

Effect of particle size and temperature on rheology and creep behavior of barley β -D-glucan concentrate dough

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ABSTRACT

Concentrated β -D-glucan has been added in the formulation of food products development that attributing human health. The purpose of this study is to assess the role of particle size (74, 105, 149, 297 and 595 μ m) of barley β -D-glucan concentrate (BGC) on two fundamental rheological properties namely oscillatory rheology and creep in a dough system (sample to water = 1:2). The water holding capacity, sediment volume fraction and protein content increased with an increase in particle size from 74 μ m to 595 μ m, which directly influences the mechanical strength and visco-elasticity of the dough. The dough exhibited predominating solid-like behavior (elastic modulus, G' > viscous modulus, G''). The G' decreased systematically with increasing temperature from 25 to 85 °C at the frequency range of 0.1–10 Hz except for the dough having particle size of 105 μ m, which could be associated with increase in protein content in the fraction. A discrete retardation spectrum is employed to the creep data to obtain retardation time and compliance parameters which varied significantly with particle size and the process temperature. All those information could be helpful to identify the particle size range of BGC that could be useful to produce a β -D-glucan enriched designed food.

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1. Introduction

People become health conscious and they choose food accordingly. Fiber-rich foods are in great demand that is associated with reduced risks of developing chronic diseases such as colon cancer, atherosclerosis, diabetes, hypertension, and obesity (Anderson, Smith, & Guftason, 1994; Tinker, Schneeman, Davis, Gallaher, & Waggoner, 1991). Cereal-derived (1 $\rightarrow$ 3)(1 $\rightarrow$ 4)- β -D-glucans are linear polymers of β -D-glucopyranosyl (β -D-Glcp) residues inter-linked via a mixture of 1 $\rightarrow$ 3 and 1 $\rightarrow$ 4 linkages and consists of consecutively (1 $\rightarrow$ 4)-linked β -D-Glcp residues in blocks that are separated by single 1 $\rightarrow$ 3 linkages (Moschakis, Lazaridou, & Biliaderis, 2014; Tosh, Brummer, Wood, Wang, & Weisz, 2004; Wood, 2007). (1 $\rightarrow$ 4)-linked sequences are mostly 3 or 4 glucose units long but sequences up to 13 in water soluble and up to 20 in water insoluble β -D-glucans have been reported (Izydorczyk, Macri, & MacGregor, 1998a, 1998b). β -D-Glucan – a soluble fiber is effective in attenuating postprandial glycemic and insulin responses and lowering blood cholesterol levels (Brownlee, 2011; Panahi, Ezatagha, Temelli, Vasanthan, & Vuksan, 2007; Wood,

2007). The Food Drugs Administration (FDA, 2005) and European Food Safety Authority (EFSA, 2010) have accepted a health claim in reduction of cholesterol levels, recommending an intake of 3 g of β -D-glucans per day to achieve the target. In order to achieve the beneficial effects of β -D-glucans, focus has been given to develop food products rich in β -D-glucan content.

Barley β -D-glucan is regarded as a non-starch polysaccharide, and it is currently sold in the concentrated form intended to use as a dietary supplement. The concentrated form is produced either by dry milling and sieving technique (Izydorczyk, Jacobs, & Dexter, 2003; Knuckles, Chiu, & Betschart, 1992) or by wet milling, sieving and solvent extraction (Bhatty, 1995; Cavallero, Empilli, Brighenti, & Stanca, 2002; Oste Triantafyllou, 2002). In fact, β -D-glucan concentrate (BGC) contains significant amount of proteins (hordein and prolamines) (Mohamed, Hojilla-Evangelista, Peterson, & Biresaw, 2007) and starch in addition to desired β -D-glucan content. In such a complex food system, β -D-glucan functionality may be affected by interactions or incompatibilities between β -D-glucan and other food constituents (Satrapai & Supphantharika, 2007). Therefore, a detailed knowledge about structure/function relationships of β -D-glucan containing systems is sought in order to exploit the real health beneficial properties of this valuable dietary fiber (Mikkelsen et al., 2010).

Mostly, β -D-glucan rich fractions or concentrates have been incorporated into breakfast cereals, bakery products (e.g., bread,

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muffin), pasta, and noodles. BGC incorporated dough significantly affect the functional properties and rheology during mixing and processing since β -D-glucan is considered as one of the best hydrocolloids for its gelling capacity and ability to impart the viscosity of aqueous solutions. The molar mass of a β -D-glucan polysaccharide plays an important role in determining the physicochemical properties especially viscosity (Ebringerová, Hromádková, & Heinze, 2005). Both oscillatory and steady flow rheology indicate that β -D-glucans solutions are non-interacting disordered polysaccharides with chain entanglements in the concentrated state. Flow behavior of β -D-glucans solutions has been studied extensively (Burkus & Temelli, 2005; Lazaridou, Biliaderis, & Izydorczyk, 2003), and it was found that the aqueous solution exhibited pseudoplastic behavior at high concentrations and Newtonian behavior at low concentrations (Agbenorhevi, Kontogiorgos, Kirby, Morris, & Tosh, 2011). Particle size has significant effect on rheology and functionality of BGC since different particle fractions have different constituents and they impart the rheology differently. However, there is a lack of information on the functionality and rheological behavior of BGC and the contribution of individual particle size into rheology and viscoelasticity.

The objectives of this present work were to evaluate the effect of (i) the particle size on functional properties of BGC from barley and (ii) subsequent effects of these fractions on the oscillatory rheology, creep behavior as function of temperature, and thermal properties.

2. Materials and methods

2.1. Materials

Barley β -D-glucan concentrate (BGC) was procured from Grain-Frac Consulting, Edmonton, Canada. According to the manufacturer, BGC was produced by an improved method of milling and sieving technology. The β -D-glucan content and starch content of the sample were provided by the manufacturer which were 20% and 28%, respectively as determined by the mixed-linkage β -D-glucan assay kit (Megazyme International Ireland Ltd., Wicklow, Ireland), and Starch assay enzyme kit (Megazyme International Ireland Ltd., Wicklow, Ireland). The molecular weight of the sample was 1030 kDa.

2.2. Sieve analysis

BGC samples were passed through a series of U.S. Standard sieve numbers 20, 30, 50, 100, 140, 200 and 230 mesh (Endecotts, London, UK), manually following the method described by Ahmed, Al-Foudari, Al-Salman, and Almusallam (2014). The fractions obtained from those sieve analysis retained by the sieve were designated as 841 (−20; +30), 595 (−30; +50), 297 (−50; +100), 149 (−100; +140), 105 (−140; +200), and 74 (−200; +230) μ m. The −ve sign represents BGC particles passed through the sieve and the retained particles are expressed through +ve sign. Fraction yields were calculated. Fractionated samples were packed in air-tight containers and stored at 5 °C till further use. The fractionation process was performed in triplicate.

2.3. Physico-chemical properties

The moisture, ash, crude fat and crude protein ($N \times 6.25$) contents of BGC (as dry basis) were determined in triplicate according to the methods of American Association of Cereal Chemists (2000). The ash and crude protein ($N \times 6.25$) for individual fractions were also determined to assess the effect of size separation. Similarly, β -D-glucan was determined for each particle fraction as per method described by McCleary and Glennie-Holmes (1985) using the mixed-linkage β -D-glucan assay kit (Megazyme International

Ireland Ltd., Wicklow, Ireland). The loose bulk density of each sieved fraction was determined by weighing the mass of the sample which freely was poured in a 100 mL graduated cylinder and expressed as weight per unit volume (kg/m^3) (ASTM D7481-09). The volume of bulk aggregate material includes the volume of the individual particles and the volume of the voids between the particles.

2.4. Water holding capacity and water solubility index

The water holding capacity (WHC) of each fraction of BGC sample was determined according to the method described by McConnell, Eastwood, and Mitchell (1974) at 25 and 70 °C, respectively. Briefly, 100 mg samples were transferred to 10 mL of deionized water in a 50 mL centrifuge tube and stirred for 6 h at 25 °C. The mixture was centrifuged at $14,000 \times g$ for 15 min and the supernatant was carefully decanted in a pre-weighed evaporating dish and subjected to dry at 105 ± 1 °C. The evaporating dish with residue was weighed further. The WHC was expressed as the volume of water held in mL/g dry matter of sample analyzed. Corrections were made for evaporation losses by measuring water uptake without sample. The water solubility index (% WSI) was calculated as the dry residue weight to original dry sample weight multiplied by 100. All measurements were performed in triplicates.

2.5. Determination of sediment volume fraction

The volume fraction of the BGC of different particle sizes was measured using a simple centrifugation method (Ahmed et al., 2014; Hemar, Lebreton, Xu, & Day, 2011) at 25 and 70 °C, respectively. Simply, 1 g of flour was dispersed in 20 mL deionized water in a graduated centrifuge tube, mixed well in a vortex and kept for 6 h for hydration followed by centrifugation (Beckman GS-6R, USA) at constant centrifugation force ($3000 \times g$) for 60 min.

After centrifugation, the total height H_T of the sample and the height of the sediment H_S were measured and the effective volume fraction, ϕ occupied by the PF particles was expressed as:

$$\phi = \frac{H_S}{H_T} \times 100 \quad (1)$$

Eq. (1) is valid only for closely packed particles without interstitial space and without damage during the packing. The volume fraction determination was performed at least in duplicate.

2.6. Tristimulus color measurement

Visual color was measured using a Hunter colorimeter model ColorFlex (Hunter Associates Laboratory, Reston, VA) in terms of L (lightness), a (redness and greenness) and b (yellowness and blueness) as following the method adopted by Canadian Grain Commission (2012) for cereal flour color measurement. The instrument ($45^\circ/0^\circ$ geometry, 10° observer) was calibrated with a standard black and white tile followed by measurement of samples. A glass cell containing the BGC sample was placed above the light source and L , a and b values were recorded. Color measurements were taken in triplicates, and average values were taken for calculation.

2.7. Rheological measurement

Oscillatory rheological and creep measurements of BGC (whole and individual fractions) samples were carried out using a Discovery Hybrid Rheometer HR-3 (TA Instruments, New Castle, DE, USA). Samples were prepared by mixing required volume of water to BGC and particle fractions (2:1 water to BGC sample), and kept for 1 h before rheological measurement at a controlled relative humidity chamber. Samples were placed in a 1500- μ m gap between two

stainless steel parallel plates (plate diameter 40 mm). The sample perimeter was covered with a thin layer of high-temperature-resistant silicone oil to prevent sample dehydration. The sample temperature was controlled by a peltier system and monitored by platinum resistance thermometer sensors (accuracy of $\pm 0.1^\circ\text{C}$) which are positioned at the upper and lower plates. The studied temperature range for oscillatory measurement was $25\text{--}85^\circ\text{C}$.

Small-amplitude oscillatory strain sweep experiments (0.001–10%) were performed, and the elastic (G') and viscous (G'') shear moduli, at a constant frequency of 0.1 Hz were monitored to determine the limit of the linear visco-elastic region (LVR). The LVR was carried out for the entire studied temperature range (data are not shown), and the measurement was carried out accordingly. Frequency sweep tests (0.01–10 Hz) were carried out in the linear regime, at constant strain (0.01) at selected temperatures. Following an initial equilibration of samples for 5 min at 10°C , ramp heating was carried out at $5^\circ\text{C}/\text{min}$ to an endpoint of 95°C (non-isothermal heating) at a frequency of 1 Hz.

Creep tests were performed at 25°C by applying an instantaneous shear stress of 20 Pa (within the LVR) for 300 s on the sample, followed by a recovery phase where the stress was suddenly removed and the sample was allowed for 600 s to recover the elastic (instantaneous and retarded) part of the deformation. To study the effect of temperature on creep behavior, only creep tests were conducted at selected temperatures: 25, 40, 55, 70 and 85°C for duration of 180 s. Creep data are presented in terms of creep compliance, J , which is defined as the strain divided by the stress applied (maintained constant during the creep test). The experimental data from creep tests can be described with the precision by a discrete retardation spectrum $\{J_k/\tau_k\}$, which is a set of $2n$ positive constants J_k and τ_k , by means of the following equation (Kaschta & Schwarzl, 1994) which is similar to the 4-parameter Burgers model:

$$J_c(t) = J_0 + \sum_{k=1}^n J_k [1 - e^{-t/\tau_k}] + \frac{t}{\eta_0} \quad (2)$$

where each retardation time τ_k is associated with a spectral compliance magnitude J_k , J_0 is the instantaneous compliance, and η_0 is the steady-state viscosity.

Similar equations were used for the recovery compliance $J_r(t)$. Since, there is no viscous flow in the recovery phase equations consist only of parameters describing the elastic response after removal of the shear stress. The data from creep tests were described by the following Burgers model:

$$J_r(t) = J_0 + \sum_{k=2}^n J_k [1 - e^{-t/\tau_k}] \quad (3)$$

All rheological measurements were carried out in triplicate and rheological parameters were obtained directly from the manufacturer supplied computer software (TRIOS, TA Instruments, New Castle, DE, USA). The parameters related to discrete retardation spectra described above were computed by TRIOS software changing the discrete number terms.

2.8. Differential scanning calorimetric (DSC) measurement

A differential scanning calorimeter (DSC) (TA Q 2000, TA Instruments, New Castle, DE, USA) was employed to measure the thermal analysis for BGC (whole and individual fractions) dough. The DSC was calibrated with indium and sapphire for temperature and heat capacity calibration. The samples (10–12 mg) were run at a $10^\circ\text{C}/\text{min}$ heating/cooling ramp in heating-cooling cycles in a nitrogen atmosphere (flow rate $50\text{ mL}/\text{min}$). The samples were heated from -20 to 160°C to detect thermal properties including, glass transition temperature (T_g), gelatinization of starch and

protein denaturation temperature in doughs. An empty pan was used as a reference. The DSC measurements were done in triplicate. Instrument software (version 4.5A, TA Instruments, New Castle, DE, USA) provides the onset temperature, end point temperature, and the change of heat flow of the glass transition region. The glass-transition midpoint value was calculated as the average of the onset and end point values and reported as the glass transition temperature.

2.9. Scanning electron microscopy

The microstructure and particle dimension of BGC particles were examined through a scanning electron microscope (SEM) (JEOL, JSM-5410LV, Tokyo, Japan). Each sample was coated with gold in a sputter coater (Structure Probe, West Chester, PA, USA) before being scanned and photographed at various magnifications ($250\times$, $1500\times$ and $4000\times$). Particle size was measured by the software attached to the instrument which allows for detailed (particle length and breadth) measurements. About 50 particles were selected randomly for the particle size measurement.

2.10. Statistical analysis

Results were expressed as mean of triplicate determinations $\pm$ standard deviation. Statistical significance (t -test: two-sample equal variance, using two-tailed distribution) was determined using the Minitab Statistical Software (Version 16; Trial Version; Minitab Corp., USA). Differences at $p < 0.05$ were considered to be significant.

3. Results and discussion

3.1. Sieve analysis

Particles obtained by size reduction of food materials showed difference in composition and functional properties, and therefore, the study of the particle size distribution plays an important role in the function of the desired final product. Furthermore, sieving of ground barley has potential to provide information on β -D-glucan-rich fraction/s that could be used for specific purposes. Particle mass (%) of BGC fraction after size distribution indicated that the maximum weight percent of particles ($\approx 48\%$) were about $297\text{ }\mu\text{m}$ followed by $149\text{ }\mu\text{m}$ ($\approx 32\%$), and $105\text{ }\mu\text{m}$ ($\approx 16\%$). Only 2.5% of the BGC sample passed through fine screen ($74\text{ }\mu\text{m}$) whereas almost 99% of the sample passed through $595\text{-}\mu\text{m}$ screen. The obtained data indicated that the BGC sample had a broader distribution of particle sizes. The results obtained in this work are comparable with the results reported by Izydorczyk, Chornick, Paulley, Edwards, and Dexter (2008) for barley. Authors compared the particle size distribution through roller milling and pin milling of fiber-rich-fraction of barley and found that roller milling produced significant amount (80%) of coarser particles ($150\text{--}300\text{ }\mu\text{m}$) whereas more ($>75\%$) smaller particles ($75\text{--}150\text{ }\mu\text{m}$) were achieved through pin milling. However, Knuckles et al. (1992) reported that more than 70% of particles passed through sieve diameter of $45\text{ }\mu\text{m}$ after dry milling and sieving of β -D-glucan enriched barley fractions.

3.2. Proximate composition

The proximate composition of the BGC sample was moisture content $6.20 \pm 0.05\%$, protein content $16.56 \pm 0.10\%$, fat content $2.36 \pm 0.07\%$, ash content $1.81 \pm 0.02\%$ and crude fiber content $2.39 \pm 0.03\%$. The ash content of each particle fraction varied significantly and ranged between 0.13 and 2.33% (Table 1). The highest value of ash content is observed for particle size of $297\text{ }\mu\text{m}$ and the lowest one was recorded for the finest powder ($74\text{ }\mu\text{m}$). However,

Table 1
Effect of particle size on physico-chemical properties of BGC samples.

	Whole	595 μm	297 μm	149 μm	105 μm	74 μm
Bulk density (kg/m^3)	279 ± 1.5	245 ± 1.7	257 ± 1.5	270 ± 1.6	326 ± 1.8	342 ± 1.6
Ash content	1.81 ± 0.1	0.82 ± 0.0	2.33 ± 0.1	1.30 ± 0.1	1.80 ± 0.1	0.13 ± 0.0
Crude protein content	15.25 ± 0.3	13.10 ± 0.2	14.58 ± 0.3	15.93 ± 0.3	18.20 ± 0.5	20.85 ± 0.4
β -D-Glucan content	19.25 ± 0.4	17.83 ± 0.3	19.67 ± 0.3	18.30 ± 0.2	16.25 ± 0.3	13.17 ± 0.2
WHC (mL/g)						
25 °C	2.58 ± 0.2	3.73 ± 0.1	2.79 ± 0.1	2.59 ± 0.2	2.45 ± 0.1	2.15 ± 0.1
70 °C	4.72 ± 0.2	8.16 ± 0.3	6.60 ± 0.1	5.40 ± 0.2	3.74 ± 0.2	3.29 ± 0.1
WSI (%)						
25 °C	8.88 ± 0.3	10.74 ± 0.4	13.37 ± 0.4	13.56 ± 0.2	13.68 ± 0.2	14.67 ± 0.3
70 °C	22.35 ± 0.4	14.22 ± 0.2	18.77 ± 0.2	18.92 ± 0.3	25.72 ± 0.3	26.39 ± 0.3
Volume fraction (%)						
25 °C	32.0 ± 0.2	36.0 ± 0.3	34.0 ± 0.2	32.0 ± 0.3	30.0 ± 0.3	26.0 ± 0.1
70 °C	40.0 ± 0.2	50.0 ± 0.3	46.0 ± 0.3	44.0 ± 0.2	40.0 ± 0.2	38.0 ± 0.2
Color value <i>L</i>	80.24 ± 1.0	78.13 ± 1.4	81.13 ± 1.1	81.92 ± 1.4	83.10 ± 1.2	85.86 ± 1.1
Color value <i>a</i>	1.89 ± 0.2	1.96 ± 0.1	1.80 ± 0.2	1.95 ± 0.1	2.60 ± 0.2	2.32 ± 0.1
Color value <i>b</i>	8.83 ± 0.7	9.41 ± 0.6	8.31 ± 0.7	9.18 ± 0.8	11.28 ± 1.0	10.87 ± 0.8

the ash content was lower compared to reported values (Bhatty, 1995). Size reduction of BGC produced fractions with enhanced crude protein contents. As a result, the 74- μm fraction had higher protein contents (20.85%) than the remaining fractions and also the starting BGC flour. The extracted crude protein (P) content followed a Power-type relationship with the particle size (S) (Eq. (4)). It is believed that the protein shift keeps a balance between protein-rich fractions and protein-weak fractions, and ultimately the total protein in the starting flour remains same. Similar observation has been reported earlier by Wu, Stringfellow, and Inglett (1994) during separation of barley fractions by sieving and air classification.

$$P = 49.08 \times S^{-0.21} \quad (R^2 = 0.94) \quad (4)$$

3.3. β -D-Glucan contents

Table 1 shows the β -D-glucan contents in BGC flours without sieving and individual fractions. The β -D-glucan contents varied with the particle size. The coarser particles showed an increase in β -D-glucan contents however, the value decreased sharply at and below 105- μm particle size. The fine fraction (74 μm) had lower β -D-glucan contents (13.2%) than other fractions. Similar observation was earlier reported by Wu et al. (1994) for fractionation of barley flour where authors observed a significant drop of β -D-glucan contents from 24.6% to 10.7% when the corresponding sieves were changed from 500 to 64 μm . Furthermore, those authors argued that the shift of β -D-glucan as the sum of the β -D-glucan shifted into the high β -D-glucan fractions and out of the low β -D-glucan fractions as a percentage of the total β -D-glucan present in the original flour. The decrease in β -D-glucan content (BG) with the particle size (S) can be described by a 2nd order polynomial equation (Eq. (5)).

$$\text{BG} = 7 \times 10^{-5}S^2 + 0.053S + 10.82 \quad (R^2 = 0.85) \quad (5)$$

3.4. Bulk density

Reduction of particle size increases the bulk density. Following the rule, the bulk density of sieved GFC fractions increased systematically from 245 to 342 kg/m^3 when the particle size reduced from 595 to 74 μm (Table 1), and followed a power-type relation (Eq. (6)). The bulk density of BGC without sieving was 279 kg/m^3 , which supports the heterogeneity of particle sizes in the sample. The above results confirmed that the particle size had a significant effect on the bulk density ($p \leq 0.05$). Similar decrease in bulk density with particle size has been reported earlier for sweet potato powder and pumpkin flour (Ahmed et al., 2014; Grabowski, Truong,

& Daubert, 2006). It could be reasoned that pore spaces among particles decreased when particle size reduced. Furthermore, the decrease can be explained by considering the stickiness of particles during dehydration and also from product agglomeration (Goula, Adamopoulos, & Kazakis, 2004). Although particles may be small when measured individually, these agglomerates take up a larger volume and, thus, would contribute to a smaller bulk density.

$$\rho_b = 669 \cdot S^{-0.16} \quad (R^2 = 0.85) \quad (6)$$

where ρ_b is the bulk density (kg/m^3) and *S* is the sieve size in μm .

3.5. Water holding capacity and water solubility index

The water holding capacity (WHC) of cereal flours/fibers is an important functional property in various food applications, and furthermore, it has implications for maintaining the product viscosity. Water holding capacity for BGC particle fractions showed a significant variability, and it ranged from 2.15 to 3.73 g/g and 3.29 to 8.16 g/g at 25 and 70 °C respectively (Table 1). The highest WHC among all the samples was found for the coarser particle size (595 μm), and the finest particle (74 μm) showed the least value. The difference in WHC has been shown to be related to the amount of soluble and insoluble fiber within the gel matrix (Robertson et al., 2000). The WHC was significantly influenced by the process temperature irrespective of the particle size. A higher WHC at 70 °C signifies more water holding capability of BGC at elevated temperature which is influenced by thermal transitions (e.g. starch gelatinization) of the constituents. WHC showed a linear relationship with the particle size (S) at 25 °C which was shifted to a logarithmic form at 70 °C (Eqs. (7)–(10)). The water solubility index (WSI) increased with fineness of the particle size and also with temperature (Table 1) and following similar trend of WHC as shown below.

$$\text{WHC}_{25^\circ\text{C}} = 0.003S + 2.07 \quad (R^2 = 0.97) \quad (7)$$

$$\text{WHC}_{70^\circ\text{C}} = 2.38 \ln S - 6.98 \quad (R^2 = 0.98) \quad (8)$$

$$\text{WSI}_{25^\circ\text{C}} = -0.006S + 142.774 \quad (R^2 = 0.88) \quad (9)$$

$$\text{WSI}_{70^\circ\text{C}} = -5.88 \ln S + 51.87 \quad (R^2 = 0.95) \quad (10)$$

3.6. Sediment volume fraction

The volume fraction (ϕ) of suspended particles in aqueous dispersions of PF at 30 and 70 °C is presented in Table 1. The volume fraction measures the particles occupancy after centrifugation, and

it mostly estimates the effective volume fraction of the particles. The coarser particle size (595 μm) exhibited the highest volume fraction whereas the least value was obtained for 74- μm BGC particles irrespective of temperature. Generally, it is expected that the magnitude of volume fraction increases with fineness of the particle size. However, the observed reverse trend could be attributed to cellular structure of BGC and its water holding capacity. It is observed that more water was absorbed by porous particles compared to finer particles. Similar results were earlier observed for pumpkin flour (Ahmed et al., 2014), where the largest hydrated particle is expanded almost two times (i.e. 8 times volume increase based on spherical uniform particle) compared to 1.5 times (volume increase 3.4 times) for smaller one. Raising the temperature to 70 °C, and isothermal heating for 30 min causes significant increase in volume fraction. This observation is contrary to earlier report on pumpkin flour where no significant increase in the volume fraction value was noticed at 70 °C. The difference could be attributed by different tissue structure and individual water hydration capacity. The volume fraction (ϕ) showed a logarithmic relationship with the particle size (S) at 25 °C, 70 °C (Eqs. (11) and (12)).

$$\phi = 5.62 \ln S + 14.35 \quad (R^2 = 0.97) \quad (11)$$

$$\phi = 4.37 \ln S + 8.82 \quad (R^2 = 0.90) \quad (12)$$

3.7. Color of particle fractions

The Hunter color values (L , a , b) of the BGC samples as function of particle size are presented in Table 1. Generally, the increase in the particle specific surface area by size reduction caused intense color values. The L value of the whole BGC sample was 80.24 which dropped to 78.13 for coarser particle size (595 μm). The L value systematically increased as the particle size was reduced from 595 to 74 μm , and the highest L value, 85.86 ± 0.78 , was recorded for the finest particles (74 μm). Among the sieved samples studied, the highest a value (2.60 ± 0.06) was obtained for the sample having particle size of 105 μm , indicating its redder hue. The color b value changed after sieving from the original sample, and the highest b value was recorded for 105 μm particles, indicating its yellower hue compared to the other fractions.

3.8. Thermal properties of particle fractions enriched dough

Thermal analysis of BGC samples revealed that only the sample without sieving exhibited the peak starch gelatinization temperature at 66 °C. For isolated barley starch, a wide range of gelatinization temperature (60–70 °C) has been reported (Bello-Pérez, Sánchez-Rivera, Núñez-Santiago, Rodríguez-Ambríz, & Román-Gutiérrez, 2010; Chavez-Murillo, Wang, & Bello-Perez, 2008). After sieving, the gelatinization peak was not detected for any of the samples, but the protein peak appeared predominantly. DSC thermograms of BGC proteins are illustrated in Fig. 6. The protein exhibited a large endothermic peak ranging between 107 and 116 °C attributed by protein denaturation, which was lower than the reported value of 125 °C (Xia, Wang, & Chen, 2011). The protein denaturation peak temperature increased systematically with decreasing particle size, and the increase in temperature is probably associated with increase in protein concentration in the finer particle fractions as observed in Table 1. The glass transition temperature, T_g provides information of molecular mobility of a material. Finer particles containing dough showed distinct T_g values at 139, 138 and 128 °C for particle size of 74, 105 and 149 μm , respectively. It indicates that finer particles are protein-rich as compared to coarser particles. Similar observation was earlier reported by Mohamed et al. (2007) for barley protein isolate, where authors have reported a T_g value of 153.3 °C at 4% moisture content and

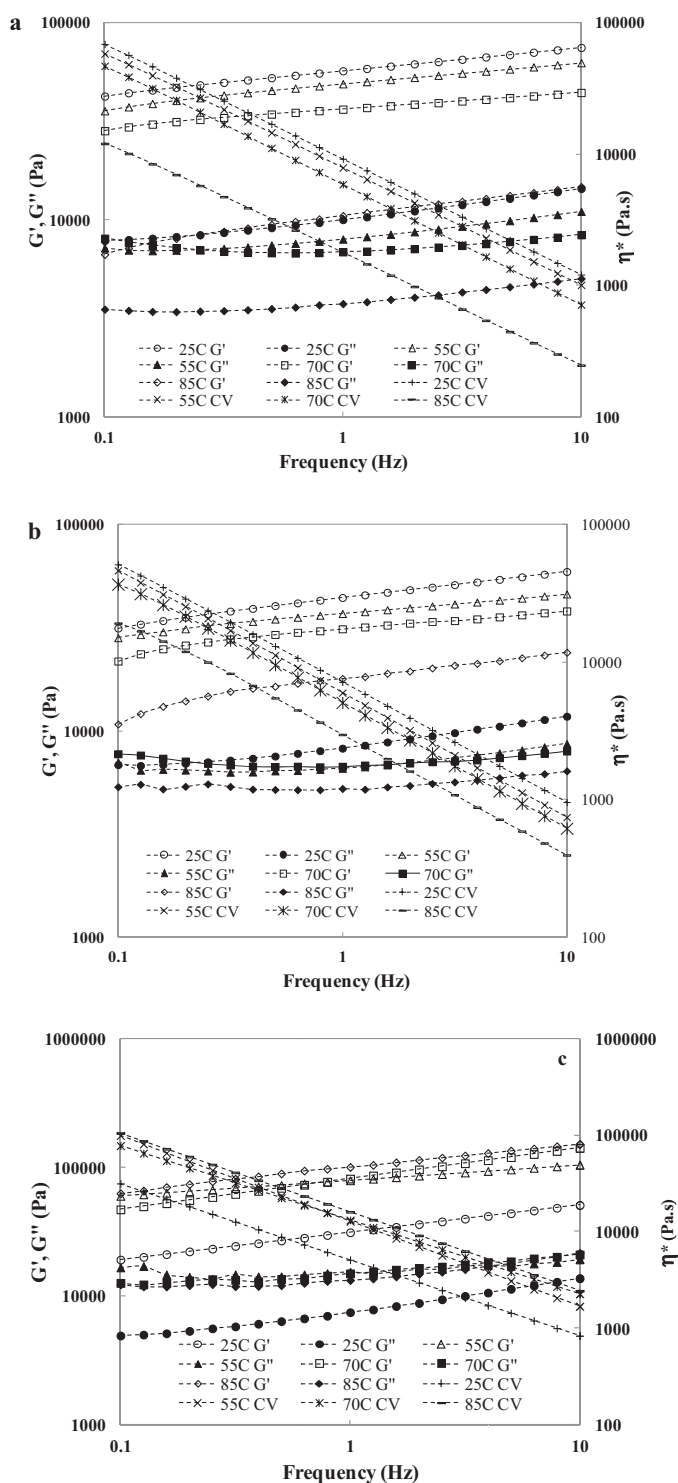


Fig. 1. Rheograms of (a) heterogeneous particles without sieving, (b) 297 μm particle size and (c) 105 μm particle sized BGC dough at selected temperatures.

136 °C when the moisture content increased to 50%. The difference in observed T_g with particle size could be attributed to the constituents (starch and proteins) of the particles itself (Ahmed et al., 2014). Furthermore, it has been reported that the protein after air classification was concentrated in fraction with fine particles and starch was concentrated in fractions with coarser particle sizes (Wu et al., 1994). Vasanathan and Bhatt (1995) studied scanning electron micrographs (SEM) of the coarse and fine barley fractions after

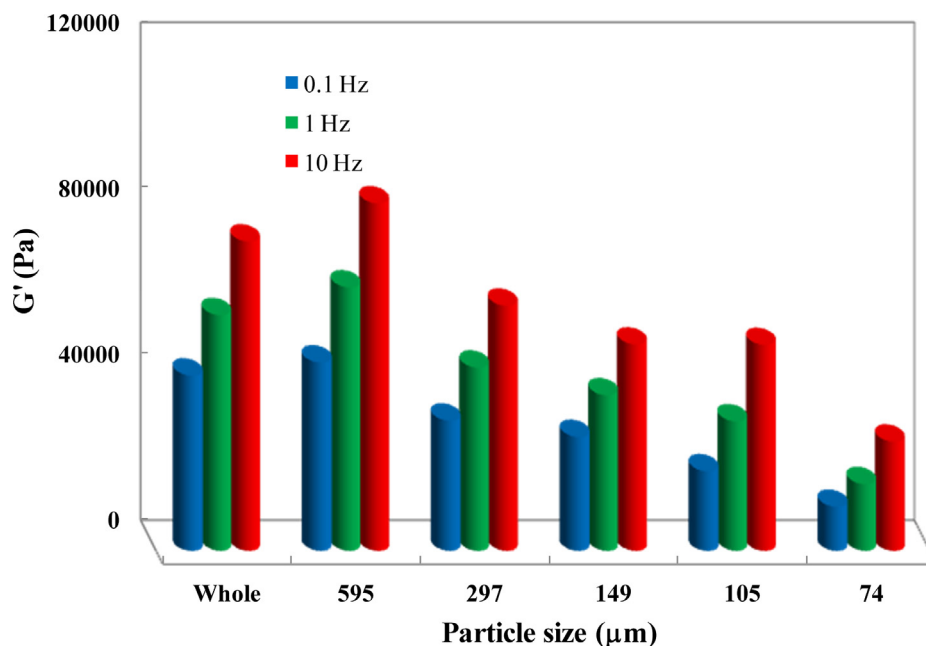


Fig. 2. Effect of particle size on mechanical rigidity of BGC dough at selected frequencies and at 25 °C.

an air classification and reported that most of the small starch granules were concentrated into finer fraction. Furthermore, coarser particles had lower protein and higher starch, and finer particles had higher protein and lower starch. Based on those above information, it could be assumed that both protein rich fraction and smaller starch granules contribute to the increase in T_g .

3.9. Rheology of BGC

The oscillatory experiments of all the BGC samples were measured by performing frequency sweeps in the linear viscoelastic region. Fig. 1a illustrates the rheological behavior of BGC dispersion without sieving. It is apparent that the elastic modulus (G') of the dough exceeds the viscous modulus (G'') significantly over the range of frequency measured (0.1–10 Hz), and confirming a predominant solid-like behavior. For example, the G' value was almost 6 times higher than G'' at 1 Hz and at 25 °C. All dynamic moduli, G' , G'' and η^* changed with frequency systematically. The $G'-\omega$ produces a smoother curve than G'' , which is probably due to the more solid-like characteristic of the dough.

3.9.1. Effect of sieving and particle size

After sieving, BGC sample was fractionated into selected particle sizes that ranged between 74 and 595 μm. Distribution of particle sizes has an important impact on dispersion/dough rheology. The mechanical strength of BGC dough samples at selected frequency range (0.1, 1 and 10 Hz) with different particle sizes at 25 °C are illustrated in Fig. 2 which clearly shows that the coarser particles (595 μm) produced the maximum mechanical rigidity (G') even exceeding the value observed for the sample without sieving (heterogeneous particles), however, the dough rigidity decreased gradually as the particle size was reduced from 595 to 74 μm. It is believed that the coarser particles (595 μm) absorbed the maximum amount of water as supported by the water holding capacity and sediment volume fraction (ϕ) values as mentioned earlier and it maintains its structure in dough during oscillatory measurement. The higher G' values of coarser particles (595 μm) have been influenced by a higher value of ϕ (36) than that of heterogeneous (32) or finer particles fragments (26–32). In addition to those effects, the

particle composition significantly affects the rheological behavior of the dough.

3.9.2. Effect of non-isothermal heating

Non-isothermal heating of BGC samples (individual particle fractions and without sieving) from 25 to 95 °C at a frequency of 1 Hz is presented in Fig. 3. Thermograms of BGC samples showed almost similar trends during temperature ramp experiment, but they differ significantly in the G' value. Dough samples with a particle size range of 149–595 μm showed an improvement in mechanical rigidity as compared to heterogeneous sample whereas the fine particle (74 μm) produced weak dough at similar condition. The G' value decreased sharply as the heating proceeds from 25 to 60 °C thereafter, a peak value was attained in the temperature range of 66–70 °C which finally dropped on further heating. The peak value of each thermogram represents the gelatinization of starch component present in the sample, and it is worth mentioning that the extent of starch gelatinization varied among those

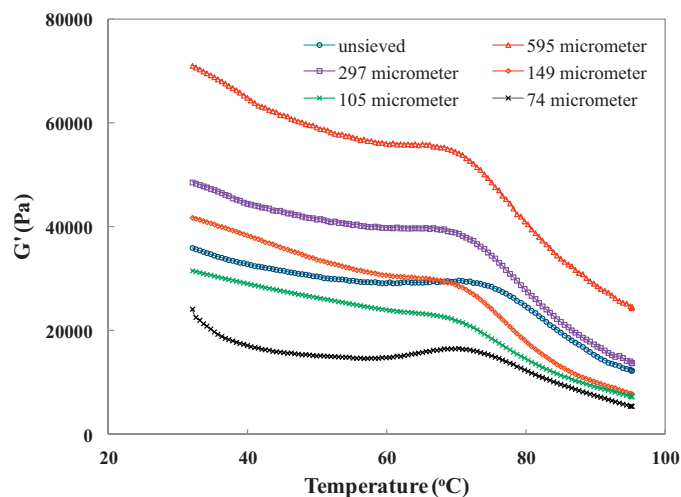


Fig. 3. Effect of temperature on mechanical spectra of BGC dough sample as affected by particle size.

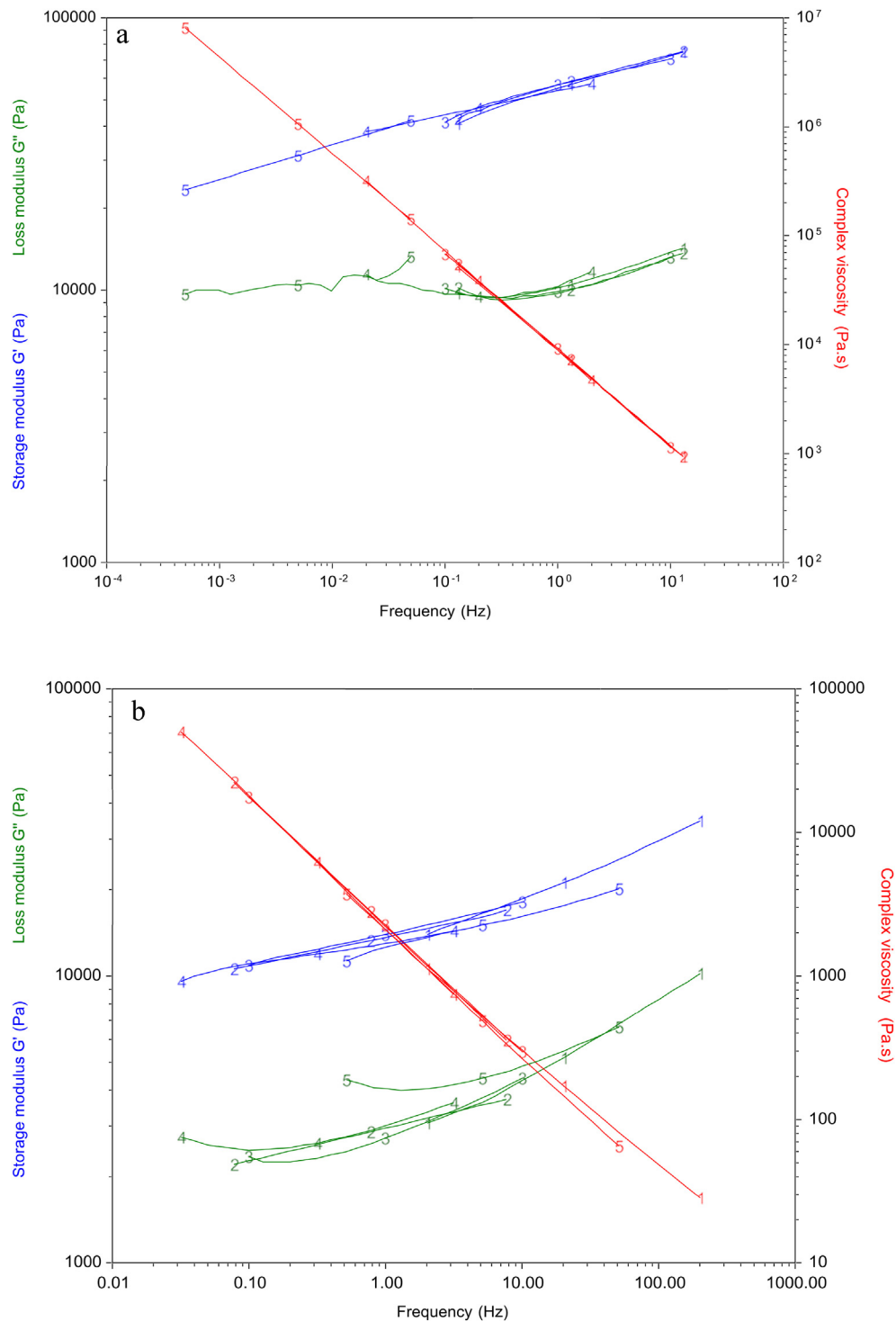


Fig. 4. Time–temperature superpositions of BGC dough (a) 595 μm and (b) 74 μm particles containing. (legends; 1–25 $^{\circ}\text{C}$, 2–40 $^{\circ}\text{C}$, 3–55 $^{\circ}\text{C}$, 4–70 $^{\circ}\text{C}$, and 5–85 $^{\circ}\text{C}$).

particles fractionates. The observed values are in accordance with the reported range of barley starch gelatinization temperatures (60–66 $^{\circ}\text{C}$) (Naguleswaran, Vasanthan, Hoover, & Bressler, 2013). Starch varied among particle fractions. Among particle fractionates, the peak value of the gelatinization curve of 105 μm particles containing dough sample was not sharp, and it could be attributed by the presence of limited amount of starch which was further substantiated by producing a weak gel. The gel strength had not improved even after non-isothermal heating followed by cooling to 25 $^{\circ}\text{C}$ (setback region) which is contrary to most of starch dough

(data not shown). This could be the nature of starch present in BGC under heating-cooling condition and also the material's inability to form a viscous paste or gel by restructuring the network.

3.9.3. Effect of particle size and temperature

Although it has been well established that the temperature affects the rheological properties of food products significantly, reports on particle size effect on food rheology are limited. Effects of temperature on rheology of selected BGC particle sizes are illustrated in Fig. 1. As shown in Fig. 1a (without sieving) and b

Table 2
Effect of temperature on viscoelastic properties of BGC as function of particle size.

	Whole	595 μm	297 μm	149 μm	105 μm	74 μm
Slope, n						
25 °C	0.12 $\pm$ 0.02	0.13 $\pm$ 0.02	0.13 $\pm$ 0.03	0.12 $\pm$ 0.04	0.21 $\pm$ 0.05	0.20 $\pm$ 0.04
40 °C	0.11 $\pm$ 0.01	0.12 $\pm$ 0.04	0.11 $\pm$ 0.02	0.10 $\pm$ 0.04	0.13 $\pm$ 0.02	0.10 $\pm$ 0.00
55 °C	0.12 $\pm$ 0.04	0.11 $\pm$ 0.04	0.10 $\pm$ 0.00	0.11 $\pm$ 0.02	0.12 $\pm$ 0.03	0.11 $\pm$ 0.02
70 °C	0.09 $\pm$ 0.00	0.09 $\pm$ 0.00	0.11 $\pm$ 0.03	0.09 $\pm$ 0.00	0.24 $\pm$ 0.05	0.08 $\pm$ 0.00
85 °C	0.16 $\pm$ 0.03	0.13 $\pm$ 0.02	0.15 $\pm$ 0.02	0.15 $\pm$ 0.02	0.19 $\pm$ 0.03	0.12 $\pm$ 0.01
Intercept, A (Pa s ^{n})						
25 °C	56,670 $\pm$ 5	63,006 $\pm$ 7	43,827 $\pm$ 5	37,459 $\pm$ 4	31,163 $\pm$ 3	17,536 $\pm$ 2
40 °C	50,716 $\pm$ 3	59,160 $\pm$ 5	40,864 $\pm$ 4	34,683 $\pm$ 4	98,420 $\pm$ 5	16,318 $\pm$ 2
55 °C	48,339 $\pm$ 5	55,882 $\pm$ 72	36,680 $\pm$ 5	31,071 $\pm$ 2	78,984 $\pm$ 4	14,003 $\pm$ 3
70 °C	36,062 $\pm$ 2	47,968 $\pm$ 4	30,516 $\pm$ 2	22,104 $\pm$ 3	81,389 $\pm$ 5	12,004 $\pm$ 4
85 °C	10,311 $\pm$ 2	32,306 $\pm$ 4	15,979 $\pm$ 2	5699 $\pm$ 1	99,608 $\pm$ 4	5203 $\pm$ 2

(297- μm particle size), all oscillatory rheological parameters, G' , G'' and η^* of dough samples decreased systematically with increasing temperature from 25 to 85 °C, and exhibited shear-thinning behavior. A significant drop in dough rigidity was observed for both samples while the temperature was increased to 85 °C which probably contributed by release of β -D-glucan in the sample at higher temperature. The rheological behavior of 105- μm particle size dough behaved differently from other two doughs (Fig. 1c). The dough rigidity started to increase gradually at and above 40 °C and such behavior clearly indicates that the composition of this particle fraction was significantly different from other fractions. The increase in rheological moduli with increasing process temperature is most probably influenced by an increase in protein content (18.2% vs 15.3% for sample without sieving). Similar observation was reported by Scanlon, Dexter, and Biliaderis (1988) while studying particle size related physical properties of wheat flour. Those researchers observed that the particle size range of 91–136 μm were protein rich fraction whereas the particle size below 91 μm contained least amount of protein.

The frequency (ω) dependence of BGC dough rigidity (G') can be described by the power-type relationship:

$$G' = A\omega^n \quad (13)$$

$$\text{After linearization, } \ln G' = \ln A + n \ln \omega \quad (13a)$$

where A is a constant and n is the frequency exponent (slope) whose value lies between 0 and 1.

The slope and intercept are calculated from the linear regression of $\ln \omega$ vs $\ln G'$. Table 2 provides magnitudes of slopes and intercepts of BGC particle fractions and sample without sieving as function of temperature. Experimental data were fitted well with R^2 values being always higher than 0.96. It is evident from the table that the temperature had a minimal effect on the solid-like properties from 25 to 55 °C as assessed by the slope, n , of BGC fractions except for the dough having particle size of 74–105 μm where temperature played a significant role by changing the mechanical rigidity. However, the slopes have markedly differed at and above 70 °C which clearly indicates a change in mechanical property of the dough. The most notable change was noticed for 74 and 105 μm particles containing dough from rest of the sample. At 25 °C both samples exhibited low solid-like property with higher n values, and the n value for 105- μm sample showed the peak value at 70 °C and slightly dropped at 85 °C. The intercept, A , of particle size showed a decreasing trend with temperature except for 105 μm .

3.9.4. Time-temperature-superposition

In order to broaden the observations horizon of viscoelastic properties, the method of superposition of time (frequency) and temperature was applied for all BGC samples (sieved and without sieving) to generate the master curves. This is a basic method for data analysis originating from rheological measurements of

synthetic polymers. For thermo-rheological materials, log-log plots of the isotherms of the elastic modulus, (G'), viscous modulus (G''), and complex viscosity (η^*) can be superimposed by horizontal shifts $\log(a_T)$, along the $\log(\omega)$ axis, and vertical shifts given by $\log(b_T)$ such that (Ahmed, Varhney, Auras, & Hwang, 2010; Ahmed, 2012, 2013):

$$b_T G'(a_T \omega, T_{\text{ref}}) = G'(\omega, T) \quad (14)$$

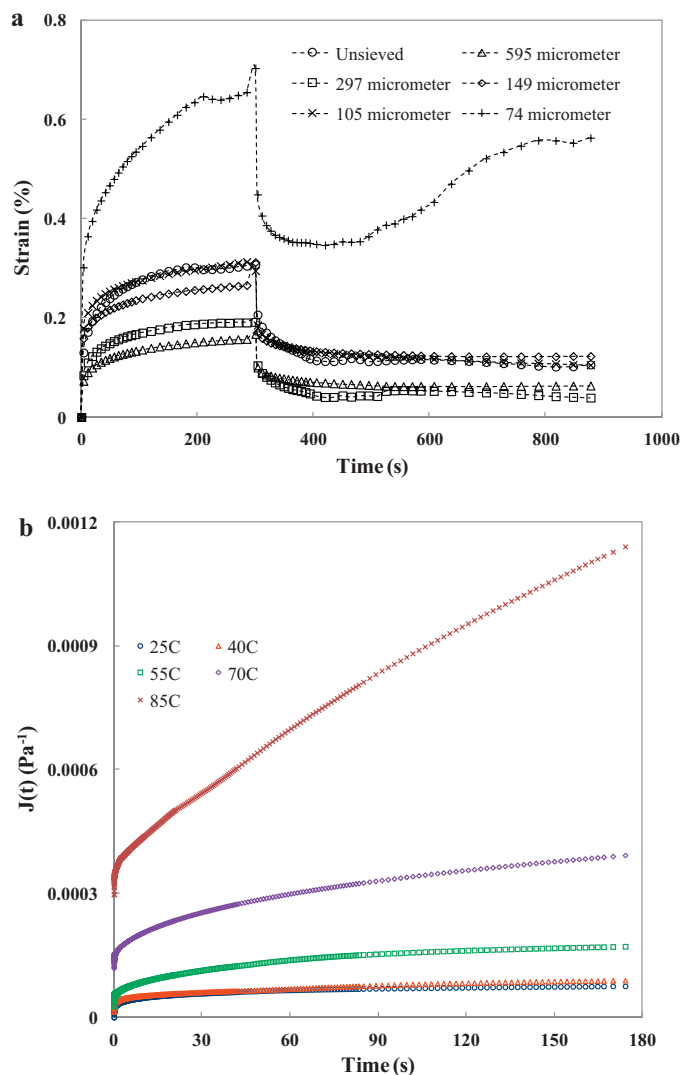


Fig. 5. (a) Creep and relaxation behavior of BGC dough at 25 °C and (b) effect of temperature on creep behavior of dough with 297 μm particles.

Table 3

Creep parameters of BGC and its fractions as function of temperature.

Temp (°C)	Creep					Recovery			
	J_0 (Pa ⁻¹) × 10 ⁻⁴	η_0 × 10 ⁶	J_1 (Pa ⁻¹) × 10 ⁻⁵	τ (s)	R^2	J_0 (Pa ⁻¹) × 10 ⁻⁴	J_1 (Pa ⁻¹) × 10 ⁻⁵	τ (s)	R^2
Whole									
25	0.16	4.25	2.56	0.72	0.91	0.17	3.26	14	0.97
40	0.32	2.92	3.33	2.36	0.95				
55	0.58	1.67	4.12	4.25	0.98				
70	1.55	0.91	7.16	8.14	0.99				
85	3.96	0.17	13.5	6.21	0.99				
595 μm									
25	0.11	2.31	3.00	2.44	0.95	0.00	2.08	37	0.91
40	0.77	3.43	5.00	13.47	0.95				
55	1.61	3.53	5.02	12.01	0.98				
70	2.45	1.63	7.21	13.41	0.99				
85	3.39	0.52	8.96	14.75	0.99				
297 μm									
25	0.02	1.50	2.06	0.10	0.81	0.02	7.35	1740	0.30
40	0.26	3.17	2.21	0.75	0.95				
55	0.46	1.64	4.50	4.54	0.98				
70	1.46	0.90	7.75	8.40	0.99				
85	3.37	0.21	5.88	22.1	0.99				
149 μm									
25	0.02	3.99	2.47	0.06	0.76	0.44	1.28	10	0.97
40	0.53	1.88	2.39	2.84	0.97				
55	2.22	1.62	5.25	0.1	0.98				
70	3.22	0.52	1.38	0.05	0.10				
85	2.98	0.18	1.43	6.03	0.99				
105 μm									
25	0.06	4.18	2.97	0.51	0.58	0.02	9.17	1740	0.55
40	0.01	4.24	2.77	0.08	0.74				
55*	0.11	5.48	2.95	0.51	0.34				
70	0.02	0.71	4.11	0.21	0.86				
85	0.85	0.18	1.23	1.32	0.97				
74 μm									
25	0.06	1.83	8.70	0.03	0.57	0.04	23.9	366	0.60
40	0.26	3.69	5.79	0.25	0.91				
55*	0.31	5.85							
70	2.57	0.88	13.3	16.15	0.97				
85	5.95	0.26	22.8	6.81	0.98				

$$b_T G''(a_T \omega, T_{\text{ref}}) = G''(\omega, T) \quad (15)$$

$$\left(\frac{b_T}{a_T} \right) |\eta^*|(a_T \omega, T_{\text{ref}}) = |\eta^*|(\omega, T) \quad (16)$$

The linear visco-elastic master curves for the BGC dough samples are generated by the time-temperature superposition (TTS) principle and shifted to a reference temperature (T_{ref}) of 55 °C with both the horizontal shift factor a_T and the vertical shift factor b_T . The reference temperature is selected based on the vicinity of barley starch gelatinization (60–66 °C) temperature. Fig. 4a shows the reduced angular frequency, $a_T \omega$ dependence of elastic modulus (G'), viscous modulus (G''), and complex viscosity (η^*) of dough having particle size of 595 μm at five selected temperatures (25, 40, 55, 70 and 85 °C). It is observed that applying both horizontal shift factor a_T and vertical shift factor b_T to the G' and η^* produced better superposition although the G'' shows some deviation (Fig. 4a). It is worth mentioning that most of the samples including the sample without sieving followed similar trends. However, applying the principle to the finest particle size (74 μm) dough (Fig. 4b), it was observed that TTS was not fitted well for $G'-\omega$ and $G''-\omega$ data, however a better superimposed was noticed for $\eta^*-\omega$ data. Calculated values of the a_T and b_T are not close to unity, and therefore scaling has significant effect on extending the frequency window (5–6 log cycles). Calculated values of a_T and b_T coefficients are varied widely with temperature especially at and above 70 °C (data not shown), which clearly indicated the failure of superimposition for $G'-\omega$ and $G''-\omega$. One of the most probable reasons for the non-applicability of TTS for $G'-\omega$ or $G''-\omega$ could be the temperature range which are

extreme from the starch gelatinization and protein denaturation temperature (especially at 70 °C). These coefficients also indicate that the starch/protein of BGC is significantly temperature dependent.

3.9.5. Creep behavior

Effect of particle size on creep and relaxation behavior of BGC dough samples is presented in Fig. 5. It is apparent from Fig. 5a that all BGC samples displayed curves typical of viscoelastic fluids due to the presence of an irrecoverable strain at the end of the test. Creep and recovery phenomenon is mainly caused by reorientation of the bonds in viscoelastic material (Onyango, Mutungi, Unbehend, & Lindhauer, 2010). The existence of such irrecoverable strain does not necessarily imply that it is the consequence of flow, but a liquid-like character (Lefebvre, Renard, & Sanchez-Gimeno, 1998). Dough with heterogeneous collection of particles (without sieving) exhibited a considerable increase in the strain, and viscous in nature. It can be seen from Fig. 5a that the creep strain decreases with an increase of the particle size at the same creep time, indicating that the coarser particles (595 μm) dough becomes stiffer when subjected to a constant 20 Pa shear stress. Further, reduction of particle size resulted decreasing in mechanical rigidity and produced viscous dough. Creep curves of 149, 105 μm particle sizes and heterogeneous particle containing dough samples are close to each other, meaning that the creep stress is below or close to the linearity limit. A significant increase in the dough characteristic was observed when the finest particle size (74 μm) was tested under constant stress. However, the dough structure with particle

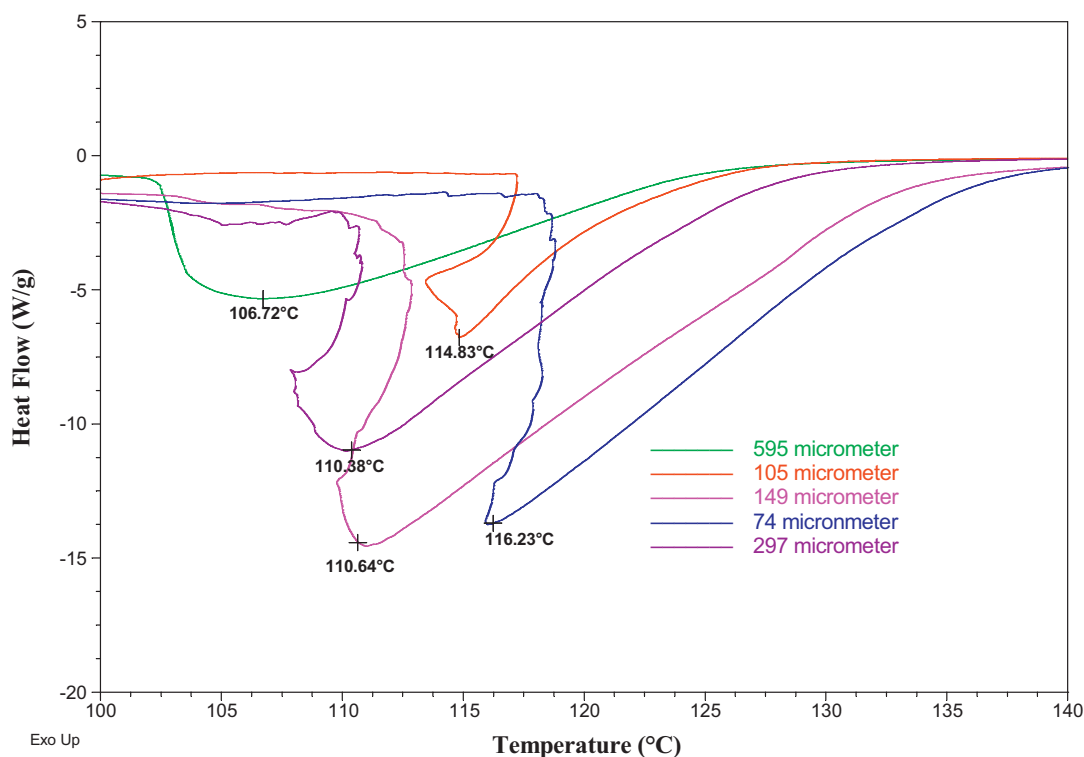


Fig. 6. DSC thermograms of BGC doughs at selected particle sizes.

size of 74 μm was broken after 200 s, which implies that the dough samples exhibited time-dependent flow behavior, and the dough structure containing the finest particle was disrupted at a constant stress of 20 Pa. Such applied stress value can be considered as a yield stress which is commonly observed for time-dependent fluid. During relaxation, 297- μm particle containing dough showed the highest recovery while the structure disruption was obvious for the finest particle containing dough.

3.9.6. Effect of temperature on the creep behavior

Temperature dependency of the creep compliance for specified particle size dough is illustrated in Fig. 5b. It can be seen from the figure that the creep compliance of 297- μm particle size dough, $J(t)$ increases with increasing temperature from 25 to 85 °C at the same creep time interval, indicating that the dough becomes viscous when subjected to a constant shear stress of 20 Pa. During thermal treatment BGC components undergoes various thermal transition resulting an increase in viscous component of the dough especially at 85 °C where the change of $J(t)$ was significant. The significant increase of $J(t)$ at 85 °C could be associated with the breakdown of the dough structure/network under stress and at elevated temperature.

During the analysis of the creep data, the discrete retardation spectrum was employed where the numbers of discrete terms have been increased from 1 to 6 to generate 4–14 elements. It is worth mentioning that the analysis followed the generalized Kelvin–Voigt model. With an increasing number of terms from 1 to 6 for dough containing 105 μm particles measured at 85 °C, it was observed a slight improvement in R^2 from 0.97 to 0.99. However, the values of instantaneous compliance (J_0), steady-shear viscosity (η_0) and retarded time (τ) were affected by changing the number of terms. For example the J_0 value changed from $8.45 \times 10^{-5} \text{ Pa}^{-1}$ to $2.27 \times 10^{-5} \text{ Pa}^{-1}$, the η_0 value increased from $1.83 \times 10^5 \text{ Pa.s}$ to $5.55 \times 10^5 \text{ Pa.s}$, and the τ value changed significantly with increasing number of terms for 1–6. It is argued that the creep compliance behavior can be well described by models combining larger

numbers of terms or elements, however from the realistic point of view 1–2 terms could be sufficient enough to represent the data. Based on this, the generalized Kelvin–Voigt model with 4 elements was selected (1-term) and used to describe the viscoelastic behavior and to compare the effect of temperature on sieved BGC dough samples. Recovery experiment was carried out only at 2585 °C and presented in Table 3. It can be seen from the Table that the recovery data were not very smooth especially for finer particles ($R^2 = 0.3\text{--}0.97$). The possible reason could be inability of the finer particles enriched dough to associate further after experiencing a sudden load.

Table 3 represents the parameters of Burger's model. Among the sample studied, heterogeneous and the coarser particle (595 μm) containing dough samples fitted the model well and the obtained parameters (J_0 and J_1) varied systematically with temperature. The highest value of J_0 was recorded in the case of dough sample with the finest particle at 85 °C while the maximum value of η_0 was observed in the case of control sample at 25 °C. Dough samples containing smaller particles (74–297 μm) exhibited a mixed response with temperature which is believed to be associated with constituents of each fraction, starch gelatinization and protein denaturation in addition to contributing effect of β -D-glucan.

3.10. Particle size analysis and SEM

Scanning electron microscopy was employed to analyze the changes in microstructure of BGC after particle fractionation. The particle sizes of the BGC samples (without sieving; 105- μm and 74- μm fractions) were observed through scanning electron microscopy (SEM). The particles were not regular in shape (Fig. 7a), and therefore, the average length and breadth of the particles were considered for the best representation. The average length and breadth of the particles without sieving were 148 ± 10 and $96 \pm 4 \mu\text{m}$, respectively (250 $\times$). The maximum and minimum length of the particles was 243 μm and 100 μm , respectively. A higher magnification (1500 $\times$ and 4000 $\times$) of 74- μm and 105 μm

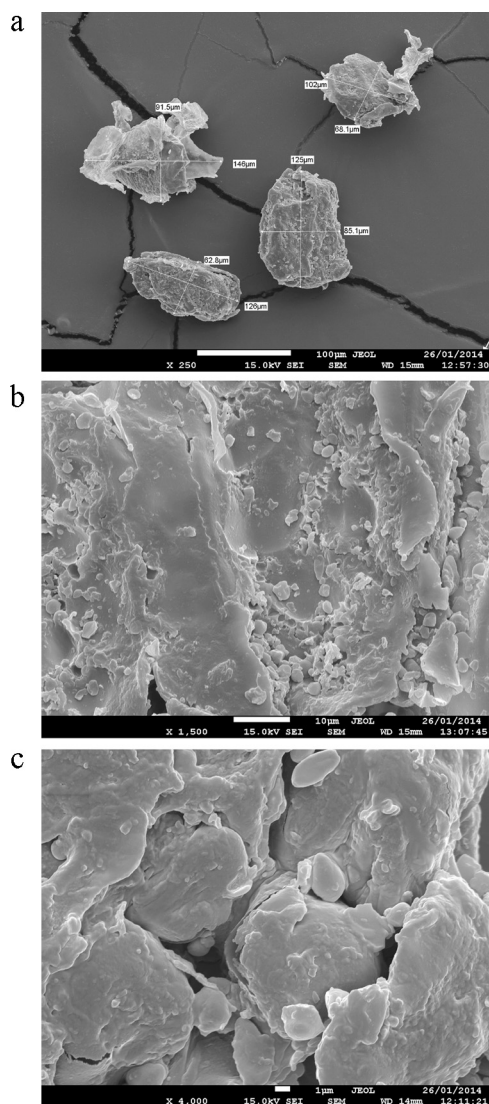


Fig. 7. Scanning electron micrographs for BGC samples. (a) Typical particle sizes, (b) 105- μ m particle size and (c) 74- μ m particle size fraction.

particles is shown in Fig. 7b and c, respectively. Micrographs revealed that the particle size has effect on distribution of protein and starch. Fig. 7c clearly shows that starch granules (round or polygonal shaped) embedded in a protein matrix and the SEM micrograph confirmed the presence of big (A-type) and small (B-type) starch granules. Similar observations were earlier reported by various researchers (Altan, 2014; Nair, Knoblauch, Ullrich, & Baik, 2011) while studying the microstructure of barley grain.

4. Conclusions

The functional properties of β -D-glucan concentrate are mostly dependent of the surface composition, size and surface characteristics of the particles. Water holding capacity, water solubility index, sedimentation volume fraction and β -D-glucan content of BGC samples varied according to the particle size. Oscillatory rheology of BGC dough is strongly influenced by the particle size, and the dough showed a predominating solid-like behavior. In general, the dough samples showed the shear-thinning behavior except the sample containing 105- μ m particle size where a shear-thickening behavior was observed. The deviation in rheological behavior is believed to be associated with the enrichment

of protein components and also by lowering of β -D-glucan content in the fraction. The finest particle (74 μ m) containing dough sample showed the maximum strain during creep test, and confirming its viscoelastic nature. Furthermore, the creep compliance of dough sample increases as function of temperature that indicates the dough becomes viscous at a constant shear stress. The coefficients of time-temperature superposition (TTS) of rheological data varied significantly from the reference temperature at 55 °C that supports wide variations in dough rigidity at different temperatures. The simultaneous utilization of two complementary rheological techniques namely rheology and creep test provided in-depth understanding of the structural phenomena of BGC induced by heat. Rheological and thermal properties of BGC provide important information for selecting specific particle size in food product development with a desired texture.

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