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Effects of high pressure homogenization on rheological properties of flaxseed gum

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ABSTRACT

The effects of high pressure homogenization on the rheological properties of flaxseed gum solution were analyzed in this study. The apparent viscosity of flaxseed gum was decreased by increasing homogenization pressure, while the temperature of the solution was gradually increased at the same time. The consistency index and the flow behavior index in the Power Law model of apparent viscosity were also modeled with Power Law and logarithm equations, respectively. The decay in parameters of storage and loss modulus model was also found along with the increasing homogenization pressure. Although no effect of pressure on conductivity and gelling temperature was significant, the clarity of the flaxseed gum was increased by increasing pressure. The activation and zeta potential of the flaxseed gum showed similar trends with the homogenization pressure, which were first increased then decreased from 30 MPa.

1. Introduction

Polysaccharide gums are commonly used as thickener, emulsifier, or carrier material in microencapsulation in the food systems (Soma, Williams, & Lo, 2009), since the polysaccharide gums often come with high viscosity, emulsifying ability, and stability. Flaxseed gum is the polysaccharide gums deprived from flaxseed, and has been paid high attention to because of not only its nutrient value as dietary fiber but also its good performance in the functional properties (Oomah, 2001; Stavro, Marchie, Kendall, Vuksan, & Jenkins, 2003). Flaxseed gum was found to be effective in reducing blood glucose and cholesterol in type 2 diabetic patients (Thakur, Mitra, Pal, & Rousseau, 2009). Moreover, the high viscosity of flaxseed gum has been reported as well as the gelling properties and emulsifying properties, which showed the potential of flaxseed gum to be used as emulsifier and stabilizer in the food industry (Mazza & Biliaderis, 1989; Chen, Xu, & Wang, 2006; Wang, Wang, Li, Xue, & Mao, 2009).

Homogenization process could provide high shear stress and inertial forces, thus is commonly used in food industry (Corredig & Dalgleish, 1996). High pressure homogenization has already been widely used in emulsion preparation and microencapsulation (Schultz, Wagner, Urban, & Ulrich, 2004; Ding & Shah, 2009). At the same time, polysaccharide gum is also widely used in the preparation of emulsion as emulsifier and stabilizer (Moschakis, Murray, & Dickinson, 2005), and in microencapsulation as carrier material (Tobitsuka, Miura, & Kobayashi, 2006).

The rheological properties of polysaccharide play a significant role when the polysaccharide was applied in food system (Dickinson, 2003). However, high pressure homogenization could significantly influence the structure and the rheological properties of polysaccharide gums, which would thus influence the application of the gums in food system. For example, the effects of high pressure and number of passes on the xanthan gum solution were both reported to produce a reduction in rheological properties. Consequently the thickening and stabilizing properties were decreased (Lagoueyte & Paquin, 1998). An exponential decay in viscosity has also been observed in the high pressure homogenization (0–300 MPa) process of three kinds of polysaccharides dispersion (alginate, *K*-carrageenan, xanthan) (Harte & Venegas, 2010). Based on the above reason, high pressure homogenization even could be used to reduce the molecular weight and modify the viscosity range of different types of pectins (Corredig & Wicker, 2001).

Flaxseed gum was reported to show emulsifying properties and has been used in the emulsion preparation together with whey protein isolate (Khalloufi, Corredig, Goff, & Alexander, 2009; Wang, Li, Wang, Li, & Adhikari, 2010). High pressure homogenization has been used in all of these studies. However, so far no research has been reported on the influence of high pressure homogenization on the rheological properties of flaxseed gum solution. Therefore, the main objective of this study was to investigate the influences of homogenization pressure on the apparent viscosity, storage and loss modulus, activation energy, and stability of

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flaxseed gum solution. This study would provide useful information for the processing of flaxseed gum, both for research and for potential industrial applications.

2. Materials and methods

2.1. Flaxseed gum extraction

Flaxseed was purchased from the Hebei province of China, with moisture content of 6.50%.

Flaxseed (100 g) was washed in water for 1 min to remove the surface dust, and then mixed with 900 mL deionized water. The flaxseed and water were then stirred for 5 h at a speed of 300 rpm/min, in a 60 °C water bath, according to the method of Cui (2001). The extracted flaxseed gum solution was filtered through 40-mesh screen.

Extracted flaxseed gum solution was precipitated with two volumes of 95% ethanol, collected by centrifugation at 3000 rpm/min for 10 min using an LG10-2.4 A machine (Beijing Medical Centrifuge Corporation, Beijing, China), according to the method of Cui, Mazza, and Biliaderis (1994) with some modification on drying method. The precipitated flaxseed gum was then dried in a hot air oven at 80 °C for 8 h.

The protein content of flaxseed gum extracted by the above method was 14.4 ± 0.2%, determined by Kjeldahl method, using a FOSS Kjeltec 2300 analyzer (FOSS Co., Höganäs Sweden). The nitrogen data were converted into protein values employing a conversion factor of 6.25. The fat content in flaxseed gum was 0.59 ± 0.13%, determined by Soxhlet standard extraction using Universal Extraction System (B-811, BÜCHI Labortechnik AG, Switzerland). And the ash content was 3.35 ± 0.12%. Analysis was performed in triplicate.

2.2. High pressure homogenization of flaxseed gum

The dried flaxseed gum was dissolved in deionized water with magnetic stirring to make the 1% (w/v) flaxseed gum solution. The solution was kept at 4 °C overnight to achieve complete hydration.

The flaxseed gum solution was then homogenized at 0, 10, 30, 50, 70, or 90 MPa for five circles, using an AH-100D homogenizer (ATS Engineering Inc., Shanghai, China) at room temperature. The outlet temperature of the solution was detected immediately after the homogenization process.

2.3. Rheological properties of processed flaxseed gum

Rheological measurements were performed using AR2000ex rheometer (TA Instruments Ltd., Crawley, UK). The temperature was controlled by a water bath connected to the Peltier system in the bottom plate. A thin layer of silicone oil was applied on the surface of the samples in order to prevent evaporation. The linear viscoelastic region was determined for each sample through strain sweeps at 1 Hz (data not shown). Viscoelastic properties (storage modulus G', loss modulus G'', and δ) of flaxseed gum solutions were determined within the linear viscoelastic region. An equilibration of 2 min was performed before each measurement.

2.3.1. Continuous shear measurements

The continuous shear tests were performed at 25 °C over the shear rate range of 0.1–100 s^{-1} to measure the apparent viscosity. Steel cone geometry (60 mm diameter, 59 μm gap) was chosen for the continuous shear measurements.

2.3.2. Activation energy measurements

The apparent viscosity was determined over the temperature range from 10 to 50 °C, in order to determine the activation energy by the Arrhenius relationship, at a constant shear rate of 10 s^{-1}. Steel cone geometry (60 mm diameter, 59 μm gap) was chosen for the activation energy measurements.

2.3.3. Frequency sweep measurements

The frequency sweep tests were performed at 25 °C over the angular frequency range of 0.01–10 rad/s. The strain amplitude for the frequency sweep measurements was selected as 1% according to the strain sweep results (data not shown) in order to be in the linear viscoelastic region for all samples. Aluminum parallel plate geometry (40 mm diameter, 1 mm gap) was chosen for the frequency sweep measurements.

2.3.4. Gelling temperature measurements

The samples were heated to 90 °C then held for 2 min. The samples were then cooled to 10 °C at 5 °C/min and kept at 10 °C. The temperatures at which G' and G'' crossed were taken as the gelling temperatures during cooling (Chen et al., 2006). Aluminum parallel plate geometry (40 mm diameter, 1 mm gap) was chosen for the gelling temperature measurements.

All rheological measurements were carried out in triplicate. The experimental rheological data were obtained directly from the TA Rheology Advantage Data Analysis software V 5.4.7 (TA Instruments Ltd., Crawley, UK). The average of the three runs was reported as the measured value with standard deviation.

2.4. Zeta potential

The zeta potential of 1% flaxseed gum solutions were measured using a Malvern Zetasizer Nano-ZS (ZEN3600; Malvern Instrument Ltd., Worcestershire, UK) across the capillary tube at 25 °C. The conductivity of samples was measured at the same time. The average of three replicates was reported.

2.5. Clarity

The clarity of the flaxseed gum solution was represented by the absorbance of the solution in this study. The absorbance of 1% flaxseed gum solution was tested at 500 nm immediately after homogenization, using an ultraviolet visible spectrophotometer (TU-1810, Beijing Purkinje General Instrument Co., Ltd., Beijing, China), according to the method of Che et al. (2009). The deionized water was used as blank.

2.6. Statistical analysis

All the experiments were done in triplicate in this study. The results reported are means ± standard deviation of three determinations excluding the flow curves. Duncan's multiple comparison tests were conducted to determine the significant effect of high pressure processes on the properties of flaxseed gum samples at $p < 0.05$ using the SAS software (SAS Institute Inc., Cary, NC, USA).

3. Results and discussion

3.1. Temperature increase

The temperature increase of flaxseed gum solution caused by the high pressure homogenization process was shown in Fig. 1. The temperature of the high pressure homogenized solution was gradually increased by the process from 30 °C to about 53 °C when the highest pressure, i.e. 90 MPa, was used. Moreover, the temperature increase was proportional to the pressure applied, and could be regressed into a straight line with the slope of 0.2564, as shown in Fig. 1. Although the apparent viscosity was found to be affected by

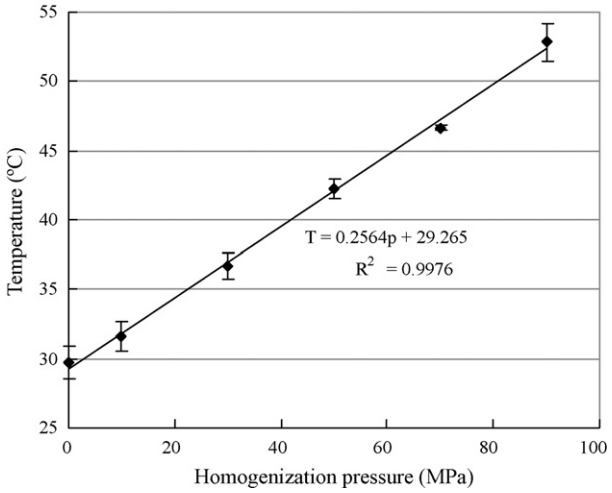

Fig. 1. Temperature increase of the flaxseed gum solutions after high pressure homogenization.

the temperature increase, few effects was introduced to the storage modulus and loss modulus in starch and flaxseed gum mixtures when the temperature increased from 25 °C to 50 °C (Wang et al., 2008). Similar trend in the temperature increase was also found in the potato and cassava starch pastes corresponding to the high pressure applied (Che et al., 2009). The slopes of the starch pastes were slightly lower (0.186 for cassava starch paste, 0.177 for potato starch paste) than flaxseed gum solutions (0.2564 in this study).

3.2. Activation energy

The influence of temperature on the apparent viscosity at a constant shear rate ($10\,s^{-1}$) of flaxseed gum solution can be calculated using the Arrhenius equation (Pongsawatmanit, Temsiripong, Ikeda, & Nishinari, 2006):

$$\eta_a = \eta_\infty \exp(E_a/RT) \quad (1)$$

where η_a is the apparent viscosity at a specific shear rate, η_∞ is the frequency factor, E_a is the activation energy (kJ/mol), R is the gas constant (8.3145 J/mol K), and T is the absolute temperature (K).

The activation energy (E_a) can be used to measure the sensitivity of apparent viscosity changes with temperature. The E_a values of flaxseed gum solutions processed by different pressures are shown in Fig. 2. These values are similar to the activation

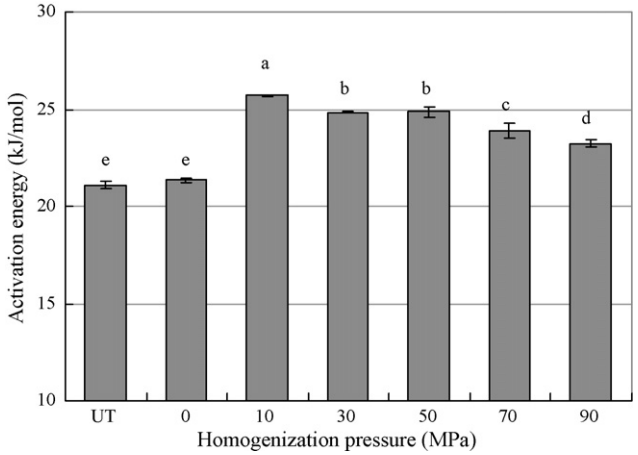

Fig. 2. Effects of high pressure homogenization on the activation energy of the flaxseed gum solutions (UT stands for untreated sample).

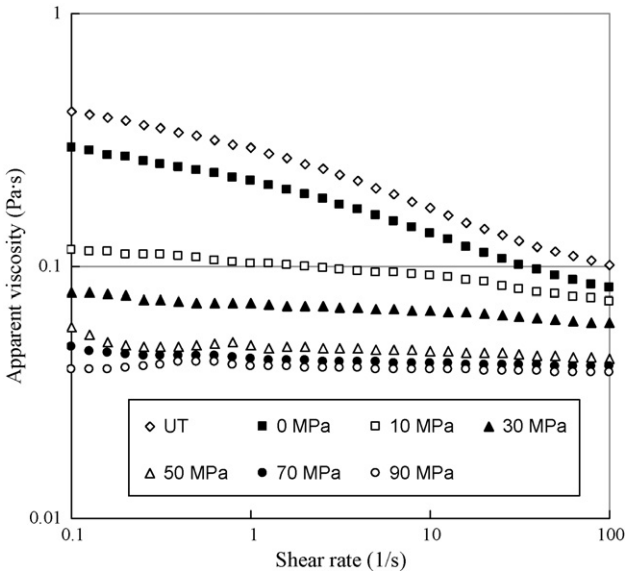

Fig. 3. Effects of high pressure homogenization on the apparent viscosity of the flaxseed gum solutions (UT stands for untreated sample).

energy of 1% xanthan gum solution (22.6 kJ/mol) at shear rate of $50\,s^{-1}$ (Pongsawatmanit et al., 2006), but higher than the E_a of 1% guar gum, which was reported to be 13.0 kJ/mol in previous study (Launay, Cuvelier, & Martinez-Reyes, 1997). All the E_a values of high pressure homogenized samples were increased compared to the untreated sample and zero pressure treated sample ($p < 0.05$). This is probably because the high pressure homogenization provides the gum molecules with more opportunity to contact each other, thus providing more opportunity to form 3D network. Because the high activation energy of flow indicates more inter-and intra-interactions between polysaccharide chains at the concentration studied (Mohammadifar, Musavi, Kiumarsi, & Williams, 2006). However, the E_a values did not increase with the increase of the homogenization pressure. On the contrary, they decreased gradually along with the pressure increase from 10 MPa to 90 MPa. This might be because that 10 MPa was sufficient to provide the contact opportunity for the molecules. But the higher pressure process (>10 MPa) broke down the molecule size of the flaxseed gum, so that both the inter-and intra-interactions of the molecules were reduced, which contributed to the decrease of E_a.

3.3. Rheological properties

The apparent viscosity of the high pressure homogenized flaxseed gum solution is shown in Fig. 3. The viscosity of the solution was decreased by all the processings, including the 0 MPa one. Since when the solution went through the homogenizer, the pump and the valve in the homogenizer still provided shearing force even without pressure applied. And a gradual decrease was found in the viscosity of the solution along with the increase of the homogenization pressure. The reduction of viscosity during high pressure homogenization was also found in pectin, because the high shear and force input during the homogenization decreased the molecular weight of the pectin (Corredig & Wicker, 2001). All the samples show shear-shinning effects. However, when processed under high pressure (50–90 MPa), the viscosity plot was almost parallel to the X axial, indicating that the shear-shinning property was almost destroyed by the homogenization. This is probably because the high shear, turbulence forces and cavitation involved in the high pressure process produced ordered-disordered conformational transition (by opening of the molecule) and poly-

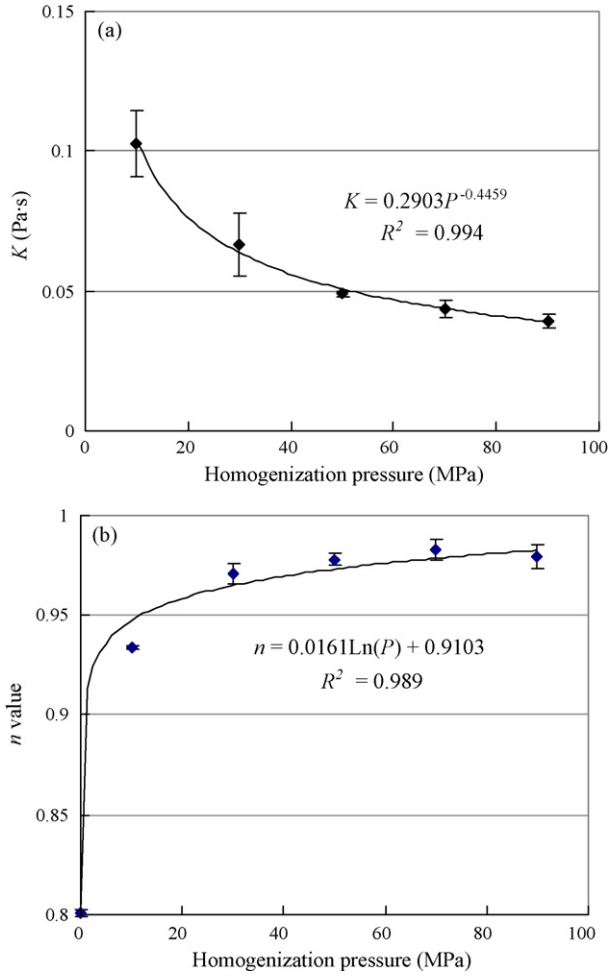

Fig. 4. Effects of homogenization pressure on the parameters in the Power Law model of the apparent viscosity plot of flaxseed gums (plot a for K value, plot b for n value; K, n, and P stand for consistency index, flow behavior index, and the homogenization pressure, separately).

mer degradation (Lagoueyte & Paquin, 1998). The opening of the molecule occurred first, followed by polymer degradation due to mechanical stress.

In order to describe the variation in the flow properties of flaxseed gum solutions under continuous shear, the Power Law model (Eq. (2)) adequately described the experimental data for the flow curves.

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

where τ is the shear stress (Pa), $\dot{\gamma}$ is the shear rate (s^{-1}), K is the consistency index (Pa s^n), and n is the flow behavior index (dimensionless). The K and n values are shown in Fig. 4. As the same trend with the apparent viscosity, the K values decrease along with the homogenization pressure increase. This might be because the high pressure broke the molecule chains of flaxseed gum, thus decreased the interaction between molecules. Similar exponential decay in viscosity was also found along with increase pressure in the high pressure homogenization of xanthan gum solution (Harte & Venegas, 2010). On the contrary, the n values increase along with the homogenization pressure, and the values also incline to one. The response of K values to the homogenization pressure was regressed into a Power Law equation, with R^2 value of 0.994. The response of n values to the homogenization pressure was also regressed into a logarithmic equation, with R^2 value of 0.989. Similar trends and values of K and n versus homogenization pressure were also found in methylcellulose emulsions (Floury, Desrumaux, Axelos, & Legrand, 2003). These two equations could be used not only to describe the tendency of the K and n values, but also to predict the K and n values response to the homogenization pressure that not included in this study.

The frequency dependence of G' and G'' for flaxseed gum solutions can be approximately described, for the frequency range studied, by the following equation (Ikeda & Nishinari, 2001):

$$G' = K' \cdot \omega^{n'} \quad (3)$$

$$G" = K" \cdot \omega^{n"} \quad (4)$$

where K' and K'' are constants and n' and n'' may be referred to as the frequency exponents, and ω is the angular frequency. The value of n' and n'' can provide useful information regarding the viscoelastic nature of food materials (Özkan, Xin, & Chen, 2002).

The Power Law parameters of G' and G'' for the different samples are presented in Table 1. The storage and loss modulus well fitted the Power Law model with the linear regression correlation coefficients (R^2) being higher than 0.909. The results indicate that the high pressure homogenization significantly changed the dynamic shear properties of gum solution compared with untreated samples. The K' values are much higher than K'' values in all the samples, which indicates that these flaxseed gum solutions are predominantly elastic than viscous. The high pressure homogenization had no significant on K' values of flaxseed gum solutions, as shown in Table 1. The K'' values of untreated sample and 0 MPa sample are higher than the others, but the others shows no significant difference between each other ($p < 0.05$). Similar trend is found for n' values, there is no significant difference caused by the different homogenization pressures, but all the processed flaxseed gum solutions have lower n' values than the untreated and the 0 MPa treated samples. The 0 MPa homogenized sample shows highest n'' value of 0.468, while the 90 MPa homogenized sample shows lowest n'' value of 0.335. Only the n'' values of flaxseed gum solutions are comparable with that of 1.5% guar gum gels, which were 0.4293–0.4789, and the K', K'', and n' values are all lower than those of 1.5% guar gum (Sandolo, Matricardi, Alhaique, & Coviello, 2007).

The effects of homogenization pressure on the gelling temperature of flaxseed gum solution was tested and shown in Fig. 5. These temperatures were similar with that of xanthan gum, which was reported to be around 50 °C (Rochefort & Middleman, 1987).

Table 1
Power Law parameters of G' and G'' of flaxseed gum solutions processed by different pressure.[a,b]

Pressure (MPa)	K'	n'	R^2	K''	n''	R^2
Untreated	1.163 ± 0.013^{A}	0.261 ± 0.006^{A}	0.994	0.695 ± 0.052^{A}	$0.426 \pm 0.016^{A,B}$	0.987
0	1.109 ± 0.186^{A}	0.245 ± 0.019^{A}	0.995	0.738 ± 0.06^{A}	0.468 ± 0.012^{A}	0.988
10	1.216 ± 0.187^{A}	0.185 ± 0.015^{B}	0.991	0.613 ± 0.046^{A}	$0.429 \pm 0.029^{A,B}$	0.953
30	0.868 ± 0.201^{A}	0.167 ± 0.012^{B}	0.975	0.376 ± 0.037^{B}	$0.368 \pm 0.029^{B,C}$	0.921
50	0.875 ± 0.21^{A}	0.156 ± 0.01^{B}	0.950	0.427 ± 0.073^{B}	$0.38 \pm 0.037^{B,C}$	0.909
70	0.734 ± 0.034^{A}	0.156 ± 0.009^{B}	0.974	0.338 ± 0.009^{B}	$0.383 \pm 0.011^{B,C}$	0.928
90	0.96 ± 0.13^{A}	0.162 ± 0.012^{B}	0.976	0.376 ± 0.028^{B}	0.335 ± 0.02^{C}	0.925

[a] Values represent the mean ± standard deviation of triplicate tests.
[b] Values in a column with different superscripts were significantly different ($p < 0.05$).

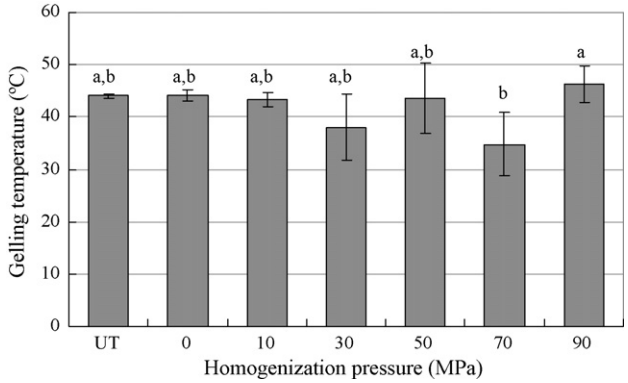

Fig. 5. Effects of high pressure homogenization on the gelling temperature of flaxseed gum (UT stands for untreated sample).

Table 2
Zeta potential and conductivity of flaxseed gum solutions prepared by different homogenization pressure.[a,b]

Pressure (MPa)	Zeta potential (mV)	Conductivity (mS/cm)
Untreated	-50.6 ± 0.4^{B}	0.769 ± 0.007^{A}
0	-51.5 ± 0.6^{B}	0.765 ± 0.006^{A}
10	-53.7 ± 0.5^{A}	0.776 ± 0.005^{A}
30	-51.7 ± 0.6^{B}	0.761 ± 0.005^{A}
50	-51.7 ± 0.4^{B}	0.771 ± 0.005^{A}
70	-48.1 ± 0.5^{C}	0.776 ± 0.005^{A}
90	-44.4 ± 0.7^{D}	0.775 ± 0.006^{A}

[a] Values represent the mean ± standard deviation of triplicate tests.
[b] Values in a column with different superscripts were significantly different ($p < 0.05$).

Although there is some difference on the gelling temperature between the 70 MPa and 90 MPa homogenized samples, no significant difference is found between the untreated sample and the 0–50 MPa homogenized samples ($p < 0.05$). It means that the high pressure homogenization process only has small effects on the gelling temperature of flaxseed gum solution especially under low homogenization pressure.

3.4. Zeta potential, conductivity, and clarity

The changes on zeta potential of flaxseed gum solution after high pressure homogenization are shown in Table 2. The zeta potential of the flaxseed gum solution was first increased when low pressure was applied, then decreased from 10 MPa with the increasing homogenization pressure. The highest zeta potential value was found in 10 MPa homogenized sample. This trend is similar with the trend of activation energy changes during homogenization, since both zeta potential and activation energy are parameters that describe the stability of the solution. So the decrease of molecule chain size of flaxseed gum caused by high pressure (>10 MPa) is probably the reason that leads to the decrease of zeta potential. Since short chains are easier to be involved in chemical reaction than the long ones. High absolute values of zeta potential mean better stability because of the mutual repulsion between the electrical double layers of macromolecules (Acedo-Carrillo et al., 2006). On the other hand when the solutions have low absolute zeta potential value then there is no force to prevent the molecules coming together. An increase in zeta potential was also found in homogenization of milk, when pressure of higher than 30 MPa was applied (Michalski & Januel, 2006). As shown in Table 2, no significant change was found among the conductivity values of the flaxseed gum solutions homogenized under different pressure ($p < 0.05$).

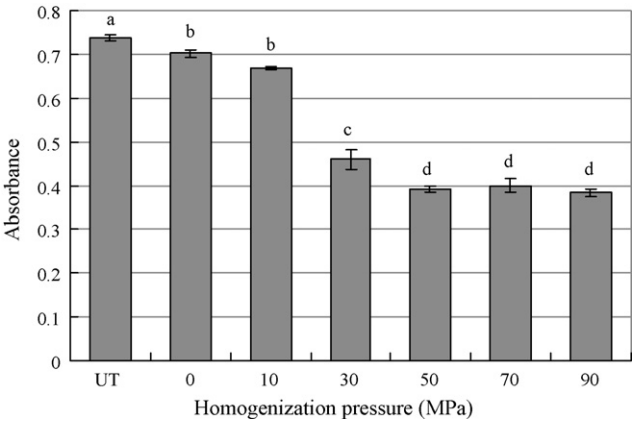

Fig. 6. Effects of high pressure homogenization on the clarity of the flaxseed gum solutions (UT stands for untreated sample).

The clarity of the flaxseed gum solution was represented by the absorbance of the solution in this study, which was shown in Fig. 6. The absorbance of the flaxseed gum solutions was gradually decreased along with the increase of the homogenization pressure, which reflected that the clarity of the solution was increased by the homogenization process. Even the zero pressure homogenization decreased the absorbance of the solution, because of the mechanical pressure applied to deliver the sample inside the homogenizer. The most significant change of absorbance was found between the 10 MPa and the 30 MPa homogenized samples, which meant that the clarity of the flaxseed gum solution was mostly changed during this pressure region. After that, the samples showed no significant difference among the samples with pressure of 50–90 MPa ($p < 0.05$). Similar method has been used in evaluating the clarity of starch paste, the most significant change in absorbance was found around 20 MPa (Che et al., 2009).

4. Conclusion

The effects of high pressure homogenization on the rheological properties of flaxseed gum solution were investigated in this study. The temperature of the solution was gradually increased by the homogenization process. The activation energy of solution first increased then decreased by increasing the homogenization pressure, with the peak reached at 10 MPa. The apparent viscosity of the flaxseed gum solution was significantly decreased along with the increase of homogenization pressure. The relation of shear stress and shear rate of the homogenized gum solution was modeled using a Power Law function. And the K and n values in this Power Law equation were further modeled to predict the flow behavior of flaxseed gum solution during homogenization. The frequency dependences of G' and G'' for flaxseed gum solutions were also modeled with a Power Law function. The homogenization processes were proved to have no significant effects on the gelling temperature or conductivity of the flaxseed gum solution. Similar to the activation energy, the zeta potential of the gum solution was first increased then decreased while the homogenization pressure increased, with the peak reached at 30 MPa. The clarity was gradually increased by the homogenization, but no significant effect was found by increasing pressure after 50 MPa. These results are useful information for the usage of flaxseed gum in present of strong mechanical changes.

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