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ORIGINAL ARTICLE

Effect of Concentration and Temperature on Flow Properties of *Alyssum homolocarpum* Seed Gum Solutions: Assessment of Time Dependency and Thixotropy

Arash Koocheki · Seyed M. A. Razavi

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Abstract Effects of different shear rates (14, 25, and 50 s^{-1}), gum concentrations (3%, 3.5%, and 4%), and temperatures (5–65 °C) on flow properties of *Alyssum homolocarpum* seed gum solutions were investigated using a rotational viscometer. The experimental data were fitted with three time-dependent rheological models, namely second-order structural kinetic model, Weltman model, and first-order stress decay model with a non-zero stress value. The rate constant and extent of viscosity strongly depended on the shear rate, gum concentration, and temperature. It was found that *A. homolocarpum* seed gum samples exhibited shear thinning and thixotropic behavior for all concentrations and temperatures. The amount of structural breakdown decreased with shear rate, but it did not have a general trend with concentration and temperature. The extent of thixotropy increased with increasing gum concentration and decreased with increasing temperature and shear rate. In this work, the decay rate constant generally increased with increasing shear rate; however, it did not have any trend with concentration and temperature.

Keywords Rheology · *Alyssum homolocarpum* · Time dependency · Thixotropy · Modeling

Introduction

Rheological studies contribute to the knowledge of the molecular structure or distribution of the molecular components of foods, as well as to predict the structural changes of the food during its manufacturing processes. Reliable and accurate rheological characterization of foodstuffs, particularly time-dependent effects, is required for the control of quality, texture, shelf life, and for the design of processing equipment[1]. When a material is sheared at a constant shear rate, the viscosity of a thixotropic material will decrease over a period of time, implying a progressive breakdown of structure[2].

In addition, the characterization of time-dependent rheological properties of food systems is important to establish relationships between structure and flow, and to correlate physical parameters with sensory evaluation[3]. Rheological characterization of flow time-dependent fluids is not easy[4]. That their apparent viscosity does not depend only on shear rate but also on the time shear is applied. In general, two approaches are used to represent the thixotropy of a solution: the transient rheological approach and the hysteresis loop approach. The transient rheological approach can be combined with a structural kinetic model to analyze the thixotropic behavior of a complex solution, where the structure parameter represents the fraction of unbroken network links in a solution[5,6].

Experimentally, the flow time dependence is clearly shown by running a hysteresis cycle. The hysteresis area will give an estimate of the magnitude of the product thixotropy[7–10]. Modeling of the thixotropic behavior of food products has been based on equations, such as the Weltman model[11], stress decay models[12], and structural kinetic models[13].

Hydrocolloids obtained from different sources have been exploited as thickeners, stabilizers, fat replacer, and emulsion stabilizers in food systems[14–16]. Razavi and Karazhiyan[17] studied the flow behavior of Salep tubers and Balangu seed gums in aqueous solutions, and concluded that both hydrocolloids exhibited thixotropic behavior. Zhang et al.[18], Mao and Chen[19], and Huei Chen and Yuu Chen[20] also investigated the thixotropic properties of

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A. Koocheki (✉) · S. M. A. Razavi
Department of Food Science and Technology,
Ferdowsi University of Mashhad (FUM),
PO Box 91775-1163, Mashhad, Iran
e-mail: koocheki@um.ac.ir

hydroxypropyl guar gum, locust bean gum, and green laver mucilage, respectively. Thixotropy of a polymer solution is quantified by the solution's ability to regain its gel structure when the solution is allowed to rest for a longer period of time, which is usually attributed to the breakdown of network links formed by the associations between polymer chains under shear[21].

Alyssum homolocarpum seed is known under the local name of Qodume shirazi in Iran, and it has been used as a traditional medicine for hundreds of years. The seeds are known to produce a large amount of mucilaginous substance when soaked in water[22]. This gum exhibits non-Newtonian, pseudoplastic behavior, and it can be used as a thickening and stabilizing agent in food industries[23,24]. However, there is no information on the time dependency of *A. homolocarpum* seed gum. Thus, the aim of the present work was to characterize the thixotropic behavior of *A. homolocarpum* seed gum and to examine different models for describing its time-dependent rheological behavior under the effect of different temperatures, concentrations, and shearing conditions.

Materials and Methods

Gum Extraction

A. homolocarpum seeds were dispersed in preheated deionized water (Milli-Q, Millipore, Bedford, USA) at a water/seed ratio of 60:1, pH 7, and temperature 55 °C. The slurry was stirred continuously with a mechanical mixing paddle for 1 h. The seeds were discarded, and the supernatant was subjected to ethanol precipitation (97% ethanol/mixture ratio of 3:1). Later, the precipitate was recovered using a sieve to allow the drainage of excess solvent and was dispersed in deionized water. The dispersion was stored overnight at 4 °C with continuous stirring. Ultimately, the dispersion was dried in a conventional oven (overnight at 45 °C), milled, and sieved using a mesh 18 sifter[22].

Sample Preparation

Gum solutions were prepared at concentrations of 3%, 3.5%, and 4% (*w*/*w*) by dispersing the required amount of gum in deionized water (Milli-Q), under slow stirring at room temperature, and stored for 18 h at 4 °C for their complete hydration prior to any assessment.

Rheological Measurement

Measurements were carried out using a rotational viscometer (Bohlin Model Visco 88, Bohlin Instruments, UK) equipped with a heating circulator (Julabo, Model F12-MC, Julabo Labortechnik, Germany). Appropriate measuring bob and cups (C14, C25, and C30) were used during viscosity measurements according to the viscosity of dispersion. Prepared samples were loaded into the cup and allowed to equilibrate for at least 10 min at desired temperature (5, 25, 45, and 65 °C). To determine the flow curves of gum, the shear rate was increased from 14 to 300 s^{-1} during 3 min and decreased from 300 to 14 s^{-1} in 3 min. In our previous work, 3-min pre-shear at 100 s^{-1} was used to obtain uniform and non-structured solution[23]. Thus, a small difference was observed between upward and downward curves, and the hysteresis area did not appear for most of the samples. In the present work, no pre-shear was used and the flow behavior index (*n*) and consistency coefficient (*k*) values were evaluated by fitting the power law model (Eq. 1):

$$\tau = k\dot{\gamma}^n \tag{1}$$

where τ is the shear stress (Pa), $\dot{\gamma}$ is the shear rate (s^{-1}), k is the consistency coefficient (Pa s^n), and n is the flow behavior index (dimensionless).

The hysteresis loop was obtained from the area between the upward and downward curves using the following equation (Eq. 2)[17]:

$$\text{Hysteresis loop area} = \int_{\dot{\gamma}_1}^{\dot{\gamma}_2} k \cdot \dot{\gamma}^n - \int_{\dot{\gamma}_1}^{\dot{\gamma}_2} k' \cdot \dot{\gamma}^{n'} \tag{2}$$

where k, k' are the consistency coefficient and n, n' are the flow behavior indices for upward and downward measurements, respectively.

For characterizing the thixotropic behavior, the gum solutions were subjected to a shearing period at constant values of shear rate to ensure that the material reached a completely destructed structure at which the rheological behavior is no longer dependent on shearing time. Based on preliminary tests in our laboratory, 500 s was sufficient for this purpose. Different concentrations of *A. homolocarpum* seed gum solutions (3%, 3.5%, and 4%) were sheared at constant shear rates, namely 14, 25, and 50 s^{-1} and different temperatures (5, 25, 45 and 65 °C), then the shear stress and apparent viscosity were measured as a function of shearing time until an equilibrium state was reached. Three models were used to describe the time-dependent flow properties of *A. homolocarpum* seed gum solution samples as follows:

1. Second-order structural kinetic model (Eq. 3):

$$\left[\frac{(\eta - \eta_\infty)}{(\eta_0 - \eta_\infty)}\right]^{1-n} = (n-1)kt + 1 \tag{3}$$

where η_0 is the initial apparent viscosity at $t=0$ (structured state) and η_∞ is the final apparent viscosity as $t \to \infty$ (equilibrium structured state). k is the rate constant of the thixotropic breakdown and it is the function of shear rate. The exponent n is the order of the breakdown reaction.

2. First-order stress decay model, with a non-zero stress value (Eq. 4):

$$\tau - \tau_{eq} = \left(\tau_0 - \tau_{eq}\right)e^{-kt} \tag{4}$$

where τ_0 is the initial shear stress value, τ_{eq} is the equilibrium stress value, and k is the breakdown rate constant.

3. Weltman model (Eq. 5):

$$\tau = A + B \ln t \tag{5}$$

In the Weltman model, parameter A represents the initial shear stress and parameter B; the time coefficient of thixotropic breakdown is “the product of rate in breakdown of thixotropic structure and time of agitation at constant rate of shear”[11].

Analysis of Data and Suitability of Model

All rheological measurements were conducted in triplicates. The extent of fit for applied models was evaluated by determining the root mean square error (RMSE, Eq. 6) and coefficient of determination (R^2) between the experimental and predicted shear stress values:

$$\mathrm{RMSE} = \sqrt{\frac{\sum_{n=1}^{N}\left(W_{experimental} - W_{calculated}\right)^2}{N}} \times 100 \tag{6}$$

where N is the number of data points and W indicates shear stress or viscosity depending on the model used.

The experiments were planned based on randomized full factorial design considering the effect of three concentrations, four temperature levels, and three shear rates. The results were analyzed using the general linear model (Minitab version 14.2) with a significance level of $P<0.05$.

Results and Discussion

Flow Curves and Thixotropy

The consistency coefficient (k) and flow behavior index (n) values obtained by fitting the power law models to the shear stress versus shear rate data as a function of gum concentration and temperature are shown in Table 1. Comparing these results with our previous work[23] showed that the consistency coefficients of samples with no pre-shear were higher than those with pre-shear (100 s^{-1} pre-shear for unification of the solution), while the flow behavior indices were lower for both upward and downward curves.

When a sample is subjected to an increasing and then decreasing shear rates, the presence of the hysteresis area between the curves representing shear stress values indicates that the sample’s flow is time dependent. According to Halmos and Tiu[25], the area encircled between the ascending and the descending curves is an index of the energy per unit of time and per unit of volume needed to eliminate the

Table 1 Power law parameters and hysteresis loop area obtained for *A. homolocarpum* seed gum solutions at different concentrations and temperatures

Concentration	Upward curve			Downward curve			Hysteresis (Pa s^{-1})
T (°C)	k	n	R^2	k	n	R^2	
3%							
5	17.60±0.35	0.34±0.01	0.99	16.96±0.75	0.32±0.01	0.99	3,453.50
25	14.35±0.50	0.35±0.00	0.97	13.95±0.45	0.33±0.02	0.99	2,784.66
45	11.81±0.12	0.35±0.01	0.97	10.24±0.43	0.35±0.02	0.99	2,527.50
65	9.54±0.54	0.37±0.02	0.95	8.95±0.68	0.36±0.01	0.98	1,829.76
3.5%							
5	24.61±0.75	0.33±0.02	0.99	21.39±0.25	0.31±0.01	0.99	7,654.67
25	22.95±0.55	0.34±0.01	0.98	19.84±0.38	0.32±0.01	0.99	7,641.72
45	20.55±0.37	0.34±0.02	0.97	17.52±0.78	0.32±0.01	0.99	7,194.72
65	17.85±0.42	0.34±0.02	0.95	14.50±0.51	0.33±0.00	0.98	6,213.74
4%							
5	31.41±0.68	0.31±0.00	0.93	25.13±0.90	0.30±0.00	0.98	8,571.40
25	29.53±0.33	0.31±0.00	0.95	24.30±0.25	0.30±0.01	0.99	8,452.22
45	26.84±0.12	0.31±0.00	0.98	21.58±0.75	0.30±0.01	0.99	8,317.118
65	25.05±1.25	0.32±0.00	0.94	20.52±1.62	0.31±0.01	0.97	7,661.69

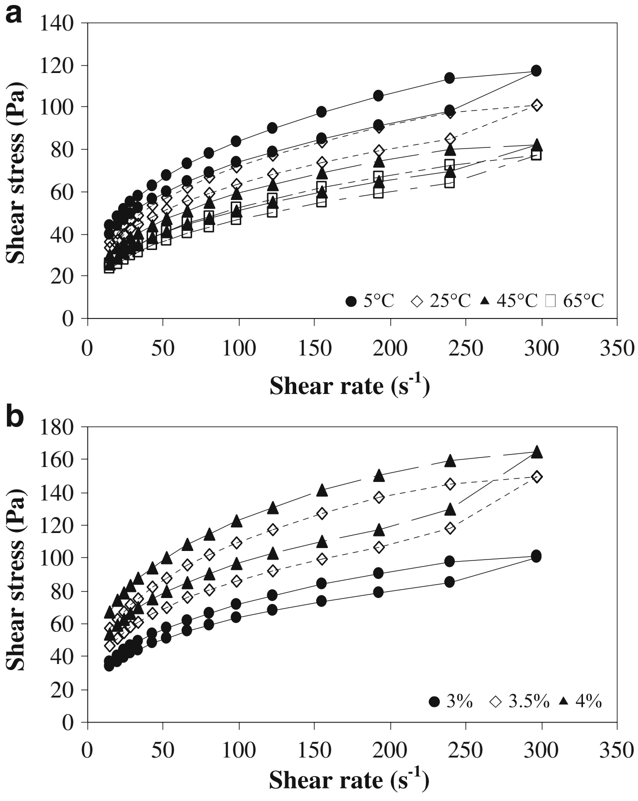

Fig. 1 Hysteresis loops obtained for *Alyssum homolocarpum* seed gum solutions at different **a** temperatures (gum concentration 3%) and **b** concentrations (temperature 25 °C)

influence of time in flow behavior. The flow curves of *A. homolocarpum* seed gum solutions at different temperatures and concentrations are shown in Figure 1. There was a hysteresis loop between the upward and downward curves, indicating thixotropic behavior. The curve for increasing rate was developed first and was at a higher position than the decreasing rate curve, showing that the gum exhibited a thinning behavior with time. The thixotropy of a polymer solution is interpreted as the continuous breakdown or rearrangement of network links formed by the associations between polymer chains under shear[19].

Thixotropic behavior was also reported for different hydrocolloids such as Salep and Balangu seed gum[17]. Some other authors determined the thixotropic behavior by evaluation of the area of the hysteresis loop between the upward and downward curves for different foods[26,27].

As expected, the loop area was significantly smaller at the higher temperature for all samples (Figure 1a). As a result, the hysteresis loop area became smaller as the temperature increased from 5 to 65 °C at a constant concentration (Table 1). This is probably an indication of the temperature effect on the molecular structure damaging of gum solution. On the other hand, the hysteresis area decreased as the concentration of *A. homolocarpum* seed gum samples decreased at a constant temperature (Figure 1b, Table 1). This indicated that the thixotropic behavior of *A. homolocarpum* seed gum solutions built up with an increase in gum concentration and might be due to the increase in the viscosity of the solution. As reported by Tarrega et al.[27], a high-viscosity thixotropic fluid may show a larger hysteresis area than a lower viscosity one even if the latter undergoes a stronger structural destruction. In our

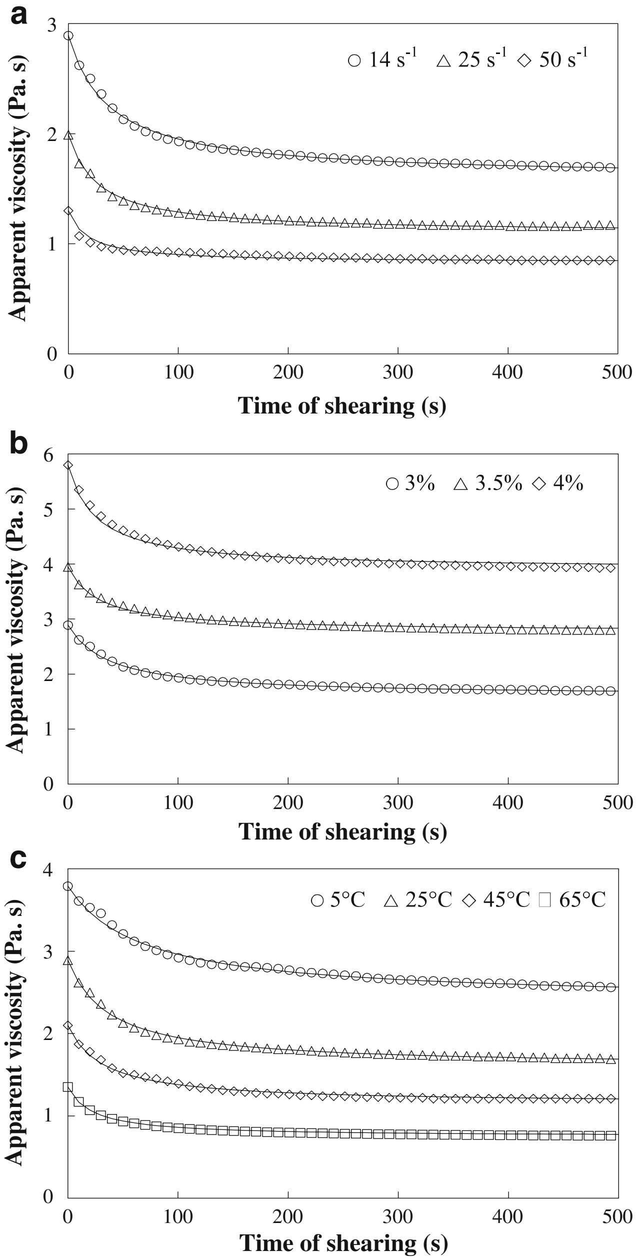

Fig. 2 Apparent viscosity data at different conditions studied: **a** shear rates (gum concentration 3%, temperature 25 °C), **b** concentrations (shear rate 14 s^{-1}, temperature 25 °C), and **c** temperatures (gum concentration 3%, shear rate 14 s^{-1}) as a function of shearing time. Experimental values and fits to the structural kinetic model

study, 4% gum solution at 5 °C had the highest hysteresis area among the samples (Table 1).

A similar trend was also observed by Zhang et al.[18] and Ghannam and Easkatoon[28] when they studied the thixotropic behavior of aqueous solution of hydroxypropyl guar gum and carboxymethylcellulose, respectively.

Comparison of straight loop areas between differently viscous systems may not render valid conclusions on the extension of time-dependent structural breakdown. Thus, different models were used to evaluate this phenomenon.

Second-Order Structural Kinetic Model

The gum solutions exhibited thixotropic behavior under all investigated conditions. Typical experiments obtained for gum solutions are shown in Figure 2. At a constant shear rate, the apparent viscosity decreased rapidly with time within the first 100 s of shearing and approached a constant value corresponding to an equilibrium state after approximately 300 s. This implies that the configuration of polysaccharides of the aqueous extracts is changed by the shearing force. Huei Chen and Yuu Chen[20] and Razavi and Karazhiyan[17] observed similar trends for green laver mucilage and Salep and Balangu seed mucilage, respectively.

The rate and extent of viscosity decay depended on the applied shear rate, gum concentration, and temperature. It can be seen that the viscosity tends to decay more rapidly at high shear rates toward an equilibrium viscosity, which was lower than that at low shear rates (Figure 2a). This fact implies that the breakdown rate of gum associations under a shear field accelerates at high shear rates.

The observed time-dependent flow behavior of *A. homolocarpum* seed gum was modeled using the structural kinetics approach, which has been successfully applied to concentrated yogurt[29,30], sweetened sesame paste[31], Salep and Balangu gum[17], and mayonnaise and tehineh[2]. The structural kinetic approach assumes that the change in the rheological behavior is associated with shear-induced breakdown of the internal structure in *A. homolocarpum* seed gum and that the rate of this breakdown during shearing depends on the kinetics of the *structured state→ non-structured state* process[2]. The results from the time-dependent measurements at constant shear rates and from the measured flow curves after long periods of shearing should prove this assumption.

For all *A. homolocarpum* seed gum solutions studied, it was found that their time-dependent apparent viscosity data at constant values of shear rate could be fitted well with a second-order structural kinetic model, i.e., with Eq. 1 using $n=2$[17]. In contrast with these results, Nguyen et al.[13] reported that transient viscosity data, for maize and waxy maize starch pastes, could be fitted to a third-order kinetic

Table 2 Adjusted mean squares for coefficients of the second-order structural kinetic, Weltman, and first-order stress decay models from the viscosity and shear stress decay data of three replicates at different concentrations, temperatures, and shear rates for *A. homolocarpum* dispersion

Source of variation	*df*	Second-order structural kinetic				Weltman				First-order stress decay					
		k (s^{-1})	P	η_0/η_∞	P	A	P	B	P	τ_0 (Pa)	P	τ_{eq} (Pa)	P	$k\times10^{-3}$ (s^{-1})	P
Concentration (*C*)	2	0.07	0.00	0.20	0.00	17,954.3	0.00	43.76	0.00	14,903.7	0.00	8,331.9	0.00	602.83	0.00
Temperature (*T*)	3	0.06	0.01	0.07	0.00	8,372.8	0.00	13.45	0.00	61.73.9	0.00	4,905.0	0.00	1,178.25	0.00
Shear rate (*R*)	2	0.09	0.00	0.47	0.01	1,866.3	0.00	19.70	0.00	3,265.8	0.00	4,649.9	0.00	798.22	0.00
C×*T*	6	0.05	0.00	0.06	0.00	1,123.3	0.01	1.49	0.01	574.8	0.00	640.6	0.00	775.50	0.01
C×*R*	4	0.05	0.00	0.02	0.01	10.2	0.12	6.04	0.00	71.8	0.01	241.1	0.01	224.1	0.00
T×*R*	6	0.05	0.01	0.01	0.01	56.3	0.00	1.97	0.00	166.8	0.00	223.3	0.01	333.35	0.01
C×*T*×*R*	12	0.05	0.01	0.02	0.00	17.2	0.01	0.65	0.03	18.4	0.03	42.4	0.00	390.16	0.00
Error	72	0.001		0.004		5.2		0.29		8.2		3.9		7.03	

model. This difference in kinetics order may be due to the complicated binding pattern in different polymers.

Both rate constant (*k*) and structural breakdown (η_0/η_∞) were significantly ($P<0.01$) affected by all main factors (concentration of hydrocolloids, temperature, and shear rate) and all interactions of these factors in the present study (Table 2). The effects of gum concentration, temperature, and shear rate on the second-order structural kinetic model's parameters of gum solution were investigated (Table 3). The rate constant, *k*, is an indication of the rate of thixotropic breakdown, i.e., the degree of thixotropy, while the ratio of initial to equilibrium viscosity, η_0/η_∞, can be considered as a relative measurement of the amount of structural breakdown, or in other words the extent of hysteresis . The high R^2 and low RMSE values for all tested samples indicated the appropriateness of the second-order structural kinetic model to describe the thixotropic behavior of the *A. homolocarpum* seed gum (Table 3). The *k* value increased with increasing shear rate, concentration, and temperature, indicating that the rupture rate of *A. homolocarpum* seed gum associations increases at high shear rates, temperatures, and concentrations. Mao and Chen[19] reported that the kinetics of structure breakdown is strongly affected by temperature. Their report also showed that the

Table 3 The parameters of second-order structural kinetic model of *A. homolocarpum* seed gum solutions

Concentration	*T* (°C)	γ (s^{-1})	*k* (s^{-1})	η_0/η_∞	R^2	RMSE
3%	5	14	0.02±0.01	1.60±0.06	0.98	2.45
		25	0.03±0.01	1.72±0.05	0.99	1.80
		50	0.05±0.01	1.61±0.05	0.98	1.90
	25	14	0.03±0.01	1.81±0.03	0.97	2.18
		25	0.04±0.00	1.80±0.06	0.97	1.72
		50	0.05±0.01	1.57±0.03	0.98	1.50
	45	14	0.03±0.01	1.83±0.04	0.98	2.24
		25	0.05±0.02	1.57±0.01	0.99	1.48
		50	0.07±0.02	1.49±0.04	0.97	1.51
	65	14	0.04±0.01	1.84±0.02	0.98	2.65
		25	0.06±0.02	1.69±0.05	0.95	4.85
		50	0.07±0.02	1.41±0.06	0.96	1.76
3.5%	5	14	0.02±0.00	1.67±0.03	0.97	1.47
		25	0.06±0.00	1.33±0.03	0.97	1.25
		50	0.07±0.01	1.27±0.01	0.89	1.75
	25	14	0.04±0.01	1.48±0.04	0.98	3.17
		25	0.07±0.00	1.48±0.02	0.98	1.07
		50	0.08±0.00	1.44±0.03	0.99	1.41
	45	14	0.04±0.01	1.71±0.02	0.91	5.72
		25	0.07±0.01	1.46±0.05	0.98	1.21
		50	0.08±0.01	1.45±0.02	0.97	1.66
	65	14	0.05±0.01	1.66±0.05	0.94	4.52
		25	0.08±0.01	1.45±0.04	0.96	1.89
		50	0.09±0.00	1.39±0.09	0.99	1.14
4%	5	14	0.04±0.00	1.63±0.01	0.98	3.14
		25	0.07±0.00	1.40±0.03	0.97	1.29
		50	0.08±0.01	1.25±0.02	0.93	1.05
	25	14	0.04±0.01	1.48±0.07	0.97	2.10
		25	0.08±0.01	1.37±0.02	0.98	1.61
		50	0.09±0.02	1.25±0.01	0.98	1.20
	45	14	0.05±0.01	1.84±0.05	0.98	2.54
		25	0.08±0.01	1.70±0.02	0.94	2.76
		50	0.09±0.03	1.33±0.01	0.96	1.13
	65	14	0.06±0.02	1.86±0.06	0.99	1.22
		25	0.08±0.02	1.72±0.07	0.97	1.68
		50	1.01±0.00	1.59±0.03	0.98	1.85

viscosity decreases more rapidly at high temperatures, which also cause a decrease in equilibrium viscosity. Similar to our results, Razavi and Karazhiyan[17] concluded that the rate constant for Salep solution increased with increasing shear rates; however, Balangu seed gum solution was shear rate independent.

Table 3 also shows that the amount of structural breakdown (η_0/η_∞) decreased with shear rate (except for 3% gum solution at 25 °C), but it did not have a general trend with concentration and temperature. The latter behavior seems to be due to the fact that the decrease of η_0 with concentration and temperature was greater than that of η_∞ (different structure as a result of shearing). Razavi and Karazhiyan[17] also reported a similar trend for Balangu seed gum at different shear rates.

Nguyen et al.[13] have suggested that the value of η_0/η_∞ may serve as a measure of the extent of hysteresis. They also found that such measure decreased with increasing shear rate. Mao and Chen[19] noted that the value of η_0/η_∞ is temperature independent. Figoni and Shoemaker[3] found that the amount of structural breakdown of mayonnaise showed no trend of either increasing or decreasing with shear rate. But this was due to the narrow range of shear rates used. This result is comparable to that for the waxy maize starch pastes[13], where η_0/η_∞ slightly varies with the shear rate.

For all shear rates, 4% gum solution at 65 °C showed higher k values than the rest of samples, indicating a faster rate of thixotropic breakdown, and a greater ratio of the initial to equilibrium viscosity (η_0/η_∞), at relative measure of the extent of thixotropy at 14 s^{-1} (Table 3). Nguyen et al.[13] reported that k values for starch pastes increased or decreased with temperature depending on the type of starch and on the applied shear rate. Abu-Jdayil[32] observed a decrease in k values with temperature in milled black cumin and an increase in milled sesame seeds[2].

Weltman Model

This section deals with the dependence of flow properties on time in terms of shear stress versus shearing time. As shown in Figure 3, the Weltman model (solid line) fits well the relationship between the shear stress and shearing time of *A. homolocarpum* seed gum solutions. Results indicate that the shear stress decreased with increasing time of shearing, which simply means that gum exhibited a thixotropic behavior. Decreases in the shear stress were more pronounced for higher-concentration solutions (Figure 3b). Similar results were observed for glucose–starch mixtures[33], low-fat sesame pastes[34], and Salep and Balangu gum solutions[17].

The coefficients of the Weltman model were also significantly affected by concentration of hydrocolloids, temperature, and shear rates. In addition, except for the effect of interaction of hydrocolloid concentration with shear rates on initial stress (A), the interaction of concentrations of hydrocolloid, temperature, and shear rate had a significant effect on the coefficients of Weltman model (Table 2).

The experimental data fitted well to the Weltman model, with R^2 values going from 0.88 to 0.97 for all conditions studied (Table 4). At higher temperatures, both Weltman

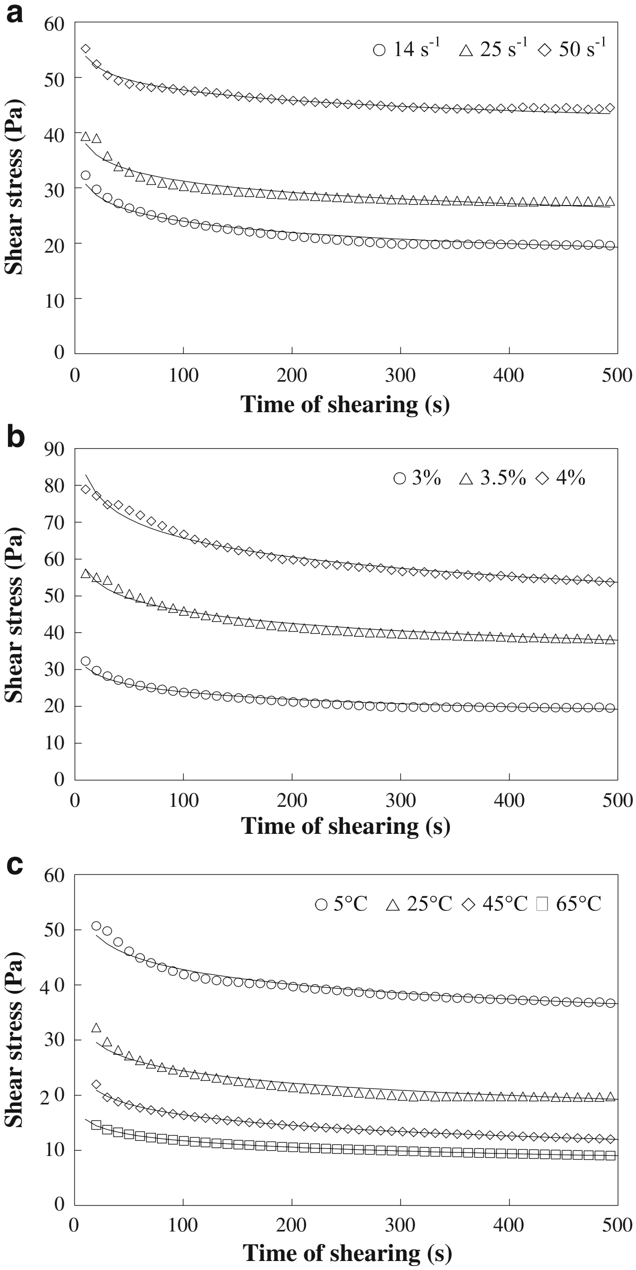

Fig. 3 Shear stress data at different shear rates (**a**—gum concentration 3%, temperature 25 °C), concentrations (**b**—shear rate 14 s^{-1}, temperature 25 °C), and temperatures (**c**—gum concentration 3%, shear rate 14 s^{-1}) as a function of shearing time. Experimental values and fits to the Weltman model

Table 4 The parameters of the Weltman model of *A. homolocarpum* seed gum solutions

Concentration	T (°C)	γ (s^{-1})	A (Pa)	$-B$ (Pa)	R^2	RMSE
3%	5	14	60.54±1.41	3.85±0.08	0.97	1.56
		25	67.59±1.85	3.54±0.15	0.97	0.43
		50	73.42±0.95	2.84±0.09	0.91	1.11
	25	14	37.41±0.78	2.92±0.02	0.95	2.79
		25	44.04±0.62	2.81±0.09	0.90	2.19
		50	59.99±0.05	2.67±0.12	0.98	0.80
	45	14	28.78±1.15	1.87±0.18	0.84	3.57
		25	32.28±0.09	1.81±0.06	0.89	2.48
		50	39.30±0.25	1.59±0.03	0.90	1.72
	65	14	19.48±0.34	1.68±0.15	0.87	3.12
		25	26.29±0.85	1.57±0.12	0.91	1.98
		50	35.62±0.93	1.42±0.04	0.91	2.01
3.5%	5	14	67.89±1.52	4.99±0.13	0.96	0.06
		25	81.42±0.36	3.89±0.75	0.94	1.09
		50	88.08±0.08	2.98±0.91	0.91	1.38
	25	14	69.04±0.24	4.89±0.16	0.97	1.93
		25	68.47±1.05	3.65±0.35	0.98	0.32
		50	86.27±1.68	3.02±0.41	0.90	1.78
	45	14	55.36±2.25	3.84±0.15	0.92	2.67
		25	60.31±1.35	3.53±0.09	0.94	1.27
		50	73.93±1.95	2.92±0.08	0.92	0.97
	65	14	45.15±1.62	3.41±0.16	0.91	2.45
		25	53.52±0.97	3.27±0.07	0.98	1.65
		50	66.38±1.41	2.85±0.16	0.97	2.52
4%	5	14	134.97±2.75	9.16±0.74	0.98	1.53
		25	141.16±2.93	8.11±0.61	0.97	1.49
		50	147.51±1.25	3.20±0.15	0.96	0.39
	25	14	102.67±3.25	7.92±0.91	0.98	1.67
		25	111.02±2.95	5.27±0.09	0.95	1.13
		50	133.32±1.95	2.97±0.85	0.98	0.19
	45	14	68.15±1.23	5.13±0.67	0.95	2.55
		25	72.98±2.26	4.95±0.59	0.86	3.76
		50	78.74±1.35	2.94±0.46	0.97	1.49
	65	14	53.47±1.62	4.42±0.15	0.87	4.35
		25	65.55±2.41	3.96±0.41	0.91	3.12
		50	70.28±1.94	2.87±0.47	0.92	2.48

parameters (A and B) showed lower values at each level of concentration or shear rate. A negative value of B measures how fast the shear stress drops from the initial value to the final equilibrium (steady-state) value[33]. The B value (indicating the extent of thixotropy) increased with increasing gum concentration at constant temperature and decreased with increasing temperature and shear rate at constant concentration. Our finding that B decreases with increasing shear rate is in agreement with that of Nguyen et al.[13]. In contrast with our results, Razavi et al.[34] and Razavi and Karazhian[17] reported that the B value for low-fat sesame paste/date syrup blends and Salep increased with shear rate, respectively. This model has also been considered appropriate to characterize the time dependence of the flow of other food products like salad dressings[35], fruit jams[36], or yogurt[37].

The *A. homolocarpum* seed gum concentration had a considerable effect on the degree of thixotropic behavior. At a constant shear rate of 14 s^{-1} and for three gum concentrations at constant temperature, not only did the initial value of shear stress (parameter A in the Weltman model) increased significantly but also the B value increased from 3.85 Pa for 3% gum concentration to 9.16 Pa for 4% hydrocolloid concentration (Table 4). In other words, increasing the *A. homolocarpum* seed gum concentration leads to an increase in the degree of thixotropy.

It can be seen in Table 4 that temperature had a significant effect on the time-dependent flow properties of *A. homolocarpum* seed gum. At constant shear rate (14 s^{-1}), for the solution containing 3% gum, the value of *B* reduced from 3.85 Pa at 5 °C to 1.68 at 65 °C. On the other hand, the solution became somewhat less thixotropic at higher temperatures. Similar results were reported by Paredes et al.[35] in commercial samples of salad dressings, for which Weltman parameters obtained at two temperatures showed lower values at the higher temperature.

At a constant shear rate of 14 s^{-1}, 4% *A. homolocarpum* seed gum solution at 5 °C showed the highest initial stress (*A*) and extent of thixotropy (*B*) values than for the rest of the samples at all concentrations, temperatures, and shear rates (Table 4). Higher values for *B* indicate its higher rate of structural breakdown by shearing.

First-Order Stress Decay Model with a Non-zero Stress Value

In this model, being the case and for the sake of inspecting the presence of a non-zero equilibrium shear stress value at a relatively infinite time, the measurements were extended up to 500 s of shearing time. Figure 4 shows that the experimental data of shear stress versus shearing time of *A. homolocarpum* seed gum solution, evaluated at different shear rates (Figure 4a), concentrations (Figure 4b), and temperatures (Figure 4c), fitted to the first-order stress decay model. It seems that this model (solid line) fits well with the relationship between the shear stress and shearing time data for all samples.

In the present study, the coefficients of the first-order stress decay model were significant at probability (*P*) levels up to 0.01 (Table 2). Further, the interaction of concentrations, temperatures, and shear rates had a significant effect on the coefficients of the time-dependent models employed in this study. Table 5 shows the parameters of the first-order stress decay with a non-zero equilibrium stress value, for a shearing period of 500 s, evaluated at different conditions studied. A good agreement (R^2>0.96 and lowest RMSE) was found between the model-fitted results and experimental shear stress data for all samples (Table 5). The equilibrium stress and initial stress values decreased with both increasing temperature and decreasing concentration (Figure 4). Besides, they increased with increasing shear rate which was similar to Balangu seed gum[17]. The decrease of the equilibrium stress (τ_{eq}) and initial stress (τ_0) with increasing temperature is typical of milled sesame[38] and Gilaboru juice[39]. This effect is greater when the solid concentration is higher[40,41].

The decay rate constant, *k*, is an indication of how fast the gum solution, under the action of shearing, reaches the equilibrium stress value beyond which the apparent viscosity remains constant. In this research work, the decay rate constant generally increased with increasing shear rate (except for 4% gum solution); however, it did not have any trend with concentration and temperature (Table 5). Razavi and Karazhian[17], Altan et al.[39], and Abu-Jdayil et al.[38] also reported that *k* increased with shear rate for Salep, Gilaboru juice, and milled sesame paste, respectively. However,

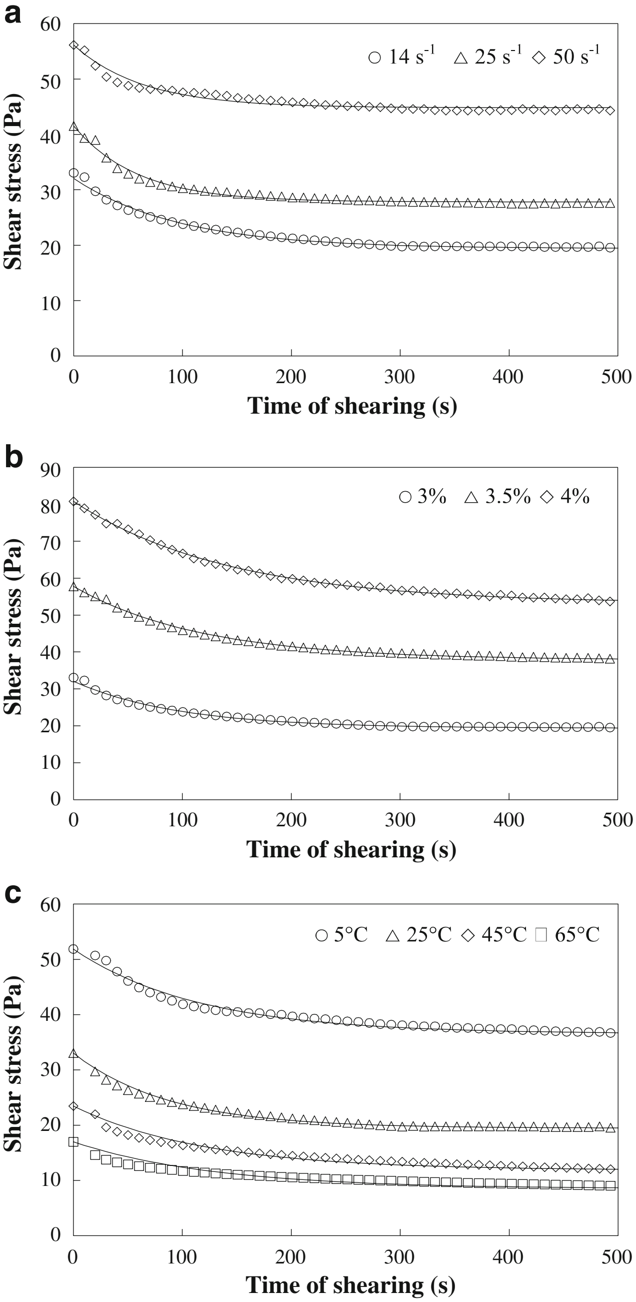

Fig. 4 Shear stress data at different shear rates (**a**—gum concentration 3%, temperature 25 °C), concentrations (**b**—shear rate 14 s^{-1}, temperature 25 °C), and temperatures (**c**—gum concentration 3%, shear rate 14 s^{-1}) as a function of shearing time. Experimental values and fits to the first-order stress decay model

Table 5 The parameters of first-order stress decay model of *A. homolocarpum* seed gum solutions

Concentration	T (°C)	γ (s^{-1})	τ_0 (Pa)	τ_{eq} (Pa)	$k\times10^{-3}$ (s^{-1})	R^2	RMSE
3%	5	14	51.88±1.95	36.55±0.98	8.6±0.52	0.98	1.32
		25	61.10±2.26	46.51±1.01	11±1.15	0.99	0.82
		50	73.59±1.64	57.56±0.85	25±2.12	0.99	0.80
	25	14	32.06±1.65	19.39±1.24	10±1.10	0.99	1.93
		25	41.52±3.45	27.82±1.85	17±2.50	0.99	1.74
		50	56.13±1.65	44.82±0.75	16±3.15	0.96	1.17
	45	14	27.61±1.15	17.41±1.10	14±2.15	0.99	0.77
		25	31.16±2.74	22.29±1.85	22±3.45	0.99	0.55
		50	39.55±1.68	30.84±0.81	29±1.85	0.99	0.62
	65	14	17.10±1.81	12.62±0.81	16±1.15	0.98	1.05
		25	24.35±1.61	18.35±1.15	18±2.95	0.99	0.98
		50	30.52±0.92	24.85±1.45	20±2.81	0.99	0.75
3.5%	5	14	58.34±1.85	38.03±1.55	11±1.35	0.98	2.44
		25	74.25±2.15	58.42±0.93	12±2.15	0.99	0.52
		50	86.04±1.21	71.92±1.95	24±1.85	0.99	0.72
	25	14	57.76±0.95	37.84±0.72	8.5±0.45	0.99	1.31
		25	63.02±2.36	46.32±1.47	11±1.25	0.98	1.36
		50	90.54±3.25	68.78±0.96	30±1.85	0.97	1.40
	45	14	48.69±1.12	31.65±1.47	10±0.95	0.99	0.74
		25	53.55±0.87	37.18±2.05	11±1.45	0.99	0.56
		50	64.25±2.85	51.66±1.74	26±1.45	0.99	0.26
	65	14	44.75±1.65	28.25±1.35	7.5±0.5	0.98	1.10
		25	49.35±2.15	32.55±1.94	15±1.26	0.99	0.87
		50	58.15±2.41	45.35±2.14	27±2.15	0.99	0.64
4%	5	14	111.16±3.35	77.14±1.36	7.1±0.48	0.99	0.80
		25	126±2.65	95.69±0.74	14±1.75	0.99	0.94
		50	142±1.25	127.90±1.15	10±3.42	0.99	0.73
	25	14	80.77±1.29	53.15±0.58	6.9±0.35	0.98	1.02
		25	92.02±1.85	71.43±1.65	2.4±0.58	0.98	0.70
		50	128±1.34	115.17±1.15	9.9±0.68	0.99	0.44
	45	14	57.37±1.98	36.22±0.79	90±1.35	0.99	0.98
		25	72.91±2.25	44.48±0.65	16±0.85	0.98	1.53
		50	80.03±1.85	63.39±1.65	49±2.25	0.96	1.66
	65	14	53.25±2.15	32.15±1.92	11±1.8	0.97	1.87
		25	65.52±1.45	39.57±1.34	39±2.22	0.98	1.45
		50	72.15±1.95	48.19±2.15	48±2.64	0.98	1.15

Razavi and Karazhian[17] concluded opposite trend for Balangu seed gum.

Though the goodness of fit as assessed by the coefficient of determination (R^2) values were often high (0.91–0.99, 0.84–0.98, and 0.96–0.99 for the second-order structural kinetic, Weltman, and first-order stress decay models, respectively), the RMSE values offer a better picture, thereby showing that R^2 values may not be a true indicator of the extent of fit, particularly when a large number of data points are analyzed.

Examination of the suitability of the time-dependent rheological models (Tables 3, 4, and 5) indicated that they were all suitable for characterizing the time-dependent rheological behavior of the *A. homolocarpum* seed gum. First-order stress decay model was marginally better than the others as it gave slightly lower RMSE values 0.44–2.44 (average 1.05) compared with those for the Weltmann (0.19–4.35, average 1.83) and second-order structural kinetic (1.05–5.72, average 2.05) models (Tables 3, 4, and 5).

Conclusions

In this work, the time dependency of *A. homolocarpum* seed gum at different concentrations, temperatures, and shear rates were studied. These solutions showed a hysteresis loop which increased with gum concentration and decreased with temperature. As far as the effect of steady shearing on the flow properties of *A. homolocarpum* seed gum is concerned, three models were used to predict the flow behavior, namely, the first-order shear stress decay, structural kinetic, and Weltman models. The *A. homolocarpum* seed gum concentration had a considerable effect on the degree of the thixotropic behavior. On the other hand, the solution became somewhat less thixotropic at higher temperatures. The rupture rate of *A. homolocarpum* seed gum association increases at high shear rates, temperatures, and concentrations. Among the three models, the thixotropic behavior of *A. homolocarpum* seed gum was described well by the first-order shear stress decay. The results on the suitability of a rheological model may be used for scaling-up purposes. However, more research is needed to analyze and identify the structural differences responsible for the effect of temperature, concentration, and shear rate on the rate of thixotropic breakdown.

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