the DHR-2 system was controlled with a Peltier plate temperature control system. Approximately, 2 mL of test samples were placed on the bottom plate of the rheometer. The stress sweep was conducted at a frequency of 1 Hz to determine the linear viscoelastic range (LVR) under oscillatory shear conditions. The frequency sweep (0.01 to 10 Hz) was conducted in a linear regime at constant stress at different temperatures (5, 25, and 50°C). New samples were reloaded for each measurement run.

2.2.3. Statistical Analysis

All statistical analyses were performed using IBM SPSS package 20 (IBM, New York, U.S.). Differences between variables were tested for significance using an analysis of variance (ANOVA) based on 5% level significance, hence Duncan's multiple range test was used to express the variability of the variable that was significant.

RESULTS AND DISCUSSION

3.1. Flow Properties of Jujube Puree

The effect of temperature on the flow properties of three varieties (Li (Li), Shuttle Worth-Wallace (SW), and Tigertooth (TT)) Jujube was tested at 5, 25, and 50°C. Hence, the flow characteristics are shown by the viscosity profile in Figure 1 indicate it is a non-Newtonian, shear thinning, and pseudoplastic biomaterial. Rao [19] reported fruit purees to be non-Newtonian fluids with yield stress. However, the properties of plant-based dispersions in foods influence the flow and viscoelastic behavior of purees [17]. Hence, the associative pectin content may have contributed to the non-Newtonian characteristics. The decrease in viscosity could be attributed to the reconstitution and restructuring of the pectin as the shear rate increase during shearing of the Jujube puree. The highest apparent viscosity index across the different shear rate ranges from $2 < \gamma < 150/s$ was observed with the Li variety. Although the temperature was observed to significantly ($p < 0.05$) affect the viscosity of the different varieties of Jujube puree when shear rate ($2 < \gamma < 70/s$) is applied, hence beyond that shear rate no significant ($p > 0.05$) variation of viscosity was observed.

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Figures 2abc shows the relationship profile when shear stress is applied at varying temperatures (5, 25, and 50°C) and the corresponding shear rates on the Jujube purees. Amongst the models tested, the Herschel-Bulkey (HB) model shows the best fit based on the R^2-values. The HB model is shown by Eq. 1:

$$\tau = \tau_0 + k\gamma^n \qquad (1)$$

where τ is the shear stress (Pa), τ0 is the yield stress (Pa), γ is the shear rate (1/s), K is the consistency coefficient (Pasn), and n is the dimensionless flow behavior index.

These parameters were used to describe the relationship between shear stress and shear rate. The n and K-values were estimated using the HB model and a good fit was obtained from the experimental data for all the Jujube purees (Table 1). By close inspection of Figure 2, the non-Newtonian characteristic of all the varieties of Jujube was exhibited within the shear rate $2 < \gamma < 150$/s, beyond which the purees structural integrity is compromised, hence become Newtonian. Lukhmana et al. [17] observed similar behavior on tart cherry puree, hence they indicated that shear thinning behavior cannot be unambiguously predicted above the shear rate of 100/s. The hydrodynamic structural modifications may cause particle dispersion and a cluster of aggregations in the food system, which offers high resistance during shearing. Hence, the flow behavior cannot be described by conducting viscosity measurement at a constant shear rate. Lukhmana et al. [17] reasoned that the high rate of particle dissociation compared to the rate of their association.

The variation of the flow behavior index over wide ranges of shear stress vs shear rate was analyzed for the Jujube purees at different temperatures shown in Table 1. The n-values << 1 (0.002 to 0.00008) for LL variety while the SW and TT were about 0.3 to 0.069 and 0.107 to 0.2, varying according to the temperature increase from 5 to 50°C, respectively, (Table 1). All the Jujube purees samples exhibit shear thinning behavior with $n < 1$. Rielly [20], reported 0.28 and 0.45 for Banana puree and Apple sauce, respectively. These are in agreement with what Lee and Yoo [21] reported on rice and Zhou et al. [22] on sweet potatoes. Maceiras, Alvarez, & Cancela [23] corroborated the results in berry fruit products during cooking. However, in contrast, Ditchfield, Tadini, Singh, & Toledo [24] reported increased flow behavioral index in the banana puree as temperature increases.

The k-value shown in Table 1 reflects the values of the viscosity of the purees for both varieties (Li and SW) decreased systematically with increasing temperatures (804 to 221 Pasn and 26.98 Pasn to 211.37 Pasn). This could be attributed to the beginning of the gelation of the carbohydrate and the pectin branches at elevated temperatures. Similar trends were reported in the study of banana puree [25, 26]. However, the contrary was observed in the TT variety (Table 1) which could be attributed to the increased intermolecular distances due to the presence and activities of the solid intraparticle mobility of the purees at elevated temperature, thus results to the ultimate reduction of viscosity [27]. Hence, Diamante & Umemoto [26], Krokida, Maroulis, & Saravacos [28] also reported the effect of temperature on the consistency of non-Newtonian semi-liquid foods.

Another important rheological parameter is yield stress, HB-mathematical model was used to estimate the behavioral characteristics for the different varieties if Jujube at different temperatures (Table 1). The nature and formation of shapes and particle sizes of the purees may create a divergence of the yield stress, hence the experimental shear stress vs shear rate data applied on the mathematical model may contribute to these variations [18, 29]. High yield stress could be attributed to the polymeric dispersion of the composite particles as a result of intermolecular hydrogen bonding and molecular entanglement. Ma and Barbosa-Canovas [30] reported the yield stress increase from 23 to 25 Pa for mayonnaise with the addition of oil and xanthan gum. The yields stress for the SW and TT Jujube puree were 62.7 and -249 Pa when conditioned at 5°C, respectively, however, in contrast, the LL-variety shows a much higher value of stress (802 Pa). The

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wide differences are probably because of the shear rate ranges used in the model. The high yield stress may be attributed to the polymeric dispersion of the composite particles because of the intermolecular hydrogen bonding and molecular entanglement.

The complex viscosity depicts the total resistance to flow as a function of angular frequency. Figure 3 shows no significant differences between the complex viscosity and dynamic viscosity between the angular frequency range ($0.1 < \omega < 0.8$) for the Li variety at 5°C. At constant temperature, a significant difference ($p < 0.05$) was observed on the complex viscosity of the different Jujube varieties, a linear relationship also exists between the complex viscosity and angular frequency as shown in Fig. 3. Ahmed et al. [31] reported increase complex viscosity with increased frequency after heat-treating insoluble date fiber in wheat flour dough.

The frequency-dependent profile of the complex viscosity of the Jujube purees is illustrated in Fig. 4abc. Both varieties of Jujube purees exhibited shear thinning behavior as discussed above. This was expected since the complex viscosity is the sum of $(G'^2 + G''^2)/\omega$. Figure 4a is the complex viscosity profile of the Li variety at different temperatures. Although, the decreasing effect of temperature is significant ($p < 0.05$) on the complex viscosity across the different ranges of angular frequency, it is an affirmation of shear thinning behavior. This could be attributed to the change in temperature on the hydrodynamic effect of the cell structure and the intrinsic composition and the melting properties of the Jujube purees. The systematic heating of the amylose starch might have influenced the degree of starch retrogradation, furthermore, the weakness of the amylose network formation due to phase separation may the cause of the decrease in the complex viscosity as a result of gel melt in the Jujube purees. The SW and TT varieties follow the same trend (Fig. 4bc). Mohamed at al. [32] reported decreasing complex viscosity of the addition of date syrup on sweet potatoes with increase angular frequency.

3.2. Viscoelastic Properties of Jujube Puree

The effect of temperatures (5, 25, and 50°C) on the viscoelastic properties of the three varieties (Li, SW, and TT) of jujube purees are shown in Fig. 5abc. The relative moduli values of the storage modulus (G') is greater than the loss modulus (G") obtained from the oscillatory test, information is vital to determine the degree of elastic or viscous behavior of the sample. The results show that G' > G" for the three varieties Li, SW, and TT at the three temperatures over the frequency period $0.01 < f < 1$Hz, hence an elastic behavior prevails, a similar trend was observed with Jaboticaba pulp [33]. Except for the Li (Fig. 5a) as the frequency increases beyond 1 Hz the purees losses its structural integrity, hence transformed into a viscous fluid as shown by the crossover point at 10 Hz for SW (Fig. 5b) and 4 Hz for TT (Fig. 5c). This structural transformation is an indication of the varietal difference between the Jujube fruits, based on their proximate composition. The crossover occurs when the full elastic structure is broken and the puree becomes more viscous in the region where G" > G' hence the viscous behavior becomes dominant. Furthermore, the depolymerization of the pectin, hemicellulose, and cellulose may have contributed to the change in elastic behavior [34]. The degree of postharvest softening could play a significant difference between the varieties used in this study. The viscoelastic properties such as the G' and G" obtained at a lower temperature (5°C) are much higher compared to those at 25 and 50°C, which is due to the stronger inter-particular interactions at low temperature. This behavior is an indication of the functionality of Jujube puree as a viscoelastic polymer.

The viscoelastic nature of polymer can further be characterized by the slopes of the linear regression data of ln (G' or G") vs ln (f) [31, 6, 35]. The slope index zero is indicative of a true gel, Ahmed et al. [31] reported a slope range from 0.32 to 0.14, hence indication the increasing gelling potential of flour dough with the addition of date fiber. In this study, the slopes obtained from the linear regression of the ln (G') and ln (G") vs ln (f) ranged from 0.102-0.204 and 0.077-0.231, respectively, (Table 2). The

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effect of temperature on the slope index is shown in Table 2 for the different Jujube varieties. Then, a decrease in the slope index is indicative of the decrease in the gelling properties hence a shear thinning tendency with increased temperature, this was also corroborated by Ahmed and Ramashamy [6] in sweet potatoes. The slope index was significant ($p < 0.05$) at different temperatures for each variety (Table 2). A yield stress value was computed but not shown in Table 2, showed that the purees can be classified as yield stress fluid.

The tendency of viscoelastic behavior with increasing frequency can be examined in more detail by considering the frequency dependence of the phase lag [18]. The phase lag (tan δ) may be calculated based on the relationship between the G′ and G″ (δ = arctan (G″/G′). A high value of tan δ show fluid tends to be more fluid-like behavior. Hence, in this study the tan δ values ranges as follows: 0.14 < tan δ < 0.24, 0.16 < tan δ < 0.33 and 0.19 < tan δ < 0.43 for the Li, SW, and TT jujube purees, respectively, hence characteristics nature supports viscoelastic behavior. The tan δ values result reported by Ahmed & Ramashwamy [6] ranges from 0.11 < tan δ < 0.285 for sweet potato puree, this corroborated the value reported in this study. The crossover frequency can be a basis as criteria for product evaluation, it occurs when G′= G″, and the point where the tan δ = π/4.

3.3. Effect of Temperature on the Rheological Properties Jujube Puree

The overall dependency of the viscosity and the consistency coefficient on temperature were derived from the Arrhenius equation. The Arrhenius-type kinetics model was used to estimate the effect on temperatures (5, 25, and 50°C) on the activation energy influence of the flow characteristics of the three Jujube variety (Li, SW, and TT) purees. A linear relationship between the ln μ and 1/T was observed for the Li, SW, and TT Jujube varieties, where the ln μ is the natural log of viscosity and 1/T is the inverse of absolute temperature. The activation energy (112.9, 102.1, and 125 kJ/mol) was computed at a constant shear rate (3/s) for the Li, SW, and TT varieties, respectively. The results show the temperature significantly ($p < 0.05$) influenced viscosity. Yanniotis et al. [36] reported 70.8 and 96.3 kJ/mol for honey, while Ahmed et al. [37] reported 164 – 276 kJ/mol for non-isothermal heating of rice and lentil starch and 10.7 to 21.7 kJ/mol for blueberry puree [5]. The activation energy reported in this study is consistent with the values report for other fruit purees.

CONCLUSIONS

The oscillatory test of Jujube purees (Li, SW, and TT) exhibited a shear thinning pseudoplastic behavior (n << 1) and likewise viscoelastic behavior with G' >> G" at 0.1< f < 1 Hz for the Li variety, while 0.1 < f < 10 Hz for the Sw and TT varieties. The low tan δ << 1 is an indication of a highly structured fluid, hence supports the existence of viscoelastic features. The complex and dynamic viscosities corroborated the results. The shear stress and shear rate of the Jujube purees well fitted the Herschel-Bulkley mathematical model and also provide the estimation of yield stress. The results show the temperature significantly ($p < 0.05$) influenced viscosity. The activation energy (112.9, 102.1, and 125 kJ/mol) was computed at a constant shear rate (3/s) for the Li, SW, and TT varieties, respectively. The information on the rheological properties of Jujube purees will provide vital designed parameters for optimize process design.

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PEER REVIEW

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FIGURES

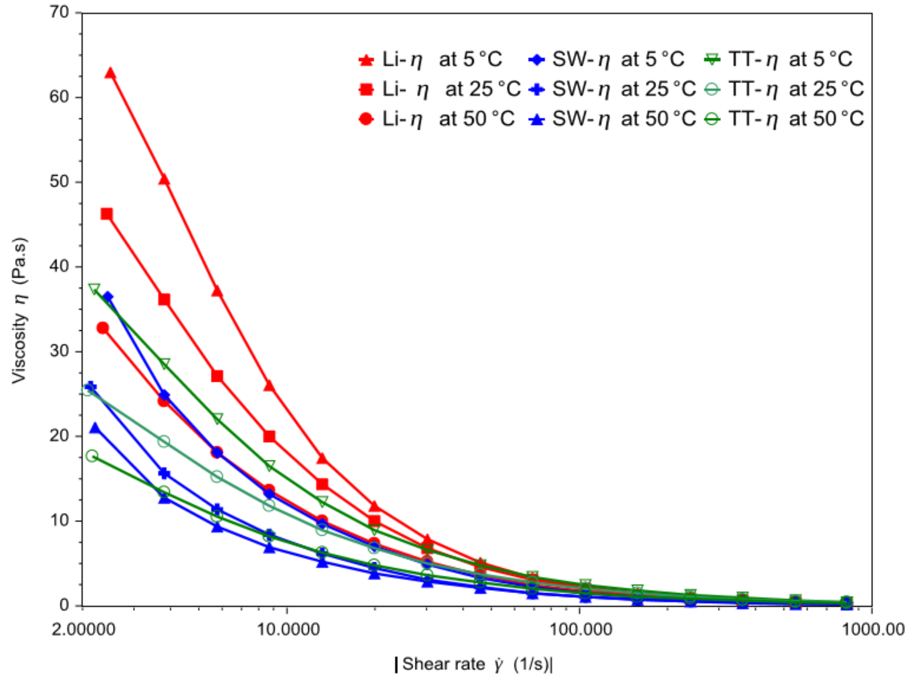
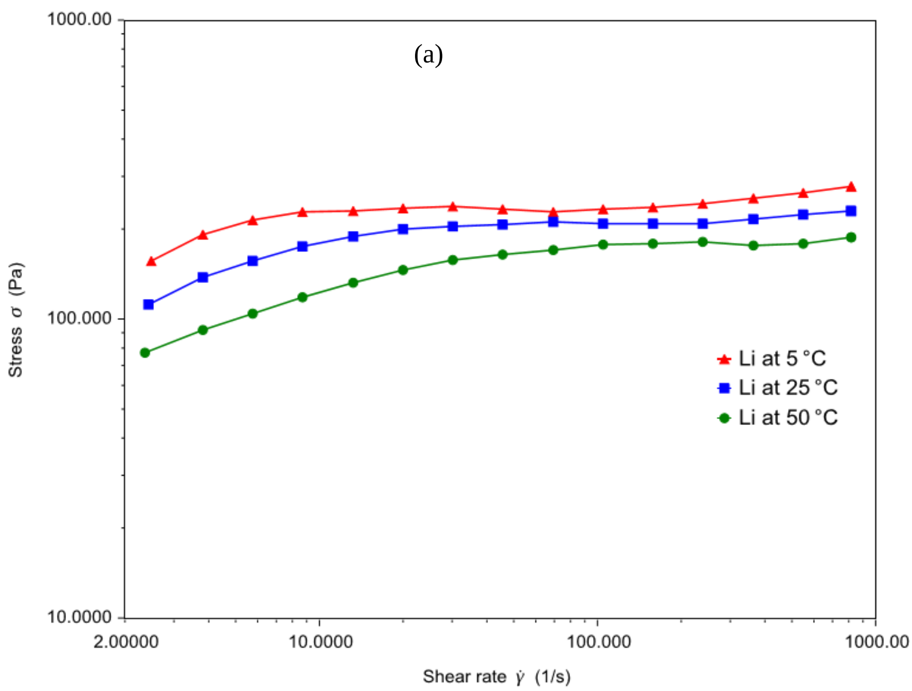
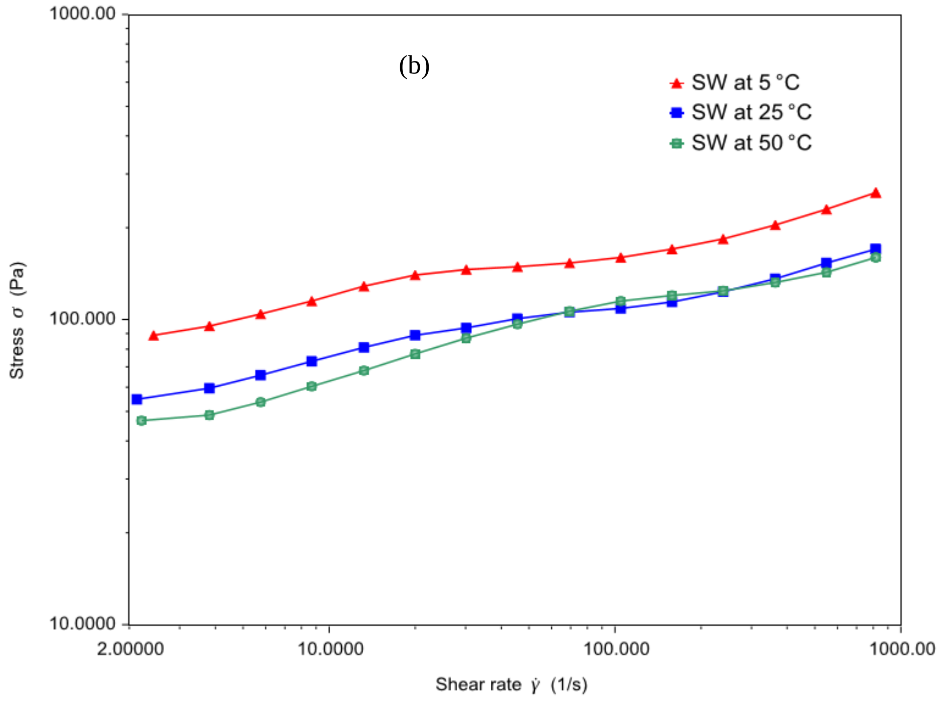


Figure 1. Comparative analysis of the viscosity profile of the Li (Li), Shuttle Worth-Wallace (SW), and Tigertooth (TT) variety Jujube puree over varying shear rate ranges 2 to 1000 /s at different temperatures 5, 25, and 50°C.





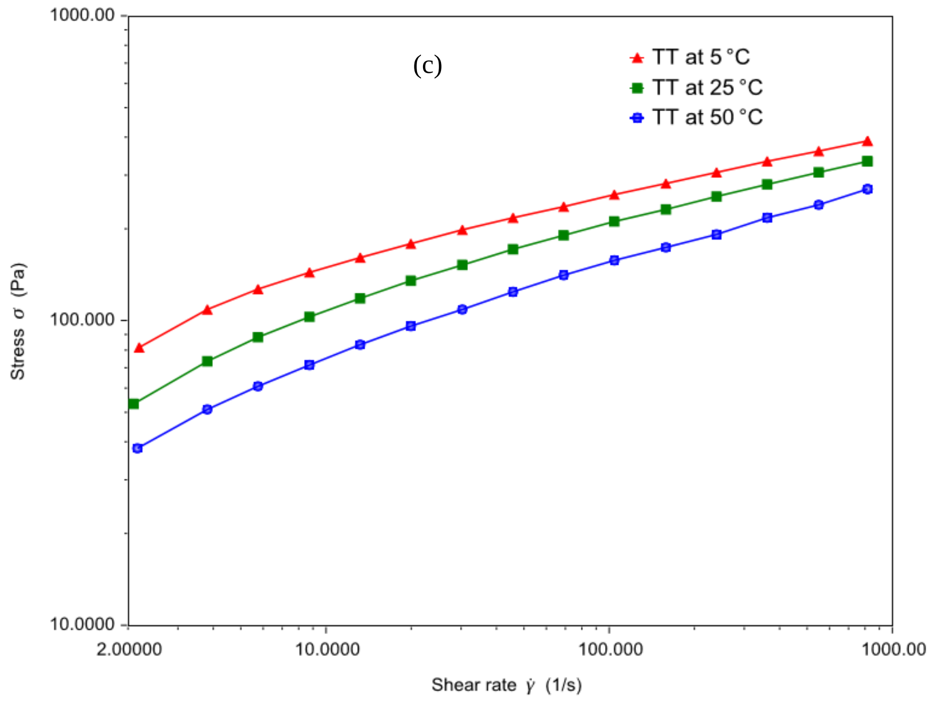


Figure 2. The effect of temperature 5°C (a), 25°C (b), and 50°C (c) on the shear stress profile of the Li (Li), Shuttle Worth-Wallace (SW), and Tigertooth (TT) variety Jujube puree.

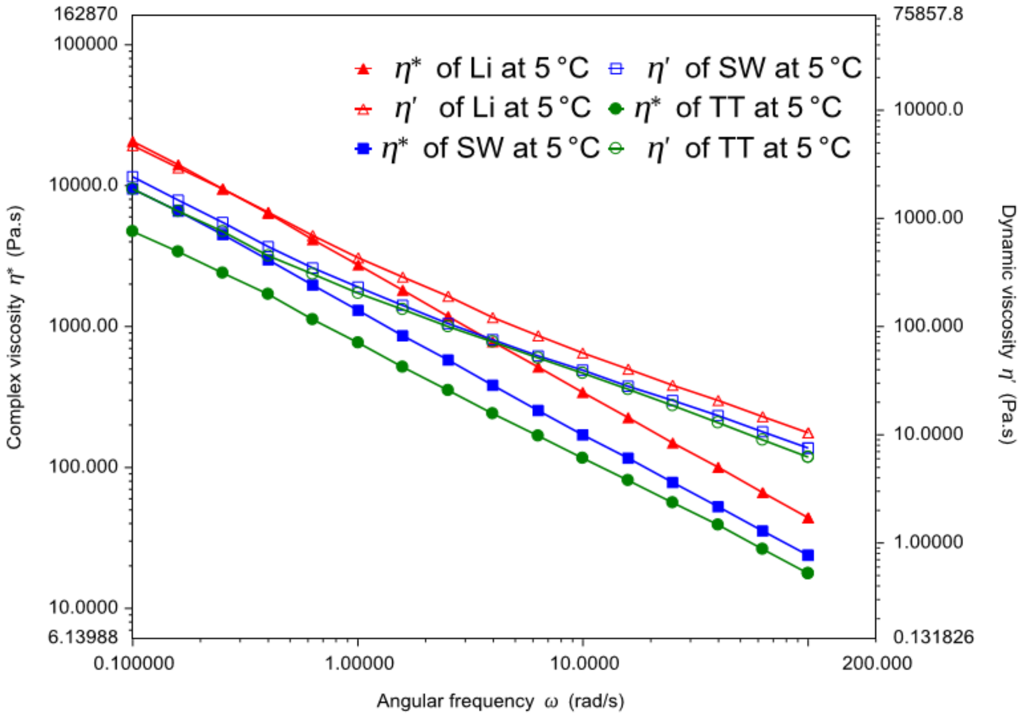
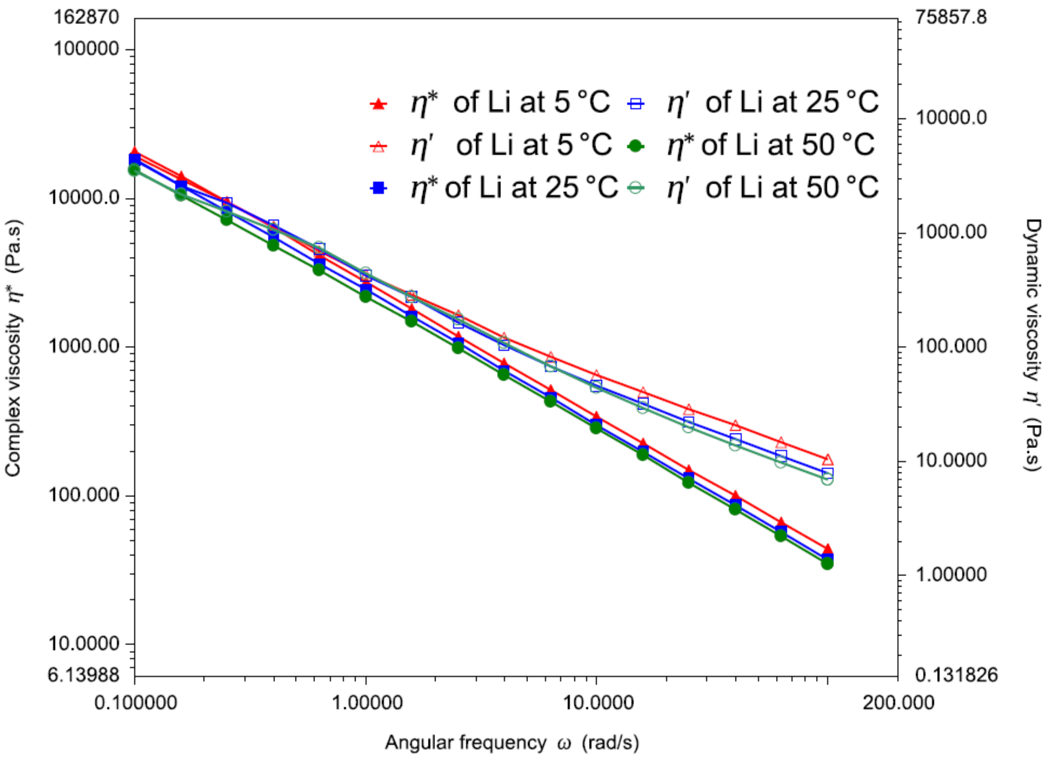
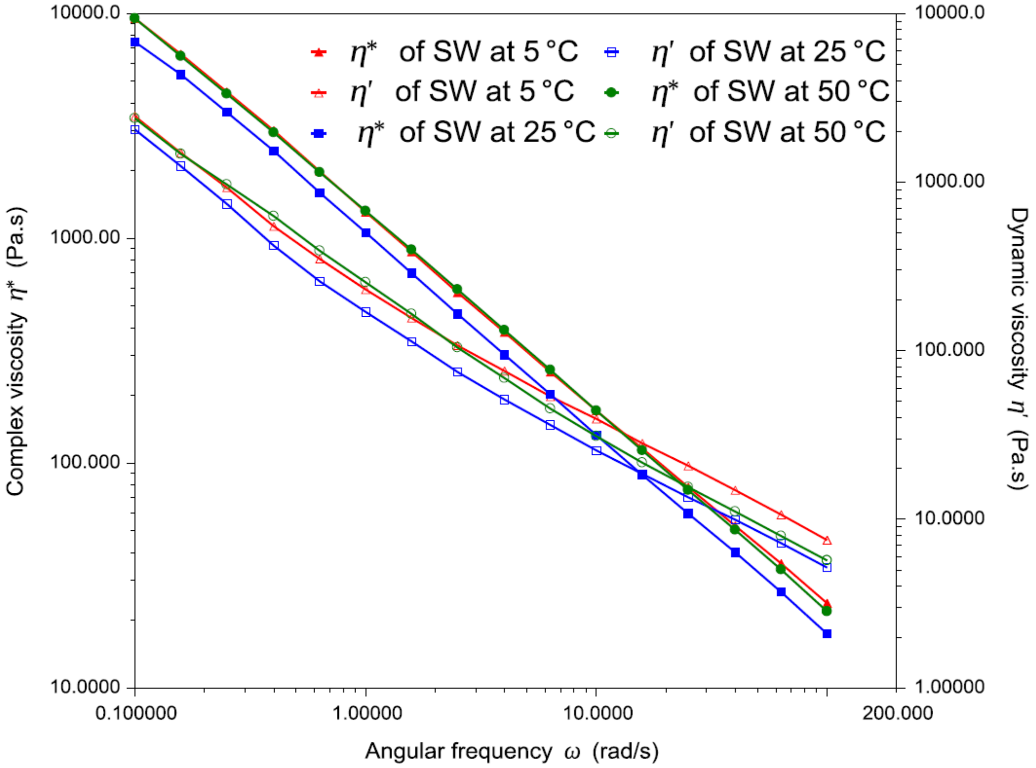


Figure 3. Comparative analysis of the Li (Li), Shuttle Worth-Wallace (SW), and Tigertooth (TT) variety Jujube puree on complex viscosity (η*) and dynamic viscosity (η') at 5°C.

(a)



(b)



(c)

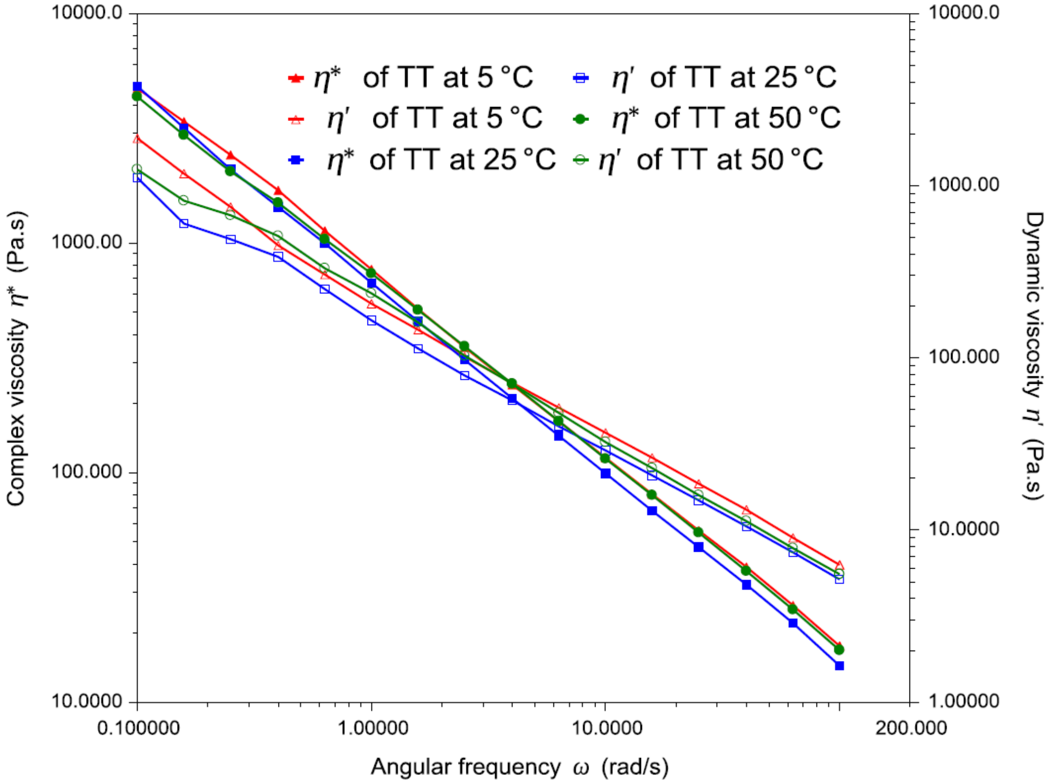


Figure 4. The effect of temperature 5°C (a), 25°C (b), and 50°C (c) on complex viscosity (η*) and dynamic viscosity (η') of the Li (Li), Shuttle Worth-Wallace (SW) and Tigertooth (TT) variety Jujube puree.

TABLES

Table 1. The effect of temperature (5°C, 25°C, and 50°C) on the Harschel Bulkey parameters of the Jujube (Li (Li), Shuttle Worth-Wallace (SW) and Tigertooth (TT)) variety puree.

Temperatures (°C)	Li			SW			TT		
	τ_0 (Pa)	K (Pas^n)	n (-)	τ_0 (Pa)	K (Pas^n)	n (-)	τ_0 (Pa)	K (Pas^n)	n (-)
5	-802	804	.00019	62.71	26.98	0.29	-249.1	309.29	0.11
25	-284	284	.000094	15.76	35.79	0.21	-179.1	211.88	0.13
50	-220.	221	.000083	-182.01	211.37	0.069	-61.14	86.08	0.19

Table 2. The effect of temperature (5°C, 25°C, and 50°C) on slopes of the linear regression of the of the relationship between ln G' versus ln frequency of three Jujube (Li (Li), Shuttle Worth-Wallace (SW) and Tigertooth (TT)) variety purees.

Jujube Variety	**Ln G'**					
	Temperature (5°C)		Temperature (25°C)		Temperature (50°C)	
	Slope	R^2	Slope	R^2	Slope	R^2
Li	0.104 ± 0.001^{a}	0.99	0.102 ± 0.002^{a}	0.98	0.118 ± 0.002^{b}	0.93
SW	0.124 ± 0.003^{a}	0.97	0.112 ± 0.001^{b}	0.99	0.117 ± 0.002^{ab}	0.99
TT	0.187 ± 0.002^{a}	0.99	0.160 ± 0.004^{b}	0.99	0.204 ± 0.004^{c}	0.99
	Ln G”					
Li	0.117 ± 0.004^{a}	0.87	0.077 ± 0.010^{b}	0.83	0.079 ± 0.010^{b}	0.91
SW	0.193 ± 0.014^{a}	0.95	0.154 ± 0.007^{b}	0.89	0.115 ± 0.005^{c}	0.85
TT	0.198 ± 0.002^{a}	0.93	0.231 ± 0.008^{b}	0.85	0.192 ± 0.010^{a}	0.98

Means (n=3) with the same letter in the same row are not significantly different ($p > 0.05$).