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Influence of different purification and drying methods on rheological properties and viscoelastic behaviour of durian seed gum

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ABSTRACT

The aim of the present study was to investigate the effects of different purification and drying methods on the viscoelastic behaviour and rheological properties of durian seed gum. The results indicated that the purified gum A (using isopropanol and ethanol) and D (using hydrochloric acid and ethanol) showed the highest and lowest viscosity, respectively. Four drying techniques included oven drying (105 °C), freeze drying, spray drying and vacuum oven drying. In the present work, all purified gums exhibited more elastic (gel-like) behaviour than the viscous (liquid-like) behaviour ($G'' < G'$). The current study revealed that all drying methods led to significantly diminish the elastic (G') and viscous modulus (G'') of durian seed gum. The freeze-dried gum and oven-dried (105 °C) gum exhibited the highest and lowest viscous modulus (G''), respectively.

1. Introduction

Plant gums serve a variety of functions in value-added products. They are water-soluble ingredients used to improve the rheological properties, extend shelf life, and encapsulate flavours. Plant gums are also used to emulsify the flavours, provide the elasticity, retain moisture, and make gel and/or thickness (Nwokocha & Williams, 2009; Williams & Phillips, 2000). The dispersion of water soluble gums in the aqueous system provides great technical importance, since they can improve the gelling or thickening properties of the final product, favouring the manufacture, distribution, storage and consumption of food products (Distler, 1999; Morris, 1991; Voragen, Pilnik, Thibault, Axelos, & Renard, 1995). Therefore, it is crucial to enclose a concept and understanding of functional characteristics of gum to select the appropriate one based on the scope of application.

The rheological characteristics play a significant role in the process design (e.g., fluid flow, pump sizing, extraction, filtration, extrusion and purification, pasteurization, evaporation and drying process; Marcotte, Taherian Hoshahili, & Ramaswamy, 2001). The thickening properties and viscoelastic behaviour are significantly influenced by several factors such as shear rate and time, frequency, temperature, and pressure (Fig. 1) (Hasan Nahid, 2010; Marcotte et al., 2001). The rheological properties and viscoelastic behaviour of natural plant gums depend on the method and condition of extraction, purification, drying and further modification processes. The physicochemical and functional properties of various plant gums have been studied by previous researchers (Cunha, de Paula, & Feitosa, 2007; Ibañez & Ferrero, 2003; Jaya & Durance, 2009; Mirhosseini & Tabatabaee Amid, 2012a; Mirhosseini & Tan, 2010a,b; Mirhosseini, Tan, Aghlara, Hamid, Yusof, & Boo, 2008; Mirhosseini, Tan, & Naghshineh, 2010; Mirhosseini, Tan, Hamid, & Yusof, 2007, 2008 a–c; Razavi & Karazhiyan, 2009; Wang, Wang, Li, Xue, & Mao, 2009).

Ibañez and Ferrero (2003) evaluated the effect of extraction and purification condition on the characteristics of the mucilage from *Prosopis flexuosa* DC seed. They reported that the thickening capacity and rheological properties of *P. flexuosa* seed gum depended on the degree of purification. The researchers reported that the crude seed gum induced a pseudoplastic behaviour even at low solids concentrations (0.9%); while the purified seed gum showed the pseudoplastic and viscoelastic behaviours only at high concentrations. Cunha et al. (2007) purified the commercial guar gum by (I) enzymatic hydrolysis with porcine pancreatin, (II) acetone and ethanol, (III) Fehling solution and (IV) the combined method II and III. The researchers found that all purification methods led to reduce the protein and mono/oligo/polysaccharide contaminants. They found that all purification methods decreased the intrinsic viscosity and molar mass.

The main goal was to investigate the influence of different purification and drying techniques on the rheological properties of purified durian seed gum. The most commonly used drying

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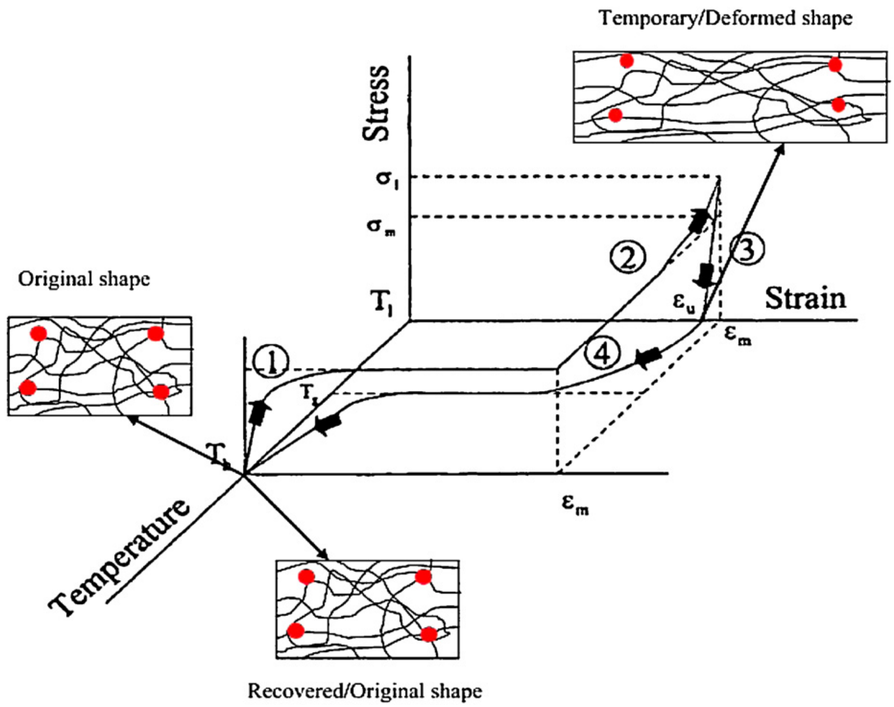

Fig. 1. Viscoelastic behaviour of a polymer as a function of stress, strain, temperature and time (http://what-when-how.com/mechanics-of-time-dependent-materials-and-processes-in-conventional-and-multifunctional-materials/degradation-of-shape-memory-polymer-due-to-water-and-diesel-fuels-part-1/).

techniques applied for plant gums included spouted bed dryers (Cunha, Maialle, & Menegalli, 2000), microwave vacuum drying (Sundaram & Durance, 2008), freeze drying (Barresi et al., 2009; Moreira, 2009), vacuum drying (Wang et al., 2009), oven drying (105 °C) (Wang, Wang, Li, & Adhikari, 2010), and spray drying (Nep & Conway, 2011). In the present study, four different drying techniques (i.e., oven drying (105 °C), spray drying, freeze drying and vacuum drying) were selected to study the rheological properties of different dried-durian seed gums. To the best of our knowledge, there is no similar published report investigating the effect of different purification and drying techniques on the rheological properties and viscoelastic behaviour of durian seed gum.

2. Materials and methods

2.1. Materials

Isopropanol, ethanol (95%), absolute ethanol (99.9%), acetone, hydrochloric acid, saturated barium hydroxide, sodium hydroxide, and acetic acid were purchased from Fisher Scientific (Pittsburgh, PA, USA). Durian (*Durio zibethinus*) fruit was purchased from the local market (Selongor, Malaysia). Ripened durian fruits were selected based on the size uniformity and free of visual defects. The fruits were then de-husked (cut open the rind), by cutting along the suture on the back of the lobules. Durian seeds were removed, cleaned and rinsed thoroughly with sterile distilled water. There is a possibility to produce "hard seed" if the moisture is reduced. Hard durian seeds with reduced moisture content will resist germination under favourable conditions, thus prolonging the storage life. The seed was partially dried by the air circulation (Tabatabaee Amid & Mirhosseini, 2012). The dried seeds were then packed in plastic bags and stored in a dry and cool place (10 ± 2 °C) until the extraction process. All the experiments were performed with deionized water.

2.2. Chemical extraction of durian seed gum

The chemical extraction was performed according to the method described by Singh, Singh, and Maurya (2010) with the minor modification. The successive steps of the defating, decolouring, solvent soaking, gum dissolution, centrifugation and precipitation, were considered for the chemical extraction. Durian seed were washed and chopped into small pieces. Then, it was air dried by using the air circulation before milling into flour. The cold extraction was used to extract the oil from the durian seed flour in order to avoid the thermal degradation. The defating process was carried out successively using hexane and isopropanol (60:40) at the room temperature (25 ± 1 °C). Our preliminary study showed that the solvent mixture containing hexane and isopropanol (60:40) was the most efficient solvent for defating process among all studied solvents (i.e., petroleum ether, hexane, isopropanol and ethanol). The solvent residue was removed by centrifugation at 1400 *g* for 15 min (Avanti J-25 Beckman Coulter Centrifuge, Krefeld, Germany). Then, defatted-durian seed flour (1 kg) was exhaustively decoloured using ethanol at the decolouring time 120 min. The decolourized seed flour was vacuum filtered and then soaked in 1% aqueous acetic acid for 1.5 h at the ambient temperature (25 ± 1 °C). Then, the slurry was filtered with Nylon cloth filter and the filtrate was precipitated with 95% ethanol. The precipitated slurry was washed three times using absolute ethanol (99.9%) to achieve very light brown amorphous crude gum. The crude gum was collected and oven dried at 40 °C (Mirhosseini and Tabatabaee Amid, 2012b).

2.3. Purification procedures

Four different purification methods, i.e., method A (isopropanol and ethanol), method B (isopropanol and acetone), method C (saturated barium hydroxide) and method D (hydrochloric acid and ethanol), were considered for the current purification study. In a series of purification techniques, successive steps of gum dissolution, centrifugation and precipitation were considered.

2.3.1. Purification using isopropanol and ethanol (Method A)

In the purification method A, the crude seed gum was purified by using hot water, ethanol and isopropanol as described by Youssef, Wang, Cui, and Barbut (2009). Initially, the gum solution (2.5%, w/v) was prepared by dissolving 25 g of the crude durian seed gum in 1 l of deionized water at 80 °C water bath for 6 h, followed by stirring at room temperature overnight. The gum solution (2.5%, w/v) was subjected to the centrifugation (Avanti J-25 Centrifuge, Beckman Coulter, Fullerton, CA, USA) for 15 min at 15,180 g. The supernatant was precipitated by the addition of absolute ethanol (1.2 l) and the supernatant was decanted. The residue was recovered and kept overnight in 100% isopropanol. Finally, the residue was dried in the oven 40 °C for overnight to prepare the purified durian seed gum.

2.3.2. Purification using isopropanol and acetone (Method B)

In the purification method B, the purification process was carried out by using isopropanol and acetone as reported by Bouzouita et al. (2007) with the minor modification. One g of the crude seed gum was precipitated by soaking into two volume excess of isopropanol, and allowing the gum-solvent slurry to stand for 30 min. The white fibrous precipitate was collected by the filtration using the screen (53 μm). Then, the collected precipitate was washed twice with isopropanol and acetone. Finally, it was dried in the oven overnight at 40 °C.

2.3.3. Purification using saturated barium hydroxide (Method C)

In the purification method C, the crude seed gum was purified through barium complexing according to the method described by Singh, Tiwari, Tripathi, and Sanghi (2005). In this method, the gum solution (2.5%, w/v) was prepared by dissolving 2.5 g of the crude durian seed gum in 100 ml of water and continuous stirring for 12 h at 60 °C. Then, the gum solution was precipitated with saturated barium hydroxide solution. The precipitate was separated by a Beckman centrifuge (Avanti J-25 Centrifuge, Fullerton, CA, USA) at 15,180 g for 15 min. Then, the precipitate was stirred with 1 M acetic acid for 8 h and again centrifuged at 15,180 g for 15 min. The supernatant was precipitated with 90% ethanol, then the precipitate was washed with 95% ethanol and oven dried at 40 °C.

2.3.4. Purification using Fehling solution (Method D)

In the purification method D, the purification was performed by using Fehling solution as reported by Cunha et al. (2007) with some modification. Initially, 1 g of the crude durian seed gum was dissolved in approximately 100 ml of water and stirred for 24 h with magnetic stirring. The prepared gum solution (1%, w/v) was precipitated by adding 5 ml of freshly prepared Fehling solution and the precipitate was collected by the glass filter (No. 3). Then, the precipitate was dissolved in 0.1 M hydrochloric acid by a magnetic stirrer for 1 h until the full solubilization. The solution was precipitated with three volumes of 95% ethanol. The precipitate was separated by the glass filter (No. 3) and washed with 95% ethanol until pH 6. Finally, the filtrate was washed with acetone and dried in the oven at 40 °C overnight.

2.4. Drying methods

In the current study, the effect of four different drying techniques on the chemical composition and functional properties of purified durian seed gum was investigated. Four drying techniques including oven drying (105 °C), freeze drying, spray drying and vacuum oven drying were selected from the previous published litratures (Massiot & Renard, 1997; Nep & Conway, 2011; Wang et al., 2010). They studied the effect of those drying methods on the physicochemical properties of apple fibre, flaxseed gum and grewia polysaccharide gum, repectively.

2.4.1. Oven drying

The purified seed gum was dried according to the method described by the previous researchers (Wang et al., 2010) with the minor modification. The seed gum solution (10%, w/v) was prepared by dissolving 10 g of the purified durian seed gum 100 ml of deionized water. The coarse gum solution was homogenized using a high pressure homogenizer (APV, Crawley, UK) for two cycles at different pressure levels (30 and 25 MPa). Then, the homogenized-gum solution (10%, w/v) was dried by using the oven dryer at 105 °C for 3 h. The dried sample was dry milled and passed through a 1.0 mm sieve. Then, the milled powder was weighed and stored in the air-tight container before further analysis (Nep & Conway, 2011).

2.4.2. Vacuum oven drying

The vacuum oven drying was employed according to the procedure described by Wang et al. (2010) with some modification. As mentioned earlier, the coarse gum solution (10%, w/v) was prepared and then homogenized using the same homogenization process (at 30 and 25 MPa). Then, the homogenized-gum solution was dried by using a vacuum-dryer at 60 °C for 24 h. The vacuum and temperature were maintained at 5 psi and 60 °C, respectively. The dried sample was then milled and passed through a 1.0 mm sieve. Finally, the milled powder was packed in the air-tight container prior to the analysis (Nep & Conway, 2011).

2.4.3. Spray drying

The spray-dried seed gum was produced according to the method described by the previous researchers (Nep & Conway, 2011; Oomah & Mazza, 2001) with minor modification. Initially, the coarse gum solution (10%) was homogenized using the same processing condition as described earlier. The homogenized-gum solution (10%) was spray-dried by using a co-current spray dryer (Niro model 2000A, Niro Atomizer, Copenhagen, Denmark) equipped with a vanes centrifugal atomizer. The spray drying was performed at the pressure, inlet and outlet temperatures of 552 kPa, 160 °C and 80–85 °C, respectively (Nep & Conway, 2011). The flow rate was controlled by adjusting the feed rate (50 ml/min) through the atomizer with a peristaltic pump (Masterflex-model 70/5, Cole-Parmer Instrument Co., Chicago, IL, USA). Finally, the spray-dried gum was collected at the bottom of the cyclone. The spray-dried seed gum was milled and passed through a 1.0 mm sieve and packed in the air-tight containers before further analysis (Nep & Conway, 2011).

2.4.4. Freeze drying

The freeze-dried seed gum was produced according to the procedure described by Nep and Conway (2011) with some modification. Initially, the coarse gum solution (10%) was homogenized using the same homogenization condition as described earlier. Then, the homogenized-gum solution (10%) was placed in Petri dishes and pre-frozen at −20 °C for 24 h prior to freeze-drying process. The freeze drying was carried out by using a freeze dryer (Labconco Freezone 18, Model 77550, MO, USA). The Petri dishes were then transferred into freeze drier chamber and frozen at −50 °C for 48 h. The freeze-dried gum was dry milled and passed through a 1.0 mm sieve and packed in the air-tight containers prior to the analysis.

2.5. Analytical tests

2.5.1. Apparent viscosity

The viscosity of crude durian seed gum was measured by using a Haake rheometer (RheoStress 600, Karlsruhe, Germany) equipped with a cone and plate probe (5.0 diameters, 0.04 radiuses) after 24 h of hydration of the gum solution. Initially, 0.5 g of crude durian seed gum was dissolved in water up to a desired concentration (0.5%). Then, it was agitated vigorously for approximately 15 min until the solution. Finally, it was stirred one overnight to form a homogenous viscous dispersion. Two ml of the gum solution (0.5%, w/w) was placed in the rheometer plate and allowed to equilibrate for 10 min at 25 °C. The viscosity was measured in triplicate by subjecting the gum solution to shear rate of 10–200 s^{-1} (Amin, Ahmad, Yin Yin, Yahya, & Ibrahim, 2007). The apparent viscosity and steady shear rate measurements were fitted to the Herschel–Bulkley and Cross models (Eqs. (1) and (2)) (Rao, 1999; Steffe, 1996).

$$\sigma = \sigma_0 + k\gamma^n \quad (1)$$

$$\eta(\gamma) = \eta_\infty + \frac{\eta_0 - \eta_\infty}{1 + (\tau\gamma)^m} \quad (2)$$

where σ is the shear stress (Pa), σ_0 is the yield stress (Pa), k is the consistency index (Pa s^n); γ is the shear rate (s^{-1}); η is the flow behaviour index; $\eta(\gamma)$ is the apparent viscosity (Pa s) at a determined shear rate; η_0 and η_∞ are the Newtonian viscosity at zero and infinite shear rate (Pa s); τ is the time constant (s); m is a rate constant.

2.5.2. Elastic modulus (G') and viscous modulus (G'')

The viscoelastic properties of crude durian seed gum were also examined by low-amplitude oscillatory measurements of storage or elastic modulus (G') and loss or viscous modulus (G'') using the same Haake rheometer (RheoStress 600, Haake, Karlsruhe, Germany). The dynamic rheometer equipped with cone and plate probe was used to test strain amplitude sweep at a fixed frequency of 1 Hz and 25 °C isothermal conditions. The amplitude of strain was swept from 0.02 to 1 Pa with the increment of a logarithmic scale (Oomah & Mazza, 2001). The oscillatory test was measured in triplicate for each sample.

2.6. Experimental design and data analysis

A completely randomized design (CRD) was considered to investigate the effects of different the purification and drying processes on the viscosity, elastic modulus (G'), viscous modulus (G'') of durian seed gum (Table 1). As shown in Table 1, all purification and drying processes were carried out in triplicate. In general, 24 treatment runs (12 purification runs + 12 drying runs) were considered in the current study (Table 1). The data was reported as means ± SD of independent trials. The crude durian seed gum was considered as the control sample to determine the effect of different purification methods on the rheological properties and viscoelastic behaviour of durian seed gum. In addition, the purified-oven dried durian seed gum (40 °C) was considered as a control sample to study the influence of different drying processes on the rheological properties of durian seed gum. The effectiveness of the purification process was evaluated by comparing the rheological properties and viscoelastic behaviour of the purified seed gum with the control sample. The data was subjected to one-way analysis of variance (ANOVA) to determine the significant ($p < 0.05$) differences among the purification and drying methods (Mirhosseini, Tan, Taherian, & Boo, 2009). All data analysis was carried out by using Minitab version 15 (Minitab Inc., PA, USA). Fisher multiple comparison test was used to evaluate significant differences ($p < 0.05$) between the different purified seed gums as compared to the control.

3. Results and discussion

3.1. Apparent viscosity

3.1.1. Effect of different purification methods on the apparent viscosity

The apparent viscosity of purified durian seed gum varied from 14.2 to 21.5 (mPa s) as compared to the crude gum (16.3 mPa s) (Figs. 2 and 3). This value was lower than the viscosity reported for durian seed gum solution (1%) (65 mPa s at the shear rate of 1000 s^{-1}) (Amin et al., 2007) and the mucilage solution (0.4%) from *P. flexuosa* seed (1900 mPa s, at the shear rate of 64 s^{-1}) (Ibañez & Ferrero, 2003). The difference could be due to different gum concentrations and shear rates. Brummer, Cui, and Wang (2003) reported different intrinsic viscosity for purified fenugreek gum, lucast bean gum and guar gum solutions (1%) ranging from 0.02 to 0.65 Pa s. Cunha et al. (2007) reported significant different viscosities for crude and purified guar gum depending on the purification method, gum concentration and shear rate. The researchers found that the purified guar gum solution (1%) had lower viscosity ranging from 30 to 1830 mPa s as compared to crude guar gum solution (1%, 1858 mPa s) at the shear rate of 5 s^{-1}; while both crude and purified guar gum solution (0.1%) exhibited almost similar viscosity (1.2–3.0) at the shear rate of 500 s^{-1}.

The purified gum A and D showed the highest and least viscosity, respectively (Fig. 3). This might be due to the different molecular mass of purified gum A and D. Cunha et al. (2007) also showed that the purified guar gum containing higher molecular mass and uronic acid contents induced more viscous solution than the one with lower molecular mass and uronic acid. The difference could be also related to the fact that the purified gum A showed the largest particle size and wide size distribution; while the purified gum D had the small particle size with narrowest size distribution (or span) among all purified gums (data not shown). The low viscosity of purified gum D could be due to the presence of Cu(II) in the complex formation induced by Fehling solution. This finding was also reported by Cunha et al. (2007). The different viscosities could be also explained by the chemical structures (i.e.,

Table 1
Experimental design for purification and drying processes.

Process	Experimental design (DOE)	References
Purification		
Methods A (isopropanol and ethanol)	Completely randomized design (CRD)	Youssef et al. (2009)
Method B (isopropanol and acetone)		Bouzouita et al. (2007)
Method C (saturated barium hydroxide)	4 Treatments × 3 replications = 12 runs	Singh et al. (2005)
Method D (Fehling solution)		Cunha et al. (2007)
Drying		
Freeze drying	Completely randomized design (CRD)	Massiot and Renard (1997)
Oven drying (105 °C)		Wang et al. (2009, 2010)
Spray drying	4 Treatments × 3 replications = 12 runs	Oomah and Mazza (2001)
Vacuum oven drying		Nep and Conway (2011)

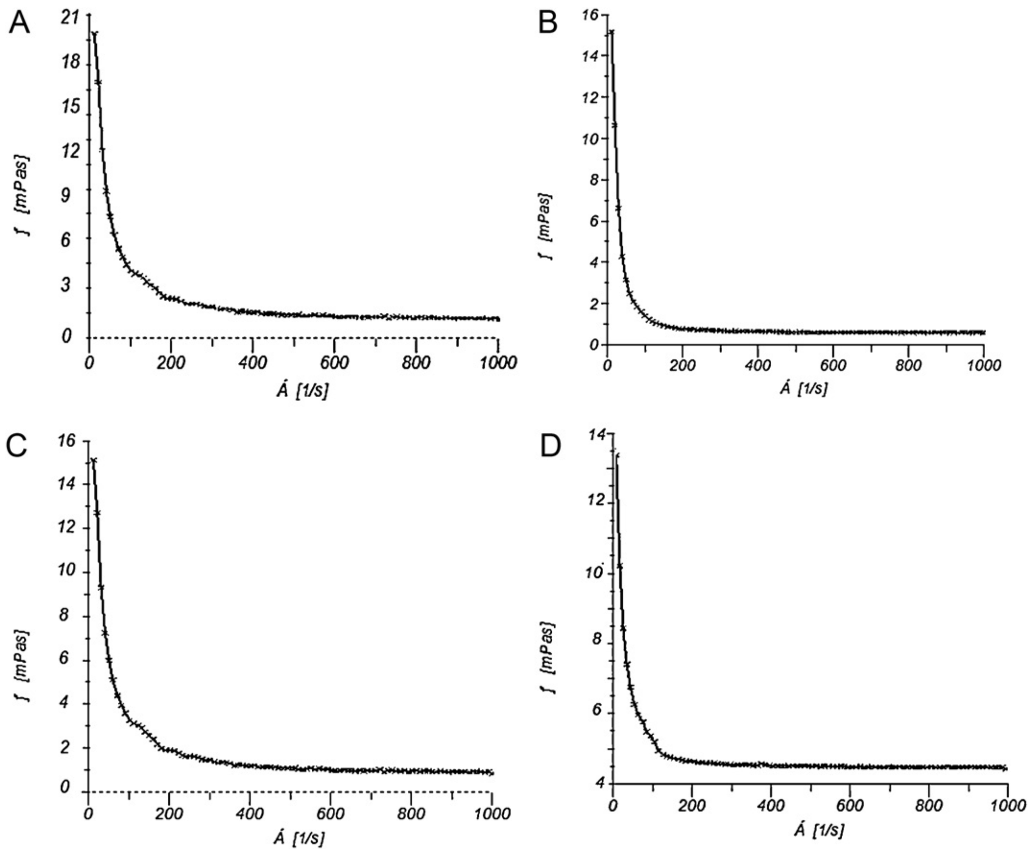

Fig. 2. Viscosity (η, ƒ) rheograms of purified durian seed gums showing the changes in viscosity as a function of shear rate (γ, Á) (s^{-1}) (A: isopropanol and ethanol; B: isopropanol and acetone; C: saturated barium hydroxide; D: Fehling solution plus hydrochloric acid and ethanol).

monosaccharide composition and protein). For example, the purified gum A and D had different galactose and protein content, thus inducing different viscosities. In addition, the length of backbone, glycosidic linkage, branching degree and side chains as well as the monosaccharide composition of polysaccharide significantly influence its rheological properties.

On the other hand, the viscosity induced by the polysaccharide depends on the electric charge. In fact, polysaccharide chains in water expand due to intra-molecular electrostatic repulsion and form high viscous solutions (Simas-Tosin et al., 2010). The purified seed gum A exhibited a significantly ($p < 0.05$) higher viscosity than the crude seed gum; while the purified seed gum D had a significantly ($p < 0.05$) lower viscosity than the crude seed gum. However, the purification method B (using isopropanol and acetone) and C (using saturated barium hydroxide) did not significantly ($p > 0.05$) influence the viscosity of crude durian seed gum (Fig. 3). It was found that the apparent viscosity of all purified durian seed gums decreased as the shear rate increased (Fig. 2). In fact, the purified

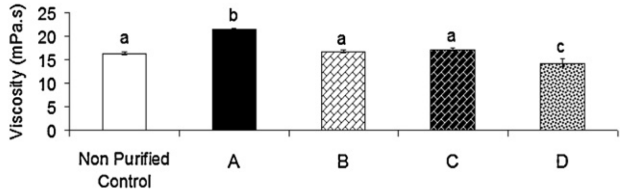

Fig. 3. Effect of different purification methods on apparent viscosity of durian seed gum (A: isopropanol and ethanol; B: isopropanol and acetone; C: saturated barium hydroxide; D: Fehling solution plus hydrochloric acid and ethanol); [a–c]significant differences at 95% confident level.

durian seed gum showed the maximum viscosity at the lowest shear rate. This indicates the pseudoplastic (or shear-thinning) flow behaviour of durian seed gum in the crude and purified forms.

The researchers observed the same rheological behaviour for the mucilage from *Monostroma nitidium*, monoi (*Cissampelos pareira*) leaves and *Mesona Blumes* (Feng, Gu, & Jin, 2007; Huei Chen & Yuu Chen, 2001; Vardhanabhuti & Ikeda, 2006). Koocheki, Taherian, and Bostan (in press) explained that the gum molecules were disarranged and partially aligned, thus resulting in a higher viscosity at a low shear rate. As the shear rate was increased, the molecules became oriented and aligned, thus reducing the inner friction and viscosity (Koocheki et al., in press). This could be explained by the reason that as the shear rate increased the droplet–droplet interaction was deformed and eventually disrupted, which resulted in the size reduction of the flocks thereby decreasing of viscosity (Peamprasart & Chiewchan, 2006). On the other hand, the integer of chain entanglements decreases at high shear rate, thus accounting for reducing the viscosity with increasing shear rate (Cui, 2005). As explained by Funami et al. (2008), the disruption of the entanglements between polymer coils (i.e., fenugreek gum) takes place simultaneously with the regeneration of molecular interacts at low shear rates, thus resulting in no reduction of viscosity; while the disentanglements become predominant at high shear rates, reducing the viscosity (Funami et al., 2008).

The high viscosity of purified gum A could be also explained by the presence of the lowest protein content. Youssef et al. (2009) also reported that the intrinsic viscosity increased when the protein level was decreased. They showed that high viscosity was related to high molecular weight. As reported by Youssef et al. (2009), the molecular weight of fenugreek gum increased with removing the attached proteins, thereby increasing the viscosity. Youssef et al.

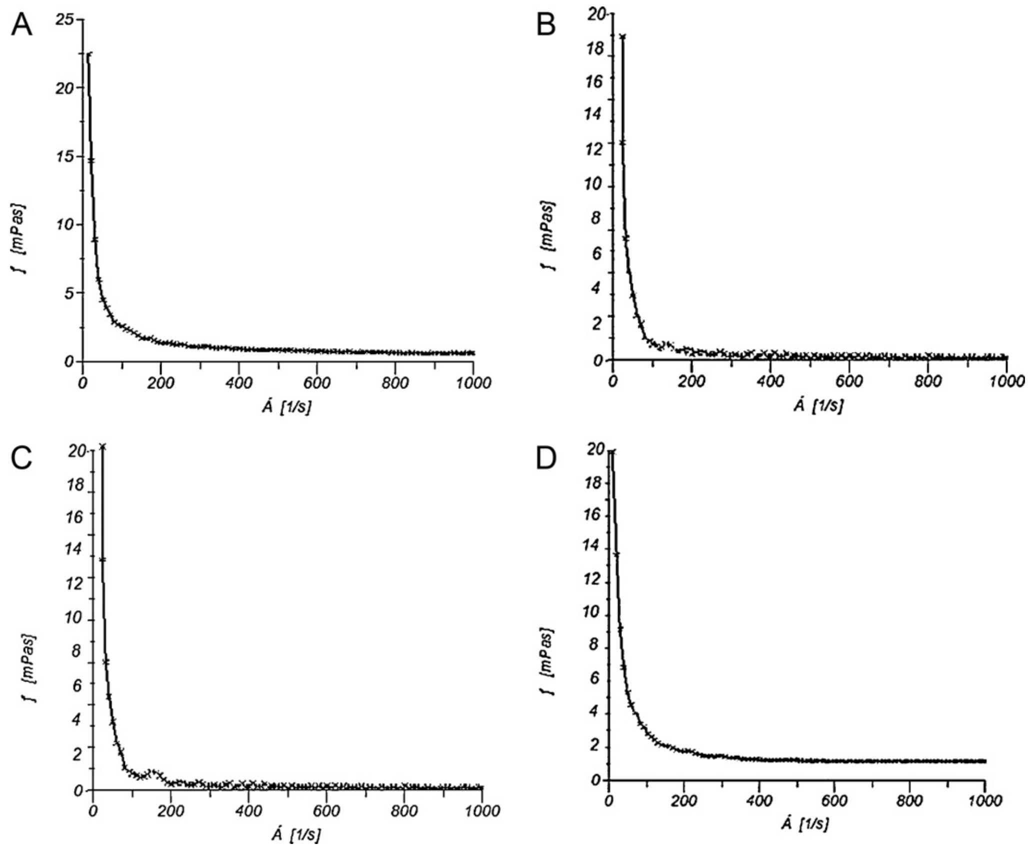

Fig. 4. Viscosity (η, ƒ) graphs of dried durian seed gums showing the changes in viscosity as a function of shear rate (γ, Á) (s^{-1}) (A: freeze drying; B: oven drying; C: spray drying; D: vacuum oven drying).

(2009) showed thet crude fenugreek followed by the purified and protein free fenugreek gums exhibited the least molecular weight inducing the highest viscosity; while protein free fenugreek had the highest molecular weight, showing the lowest viscosity. As reported by Youssef et al. (2009), the molecular weight increased after the initial purification using phenol treatment due to the efficient removal of the protein fraction. However, they reported that the crude fenugreek gum exhibited lower intrinsic viscosity as compared to the purified and protein free fenugreek gums.

3.1.2. Effect of different drying techniques on the apparent viscosity

The functional properties (e.g., viscosity and rheological properties) of gums are greatly sensitive to the drying processes (Jaya & Durance, 2009). The present study also demonstrated that the viscosity of durian seed gum was significantly ($p < 0.05$) influenced by the drying process. This observation was also reported by previous researchers (Jaya & Durance, 2009; Jimoh, Olurin, & Aina, 2009; Nep & Conway, 2011). In the present study, the viscosity of durian seed gum fluctuated from 17.8 to 21.5 mPa s depending on the drying method (Figs. 4 and 5). In general, the drying process led to increase the viscosity of durian seed gum (Fig. 5). As stated by Onweluzo, Obanu, and Onuoha (1994), the increase in viscosity may be due to additional intermolecular interactions that naturally affect the viscosity depending on the type of interaction. Wang et al. (2009) also compared the viscosity of flaxseed gum dried by different techniques. They reported that the viscosity of flaxseed gum varied from 1.382 to 5.087 Pa s. Nep and Conway (2011) also demosntrated that the grewia gum showed different degree of viscosity varying from 0.20 to 0.32 Pa s depending on the drying method. They reported that air-dried grewia gum showed higher viscosity than spray-dried and freeze-dried grewia gum.

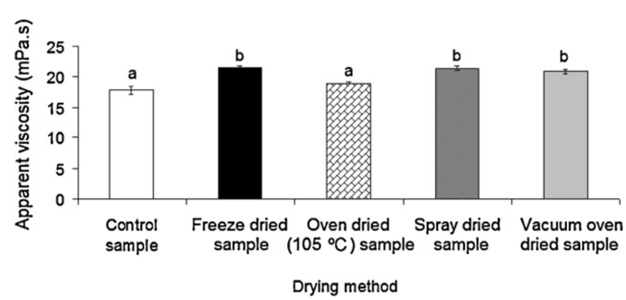

Fig. 5. Effect of different drying techniques on apparent viscosity of durian seed gum; [a,b]significant differences at 95% confident level.

The significant changes of apparent viscosity could be due to the substantial impact of drying process on the chemical composition of durian seed gum. As also explained by Simas-Tosin et al. (2010), the presence of free, reducing oligosaccharides, phenolics and inorganic salts, beyond polysaccharide in the gum structure gives a more viscous solution. The results indicated that the freeze-dried gum exhibited the highest viscosity among all dried durian seed gums (Fig. 5). However, there was no significant ($p > 0.05$) difference among the viscosity of freeze-dried, spray-dried and vacuum oven-dried gums. In addition, the control and oven-dried samples also did not exhibit a significant ($p > 0.05$) different viscosity (Fig. 5). The viscosity of polysaccharide gums is largely influenced by the particle size and distribution, molecular weight and ratio of soluble to insoluble matters (Larrauri, Rodríguez, Fernández, & Borroto, 1994; Wang & Cui, 2005). The drying process can provide a broad range of molecular weight depending on the type and condition of drying, thus varying the viscosity (Nep & Conway, 2011). In the current study, the significant differece among the viscosity of all seed gums could be also due to the difference between their molecular weights.

It was found that all dried durian seed gums showed pseudoplastic (or shear-thinning) flow behaviour (Fig. 4). Wang et al. (2009) also reported the similar pseudoplastic (or shear-thinning) flow behaviour for freeze-dried, oven dried, spray-dried and vacuum oven-dried flaxseed gums, respectively. Previous researchers (Simas-Tosin et al., 2010) also found that freeze-dried peach tree gum exudate exhibited the pseudoplastic (or shear-thinning) flow behaviour at different concentrations. As reported by Cui (2005), the viscosity of gum dispersions decreased with increasing the shear rate due to a decreasing number of chain entanglements at high shear rates. The effect of different drying processes on the viscosity of polysaccharide gum could be due to different proportions of soluble to insoluble matters. The drying processes significantly ($p < 0.05$) influence the ratio of soluble to insoluble matters depending on the drying condition, thereby changing the viscosity of powder.

3.2. Rheological properties

3.2.1. Effect of different purification methods on the viscoelastic behaviour

The viscoelastic property of the mucilage is a critical rheological behaviour due to its multiple industrial applications (Medina-Torres, Brito-De La Fuente, Torrestiana-Sánchez, & Katthain, 2000). The polymer posses both liquid (viscous) and solid (gel)-like behaviours termed as 'viscoelastic substance' (Hasan Nahid, 2010). Oscillatory test is a dynamic method for determining the viscoelastic behaviour. In the oscillatory test, the observation for a gel exhibits a highly elastic (solid-like) behaviour; while the steady shear property for the concentrated solution shows the viscoelastic behaviour (Steffe, 1996). In the current study, the viscoelastic behaviour of durian seed gum was analyzed at the constant frequency. The results indicated that the elastic modulus (G') of purified durian seed gum was higher than its viscous modulus (G''), representing the elastic behaviour at the low frequency. As explained by previous researchers (Funami et al., 2008; Mo, Takaya, Nishinari, Kubota, & Okamoto, 1999), polymer (i.e., fenugreek gum) coils disentangle at low frequencies (rather than high frequencies) during the long period of oscillation, thus resulting in $G' > G''$. This is due to the entanglement forms a temporary network structure.

The results indicated that the purification process significantly ($p < 0.05$) influenced both elastic modulus (G') and viscous modulus (G'') of durian seed gum (Fig. 6a and b). This could be explained by the significant effect of purification process on the chemical composition of the crude gum. For example, all the purification process significantly affected the protein content and monosaccharide composition of durian seed gum, thus affecting its rheological properties. Youssef et al. (2009) also reported that the protein fraction significantly influenced the apparent viscosity, elastic modulus (G') and viscous modulus (G'') of fenugreek gum. In the current study, the crude and purified durian seed gums contained significant ($p < 0.05$) different contents of protein and monosaccharide composition, thus reflecting different rheological properties. Youssef et al. (2009) also revealed that the purified and crude fenugreek gum containing different protein content showed different rheological properties. As shown in Fig. 6a, the elastic modulus (G') of purified durian seed gum significantly ($p < 0.05$) depended on the purification method ranging from 5.8 to 31.9 Pa. On the other hand, the viscous modulus (G'') of purified durian seed gum varied from 1.3 to 10.9 Pa as compared to the control sample (2 Pa) (Fig. 6b).

The results showed that the purification using saturated barium hydroxide resulted in the significant ($p < 0.05$) strongest elastic behaviour (G'); while the purification using Fehling solution (method D) led to induce the weakest elastic behaviour (G') (Fig. 6a).

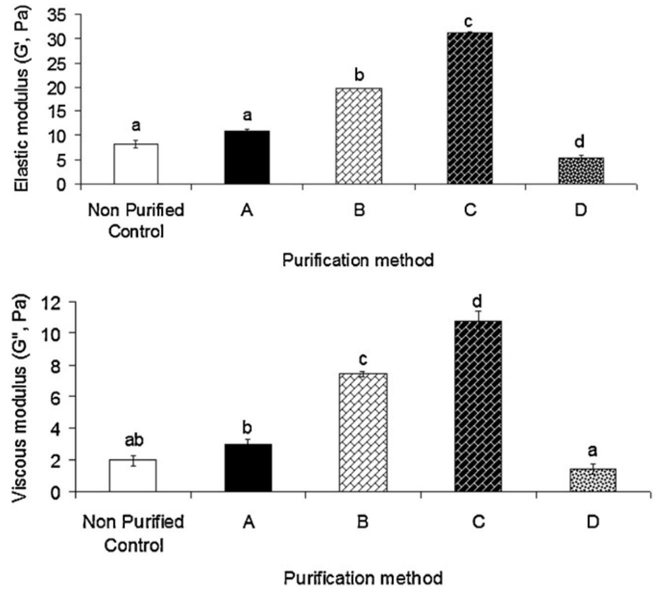

Fig. 6. Effect of different purification methods on the elastic modulus (G', a) and viscous modulus (G'', b) of durian seed gum (A: isopropanol and ethanol; B: isopropanol and acetone; C: saturated barium hydroxide; D: Fehling solution plus hydrochloric acid and ethanol); [a–d]significant differences at 95% confident level.

The purified gum C and D also showed the strongest and weakest viscous behaviour, respectively (Fig. 6b). All purification techniques (except for method D) significantly ($p < 0.05$) increased the elastic behaviour (G'). The same trend was observed for viscous modulus (G'') (Fig. 6b). Paulsson, Hagerstrom, and Edsman (1999) reported that the elastic modulus increased substantially when the ionic content increases. However, the elastic modulus (G') was found to be higher than the viscous modulus (G'') until a crossover frequency. After this point, the behaviour was reversed and the elastic response prevailed. This observation was also reported by Ibañez and Ferrero (2003). This mechanical spectrum corresponds to the entangled macromolecules since gels would show the high elastic modulus (G') throughout the frequency range (Ross-Murphy, 1995; Steffe, 1996).

3.2.2. Effect of different drying techniques on the viscoelastic behaviour

The results of dynamic oscillation tests are presented in Fig. 7a–e. In the current study, the oven dried (105 °C) samples exhibited different viscoelastic behaviour as compared to the freeze dried, spray dried and vacuum oven dried samples (Fig. 7b–e). In general, all drying techniques significantly ($p < 0.05$) changed the dynamic viscoelastic properties of different dried durian seed gums as compared to the control gum (Fig. 8a and b). The results indicated that all drying methods resulted in the significant ($p < 0.05$) reduction of both elastic (G') and viscous modulus (G''). As shown in Fig. 8a and b, the oven drying (105 °C) led to the highest significant ($p < 0.05$) reduction of both elastic (G') and viscous modulus (G'') among all drying techniques. The similar reduction trend was reported by Wang et al. (2009) who studied the effect of freeze drying, oven drying (80 °C and 105 °C), spray drying and vacuum drying on the rheological properties of flaxseed gum. They reported that all drying methods decreased the elastic behaviour (i.e., storage modulus, G′) and viscous behaviour (i.e., loss modulus, G'') of flaxseed gum in frequency sweep test. As explained by Wang et al. (2009), the reduction of elasticity might be attributed to the thermal decomposition of some components at the high temperature, thus resulting in the elasticity reduction of gum. They explained that all drying methods almost destroyed the structure of flaxseed

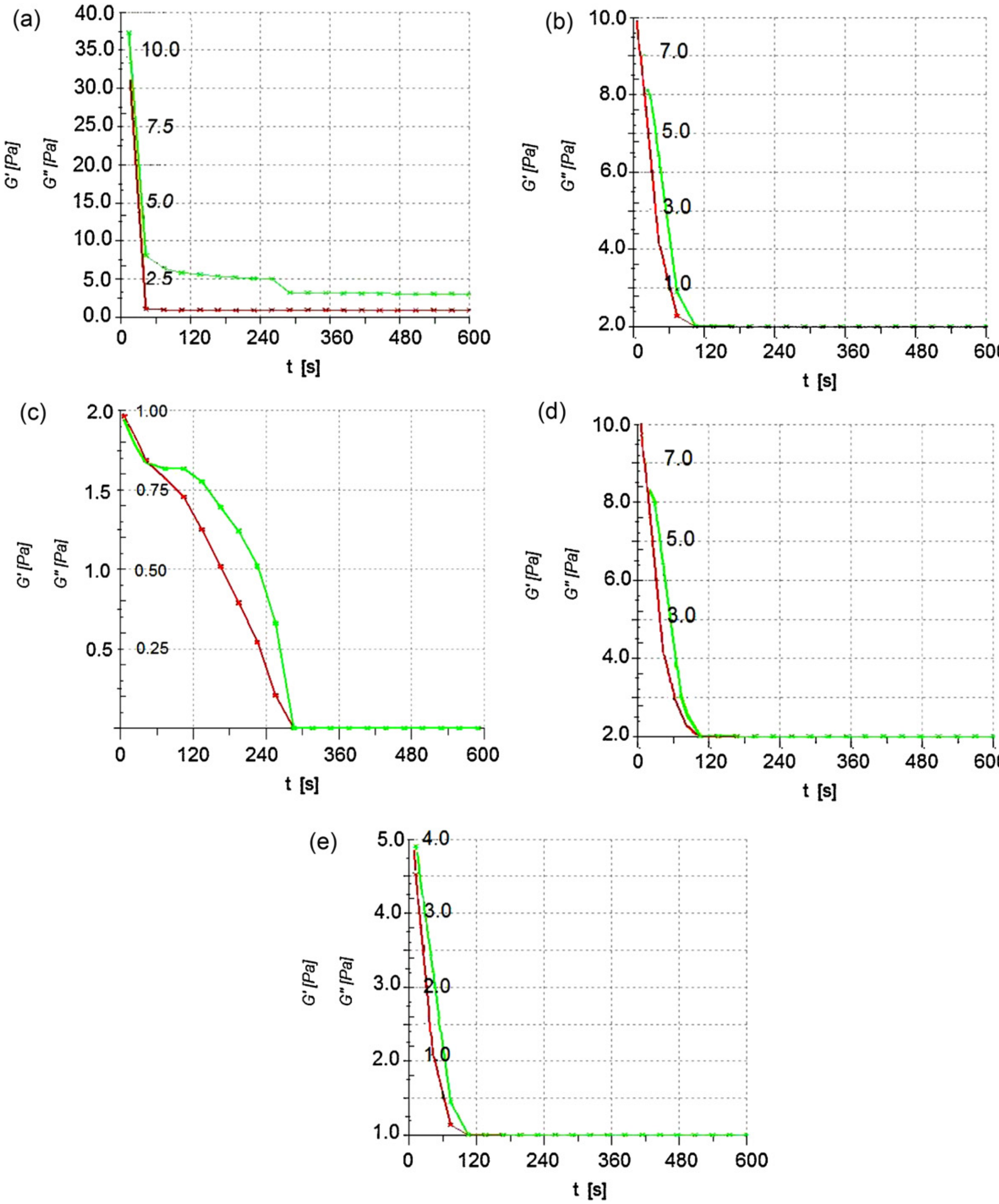

Fig. 7. The results of dynamic oscillation tests showing the elastic modulus (G') and viscous modulus (G'') of all dried durian seed gums (a: control sample; b: freeze dried gum; c: oven dried gum; d: spray dried gum; e: vacuum oven dried gum).

gum solutions more or less depending on the drying method. In fact, the effect of drying process on the chemical composition and molecular structure of durian seed gum resulted in the significant ($p < 0.05$) changes of dynamic viscoelastic properties of gum.

In the current study, the degree of elastic modulus (G') ranged from 1.57 to 11.15 Pa, lower than G′ (32.90 Pa) of the control sample (Fig. 8a). This range was comparable with the elastic modulus (G', ∼1.0–5.3 Pa) reported for the control and different-dried flaxseed gums at the low frequency 0.1 rad/s (Wang et al., 2009). The different-dried durian seed gums showed the lower elastic modulus (G') and viscous modulus (G'') than the control gum. Krokida, Maroulis, and Saravacos (2001) also reported that all dehydrated fruit and vegetable puree had lower elastic modulus (G') than the fresh samples. The results exhibited that the spray-dried and freeze-dried gums showed more significant ($p < 0.05$) elastic behaviour (G') than oven-dried (105 °C) and vacuum oven-dried gums. Conversely, the oven-dried gum followed by vacuum oven-dried gum had the least significant ($p < 0.05$) degree of elastic behaviour (G') among all dried durian seed gums (Fig. 8a). However, only low elasticity (G') does not always reflect the desirable rheological behaviour. For instance, the thermal process may decompose the molecular structure of gum, resulting in lower elasticity (G'), but the reduced elasticity caused by thermal degradation does not reflect the appropriate rheological properties. Bárcenas, O-Keller, and Rosell (2009) investigated the effect of Arabic gum, pectin and hydroxypropyl methylcellulose (HPMC) on the

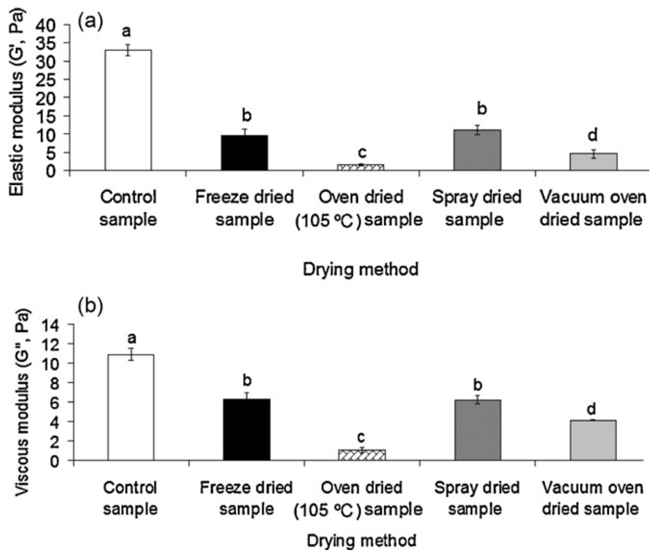

Fig. 8. Effect of different drying techniques on the elastic modulus (G', a) and viscous modulus (G'', b) of durian seed gum; [a–d] significant differences at 95% confident level.

viscoelastic behaviour of gluten. They found that the elastic modulus reduced with the heating process. As reported by Bárcenas et al. (2009), the presence of pectin and hydroxypropyl methylcellulose (HPMC) led to decrease the viscoelastic modulus during the heating process.

The results showed that the degree of viscous modulus (G'') ranged from 1.50 to 6.23 Pa, lower than G'' (10.92 Pa) of the control sample (Fig. 8b). This liquid behaviour was greater than the degree of viscous modulus (G'', ~0.55–0.92 Pa) reported for the control and different-dried flaxseed gums at the low frequency 0.1 rad/s (Wang et al., 2009). The comparison analysis shows that durian seed gum has relatively higher degree of elastic and viscous behaviour than flaxseed gum at the same frequency and gum concentration. The degree of elastic modulus (G') of durian seed gum was greater than its viscous modulus (G''), thus indicating the gel (or solid-like) behaviour of different dried durian seed gums. In fact, the solutions containing all dried durian seed gums showed predominantly elastic (gel- or solid-like) behaviour rather than viscous (liquid-like) behaviour at the low frequency. The elastic modulus (G') is related to the solid response of the material and viscous modulus (G'') is related to the fluid response of the material (Simas-Tosin et al., 2010). Therefore, sample with higher elastic value (G') has more rigidity than the sample with lower G′ (Mandala, Brito-De La Fuente, Torrestiana-Sánchez, & Katthain, 2004). If the elastic modulus (G') is lower than the viscous modulus (G''), the energy used to deform the material is dissipated viscously and the sample behaves like a liquid (Tabilo-Munizaga & Barbosa-Canovas, 2005).

Wang et al. (2009) also observed that the storage modulus (or elastic modulus, G') of flaxseed gum was higher than its loss modulus (or viscous modulus, G'') for all different dried flaxseed gums throughout the frequency range. This could be due to the low frequency applied for the oscillatory test. At low frequencies, there is sufficient time to break and reform the bonds between biopolymers during one period of oscillation. Therefore, the material shows a solid-like (or gel-like) behaviour, but some bonds have no enough time to form new linkages as the frequency increases, thus the material exhibits a liquid-like behaviour at the elevated frequency (Everett & McLeod, 2005). In the current study, the similar trend as elastic modulus (G') was observed for the viscous modulus (G''). It was found that the freeze-dried gum had insignificant ($p > 0.05$) higher degree of viscous modulus (G'') than the spray-dried, Oven-dried and vacuum oven-dried gums (Fig. 8b). The results indicated that different dried durian seed gums did not show particular elastic or viscous behaviour. In fact, viscoelastic properties of durian seed gum significantly ($p < 0.05$) mainly depended upon the drying process. As also stated by Hasan Nahid (2010), polymers do not exhibit neither as perfectly elastic nor perfectly viscous substance, but there are two deviations: (i) the strain (in a solid) or the rate of strain (in a liquid) is not directly proportional to stress; (ii) the stress may be dependent upon the value, time and rate of strain.

4. Conclusion

In the current work, the effect of different purification and drying processes on the rheological properties and viscoelastic behaviour of durian seed gum. The results indicated that all purified and dried durian seed gums showed the pseudoplastic (shear-thinning) flow behaviour. The present study revealed that durian seed gum exhibited neither as a perfectly elastic nor as a perfectly viscous behaviour. The current study revealed that the purification using saturated barium hydroxide followed by freeze drying resulted in the most desirable rheological properties for durian seed gum. Although, the oven drying is a low cost drying technique as compared to spray drying or freeze drying, but the present study reveals that the oven drying results in the weak viscoelastic behaviour. This might be due to the thermal degradation (at high temperature 105 °C) of the side chains present in the molecular structure of the durian seed gum. The present work recommends further optimization of purification using saturated barium hydroxide and freeze drying processes to establish the consistancy of rheological properties of final product. In addition, further modification process using octenyl succinic acid and/or cross linking modification is recommended to improve the rheological properties and viscoelastic behaviour of durian seed gum.

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