



# The effect of addition of flaxseed gum on the rheological behavior of mixed flaxseed gum–casein gels

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## ABSTRACT

The effect of addition of flaxseed gum on the rheological properties of mixed flaxseed gum–casein gels (flaxseed gum concentration: 0.1–0.5%, w/w and casein concentration: 15–23%, w/w) was studied using both small amplitude oscillatory and steady shear measurements. It was found that the storage ( $G'$ ) and loss ( $G''$ ) moduli increased with the increase in the flaxseed gum concentration. Both the experimentally determined  $G'$  and  $G''$  values were fitted well by Power Law model. The addition of flaxseed gum increased the Power Law constants ( $K'$  and  $K''$ ) at the same time the frequency exponents ( $n'$  and  $n''$ ) decreased. The  $G'$ ,  $G''$  of the mixed flaxseed gum–casein gels decreased with increase in temperature. The gelling temperature was found to increase linearly with the increase in flaxseed gum concentration. The apparent viscosities increased with increase in the flaxseed gum and casein concentrations, and were also fitted well by Power Law model.

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## 1. Introduction

Proteins and polysaccharides are widely used in food industry because of their unique characteristics of improving texture, stability and nutritional aspects of the products (De Kruif & Tuinier, 2001). Polysaccharides are usually added into proteins to increase the viscosity of the continuous phase. The rheological and structural properties of protein–polysaccharide mixtures depend on the interactions of these two biopolymers. The interactions between the proteins and polysaccharides depend on the molecular structure of these biopolymers and their concentration (Bunka et al., 2007). Today, increasingly large number of manufactured products contain proteins and polysaccharides in their formulation in order to control the processability, stability during storage, release kinetics of active compounds, nutritional value and mouthfeel/texture (Morris, 2004). The interactions occurring in polysaccharide–protein systems may affect textural and other quality attributes of new products during manufacture. The texture and stability of polysaccharide–protein mixture can be quantified by using rheological tests, such as small amplitude oscillatory and steady shear measurements.

Casein is one of the most commonly used proteins in food industry. It is the major milk protein component, of which about

80% of bovine milk protein is made up (Swaisgood, 1993). In milk, the casein aggregates into micelles, containing so-called colloidal calcium phosphate (CCP) and forms a continuous network in a large variety of dairy products (Nono, Nicolai, & Durand, 2011; Zhong, Daubert, & Velez, 2004a). Casein is insoluble in water and can be destabilized in different ways (Tuinier, Rieger, & De Kruif, 2003). When CCP is removed, the micelles fall apart and the different casein molecules regroup into small particles (HadjSadok, Pitkowski, Nicolai, Benyahia, & Moulay-Mostefa, 2007). The addition of emulsifying salts, for example, polyphosphate, monosodium phosphate, and disodium phosphate helps sequester the calcium inside the casein micelles thereby disintegrating the casein micelles into smaller units and increasing protein solubility (Bowland & Foegeding, 2001; Panouillé, Nicolai, & Durand, 2004).

Flaxseed gum is a polysaccharide gum derived from flaxseed. Recently it has attracted increasing research interests because of its unique nutritive value as a dietary fiber and also because of its emulsion-stabilizing properties (Oomah, 2001; Wang, Li, Wang, & Xue, 2011). As a kind of water soluble dietary fiber, flaxseed gum is effective in reducing blood glucose and cholesterol in type II diabetic patients and also found to be beneficial to heart disease and colorectal cancer (Cunnane et al., 1993; Tarpila, Wennberg, & Tarpila, 2005; Thakur, Mitra, Pal, & Rousseau, 2009). It has been reported that the flaxseed gum has high viscosity, high water-holding capacity, good emulsifying properties and weak gel-forming properties (Chen, Xu, & Wang, 2006; Fedeniuk & Biliaderis, 1994). Thus, it can be used as emulsifier and stabilizer to replace

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### Nomenclature

$G'$	storage modulus, Pa
$G''$	loss modulus, Pa
$K$	consistency index, Pa·s <sup>n</sup>
$K'$	Power Law model constant, Pa s <sup>n</sup>
$K''$	Power Law model constant, Pa s <sup>n</sup>
$n$	flow behavior index (dimensionless)
$n'$	frequency exponent (dimensionless)
$n''$	frequency exponent (dimensionless)
$R^2$	correlation coefficient (dimensionless)
$\dot{\gamma}$	shear rate, s <sup>-1</sup>
$\eta$	apparent viscosity, Pa·s
$\tau$	shear stress, Pa
$\omega$	angular frequency, rad/s
$\mu_a$	shear dependent apparent viscosity, Pa·s

most of the non-gelling gums currently in use in food manufacturing (Chen et al., 2006; Cui & Mazza, 1996; Wang, Wang, Li, Xue, & Mao, 2009).

Polysaccharides (pectin, modified starch, xanthan gum, locust bean gum, guar gum, algin, etc.) are often added in dairy products to stabilize their structure, to enhance viscosity and to alter textural characteristics (Simeone, Alfani, & Guido, 2004; Thaiudom & Goff, 2003). The effect of the addition of polysaccharides in casein or in other dairy products has been studied extensively. For example,  $\beta$ -glucan was found to interact with caseinate to affect overall viscoelasticity and texture of products (Polyakov, Grinberg, & Tolstoguzov, 1997; Tolstoguzov, 1997). Kontogiorgos, Ritzoulis, Biliaderis, and Kasapis (2006) studied the microstructural and mechanical properties of acid-set mixtures of sodium caseinate/barley  $\beta$ -glucans in an effort to evaluate the effect of these polysaccharides on the texture of fermented dairy products.  $\kappa$ -Carrageenan is often added to dairy products to prevent phase separation and increase the elasticity of the mixtures. Pectin is also found to affect the rheological properties of casein gels (Matia-Merino, Lau, & Dickinson, 2004). All of these beneficial characteristics result from the interactions between casein micelles and the above mentioned hydrocolloids (Ji, Corredig, & Goff, 2008; Langendorff et al., 2000; Nono et al., 2011; Schorsch, Jones, & Norton, 2000).

To the best of our knowledge, there is no published study on the interaction between flaxseed gum and casein. The objective of this work was to study the effect of addition of flaxseed gum on the rheological properties of mixed flaxseed gum–casein gels. To meet this objective, we performed series of rheological experiments where flaxseed gum in varying concentration was added to the casein. Both the small amplitude oscillatory and steady shear measurements were carried out. This study will provide better understanding of the behavior of mixed flaxseed gum–casein gels and the way these interactions can be exploited in food manufacturing.

## 2. Materials and methods

### 2.1. Materials

Flaxseed (6.5%, w/w moisture content) was obtained from the Hebei province of China. Casein (BR) was purchased from Sinopharm Chemical Reagent Co., Ltd. (Beijing, China). This casein powder had a nitrogen content of 13.5%, and therefore, the protein concentration was calculated to be 84.4% using a standard conversion factor of 6.25. Analytical grade sodium chloride, monosodium and disodium phosphates were purchased from a local vendor.

### 2.2. Flaxseed gum extraction

Hundred grams of flaxseed was weighed and subsequently washed with water in a sieve to remove the dust and soil. Any other foreign matter entrapped in flaxseed sample was removed manually. The flaxseed was then soaked in de-ionized water (900 mL). The flaxseed–water mixture was stirred in a 60 °C water bath, at 300 rpm for 5 h using a magnetic stirrer (Kexi Instruments Company, Jintan, China) following a previously reported extraction method (Cui, 2001; Wang et al., 2009). The extracted flaxseed gum solution was filtered through 40-mesh screen, precipitated using two volumes of 95% ethanol and then collected by using a glass rod with gentle stirring as suggested by Wang et al. (2011). The precipitated flaxseed gum was intertwined around the glass rod and allowed itself to be collected. The flaxseed gum extracted in this way was subsequently dried in a hot air oven (Model 101-3, Shanghai Luda Experimental Instrument Co., Shanghai, China) at 80 °C for 8 h. The protein content of flaxseed gum extracted in this way was 14.4% (w/w) as determined by Kjeldahl method using a FOSS Kjeltac 2300 analyzer (FOSS Co., Höganäs, Sweden). The nitrogen content was converted into protein value using a conversion factor of 6.25.

### 2.3. Sample preparation

The flaxseed gum (0.1–0.5%, w/w)–casein (15–23%, w/w) gels were prepared according to the method suggested by Zhong et al. (2004a). First, suspension of suitable amount flaxseed gum in water was prepared and heated to 50 °C. Then, other chemicals (1.25 g Na<sub>2</sub>HPO<sub>4</sub>, 0.195 g NaH<sub>2</sub>PO<sub>4</sub>, and 2 g NaCl per 50 g solution) were added while stirring to make complete dissolution. Finally casein (15–23%, w/w) was added while stirring. At this point, the mixed sample appeared to be a viscous paste. This viscous sample was then stored in a refrigerator at 4 °C to form a gel. The samples were equilibrated at 4 °C over night before the rheological tests. The casein gel (15–23%, w/w) without gum was prepared in the same way.

### 2.4. Rheological study of flaxseed gum–casein mixtures

Rheological measurements were carried out using AR2000ex Rheometer (TA Instruments Ltd., Crawley, UK). The temperature was controlled by a water bath connected to the Peltier system in the bottom plate. The linear viscoelastic region was determined for each sample through strain sweeps at 1 Hz. The viscoelastic properties (storage modulus  $G'$ , loss modulus  $G''$ , and phase angle  $\delta$ ) of flaxseed gum–casein mixtures were measured within the linear viscoelastic region. Before each following experiment, the sample was placed on the bottom plate, which was set to 20 °C. This temperature could insure the quick melt of the sample. After melt, the parallel plate was immediately put down and suitable amount of silicone oil was used to prevent evaporation. Then the temperature of the bottom plate was set to designed experimental temperature. An equilibration time of 3 min was maintained before the measurement.

#### 2.4.1. Frequency sweep measurements

The frequency sweep tests were performed at 5 °C over the angular frequency range of 1–100 rad/s. The strain amplitude for the frequency sweep measurements was selected as 2% based on the strain sweep results in order to be in the linear viscoelastic region for all the samples. An aluminum parallel plate geometry (40 mm diameter, 1 mm gap, 1° 0' 40'') was chosen for the frequency sweep measurements.

In order to describe the frequency dependence of  $G'$  and  $G''$  for the gels, the following equation was used in the prevailing frequency range (Ikeda & Nishinari, 2001):

$$G' = K' \cdot \omega^{n'} \quad (1)$$

$$G'' = K'' \cdot \omega^{n''} \quad (2)$$

where,  $\omega$  is the angular frequency (rad/s),  $K'$  and  $K''$  are Power Law constants and  $n'$  and  $n''$  can be referred to as the frequency exponents. The value of  $n'$  and  $n''$  can provide useful information with respect to the viscoelastic nature of food materials (Özkan, Xin, & Chen, 2002).

#### 2.4.2. Temperature ramp measurements and gelling temperature measurements

The temperature ramp tests were performed from 70 °C to 5 °C using a ramp-down rate of 2.0 °C/min. In this tests the oscillation stress of 0.7958 Pa under single angular frequency of 6.283 rad/s were used. An equilibration time of 3 min was maintained before each measurement. An aluminum parallel plate geometry (40 mm diameter, 1 mm gap) was chosen for these temperature ramp measurements. The temperatures at which  $G'$  and  $G''$  crossed were taken as the gelling temperatures during cooling (Chen et al., 2006).

#### 2.4.3. Continuous shear measurements

The continuous shear tests were carried out at 60 °C over the shear rate range of 1–100 s<sup>-1</sup> to measure apparent viscosity. Steel cone geometry (40 mm diameter, 26 μm gap, 1°0'40" degree) was chosen for these continuous shear measurements.

In order to describe the variation in the flow properties of flaxseed gum–casein mixtures under continuous shear, the Power Law model (Eq. (3)) was used to represent the experimental data. The Power Law model is extensively used to model and interpret the flow properties of non-Newtonian liquids in theoretical analysis as well as in practical engineering applications (Barñes, Hutton, & Walters, 1989).

$$\tau = K \dot{\gamma}^n \quad (3)$$

where,  $\dot{\gamma}$  is the shear rate (s<sup>-1</sup>),  $\tau$  is the shear stress (Pa),  $n$  is the flow behavior index (dimensionless), and  $K$  is the consistency index (Pa s<sup>n</sup>). The exponent  $n$  is the flow behavior index and reflects the Newtonian fluids ( $n=1$ ), non-Newtonian shear-thinning or pseudoplastic ( $0 < n < 1$ ) and non-Newtonian shear-thickening fluids ( $n > 1$ ). The consistency coefficient  $K$  is related to apparent viscosity as shown by Eq. (4) (Corzo-Martínez, Moreno, Villamiel, & Harte, 2010).

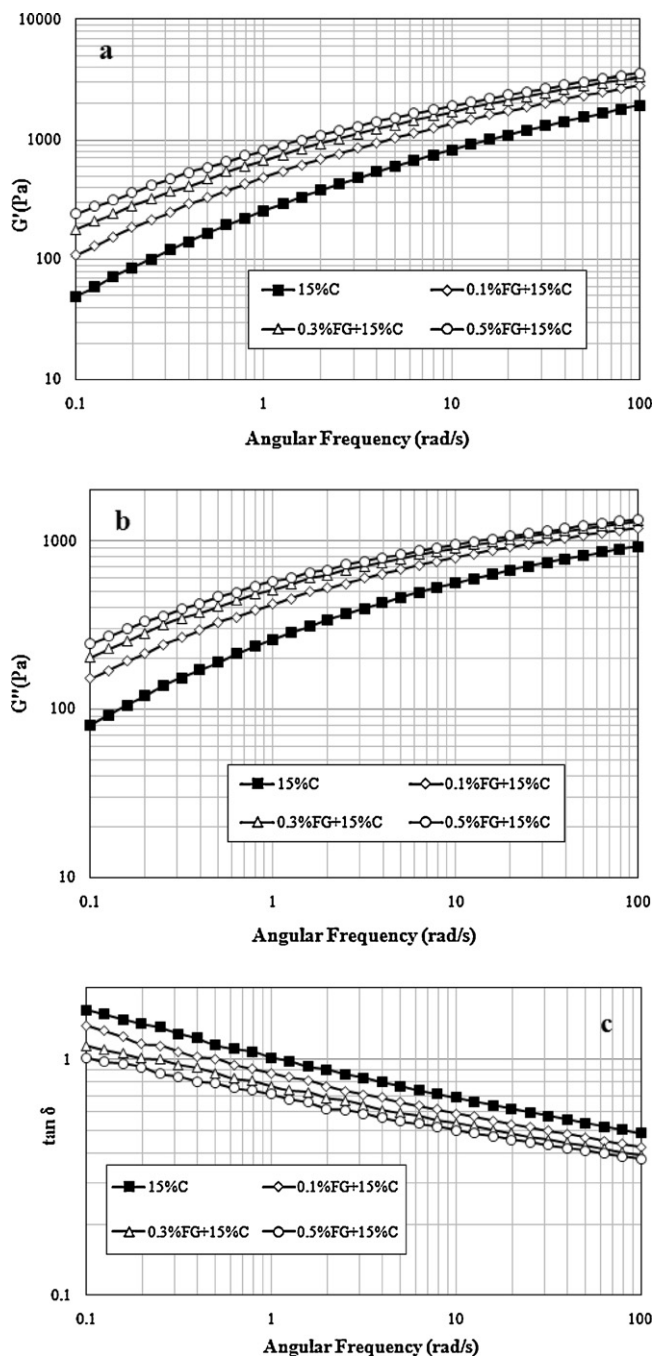
$$K = \mu_a (\dot{\gamma})^{1-n} \quad (4)$$

where,  $\mu_a$  is shear dependent apparent viscosity (Pa s).

#### 2.5. Statistical analysis

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

The results were evaluated by analysis of variance (ANOVA), and significant differences ( $p < 0.05$ ) between treatments were evaluated by Duncan's multiple comparison using the SAS software (SAS Institute Inc., Cary, NC, USA). The continuous shear data and frequency sweep data were modeled according to Power Law model, using the non-linear regression feature in SPSS 13.0 (SPSS Inc., Chicago, USA).



**Fig. 1.** The  $G'$  (panel a),  $G''$  (panel b),  $\tan \delta$  (panel c) as a function of angular frequency at 5 °C for 15% casein gel, and flaxseed gum–15% casein mixed gels (C stands for casein, FG stands for flaxseed gum).

### 3. Results and discussions

#### 3.1. Frequency sweep measurements

Fig. 1 shows the results of storage modulus ( $G'$ ), loss modulus ( $G''$ ), and loss tangent ( $\tan \delta$ ) as a function of frequency at 5 °C and a strain amplitude of 2.0% for 15% casein, and the flaxseed gum–casein mixtures (0.1–0.5% flaxseed gum concentration). It can be observed from this figure that the moduli ( $G'$ ,  $G''$ ) of all samples increased with increase in the angular frequency. Panouillé, Benyahia, Durand, and Nicolai (2005) reported similar frequency dependency in the moduli ( $G'$ ,  $G''$ ) of casein suspensions at 20 °C.  $G'$  and  $G''$  increased along with the increase in flaxseed gum

**Table 1**The variation of  $K'$ ,  $K''$ ,  $n'$  and  $n''$  of mixed flaxseed gum–casein gels as a function of flaxseed gum and protein concentration (C stands for casein, FG stands for flaxseed gum).<sup>A,B</sup>

FG conc. (%)	C conc. (%)	$G' = K' \cdot \omega^{n'}$			$G'' = K'' \cdot \omega^{n''}$		
		$n'$	$K'$	$R^2$	$n''$	$K''$	$R^2$
0	15	0.535 ± 0.028 <sup>a</sup>	213.4 ± 21.0 <sup>d</sup>	0.975	0.351 ± 0.012 <sup>a</sup>	230.4 ± 6.6 <sup>c</sup>	0.968
0.1	15	0.469 ± 0.014 <sup>b</sup>	415.0 ± 35.5 <sup>c</sup>	0.978	0.294 ± 0.009 <sup>b</sup>	376.8 ± 21.7 <sup>b</sup>	0.964
0.3	15	0.430 ± 0.013 <sup>c</sup>	552.7 ± 41.8 <sup>b</sup>	0.979	0.268 ± 0.008 <sup>c</sup>	450.0 ± 16.4 <sup>a</sup>	0.963
0.5	15	0.383 ± 0.015 <sup>d</sup>	622.1 ± 62.8 <sup>a</sup>	0.981	0.245 ± 0.007 <sup>d</sup>	466.0 ± 51.3 <sup>a</sup>	0.968
0	17	0.393 ± 0.005 <sup>a</sup>	983.1 ± 42.7 <sup>b</sup>	0.974	0.219 ± 0.002 <sup>a</sup>	726.3 ± 20.3 <sup>a</sup>	0.957
0.1	17	0.385 ± 0.001 <sup>a</sup>	1069.1 ± 4.9 <sup>b</sup>	0.970	0.208 ± 0.001 <sup>b</sup>	773.9 ± 8.0 <sup>a</sup>	0.954
0.3	17	0.353 ± 0.011 <sup>b</sup>	1510.4 ± 182.2 <sup>a</sup>	0.968	0.180 ± 0.008 <sup>c</sup>	1004.4 ± 80.4 <sup>b</sup>	0.957
0.5	17	0.328 ± 0.014 <sup>c</sup>	1751.7 ± 207.3 <sup>a</sup>	0.972	0.168 ± 0.007 <sup>d</sup>	1060.5 ± 79.9 <sup>b</sup>	0.964
0	19	0.362 ± 0.005 <sup>a</sup>	1504.5 ± 30.4 <sup>c</sup>	0.967	0.186 ± 0.004 <sup>a</sup>	1021.0 ± 16.4 <sup>c</sup>	0.961
0.1	19	0.303 ± 0.010 <sup>b</sup>	2725.8 ± 243.8 <sup>b</sup>	0.970	0.144 ± 0.005 <sup>b</sup>	1449.2 ± 146.3 <sup>b</sup>	0.979
0.3	19	0.286 ± 0.002 <sup>b,c</sup>	3164.8 ± 23.5 <sup>a</sup>	0.973	0.136 ± 0.001 <sup>b</sup>	1668.6 ± 19.8 <sup>a</sup>	0.980
0.5	19	0.265 ± 0.027 <sup>c</sup>	3155.0 ± 19.2 <sup>a</sup>	0.957	0.121 ± 0.025 <sup>b</sup>	1618.3 ± 33.0 <sup>a</sup>	0.939
0	21	0.264 ± 0.004 <sup>a</sup>	4134.9 ± 232.4 <sup>b</sup>	0.977	0.130 ± 0.005 <sup>a</sup>	1990.7 ± 96.6 <sup>b</sup>	0.983
0.1	21	0.243 ± 0.010 <sup>b</sup>	5725.1 ± 904.8 <sup>a</sup>	0.980	0.120 ± 0.005 <sup>b</sup>	2520.6 ± 301.0 <sup>a</sup>	0.996
0.3	21	0.236 ± 0.007 <sup>b,c</sup>	5846.8 ± 550.5 <sup>a</sup>	0.984	0.117 ± 0.003 <sup>b</sup>	2495.9 ± 156.4 <sup>a</sup>	0.997
0.5	21	0.225 ± 0.002 <sup>c</sup>	6798.4 ± 152.6 <sup>a</sup>	0.986	0.115 ± 0.003 <sup>b</sup>	2780.6 ± 85.3 <sup>a</sup>	0.992
0	23	0.254 ± 0.009 <sup>a</sup>	5617.7 ± 591.9 <sup>b</sup>	0.981	0.128 ± 0.006 <sup>a</sup>	2580.8 ± 189.3 <sup>b</sup>	0.989
0.1	23	0.223 ± 0.002 <sup>b</sup>	7977.7 ± 305.3 <sup>a</sup>	0.988	0.116 ± 0.003 <sup>b</sup>	3195.3 ± 113.7 <sup>a</sup>	1.000
0.3	23	0.221 ± 0.003 <sup>b</sup>	8374.5 ± 373.7 <sup>a</sup>	0.988	0.120 ± 0.002 <sup>b</sup>	3284.2 ± 84.6 <sup>a</sup>	0.996
0.5	23	0.227 ± 0.004 <sup>b</sup>	7887.1 ± 326.7 <sup>a</sup>	0.987	0.123 ± 0.004 <sup>a,b</sup>	3190.8 ± 124.6 <sup>a</sup>	0.996

<sup>A</sup> Values represent the mean ± standard deviation of triplicate tests.<sup>B</sup> Values in a column with different superscripts were significantly different ( $p < 0.05$ ).

concentration. The  $G'$  values were lower than  $G''$  values in the first place, however, the  $G'$  values exceeded the  $G''$  values after 1 rad/s within the experimental range for all the samples. The trends of variation of the  $G'$  and  $G''$  with frequency for the casein only gel and the mixed flaxseed gum–casein gels were similar. These observations suggested that both the casein gel and the mixed flaxseed gum–casein gel had a similar biopolymer gel network (Mohammed, Hember, Richardson, & Morris, 1998). However, the addition of flaxseed gum significantly increased the magnitude of  $G'$  and  $G''$  values, indicating that the network of the mixed flaxseed gum–casein gel is much stronger than the casein only gel. It implies that the addition of flaxseed gum increases the moduli of the flaxseed gum–casein gels by modifying their structures. The  $\tan \delta = (G''/G')$  values decreased with the increase in the frequency in all the samples, suggesting that the frequency dependence of  $G'$  was considerably greater than that of  $G''$ .

The effect of flaxseed gum concentration and casein concentration on  $K'$ ,  $K''$ ,  $n'$ , and  $n''$  parameters for the mixed flaxseed gum–casein gels is summarized in Table 1. The storage and loss moduli obeyed the Power Law model with correlation coefficients ( $R^2$ ) being higher than 0.954. These results indicated that the addition of flaxseed gum significantly changes the dynamic shear properties of the flaxseed gum–casein mixtures compared with casein only gels at all the casein concentration tested. As to 15% (w/w) casein concentration, the addition of all the tested flaxseed gum concentrations effectively increased the  $K'$  and  $K''$  values (or  $G'$  and  $G''$  values). When casein concentration was maintained at 17% (w/w), there was no obvious difference between  $K'$  and  $K''$  values (or  $G'$  and  $G''$  values) of flaxseed gum–casein mixed gels containing 0.3% (w/w) and 0.5% (w/w) flaxseed gum. When the casein concentration increased above 19% (w/w), the increase in the gum concentration hardly made any impact on the  $K'$  and  $K''$  values. However, for all of these experiments it was observed that the  $K'$  and  $K''$  (or  $G'$  and  $G''$ ) values of the flaxseed gum–casein mixed gels were greater than those of the casein only gels at all the casein concentrations tested. This can be attributed to the formation of stronger network due to the presence of flaxseed gum. In similar studies, Trčková, Štetina, and Kánský (2004) found that the elasticity of carrageenan–casein gels increased linearly with the

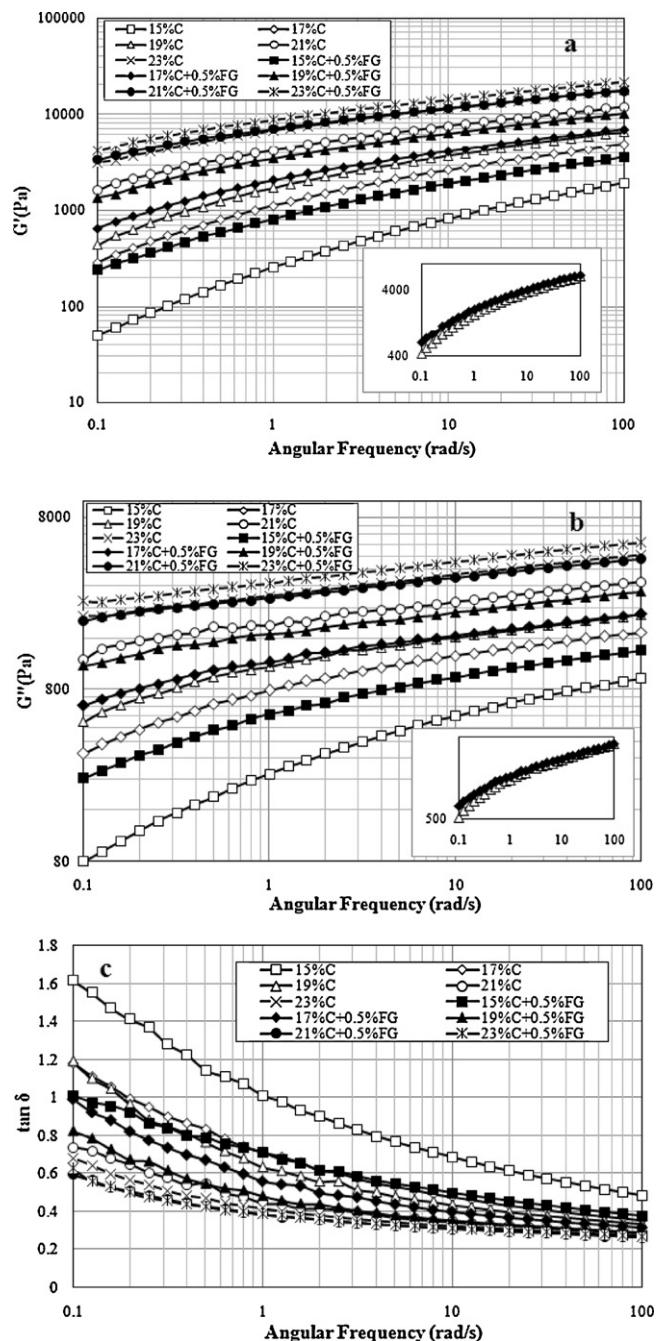
increase in the carrageenan concentration and quadratically with the increase in the casein concentration. These authors also noted some deviation from the above observations at higher concentration level of both components. It has also been reported that the increase in the locust bean gum content resulted into an overall increase in both  $G'$  and  $G''$  of samples (Perrechil, Braga, & Cunha, 2009) at a fixed caseinate concentration of 0.5% (w/v). Similarly, it has now established that the exopolysaccharides can also increase the firmness of casein gels (Girard & Schaffer-Lequart, 2007). For  $n'$  and  $n''$  values, the casein gels had a relatively higher value than the flaxseed gum–casein gels in all the tested casein concentrations. These observations suggest that the addition of flaxseed gum decreases the frequency sensitivity of flaxseed gum–casein gels compared to casein only gels.

Fig. 2 presents the variation of  $G'$ ,  $G''$ , and  $\tan \delta$  as a function of angular frequency for casein only gels and 0.5% (w/w) flaxseed gum–casein mixed gels. At all the flaxseed gum concentration tested (data was not shown), the  $G'$  and  $G''$  values increased with increase in casein concentration. This is due to the formation of increasingly complex structure with higher protein concentration at same volume. However, as can be seen from the inset graph, the 17% (w/w) casein–0.5% (w/w) flaxseed gum gel was stiffer than 19% (w/w) casein gel. This observation suggests that the addition of 0.5% (w/w) flaxseed gum is more effective in increasing both the moduli in the gel than 2% (w/w) casein. Thus, in order to enhance the solid-like behavior (elasticity) of casein gels, the addition of flaxseed gum is preferable. This means that by adding very small amount of flaxseed gum amount of casein required can be reduced. The  $\tan \delta$  values of casein only gels and 0.5% (w/w) flaxseed gum–casein gels all decreased with the increasing in the casein concentration.

### 3.2. Temperature ramp measurements and gelling properties

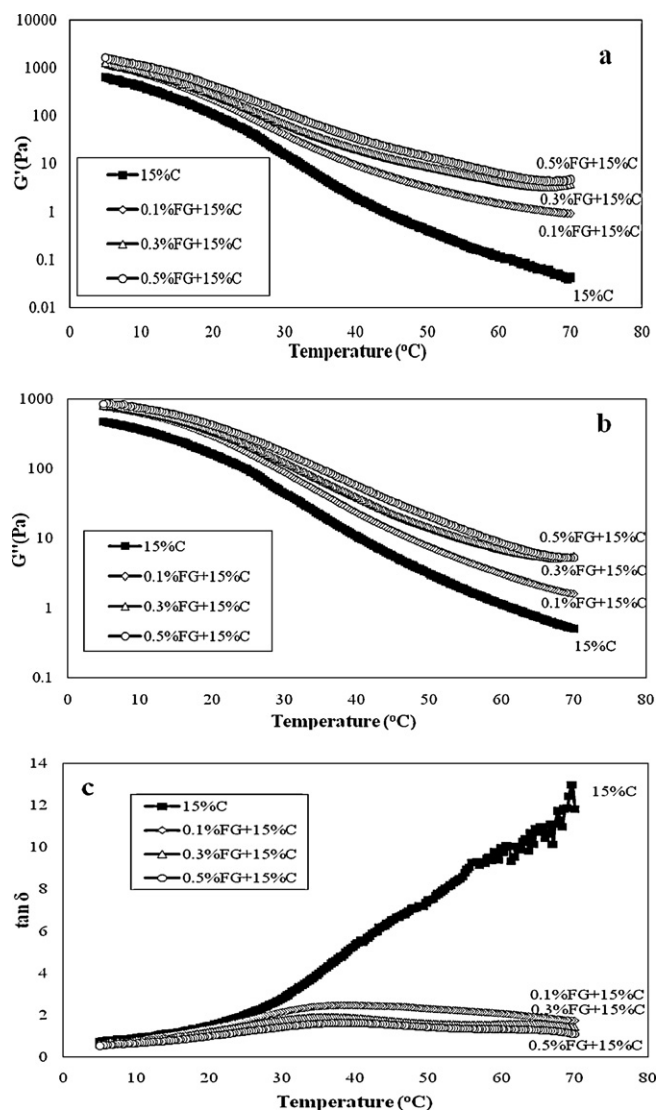
The rheological data for 15% (w/w) casein gels in the presence of varying flaxseed gum concentration from 0.1 to 0.5% (w/w) as a function of temperature are presented in Fig. 3. As can be seen from this figure that both the moduli increased with increase in the flaxseed gum concentration Fig. 3 (panel a and b) and decreased with increase in the sample temperature. Similar trend of decrease





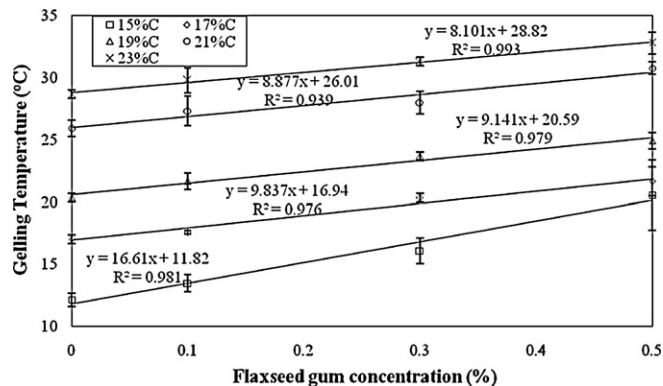
**Fig. 2.** The  $G'$  (panel a),  $G''$  (panel b), and  $\tan \delta$  (panel c) as a function of angular frequency at 5 °C for casein gel (15–23%), and 0.5% flaxseed gum–casein (15–23%) mixtures (C stands for casein, FG stands for flaxseed gum).

in moduli values with increase in temperature were observed and reported by Zhong, Daubert, and Velev (2004b) and Panouillé et al. (2005). The trends of variation in phase angle are quite different among casein only gels and flaxseed gum–casein mixed gels. For example, in the case of 15% (w/w) casein only gel, the curve decreased quite rapidly from 70 °C to 20 °C, then decreased gradually below about 20 °C. The  $\tan \delta$  curve of 15% (w/w) casein–flaxseed gum gel exhibited no obvious change while cooling from 70 °C to about 20 °C. Subsequently, it decreased slightly, indicating that the rate of increase in the  $G'$  and  $G''$  is similar in the first stage, however, the  $G''$  increased faster than  $G'$  in the subsequent stage. Thus these results indicate that the presence of flaxseed gum lessen the sensitivity of  $G'$  to temperature.

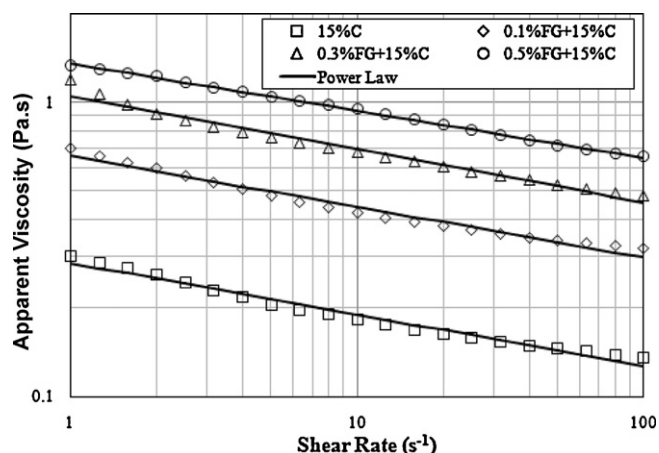


**Fig. 3.** The variation of  $G'$  (panel a),  $G''$  (panel b) and  $\tan \delta$  (panel c) with temperature from 70 °C to 5 °C for 15% casein gel, and flaxseed gum–casein mixed gels (C stands for casein, FG stands for flaxseed gum).

The effect of addition of flaxseed gum on the gelling temperature of flaxseed gum–casein gels was measured and is shown in Fig. 4. As shown in this figure, the temperature at which the protein–gum mixture solutions gelled (gelling temperature) increased along



**Fig. 4.** The effect of flaxseed gum concentration on gelling temperature of flaxseed gum–casein mixtures. Symbols represent the experimental data and the solid lines represent the linear regression results (C stands for casein).



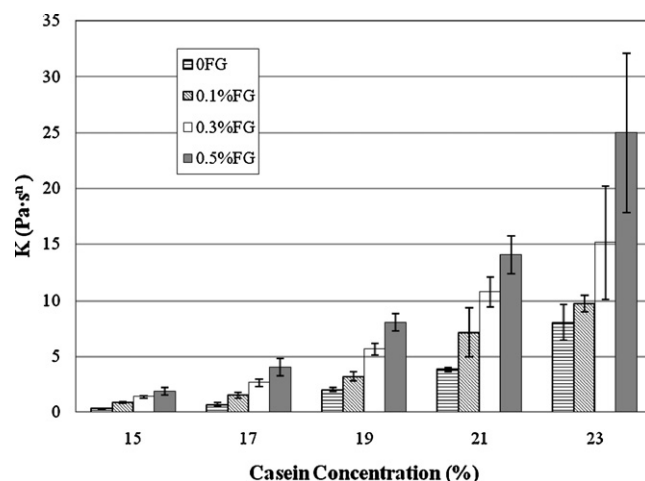
**Fig. 5.** The effect of flaxseed gum concentration on the apparent viscosity of flaxseed gum-casein mixed gels at 60°C (C stands for casein, FG stands for flaxseed gum). Symbols represent the experimental data and the solid lines represent the fitted Power Law model lines.

with the increase in the flaxseed gum concentration at all the tested casein concentrations. This behavior might have been resulted from the interaction between the adsorbed flaxseed gum and casein micelles. The flaxseed gum adsorbed onto the casein micelles leading to the increase in the mean size of the aggregates. These bigger aggregates facilitated the formation of the continuous network (Langendorff et al., 2000; Nono et al., 2011). Similar increase in the gelling temperature as a function of the increase in the concentration of polysaccharide in polysaccharide-protein mixtures has been reported by Braga and Cunha (2004), Oakenfull, Miyoshi, Nishinari, and Scott (1999), Olsson, Langton, and Hermansson (2002), Tavares, Monteiro, Moreno, and Lopes Da Silva (2005). The gelling temperature of the gum-casein mixed gel was also found to increase with increase in the casein concentration. Oakenfull et al. (1999) found that the gelling temperature of carageenan-casein mixed gels first decreased with increase in the sodium caseinate concentration (<10 g/kg) in caseinnate- $\kappa$ -carrageenan mixed gels. The gelling temperature subsequently increased with increase in the sodium caseinate concentration (10–50 g/kg).

A regression analysis was carried out to relate the gelling temperature with the flaxseed gum concentrations using linear model. The linear model fitted the experimental data with coefficient of determination ( $R^2$ ) being greater than 0.995. The linear models were found to be adequate not only to describe the tendency of the gelling temperatures, but also to predict the gelling temperature with the concentration of the flaxseed gum which is not included in this study. The slope of gelling temperature decreased along with increase in the casein concentration. This observation suggests that at higher casein concentration the addition of flaxseed gum will have less and less effect in gelling properties of flaxseed gum-casein mixtures. However, within the casein concentration range of 15–23% (w/w), the addition of flaxseed gum can significantly increase the gelling temperature, indicating that the flaxseed gum is capable of altering the gelling properties of flaxseed gum-casein mixed gels.

### 3.3. Continuous shear measurements

The flow curves for flaxseed gum-casein (15%, w/w concentration) mixed gels at 60°C are provided in Fig. 5. As can be seen from this figure, the apparent viscosity of all the samples decreased with increase in the shear rate. This indicates that these mixed gel samples exhibit shear-thinning behavior. The addition of flaxseed gum did not change the shear-thinning behavior of the mixed



**Fig. 6.** The effect of flaxseed gum concentration and casein concentration on the consistency indices ( $K$ ) of the flaxseed gum-casein mixtures obtained by applying the Power Law model to the steady shear data (C stands for casein, FG stands for flaxseed gum).

gels as indicated by the almost parallel flow curves. The increase in the gum concentration also increased the apparent viscosity of the mixed gels significantly ( $p < 0.05$ ) compared to the control (casein gel with no flaxseed gum). This can be attributed to the interaction between flaxseed gum and casein molecules. In similar note, it has been reported that the addition of locust bean gum in sodium caseinate had increased the apparent viscosity in locust bean gum-sodium caseinate systems and those mixed gels also exhibited shear-thinning behavior (Perrechil et al., 2009). A similar trend of increase in the apparent viscosity was observed when xanthan gum was added to sodium caseinate (Hadjsadok, Moulai-Mostefa, & Rebiha, 2010).

The Power Law model was used to predict the experimental apparent viscosity values as a function of shear rate. The coefficient of determination ( $R^2$ ) in these predictions were higher than 0.976 and are shown in Fig. 5 as solid lines. The experimental shear stress versus shear rate data for the flaxseed gum-casein mixed gels at different flaxseed gum concentrations and different casein concentrations were also fitted well with the Power Law model ( $R^2 > 0.976$ ). The effect of flaxseed gum concentration and casein concentration on the consistency indices ( $K$ ) of flaxseed gum-casein mixed gels is shown in Fig. 6. The consistency indices ( $K$ ) values significantly ( $p < 0.05$ ) increased with the addition of flaxseed gum at any fixed casein concentration tested. The consistency indices ( $K$ ) also increased with the increase in the flaxseed gum concentration. Similar trend of increase in  $K$  values has been previously reported with the increase in the concentration of gellan concentration in gellan-milk protein blends (Kiani, Ebrahimzadeh-Mousavi, Djomeh, & Yarmand, 2008).

### 4. Conclusion

The rheological properties of flaxseed gum-casein mixed gels were studied using small amplitude oscillatory and steady shear measurements. The storage ( $G'$ ) and loss ( $G''$ ) moduli were measured using frequency sweep tests and modeled using Power Law equation. At 15% (w/w) casein concentration, both the  $G'$  and  $G''$  increased with increase in the flaxseed gum concentration (from 0.1 to 0.5%, w/w). The addition of flaxseed gum significantly ( $p < 0.05$ ) increased the Power Law model constants ( $K'$  and  $K''$ ) and decreased frequency exponents ( $n'$  and  $n''$ ) values within the casein concentration range tested. In the mixed flaxseed gum-casein gels, the increase in the casein concentration led to increase in the  $G'$  and

$G''$  values, however the increase in these value due to increase in flaxseed gum concentration was much higher. The  $G'$ ,  $G''$  of the mixed flaxseed gum–casein gels decreased with increase in temperature; however, both of these values at higher flaxseed gum concentrations were always higher at any given gel temperature. The gelling temperature was found to increase linearly with the increase in flaxseed gum concentration. However, the slope of this linear line decreased with the increase in the casein concentration. It was found that the apparent viscosity of mixed flaxseed gum–casein gels increased with the increase in the flaxseed gum concentration (from 0.1 to 0.5%, w/w) within the casein concentration range (from 15 to 23%, w/w) tested. The Power Law model was suitable for representing the apparent viscosity of the mixed flaxseed gum–casein mixtures measured as a function of shear rate.

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