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Rheological properties of barley β-glucan

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Abstract

Health benefits of cereal β-glucan are linked to its high viscosity. Although viscosity of β-glucan gum solutions has been reported previously, there are conflicting reports about its behavior at elevated temperatures. Therefore, the viscosity behavior of barley β-glucan gum obtained in a pilot plant (PP) or in a laboratory (LAB) was determined at different shear rates (1.29–129 s^{-1}) and temperatures (0.1–75 °C) in this study. Viscosity decrease with temperature was demonstrated for both gums and activation energy E_a was calculated from the Arrhenius equation. None of the fresh gum solutions exhibited thixotropic behavior at $\leq 1\%$ (w/w) concentration, but the measurement demonstrated that increased shear rate is not applicable to polymer solutions of low viscosity. Information about rheological properties of β-glucan will lead to better understanding of its behavior under physiological and processing conditions.

Keywords: Activation energy; Barley; β-Glucan; Rheology; Thixotropy; Viscosity

1. Introduction

β-Glucan is a trivial name for the glucose polymer found in the endosperm cell walls of barley and oats, usually at a level of 3–7% of grain weight. Structurally, it is $(1 \rightarrow 3)$ $(1 \rightarrow 4)$-β-D-glucan with cellulose-like portions linked through $(1 \rightarrow 3)$ glycosidic bonds, which makes it more soluble. The β bond is not digestible by enzymes in human gastrointestinal tract, resulting in the classification of β-glucan as a soluble dietary fiber. Native β-glucan has high viscosity that is related to its health promoting properties. This high native viscosity is very often degraded during extraction and further processing of β-glucan. Wood et al. (1994) found a significant inverse relationship between the glycaemic and insulin response and the logarithm of oat β-glucan viscosity. Since viscosity is a function of molecular weight and concentration, a similar relationship was confirmed between plasma glucose and the logarithm of concentration times molecular weight (Wood, Beer, & Butler, 2000).

Barley and oat β-glucan (BBG and OBG, respectively) show similarities in structure, both having about 90% cellotriosyl and cellotetraosyl units. Oat β-glucan usually exhibits somewhat higher viscosity due to its longer molecular chains (Beer, Wood, & Weisz, 1997; Wood, Weisz, & Mahn, 1991), but it may be expected that both β-glucans would behave alike at similar molecular weights. Thus, the following discussion highlights rheological properties of both β-glucan gums.

The pseudoplasticity of β-glucan gums is already an established fact with high viscosity (HV) gums having a high consistency coefficient and low ($\ll 1$) flow behavior index (parameters of the Power Law equation, Eq. (1)). Bhatty (1995) and Burkus (1996) reported a flow behavior index >0.7 for both BBG and OBG gums. Flow behavior index values closer to one implies good fat replacing properties (Szczesniak & Farkas, 1962). However, Autio, Myllymaki, and Malkki (1987) found that very high viscosity oat gum at concentrations $\geq 1\%$ (w/w) had a flow behavior index <0.3, which means that the recommended concentration of such gums for potential fat replacement may be $\leq 0.5\%$. Low viscosity (LV) gums and HV gums at low concentrations behave like Newtonian fluids (Burkus, 1996). The concentration of HV gums at which they exhibit pseudoplasticity may be as low as 0.2% (Autio et al., 1987).

Temperature causes reversible changes in the viscosity of β-glucan gums (Dawkins & Nnanna, 1995).

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Generally, viscosity decreases with temperature, but the expected viscosity decrease reportedly may show some deviation for BBG with increasing temperature. For example, Dawkins and Nnanna (1995) found that the viscosity of OBG gum (0.5%, w/w) increased with temperature from 25 to 37 °C, while viscosity at 61 °C was slightly below that of the control at 25 °C. At 100 °C, viscosity was much lower than that at 25 °C. Gomez, Navarro, Garnier, Horta, and Carbonell (1997) determined the viscosity of 1.5% BBG solution at low shear rates ($<3\ \mathrm{s}^{-1}$) and 70 °C as higher than that at 25 °C. While their conclusion about β-glucan aggregation at high temperatures is somewhat questionable, some viscosity deviation due to structural changes of β-glucan micelles may be possible. Therefore, there is a need for further elucidation of BBG behavior at elevated temperatures, especially under conditions that may have industrial processing implications.

Thixotropy is the decrease in viscosity under constant shear rate over a period of time. Thixotropy of BBG from beer was investigated by Linemann and Kruger (1997). The procedure applied was flow curve determination where shear rate was increased from 0 to $120\ \mathrm{s}^{-1}$ and then decreased back to $0\ \mathrm{s}^{-1}$ in 8 min. While quick gelation of the hydrolyzed beer BBG in the samples of Linemann and Kruger (1997) and dispersion of gel particles upon shearing cannot be completely precluded as a source of thixotropy, inertia of the measurement system may also contribute to such behavior. Inappropriateness of flow curve measurements for such viscosity tests was already demonstrated by Burkus (1996); therefore, the only relevant test would be a step-wise test, which is in agreement with Speers (1999). Linemann and Kruger (1997) also found that thixotropy disappeared at 55 °C, which could be due to melting of the micelle structure (Burkus & Temelli, 2000; Morgan & Ofman, 1998), and a drop in viscosity with increased temperature. Conversely, Autio et al. (1987, 1992), Wikstrom, Lindahl, Andersson, and Westerlund (1994), and Wood, Weisz, Fedec, and Burrows (1989) did not observe thixotropy in the behavior of OBG gum.

Therefore, the objectives of this study were: (a) to investigate the effects of temperature and shear rate on the viscosity of laboratory (LAB) and pilot plant (PP) extracted BBG gums and to use the Arrhenius and Power Law equations to describe their respective effects, and (b) to confirm thixotropic behavior of BBG gums.

2. Materials and methods

2.1. Materials

Pilot Plant (PP) and LAB BBG gums were extracted from Bly Blend waxy barley (a mixture of two experimental cultivars, SB89528 and SB89497), as previously described by Burkus (2001) and Burkus and Temelli (2000). β-Glucan isolation procedure is an aqueous alkali extraction at pH 9.4 and 53–55 °C, followed by starch separation, cooking to stabilize the β-glucan extract and digest residual starch, acidic protein precipitation, and precipitation of β-glucan by ethanol. Different processing equipment and longer processing time at every step in pilot plant resulted in higher purity, but lower yield of PP gum.

Table 1
Proximate composition (%, db) of PP and LAB gum

Component	PP gum	LAB gum
β-Glucan	83.3	78.0
Protein	1.5	3.6
Starch	1.2	0.5
Pentosans	1.7	4.1
Ash	4.1	4.1
Lipid	0	0

β-Glucan content of PP and LAB gums (78.9 and 71.1% as is, respectively) was determined according to McCleary and Glennie-Holmes (1985). Proximate composition of these two gums was determined as described by Burkus and Temelli (1998) and presented in Table 1. Starch and pentosans were quantitated acoording to Hashimoto, Shogren, and Pomeranz (1987) and Holm et al. (1986), respectively. Proteins were measured with a Leco Nitrogen Analyzer (FP-428 Leco Corp., St Joseph, MI). Ash content was determined according to Approved Method 08-01 (AACC, 1995).

Molecular weight (MW) of PP and LAB gums was determined by Burkus and Temelli (2003) and found to be 198 and 598 K, respectively. This large difference in MW was the consequence of high shear in pilot plant equipment, mostly in centrifuges used for starch and protein separation.

2.2. Rheological measurements

β-Glucan gum solutions were prepared in duplicate in the desired concentration (0.25 or 1.0% of gum, w/w) according to Burkus and Temelli (1998) using distilled water from the local supply. Viscosity was determined in duplicate for each solution by consecutive fixed speed tests at shear rates $1.29–129\ \mathrm{s}^{-1}$ using a PAAR Physica UDS 200 rheometer (Glenn Allen, VA) equipped with a Peltier heating system. Tests were performed at the desired temperature (±0.03 °C) using the DG 27 cup and bob geometry with double gap and a 7 mL sample size. Sample size was not measured by volume but by weight. The clean DG 27 cup was placed on the balance, tared and 7.01±0.005 g of sample was weighed directly into the cup. In order to prevent evaporation during high temperature testing, the sample was covered with approximately 0.4 ml of low viscosity S3 standard oil (3.408 mPa s at 25 °C, Cannon Instrument Co., State College, PA). Shear rate data were reported as rpm or, after multiplication by the conversion factor of 1.29, as s^{-1} according to the manufacturers specifications. The instrument was calibrated with S3 standard oil for LV

Table 2
Parameters in the Measurement Window of PAAR Physica UDS 200 rheometer used for the testing of PP and LAB gum thixotropy at 1–3000 rpm (1.29–3870 s^{-1}) and 20 °C

	Interval								
	1	2	3	4	5	6	7	8	9
LAB 1000 rpm									
Rotation (rpm)	1	5	10	20	50	100	1000	1	–
Measuring points, #	7	7	7	7	7	7	20	8	–
Meas. point duration	1 min	12 s	6 s	3 s	3 s	3 s	3 s	1 min	–
Raw values/point	20,000	4000	2000	1000	Automatic	Automatic	Automatic	20,000	–
LAB 3000 rpm									
Rotation (rpm)	1	5	10	20	50	100	1000	3000	1
Measuring points, #	4	7	7	7	7	7	7	20	10
Meas. point duration	1 min	12 s	6 s	3 s	3 s	3 s	3 s	3 s	1 min
Raw values/point	20,000	4000	2000	1000	Automatic	Automatic	Automatic	Automatic	20,000
PP 1000 rpm									
Rotation (rpm)	5	10	20	50	100	1000	5	–	–
Measuring points, #	7	7	7	7	7	20	10	–	–
Meas. Point duration	12 s	6 s	3 s	3 s	3 s	3 s	12 s	–	–
Raw values/point	4000	2000	1000	Automatic	Automatic	Automatic	4000	–	–

measurements and Brookfield Viscosity Standard—Fluid 500 standard oil (482 mPa s at 25 °C, Brookfield Engineering Laboratories, Inc., Middleboro, MA) for HV measurements.

Pseudoplastic behavior was described by the Power Law model (Eq. (1))

$$\mathrm{SS} = \mathrm{c}\ \mathrm{SR}^{n} \tag{1}$$

where SS is shear stress (N/m^2), SR is shear rate (s^{-1}), c is the consistency coefficient and n is the flow behavior index.

The viscosity of 1% (w/w) LAB and PP gum solutions was determined at temperatures ranging from 0.1 to 75 °C. The effect of temperature on viscosity is described by the Arrhenius equation

$$\eta_a = A\ e^{(-E_a/RT)} \tag{2}$$

where η_a is apparent viscosity, A is a constant, E_a is activation energy (J/mol), R is the gas constant (8.314 J/mol K) and T is temperature (K). Activation energy can be determined from the slope ($-E_a/R$) of ln η_a vs. $1/T$ plot. Since E_a has been reported to be both concentration and shear dependent (Autio et al., 1987), E_a was determined over the shear rate range 1.29–25.8 s^{-1}.

Classical thixotropy test is usually performed at a constant shear rate (e.g. 20 s^{-1}) over a period of time (e.g. 15 min). Since none of the samples exhibited viscosity loss in such tests, a different, somewhat more complex but far more informative approach was taken. Thixotropy was investigated by testing viscosity recovery after exposing 1% (w/w) PP and LAB gum solutions and 0.25% (w/w) LAB gum solutions (w/w) to high shear. Preset variables such as time, temperature, shear rate or speed, the number of measurement points and raw values per point, which were entered into the Measurement window of US200-V.2.04 software, are shown in Table 2. One of the advantages of this modified thixotropy protocol is that it is possible to collect data for the evaluation of flow behavior during the same experiment.

3. Results and discussion

3.1. Effect of temperature and shear on viscosity

The viscosity of LAB and PP gum solutions (1%, w/w) as a function of shear rate at temperatures of 0.1–75 °C is shown in Figs. 1 and 2, respectively, and Power Law

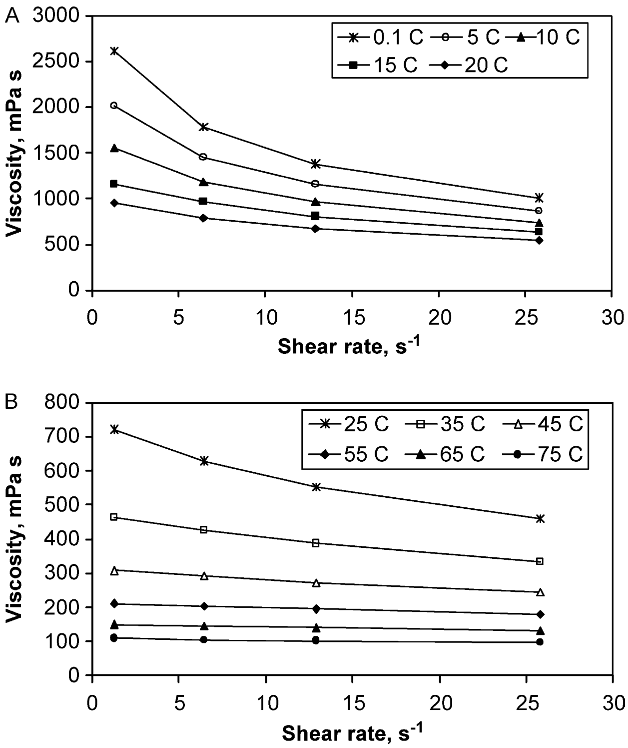

Fig. 1. Viscosity of 1% (w/w) LAB gum solution at 0.1–20 °C (A) and 25–75 °C (B) determined at 1–20 rpm (1.29–25.8 s^{-1}).

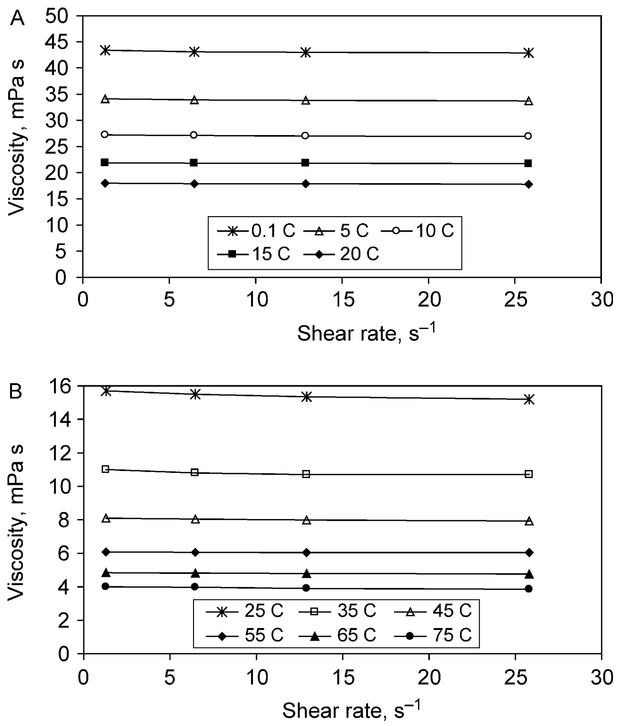

Fig. 2. Viscosity of 1% (w/w) PP gum solution at 0.1–20 °C (A) and 25–75 °C (B) determined at 1–20 rpm (1.29–25.8 s^{-1}).

coefficients for viscosity curves in the range 0.1–75 °C are presented in Tables 3 and 4, respectively. Viscosity behavior of β-glucan solutions over such a wide temperature range has not been reported previously.

As expected, over the whole range of investigated temperatures (0.1–75 °C) (Figs. 1 and 2) for both PP and LAB gum solutions viscosity decreased with increasing temperature without any of the unusual behavior described by Dawkins and Nnanna (1995) or Gomez et al. (1997). A possible reason for the deviation in the behavior described by Dawkins and Nnanna (1995) may be the initial incomplete hydration of HV oat gum and further swelling of β-glucan micelles upon heating to 37 °C. That trend continued up to 61 °C in the data of Dawkins and Nnanna (1995), offsetting the drop in viscosity due to temperature rise, while further heating to 100 °C resulted in a normal viscosity decrease.

Table 3
Power Law constants for 1% (w/w) LAB gum viscosity curves in the range 1–20 rpm (1.29–25.8 s^{-1}) at 0.1–75 °C

Temperature (°C)	Consistency coefficient, *c*	Flow behavior index, *n*	R^2
0.1	2.9503	0.6899	0.9945
5	2.2486	0.7264	0.9952
10	1.7192	0.7607	0.9957
15	1.2704	0.8075	0.9958
20	1.0324	0.8197	0.9968
25	0.7745	0.8571	0.9977
35	0.491	0.8967	0.9984
45	0.3132	0.9364	0.9988
55	0.2176	0.9494	0.9994
65	0.1544	0.9557	0.9998
75	0.1103	0.9659	0.9999

Table 4
Power law constants for 1% (w/w) PP gum viscosity curves in the range 1–20 rpm (1.29–25.8 s^{-1}) at 0.1–75 °C

Temperature (°C)	Consistency coefficient, *c*	Flow behavior index, *n*	R^2
0.1	0.0434	0.9961	1
5	0.0341	0.9961	1
10	0.0272	0.9964	1
15	0.0219	0.9972	1
20	0.018	0.9966	1
25	0.0145	0.9974	1
35	0.0105	0.9955	1
45	0.0078	0.9952	1
55	0.0058	0.9977	1
65	0.0048	0.9945	1
75	0.0039	0.9865	1

Higher viscosity of BBG at low shear rates and 70 °C, as described by Gomez et al. (1997), is more of an anomaly and could be due to experimental error. Whether their instrument was equipped with the Automatic Gap Control (AGC) feature was not indicated. This feature (AGC) automatically adjusts the position of measuring elements that may have changed due to thermal expansion. As well, Gomez et al. (1997) did not indicate taking any precautions to prevent sample evaporation on the cone edge, which is the most probable reason for the deviation. At elevated temperatures, evaporation of β-glucan solution creates a skin-like layer if the solution is not mixed vigorously and kept in a high humidity environment. This skin-like layer probably contributed to higher viscosity readings at low shear rates, while at higher shear it was disrupted and viscosity readings were closer to expected values. Therefore, the sample must be covered with oil for tests at elevated temperatures as well as for tests with a longer time component, even at room temperature, as was done in this study.

The observed reversible drop in viscosity with temperature may be useful in industrial processing of β-glucan since it enables easier pumping and stirring leading to reduced energy consumption. Despite the change in temperature, the flow behavior index was >0.986 for PP gum solutions in the temperature range 0.1–75 °C (Table 4), indicating essentially Newtonian behavior, while for 1% LAB gum solutions flow behavior index increased from 0.69 at 0.1 °C to 0.966 at 75 °C (Table 3), demonstrating transition from pseudoplastic to nearly Newtonian behavior. At concentration of 0.25%, LAB gum exhibited Newtonian behavior with flow behavior index >0.99. Viscosity of PP gum solutions was substantially lower than that of LAB gum due to higher shear rates during extraction in pilot plant.

Arrhenius plot of the viscosity of a 1% LAB gum solution is shown in Fig. 3, while activation energies at different shear rates over the temperature range 0.1–75 °C

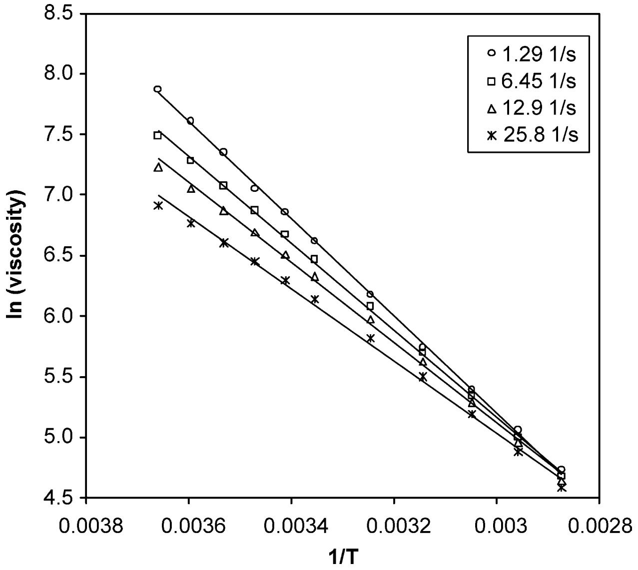

Fig. 3. Arrhenius plot for viscosity of 1% (w/w) LAB gum solution over the temperature range of 0.1–75 °C and at four shear rates (1.29–25.8 s^{-1}).

are presented in Table 5 for both LAB and PP gums. The decrease in E_a with shear rate for HV LAB gum was in agreement with Autio et al. (1987). The value of E_a in this study is somewhat higher, but the shear rate range is much lower than that in Autio et al. (1987) ($E_a = 3.1$ kcal/mol at 92.3 s^{-1}). Pilot Plant gum solution had a constant E_a independent of shear rate, since it exhibits Newtonian fluid behavior (Fig. 2). Good agreement (R^2) with linearity was found. Higher E_a means higher viscosity dependence on temperature changes. The activation energy of β-glucan is concentration and shear dependent (Autio et al., 1987) and both variables should be reported.

3.2. Thixotropy

Thixotropy was not detected for either BBG gum despite the very high shear being applied, which reached approximately 3800 s^{-1} for LAB gum and approximately 1300 s^{-1} for PP gum (Figs. 4 and 5, respectively), which is in agreement with Autio et al. (1987) and Wikstrom et al. (1994). The time required for the recovery of LAB samples from 3000 rpm to the starting viscosity at 1 rpm exceeded 4 min without any resting, which confirms the necessity for step wise measurements, especially for HV samples. Pilot Plant gum recovered its original viscosity almost instantly (in <12 s), which is necessary for the next measurement point at 5 rpm. Therefore, the conclusion of Linemann and Kruger (1997) about thixotropy of beer β-glucan should be accepted with great caution and preferably called gel or network destruction. Their findings should be confirmed using step-wise measurements.

Table 5
Activation energy and coefficients calculated from Arrhenius plot for 1% PP and LAB gums at 0.1–75 °C

Shear rate (s^{-1})	E_a (kJ/mol)	A (mPa s)	R^2
LAB			
1.29	33.2	1.13×10^{-3}	0.9995
6.45	29.8	3.75×10^{-3}	0.9992
12.9	27.4	8.50×10^{-3}	0.9979
25.8	24.6	21.3×10^{-3}	0.9963
PP			
1.29	25.8	4.75×10^{-4}	0.9956
6.45	25.8	4.66×10^{-4}	0.9959
12.9	25.9	4.44×10^{-4}	0.9963
25.8	26.0	4.33×10^{-4}	0.9964

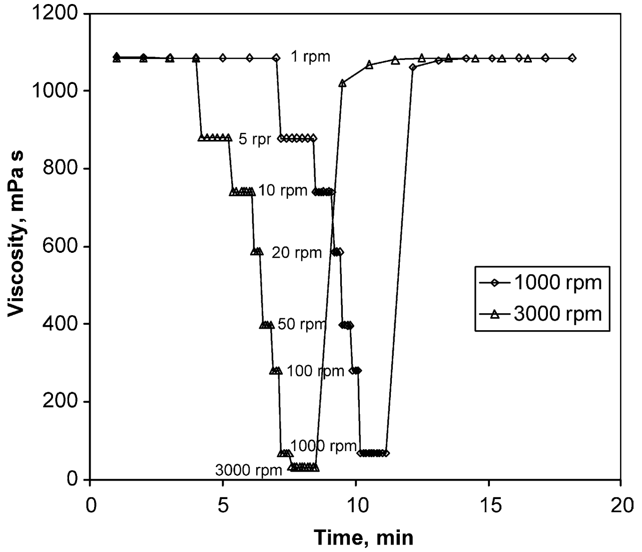

Fig. 4. Thixotropy of 1% (w/w) LAB gum solution after shearing at 1000 or 3000 rpm at 20 °C.

Neither PP gum at 1% nor LAB gum at 0.25% concentration could be tested for thixotropy at 3000 rpm (Fig. 6). Laboratory gum (0.25%) was apparently more viscous already at 1000 rpm. When shear rate was adjusted to the initial value of 5 rpm (6.45 s^{-1}), viscosity curves for both samples shot upward, probably because of low friction in the sample and the occurrence of turbulent flow when

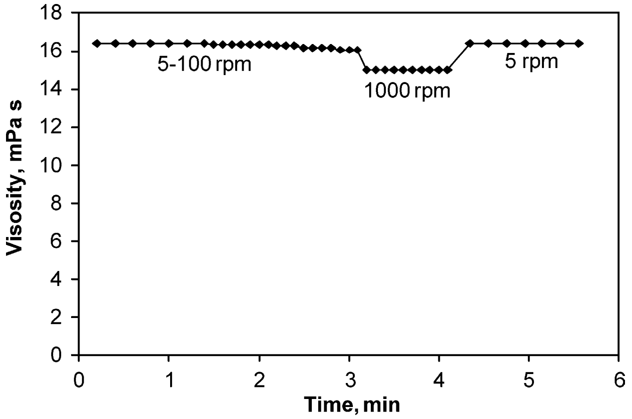

Fig. 5. Thixotropy of 1% (w/w) PP gum solution after shearing at 1000 rpm and 20 °C.

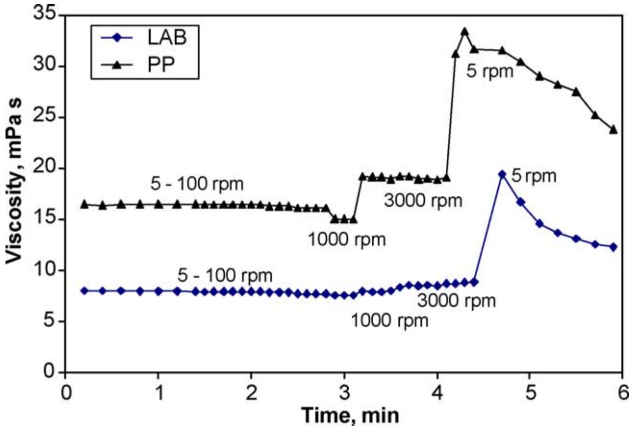

Fig. 6. Thixotropy of 1% PP gum and 0.25% LAB gum solutions after shearing at 1000 and 3000 rpm and 20 °C.

the shear rate was increased from 1000 to 3000 rpm or was abruptly slowed down. This attempt to test thixotropy under these conditions gave some insight into proper determination of viscosity. Obviously, LV samples have an upper shear rate limit at $<1000\ \mathrm{s}^{-1}$ for proper viscosity determination. For samples that have even lower viscosity, (e.g. close to that of water, ~1 mPa s) upper shear rate limit is approximately $100\ \mathrm{s}^{-1}$ when using the double gap geometry for rheological measurements. Therefore, some other types of viscometers that produce shear rate in excess of $100\ \mathrm{s}^{-1}$ may be giving erroneously high readings. This is especially important if the objective is to determine intrinsic viscosity. Even a small error in the range of 0.1–0.2 mPa s would lead to a greatly increased value of reduced viscosity due to the small denominator representing a low solute concentration. In the end, it leads to overestimation of intrinsic viscosity.

4. Conclusions

High viscosity LAB gum at 1% (w/w) concentration is highly pseudoplastic but with a relatively high flow behavior index of >0.8 above 15 °C, which means that HV β-glucan gum may be a good fat replacer. For HV LAB gum, activation energy decreased with increasing shear rate showing lower temperature dependence of viscosity at increased shear rates. Conversely, PP gum was Newtonian with a flow behavior index >0.986 for temperatures of 0.1–75 °C. The activation energy for PP gum was independent of shear rate.

Fresh solutions of β-glucan were not thixotropic at either 0.25 or 1% concentration. Low viscosity β-glucan solutions have a shear rate limit after which viscosity readings become higher than true values, probably due to turbulent flow. This may be important for proper determination of intrinsic viscosity since this shear rate limit may be exceeded in certain types of viscometers.

This study does not confirm some of the previously reported anomalous results that may be due to incomplete hydration of β-glucan samples and problems during rheological testing. For accurate rheological measurements, complete hydration of the gum tested, e.g. ≥30 min at 80–85 °C for β-glucan is absolutely essential. The occurrence of lumps or any visible particle requires even longer hydration time. Evaporation due to increased temperature or longer time tests and possible formation of a skin-like layer should be prevented by keeping the sample in a high-humidity environment, or by covering with a layer of a light mineral oil of low viscosity, which is a more reliable approach. Since the inertia of the measuring system or of the sample may create a delay in stable viscosity reading, step-wise viscosity measurement is absolutely essential.

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