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Rheological study and fractal analysis of flaxseed gum gels

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ABSTRACT

The rheological properties and fractal dimensions of flaxseed gum gels were analyzed in various ionic strength values ranging between 0 and 1000 mM. The rheological properties of flaxseed gum were significantly influenced by the ionic strength. The gel strength first increased then decreased with the increasing of ionic strength and the strongest gel was obtained when the ionic strength was 400 mM. The calculated fractal dimensions of the flaxseed gum gels were 2.06–2.49 or 1.42–2.18, based on the model selected and the ionic strength applied. These results would help to understand the microstructure of the flaxseed gum gels and the influence of ionic strength on that. However, the difference in results between the two models implied that further study is needed to find suitable model for flaxseed gum gels.

1. Introduction

Fractal structure is an illustration of the initial structure of macro molecules (Iannaccone & Khokha, 1996). The fractal structure was commonly used to describe the branches complexity of the molecules, and also reflect the type of organization of those branches. The fractal dimension, referred as d_f, is introduced to describe this kind of complexity. Fractal analysis is now widely used in the studies on the microstructure of biomacromolecules, such as proteins, carbohydrate polysaccharides, and DNA (Su, Wu, & Wang, 2009).

Several methods have been used to study the fractal structure and calculate the d_f value. Those methods could basically be classified as direct methods (e.g. confocal scanning laser microscopy, dynamic light scattering, small-angle X-ray scattering), and indirect methods (e.g. rheological and acoustic methods) (Hagiwara, Kumagai, Matsunaga, & Nakamura, 1997; Hagiwara, Kumagai, & Nakamura, 1996; Matsumoto, Kawai, & Masuda, 1992; Wu, Xie, Lattuada, & Morbidelli, 2005). Rheological methods are commonly used in fractal analysis on gels, as it reflects the gel microstructure based on the macro mechanical properties, which is easily measured. Several rheological models have been developed to calculate the d_f value of gels.

Cates (1984) has developed a model related complex viscosity with the fractal dimension, in the absence of excluded-volume and entanglement effects. This model could be widely applied to the statics and dynamics of a large range of physically interesting fractals, such as gelation clusters, branched polymers, and flexible types of fractal aggregates. Bremer, Vliet, and Walstra (1989) have developed a model to describe gel formation and structure using the concept of fractal geometry. The model was in good agreement with the dependence of the storage modulus of the gels on the volume fraction, thus was successfully used in fractal analysis of acid casein gels.

Shih, Shih, Kim, Liu, and Aksay (1990) have developed a model to calculate the fractal dimension based on the critical strain and the corresponding storage modulus. The gels were characterized into strong-link regime (the link between molecules are stronger than the links inner molecules) and weak-link regime. So that suitable models were chosen before applying the data to the model. This model has been applied to whey protein isolate, caseinate gel, soybean globulin gel, and β-lactoglobulin gel. The calculated d_f of those gels was within 1.5–2.82 (Hagiwara et al., 1997; Pouzot, Nicolai, Durand, & Benyahia, 2004; Vreeker, Hockstra, den Boer, & Agterof, 1992). Wu and Morbidelli (2001) further developed the model of Shih et al. (1990) by introducing new parameters α, β, and x, to describe the link type of the gels. The gels were then assorted into three group based on the α value, the strong-link regime, the weak-link regime, and the transition regime. This model has been successfully used in the analysis of whey protein isolate, sodium caseinate aggregates, soybean protein, β-lactoglobulin gel, and bovine serum albumin gel, the d_f of these gels

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were within 1.73–2.87 (Caillard, Remondetto, & Subirade, 2010; Hagiwara, Kumagai, & Nakamura, 1998; Maltais, Remondetto, & Subirade, 2008).

Flaxseed gum is natural carbohydrate polysaccharide extracted from flaxseed (*Linum usitatissimum*), which makes up about 8% of the seed weight (Mazza & Biliaderis, 1989). Flaxseed gum has shown good performance in viscosity, emulsion property, and gelling properties, thus has the potential to be used in the food industry as thickener, emulsifier, stabilizer, etc. (Chen, Xu, & Wang, 2006; Wang et al., 2008; Wang, Wang, Li, Xue, & Mao, 2009). The ionic strength is known to influence the gelling properties of flaxseed gum (Chen et al., 2006). Thus the microstructure of flaxseed gum gels might be altered by the ions. Generally, gels tend to be formed with linear aggregates with a low degree of branching, while at a high ionic strength, a random aggregation occurs, leading to the formation of an opaque particulate network (Ikeda, Foegeding, & Hagiwara, 1999).

To the best of our knowledge, no research has been conducted on the fractal structure of flaxseed gum gel. In this study, flaxseed gum gels were analyzed for the scaling behavior and the fractal dimension via the rheological method. Different models were applied to compare the results and to find the suitable fractal model for flaxseed gum gel. The influence of ionic strength on the rheological properties and the fractal dimension was also considered in this study.

2. Materials and methods

2.1. Flaxseed gum extraction

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

Flaxseed (100 g) was washed in water for 1 min to remove the surface dust, and then mixed with 900 mL deionized water. The flaxseed and water were then stirred for 5 h at a speed of 300 rpm in a 60 °C water bath, according to the method of Cui (2001). The temperature of 60 °C was chose for flaxseed gum extraction is because of the mild temperature could lowered impurity (protein contents) in extracts, even although a higher yield of gum could be obtained at high temperature but increase impurity (protein contents) simultaneously. The extracted flaxseed gum solution was filtered through 40-mesh screen. After that the extracted flaxseed gum solution was precipitated with two volumes of 95% ethanol, collected by centrifugation at 3000 r/min for 10 min using an LG10-2.4 A machine (Beijing Medical Centrifuge Corporation, Beijing, China), according to the method of Cui, Mazza, and Biliaderis (1994) with some modifications on drying method. The precipitated flaxseed gum was then dried in a hot air oven at 80 °C for 4 h.

2.2. Solution preparation

For rheological tests, the flaxseed gum was dissolved in deionized water (pH 6.4–7.0) using a magnetic stirrer for 30 min at 25 °C to make 1–2% flaxseed gum solutions (dry base, w/w). Sodium chloride was then added to the flaxseed gum solution to reach the ionic strength values between 200 mM/L and 1000 mM/L.

2.3. Rheological tests

Rheological properties of the flaxseed gum solutions were measured using AR2000ex rheometer (TA Instruments Ltd., Crawley, UK). Aluminum parallel plate geometry (40 mm diameter, 1 mm gap) was chosen for the gel strength measurements. A thin layer of low viscosity silicone oil was applied on the surface of the samples in order to prevent evaporation. The linear viscoelastic region was determined for each sample through strain sweeps at 1 Hz (data not shown). Viscoelastic properties (storage modulus (G') and loss modulus (G'')) of the solutions were determined within the linear viscoelastic region.

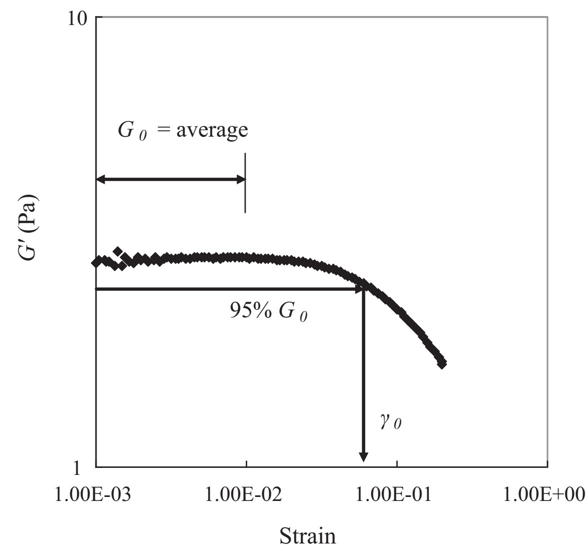

Fig. 1. Illustration for the estimation of the initial storage modulus (G_0) and the critical strain (γ_0).

The solutions were heated to 90 °C and held at this temperature for 5 min. The solutions were subsequently cooled to 25 °C and held for 30 min for the gel formation. The G' and G'' values were recorded during cooling using constant angular frequency of 6.283 rad/s and a strain of 0.5%.

Afterward, the gels were subjected to the strain sweep test, in which the strain was varied from 0.001 to 0.2. The data was collected at 40 points per decade. The average value of G' at the strain values between 0.001 and 0.01 was calculated as the initial value (G_0). And the point where the G' reduced to 95% of G_0 was taken as the critical point of the gel (see Fig. 1). The strain at the critical point was defined as the critical strain (γ_0). The G' and the critical strain points of the gels were used to calculate the fractal dimension of the gels.

2.4. Theory

A scaling model has been developed by Shih et al. (1990) to relate the storage modulus (G') and the critical strain (γ_0) to the volume fraction (ϕ) for a colloidal gel. Based on the strength of the inter- and intra-floc links, two regimes were defined: strong-link regime (inter-floc links are stronger than intra-floc links) and weak-link regime (inter-floc links are weaker than intra-floc links).

In the strong-link regime:

$$G' \propto \phi^{(d+x)/(d-d_f)} \tag{1}$$

$$\gamma_0 \propto \phi^{-(1+x)/(d-d_f)} \tag{2}$$

In the weak-link regime:

$$G' \propto \phi^{1/(d-d_f)} \tag{3}$$

$$\gamma_0 \propto \phi^{1/(d-d_f)} \tag{4}$$

where d is the Euclidean dimension, d_f is the fractal dimension of the flocs and x is the fractal dimension of the floc backbone ($1 \le x \le d_f$).

Wu and Morbidelli (2001) have modified the model suggested by Shih et al. (1990) and they related G' and γ_0 of the gel to the volume fraction of primary particles. A constant (α) (where $0 \le \alpha \le 1$) was introduced to account for the elastic contributions of both

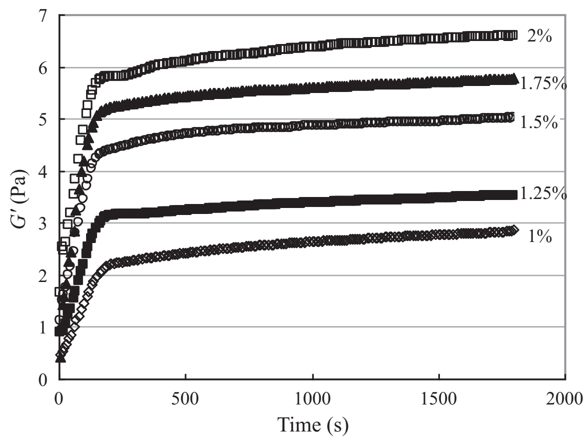

Fig. 2. The gelation profile of the flaxseed gum solutions with various concentrations at zero ionic strength.

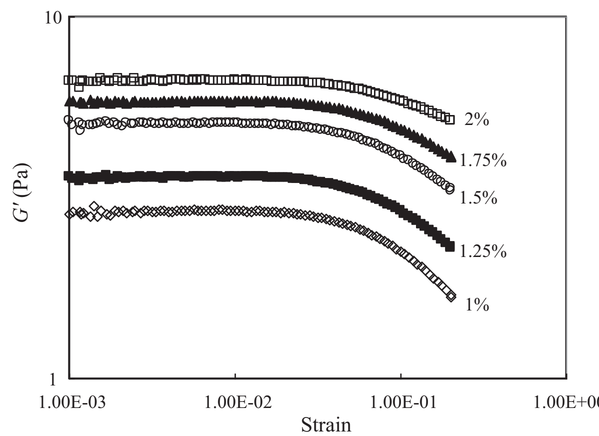

Fig. 3. The storage modulus as a function of strain (strain sweep) for the flaxseed gum gel with various concentrations at zero ionic strength.

inter- and intra-floc links. It allows identifying the gelation regimes prevailing in the system, and serves as indicator of the relative importance of the two links type.

$$G' \propto \phi^{\beta/(d-d_f)} \tag{5}$$

$$\gamma_0 \propto \phi^{(d-\beta-1)/(d-d_f)} \tag{6}$$

$$\beta = (d-2) + (2+x)(1-\alpha) \tag{7}$$

where x is the fractal dimension of the floc backbone ($1 \le x \le d_f$) as in the Shih et al. (1990) model.

2.5. Determination of fractal dimension

The volume fraction of particles (ϕ) in the gels was assumed to be proportional to the gum concentration (C). Fractal dimension values of flaxseed gum gels were calculated using the slope values of $\log G'$ versus $\log C$ and of log critical strain γ_0 versus $\log C$, according to the models listed above (Eqs. (3)–(6)).

3. Results and discussions

3.1. Gelation process of flaxseed gum

The effects of concentration on the gelation process of flaxseed gum were shown in Fig. 2. The G' of the gels increased rapidly during the first 200 s, and then the G' increased slowly as a function of time, suggesting that the gelation occurred very quickly. Furthermore, the storage modulus increased with the increasing of flaxseed gum concentration. High gum concentration may help to form more solid-like structure with high density, which could also hold the water better so that the storage modulus was increased. The same trends were also observed when the ionic strength of the flaxseed gum solutions was altered by adding 200–1000 mM NaCl (data not shown). Similar gelation trend and concentration effect were also found in gellan gum and xanthan gum (Iseki, Takahashi, Hattori, Hatakeyama, & Hatakeyama, 2001; Tako, Teruya, Tamaki, & Konishi, 2009).

3.2. Influence of strain on G'

The strain sweep test, which was carried out to find the influence of strain on the G' of the gels produced without the addition of NaCl, was presented in Fig. 3. All the samples show similar trends. Firstly the G' values are almost constant up to a certain strain, and then they began to decrease suddenly when the strain was further increased. The sudden decrease in the G' may indicate the breaking of bonds within the gel network and a transition from a linear to a non-linear behavior (Ould Eleya, Ko, & Gunasekaran, 2004). That is why the point where the G' reduced to 95% of G_0 was taken as the critical point of the gel, since the gel has experience significant change in structure when the G' reduced to 95% of G_0. The flaxseed gum gels with various ionic strength values (200–1000 mM NaCl) exhibited similar trend with the gels shown in Fig. 3 (results not shown).

3.3. Effects of ionic strength on G'

The effects of ionic strength on the G' values of 1% flaxseed gum gels are shown in Fig. 4. The G' values first increase with the ionic strength increase, and then they start to decrease from the ionic strength of 400 mM. It reflects that the increase of gel strength of flaxseed gum could be achieved by adding suitable amount of salt. Since the electrical charges of ions could act as the media of the cross-linking during the gelling process. With the increasing of salt concentration, attractive interactions between gum molecules become stronger, as a result the rigidity of the gels increased. Further increases in the salt concentration cause attractive interactions to dominate repulsive ones. Aggregation becomes increasingly random and gel rigidity decreases (Ikeda et al., 1999). Similar trends were observed when $CaCl_2$ were used for changing the ionic strength of the gels (Chen et al., 2006). The storage modulus values of 0.8% gellan gum gel and 0.8% κ-carrageenan gum gel were also dramatically

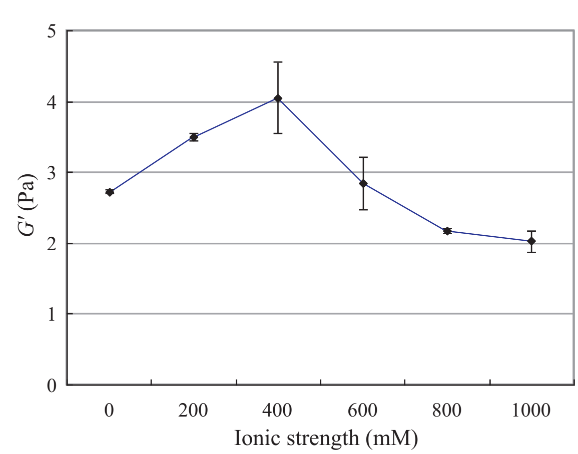

Fig. 4. Effects of the ionic strength on the storage modulus of flaxseed gum gel at the concentration of 1%.

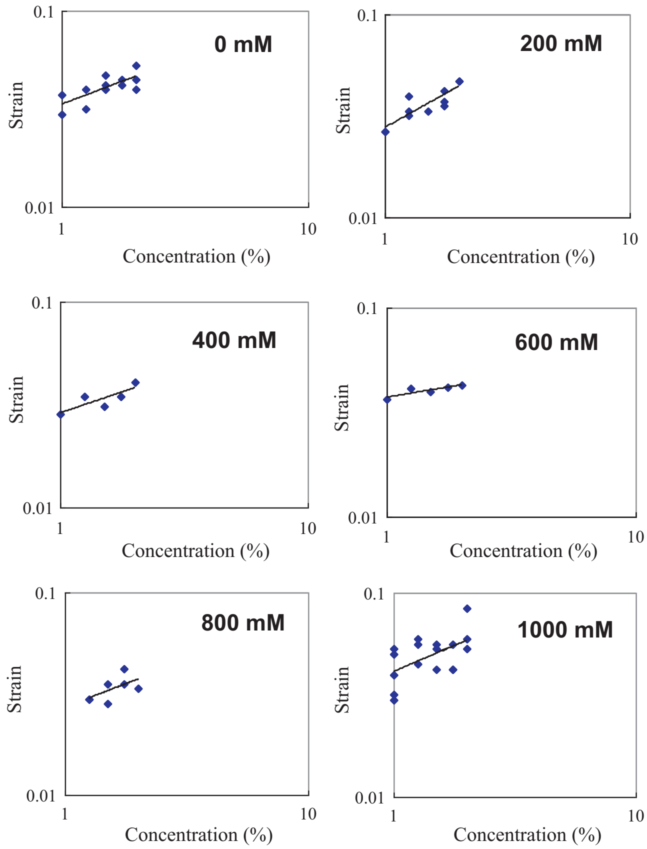

Fig. 5. Effects of concentration and ionic strength on the scaling behavior of the critical strain of flaxseed gum gels.

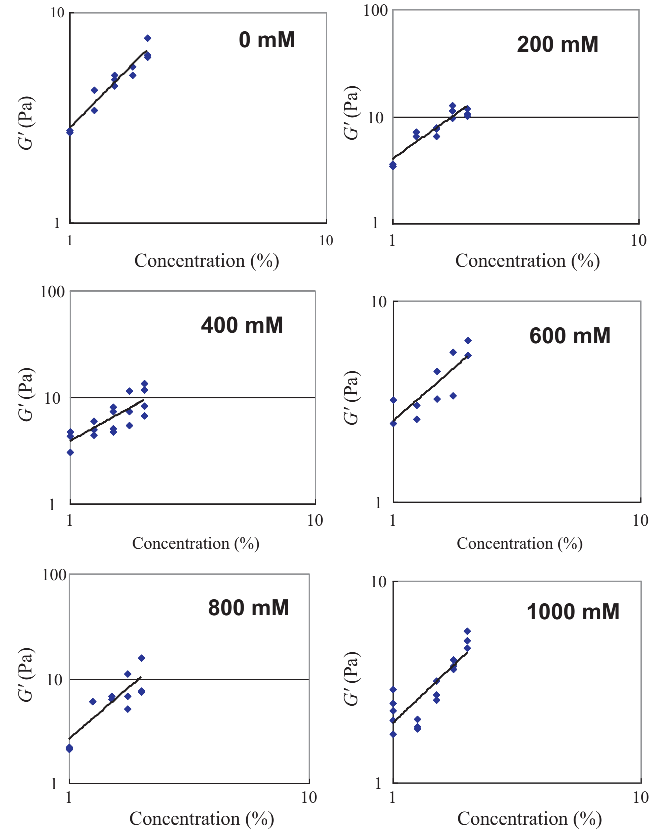

Fig. 6. Effects of concentration and ionic strength on the scaling behavior of the storage modulus of flaxseed gum gels.

increased by adding 0.1–0.5% NaCl (Moritaka, Takahashi, & Kubota, 2007).

3.4. Scaling behavior of flaxseed gum gels

The scaling behaviors of strain of flaxseed gum gels as a function of gum concentration are shown in Fig. 5. For all the ionic strength in this study, the critical strain γ_0 of flaxseed gum gels shows a power–law relationship, i.e. $\gamma_0 \sim C^m$, where m is the power–law exponent. The m values for all the gels are positive, as shown in Table 1. The m values were strongly influenced by the ionic strength. The highest value was obtained when ionic strength was 200 mM (m = 0.68), while the lowest value was at 600 mM ionic strength (m = 0.20).

The effects of ionic strength on the scaling behavior of G' of flaxseed gum gels are shown in Fig. 6. The G' values of all the samples show power–law behavior with the increase of concentration, which can be fitted to the form: $G' \sim C^n$, where n is the power–law exponent. Similar with the m value in critical strain scaling, the n values were also sensitive to the ionic strength. The highest n was 1.97 at ionic when the ionic strength was 800 mM, whereas the lowest n value was 1.07 at ionic strength of 600 mM.

3.5. Fractal analysis

The fractal dimensions of flaxseed gum gels in different ionic strength were estimated from the power–law exponents n and m, based on the model of Shih et al. (1990) and Wu and Morbidelli (2001). The calculated d_f values are shown in Table 1. For all the ionic strength conditions, the m values have positive values, suggesting that the flaxseed gum gels can be considered as weak-link gel according to the model of Shih et al. (1990).

Table 1
Effect of the ionic strength on the fractal dimension of flaxseed gum gels calculated by two different models.

Ionic strength (mM)	Power–law exponents		Model of Shih et al. (1990)	Model of Wu and Morbidelli (2001)				Regime
	n[a]	m[b]	d_f[c]	d_f[d]	β[d]	α[d] at x = 1	α[d] at x = 1.3	
0	1.23	0.47	2.19	1.83	1.45	0.85	0.86	Transition gel
200	1.63	0.68	2.39	2.13	1.41	0.86	0.87	Transition gel
400	1.28	0.40	2.22	1.81	1.52	0.83	0.84	Transition gel
600	1.07	0.20	2.06	1.42	1.69	0.77	0.79	Transition gel
800	1.97	0.46	2.49	2.18	1.62	0.79	0.81	Transition gel
1000	1.17	0.51	2.15	1.81	1.40	0.87	0.88	Transition gel

[a] Power–law exponent relating G' to concentration: $G' \sim C^n$.
[b] Power–law exponent relating γ_0 to concentration: $\gamma_0 \sim C^m$.
[c] Values of fractal dimensions d_f based on the model of Shih et al. (1990).
[d] Value of d_f, β, and α parameters based on the model of Wu and Morbidelli (2001).

Thus, Eqs. (3) and (4) were used in this study to calculate d_f. The d_f values were between 2.06 and 2.49, depending on the ionic strength of the gels. These values are similar with the fractal dimensions of guar gum/glutaraldehyde hydrogels, which were found to be between 2.09 and 2.28, according to the different value of guar gum/glutaraldehyde ratio (Sandolo, Matricardi, Alhaique, & Coviello, 2009).

Wu and Morbidelli (2001) have modified the model described by Shih et al. (1990) by introducing a new parameter, α, to find out the type (strong-link, weak-link, or transition gel) of the gels. The fractal dimension of flaxseed gum gels were also evaluated using Eqs. (5)–(7) and results were shown in Table 1. The d_f values determined from the modified model were between 1.42 and 2.18, which were lower than the fractal dimension values calculated from the model of Shih et al. (1990). But the d_f values obtained from these different models showed similar trend with the increasing of ionic strength. The difference in fractal dimension values calculated by these two models was also found in whey protein isolate gels and β-lactoglobulin gels, which were all transition gels (Wu & Morbidelli, 2001).

The model of Wu and Morbidelli (2001) provides two additional parameters: α and β (see Eqs. (6) and (7)). The parameter α indicates the relative importance of the elastic contributions of both inter- and intra-flocculation links. The parameter α also allows to identify the different gelation regime prevailing in the system (i.e. strong-link, transition, or weak-link). Strong-link and weak-link regimes are found for $\alpha = 0$ and $\alpha = 1$, respectively, and transition regimes are found for $0 < \alpha < 1$. In this study, the parameter α was estimated at two x values, 1 and 1.3, which were commonly considered to be a good approximation of the fractal dimension of the backbone of colloidal aggregates (Ould Eleya et al., 2004). In both cases, the α values were between 0 and 1, as shown in Table 1. It means that all the flaxseed gum gels could be classified as the transition gels. It should be mentioned that all the α values are very high, between 0.77 and 0.88. The flaxseed gum gel in 1000 mM ionic strength with α value of 0.88 was very near to the weak-link regime. It also explains why all the flaxseed gum gels were classified as the weak-link gel, when using the model of Shih et al. (1990).

The ionic strength has significant effects on the fractal dimensions of the flaxseed gum gels, as shown in Table 1. When the ionic strength was 800 mM, the highest d_f values of 2.49 and 2.18 were obtained according to the models of Shih et al. (1990) and Wu and Morbidelli (2001), respectively. The high d_f value suggests a more "tight" structure of the network of flaxseed gum gel in 800 mM ionic strength (Sandolo, Matricradi, Alhaique, & Coviello, 2007). When the ionic strength was 600 mM, the lowest d_f values of 2.06 and 1.42 was found at 600 mM ionic strength for 2.06 and 1.42 according to the models of Shih et al. (1990) and Wu and Morbidelli (2001), respectively. The d_f values of flaxseed gum gels were between 1.42 and 2.49, depending on the ionic strength of the gels. This is similar with the fractal dimensions of silica gels which were between 1.4 and 2.5, based on the preparation temperature and mechanical shear (Rueb & Zukoski, 1997). The ionic strength may affect the d_f by modifying the structure of the flaxseed gum gels by changing the gum connections (ionic bond). Then the viscoelastic properties were significantly changed by the ionic strength (see Fig. 4). Finally, the changes of ionic strength resulted in the changes in fractal structure of flaxseed gum gels.

Typically, the fractal dimensions of hydrocolloid gels were less than 2. The fractal dimensions of gellan gums have been found to be between 1.6 and 2.1, which is similar with the d_f of the flaxseed gums calculated from Wu and Morbidelli's (2001) model (Dai, Liu, Liu, & Tong, 2008). The fractal dimension of alginate gel induced by calcium cations was found to be between 1.4 and 2.2, for the calcium concentration ranging from 2% to 6% (Lu, Liu, Dai, & Tong, 2005). The d_f of alginate gel induced by cupric cations was reported to be between 1.6 and 2.3, depending on the ionic strength and the alginate concentration (Lu, Liu, & Tong, 2006). The fractal dimensions of agar, gellen, and κ-carrageenan gels were all in the range of 1.56–1.76 (Nussinovitch, Corradini, Normand, & Peleg, 2000). Based on these fractal dimensions from hydrocolloid gels, it might be concluded that the model of Wu and Morbidelli (2001) was more suitable for flaxseed gum gels. However, further testing methods should be applied to verify this assumption.

4. Conclusions

The fractal analysis of flaxseed gum gels were studied in the ionic strength range of 0–1000 mM. The ionic strength significantly influenced the rheological properties of the flaxseed gum gels. The highest storage modulus of the gels was observed at 400 mM. Both storage modulus and critical strain showed the scaling behavior (i.e. the power–law relationship with the gum concentration). Two different models were used to estimate the fractal dimension of the gels. The fractal dimension values were 1.42–2.18 and 2.06–2.49 based on the model selected and the ionic strength applied. These values were in good agreement with the fractal dimensions of other hydrocolloid gels. The fractal dimension values were found to be strongly dependent on the ionic strength. All of the flaxseed gum gels were classified as the weak-link gel based on the model of Shih et al. (1990), while the gels were classified in the transition regime based on the model of Wu and Morbidelli (2001). This study provides useful information on the fractal dimensions of flaxseed gum gels, which also help understand the microstructure of the gels.

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