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Mechanical properties of gels formed by mixtures of mucilage gum (*Opuntia ficus indica*) and carrageenans

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Abstract

The mechanical properties of gels formed by either mixtures of mucilage gum from *Opuntia ficus indica* and κ-carrageenan or i-carrageenan have been examined using dynamic shear and uniaxial compression measurements. A total polymer concentration of 2% (w/w) was used, the proportion of mucilage gum varying from 0 to 80% (w/w) and KCl or $CaCl_2$ in the range from 12 to 60 mM. For the mixed gels of i-carrageenan and mucilage gum, no enhancement of the mechanical properties with respect to the pure i-carrageenan was observed. For the κ-carrageenan/mucilage gum system, at an 80/20 ratio and 12 mM KCl, higher gel rigidity than the pure κ-carrageenan gels was observed and failure stress and strain increases as mucilage gum concentration increases. A peak in the Young's modulus was also observed for these last conditions suggesting a synergistic interaction. The main feature of the mucilage is to enhance elasticity on the final gels.

Keywords: Rheology; Gels mechanical properties; Mucilage; *Opuntia ficus indica*; Iota-carrageenan; Kappa-carrageenan

1. Introduction

Foods are multicomponent in nature and many of their functional characteristics depend on their macromolecular composition. Because of this active role, macromolecules in foods have been studied as individual species or interacting with the surroundings. In many cases, the macromolecules responsible for the mechanical and physicochemical properties of many food systems are polysaccharides (i.e. hydrocolloids). The ability that these macromolecules possess in being part of the structural function of foods is known as functional property, and it is expressed among others as gelation capacity, viscosity modification, and stabilization of suspensions, emulsification, and ability to retain water. It must be pointed out that in industry, hydrocolloids are quite often used as pure compounds or as mixtures of different compounds. This last is motivated by a constant search of new functional properties in the final products.

In recent years, the use of mixtures of these macromolecules has opened the possibility of obtaining a wide range of textures and rheological properties (Whistler, 1983). For example, the iota (i) and kappa (κ) carrageenans form thermo reversible gels in water solutions and in the presence of cations (Stading & Hermansson, 1993; Therkelsen, 1983). However, it is well known that gels made from these carrageenans show water exudation (i.e. syneresis) and thus significant changes in their textural properties. On the other hand, the recently studied gum or mucilage from *Opuntia ficus indicia* (*Ofi-m*), displays elastic properties but not gelation (Medina-Torres, Brito-De La Fuente, Torrestiana–Sanchez, & Katthain, 2000). A mixture of these two different types of hydrocolloids may result in interesting properties from the functional point of view.

The carrageenan is a class of galactan polysaccharides that occur as an intercellular matrix material in numerous species of red seaweeds (Rhodophyceae) of the order Gigartinales. Carrageenans serve a function in the structure of the plant, analogous to, but differing from that of cellulose in the plants. Whereas land plants require a rigid structure capable of withstanding the constant pull of gravity, marine plants need a more flexible structure to accommodate the varying stresses of currents and wave motion (Christensen & Trudsoe, 1980). They have adapted accordingly by developing hydrophilic, gelatinous

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structural materials having the necessary compliancy. The carrageenans are important classes of this type of material.

The carrageenans are linear polysaccharides their molecule being made up of some 1000 residues; there are many possibilities for structural variations. The carrageenans are made up of alternating beta-1,3- and alpha-1,4-linked galactose residues. Thus, the repeating units are disaccharides. The 1,4-linked residues are commonly, but not invariably, present as the 3,6-anhydride. This last type is known as lambda and in its pure form, it does not form gels as compared with the kappa and iota forms. Even though potassium and calcium ions induce gelation, the latter results in stronger gels (Stading & Hermansson, 1993; Stanley, 1990). The gelation mechanism is controversial, and the domain model has been suggested to explain gelation in terms of coil-to helix transition followed by aggregation of domains of double helices in the presence of gel-inducing ions (Picullel, Nilsson, & Muhrbeck, 1992). The i-carrageenan displays a greater hydrophilic character than the κ-carrageenan because of the presence of additional sulphate group in the anhydrous galactose residue. This also enhances its ability to inhibit syneresis, even though its gels are less rigid than those of κ-carrageenan owing to its lower aggregation capacity (Stanley, 1990).

Synergistic interactions between carrageenans have been studied through small strain oscillatory rheological tests. In this case, higher storage [G'] and loss modulus [G''] for the mixtures of carrageenans than that observed for each component alone have been reported (Stading & Hermansson, 1993). This has motivated more studies on such systems as well as on other polysaccharide blends formed either by two gelling polysaccharides or by one gelling and a non-gelling one. Rheological studies on carrageenans and their mixtures with others hydrocolloids have been reported by Fernandes, Goncalves, and Doublier (1994a,b). On the other hand, studies on gel formation have also been discussed by Glicksman (1982) and Morris, Cutler, Ross-Murphy, Rees, and Price (1983).

As it was mentioned above, the rheological properties of a non-gelling hydrocolloid from the mucilage of *Opuntia ficus indica* have been reported recently (Medina-Torres et al., 2000). This hydrocolloid is a polysaccharide with a molecular weight in the order of 2.3×10^4 that contains residues of arabinose, rhamnose, xylose, galactose and galacturonic acid. Even though it does not form gels, the polymer showed high elastic properties similar to those of synthetic polymers like polyisobutylene. Furthermore, it also showed a high capacity to modify the viscosity of the aqueous systems formulated with it. Therefore, a rheological study of mixtures of carrageenans and mucilage gum from *Opuntia ficus indica* would be interesting considering the possibility of solving the syneresis problem as well as the changes in textural properties of the resulting gels, if a synergistic effect exist.

Following our earlier work in this field, the aim of the present study is to gain insight on the mechanical properties of mixtures of the *Opuntia ficus indica* gum with iota or kappa-carrageenan gums. The rheological behavior of these binary systems as well as the possible interactions between the gums is characterized using both small amplitude oscillatory shear flows and uniaxial compression measurements.

2. Materials and methods

Stems from *Opuntia ficus indica* were selected from Milpa Alta (Mexico) and from the same batch. The isolation and purification techniques of the mucilage have been reported elsewhere (Medina-Torres et al., 2000). The main sugar component of mucilage is arabinose (44.04 wt%) followed by xylose (22.13 wt%). A molecular weight in the order of 2.3×10^4 was estimated from elution profiles by HPLC chromatography, equipped with a G 2000 SW (TSK-GEL) column, a refraction index detector, IR (Perkin Elmer series 200) and an integrator (Spectra-Physics, model SP4270) in a previous work (Medina-Torres et al., 2000). Dextrans (commercial grades, Sigma USA, Leuconostoc mesenteroides, strain B-152 and Dextran T70 produced by Pharmacia Biotechnology, Sweden) with molecular weight from 5000 to 87,000 were used as standards.

Food grade commercial preparation of i-carrageenan (Genuvisco, Germantown, Co.) and κ-carrageenan (FMC-911), were used. The study was performed at a 2% (w/w) of total hydrocolloids concentration, the proportion of gum varying from 0 to 80% (w/w) in the mixture. Previous experiments showed that 100% mucilage does not form gels (Medina-Torres et al., 2000). Either KCl or $CaCl_2$ were used at a given concentration such to obtain the desired ionic strength (I) in the range from 12 to 60 mM.

2.1. Gel preparation

Mixtures of *Opuntia ficus indica*–κ-carrageenan and *Opuntia ficus indica*–i-carrageenan were prepared at a constant total polysaccharide concentration of 2% (w/w). The preparations contained from 0.012 to 0.12 mol/kg of KCl in the case of the mixtures formed by *Opuntia ficus indica*–κ-carrageenan. On the other hand, $CaCl_2$ was used in the case of mixtures formed by *Opuntia ficus indica*–i-carrageenan system. The following carrageenan/mucilage ratios were then prepared: 100/0, 80/20, 60/40, 50/50, 40/60, and 20/80 (w/w%).

The mixture of polysaccharides was dispersed at room temperature under magnetic stirring during 10 min and then heated up to 75 °C with agitation and maintained so during 20 min. After this, the proper amount of ions was added to the hot solutions to provide the targeted ionic strength. The samples were next poured into cylindrical plastic tubes (internal diameter 20.3 mm), stoppered, and cooled to 25 °C. Then, the gels were refrigerated at 13 °C during 48 h to allow equilibrium and subsequently setup at room temperature. Gel cylinders of 20.3 mm diameter and 10.0 mm height were prepared for the rheological and compression tests.

2.2. Rheological measurements

All the rheological measurements were performed in a rotational rheometer (Haake, Germany, Mod. CV20N) using parallel plates (diameter = 19.25 mm; gap = 2 mm) with a temperature control bath (Haake, Germany, Mod. F3T). The parallel plates were covered with fine sand (mesh 100) mixed previously with glue to avoid slippage during oscillation.

The viscoelastic properties, storage modulus [G'] and loss modules [G''] were determined through small amplitude oscillatory shear flows at frequencies ranging from 1.36 to 13.6 rad/s. From previous strain, sweep runs the upper limit of the linear viscoelasticity zone was located at 0.05 strain. In this domain, the experimental tests are essentially non-destructive. All tests were carried out at 25 °C at least by duplicate on fresh cut samples from different cylindrical preparations.

2.3. Resistance to compression measurements

The resistance to compression test was performed using a MTS Sintech 1/S Universal Machine (MTS, USA), with a 100 N load cell. The runs were carried out with a 2.9 cm diameter cylindrical probe at a compression rate of 15 mm/min. The contact surfaces were lubricated with silicone oil to avoid evaporation of moisture and to reduce friction (Chen, Liao, Boger, & Dunstan, 2001). All tests were performed at 25 °C at least by duplicate on fresh cut samples from different cylindrical preparations.

Preliminary stress–strain tests on the gels showed that the breaking strength was located at deformations below 80%. The experiments were divided into two parts: small and large strains. At small strains, the Young's modulus remained essentially constant below 20% strain and this was considered as the upper limit of the viscoelastic region. Then, a strain of 15% was used for Young's modulus calculation purposes. The following large strain stage (compression up to 80% deformation) was recorded in order to obtain failure stresses and strains.

From the uniaxial compression curves, the engineering stress [σ_{eng} = force/original cross-section area] and strain [$\varepsilon_{\mathrm{eng}}$ = reduction of length/original length] curves were obtained. Considering that the cross-sectional area of the gels increased significantly under large compression before failure, the Hencky's normal strain (ε_{h}) and the corrected stress (σ_{c}) were used in order to estimate the true gel properties. Hencky's strain and the corrected stress were converted from engineering strain and stress by the following equations (Tang, Tung, & Zeng, 1996):

$$\varepsilon_{\mathrm{h}} = -\ln(1 - \varepsilon_{\mathrm{eng}}) \tag{1}$$

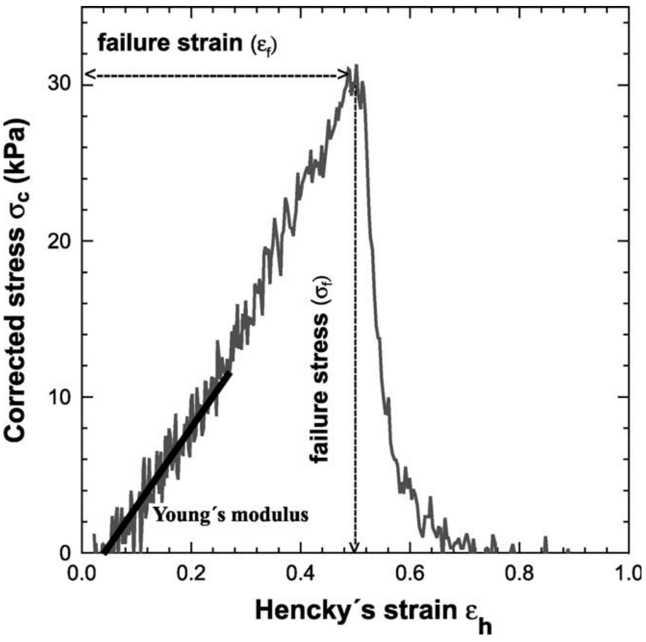

Fig. 1. Determination of the Young's modulus, failure stress and strain from uniaxial compression tests.

$$\sigma_{\mathrm{c}} = \sigma_{\mathrm{eng}}(1 - \varepsilon_{\mathrm{eng}}) \tag{2}$$

The failure strain (ε_{f}) and the failure stress (σ_{f}) were also determined from compression curves as shown in Fig. 1. As it is well known, a large failure strain means a high deformable gel and a large failure stress indicates a strong gel. On the other hand, the gel firmness was also characterized through the initial Young's modulus, estimated from the linear portion of the corrected stress versus Hencky's normal strain curve (see Fig. 1).

3. Results and discussion

3.1. Steady oscillatory flow measurements

The mechanical spectrum as a function of mucilage concentration in deionized water and at 25 °C has been previously reported (Medina-Torres et al., 2000). It was shown the dependency of G' and G'' on the mucilage gum concentration. At low mucilage concentration ($<3\%$), G'' is always greater than G' meaning that mucilage solutions will show mainly viscous properties. On the other hand, increasing mucilage concentration, $G' > G''$ indicating a clear tendency to form macromolecular networks with important elastic properties.

In this study, the *Opuntia ficus indica* was added to mixtures of carrageenan in order to see stabilization and a probably reinforcement of the carrageenan network. A search for synergistic effects was then conducted through the analysis of any modification of the storage modulus G'.

Figs. 2 and 3 show the frequency sweep results on 2% (w/w) ι-carrageenan and κ-carrageenan gels, respectively,

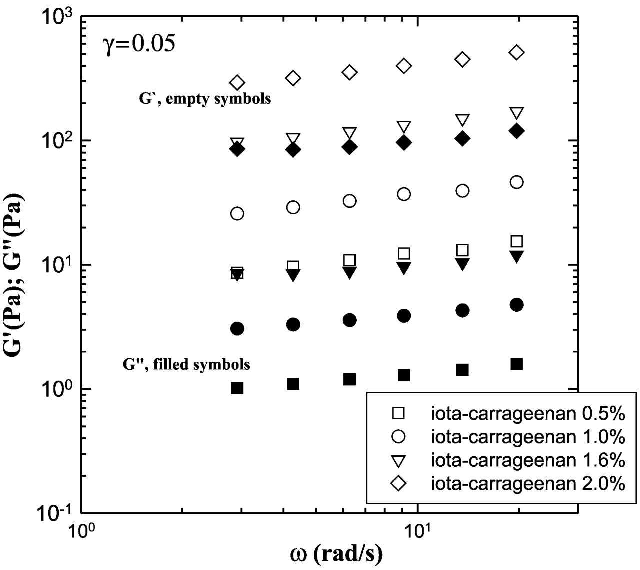

Fig. 2. Mechanical spectra of frequency dependence of the storage modulus G' and loss modulus G'' for different concentrations of iota-carrageenan gels in 12 mM $CaCl_2$.

no mucilage present. As it can be observed, for a given polymer concentration, the dynamic storage modulus, G', shows a weak dependency on the frequency and it is always superior to G''. If both moduli are virtually independent of frequency, they represent the behavior of a self-standing gel. Then, the mechanical spectrum shown in Figs. 2 and 3 represents a behavior that is predominantly solid rather than liquid like (Mitchell, 1976). This behavior is clearly dependent on the carrageenan concentration. This means, as the polysaccharide concentration increases, G' increases in both cases. The curves also show that i-carrageenan gels are much less rigid and less brittle with a considerable lower G' modulus than the κ-carrageenans gels. Arnaud, Choplin, and Lacroix (1989) in mixtures of κ-carrageenan–galactomannans reported a similar tendency.

The mechanical spectra of mixtures of i-carrageenan and *Ofi-m* as well as of κ-carrageenan–*Ofi-m*, are shown in Figs. 4 and 5, respectively. As it can be seen, the dynamic modulus G'' shows a weak dependency on frequency, even lower than the results shown in Figs. 2 and 3, confirming the true gel character of these mixtures.

As Fig. 4 suggest, for the gels formed with mixtures of i-carrageenan–*Ofi-m* no enhancement of the mechanical properties with respect to the i-carrageenan alone was observed. Thus, these results suggest that under these conditions there are not synergistic effects. On the other hand, some gels formed with mixtures of κ-carrageenan–*Ofi-m*, in particular those formed with the ratios 80/20 and 60/40, showed higher moduli than the κ-carrageenan gels alone at equal concentration (see Fig. 5). This is a clear indication that these mixtures exhibited a synergistic effect.

Several authors have reported synergistic effects between κ-carrageenan and galactomannans (Stading & Hermansson, 1993; Tako, Asato, & Nakamura, 1984). It is well known that κ-carrageenan forms a gel on cooling. Galactomannan does not form a gel by itself. Mixtures of

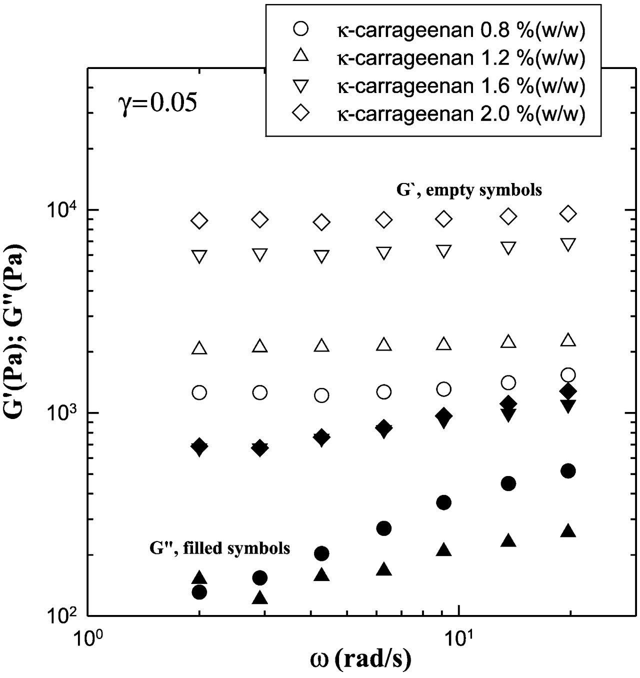

Fig. 3. Mechanical spectra of frequency dependence of the storage modulus G' and loss modulus G'' for different concentrations of κ-carrageenan gels in 12 mM KCl.

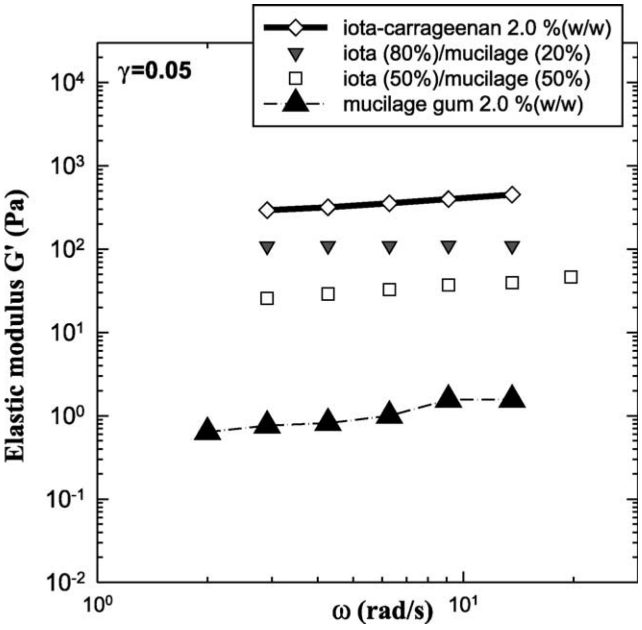

Fig. 4. Mechanical spectra of frequency dependence of the storage modulus G' for different concentrations of iota-carrageenan–mucilage gels at 25 °C and 12 mM $CaCl_2$.

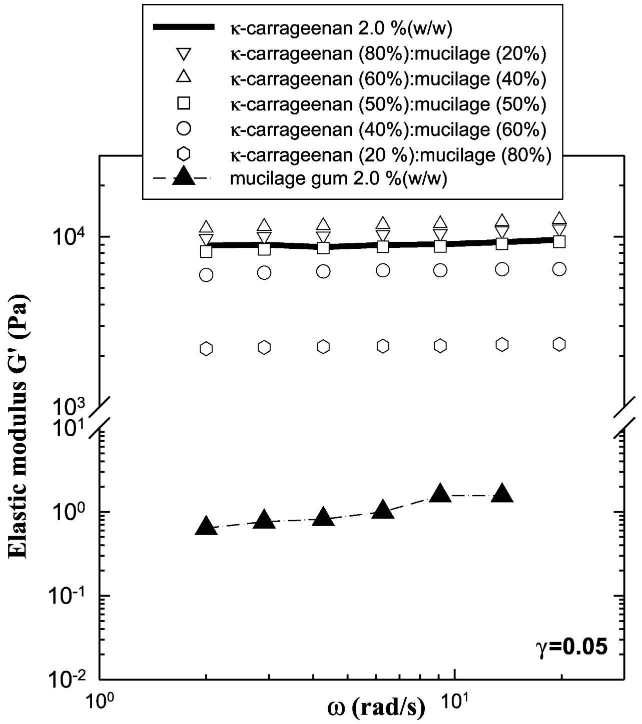

Fig. 5. Mechanical spectra of frequency dependence of the storage modulus G' for different concentrations of κ-carrageenan–mucilage gels at 25 °C and 12 mM KCl.

these two polysaccharides may form gels under conditions in which pure κ-carrageenan does not, and the gel strength may be enhanced as shown by Stading and Hermansson (1993) in the case of mixtures of κ-carrageenan–locust bean gum. These authors reported that G' had a maximum for a κ-carrageenan/locust bean gum ratio of 80/20 in 8 mM KCl and then concluded that at this conditions, the two polysaccharides show a strong synergistic effect. Fernandes et al. (1994a,b) and Tako et al. (1984) reported similar results for the same mixture.

In our study, the mucilage is a galactomannan, which does not form a gel by itself (Medina-Torres et al., 2000) but at a given κ-carrageenan/mucilage ratio does form gels with enhanced strength as compared with those formed with pure κ-carrageenan (Fig. 6). In this figure, it is clear that a maximum in G' is obtained for a κ-carrageenan/mucilage ratio of 60/40. Furthermore, for a ratio of 80/20, the G' value is also higher than the one shown by κ-carrageenan pure. These results suggest that under the above conditions, the mucilage and the κ-carrageenan show a synergistic effect. It is possible that the mucilage could aggregate and therefore yield a secondary network producing a stabilization effect and thus an enhancement of the gel strength, as proposed by Fernandes et al. (1994a,b). However, studies on the structure of the gels formed are needed in order to better understand the role of the mucilage in the κ-carrageenan network.

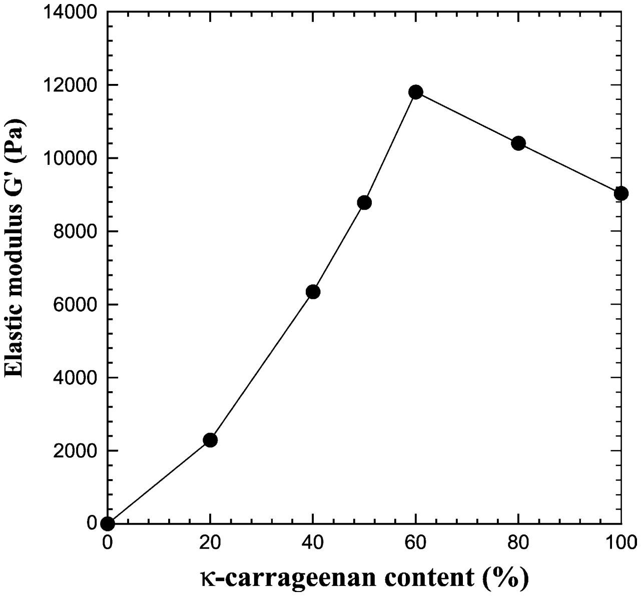

Fig. 6. Elastic modulus as a function of κ-carrageenan fraction in the mixed gel with $I = 12$ mM KCl and total polysaccharide concentration of 2% (w/w). Data at $\omega = 10$ rad/s.

3.2. Resistance to compression

Typical results regarding failure normal stresses and strains for the gels formed with mixtures of κ-carrageenan and *Ofi-m* at $I = 12$ mM KCl are shown in Fig. 7. Table 1 lists the failure properties of these gels, together with those

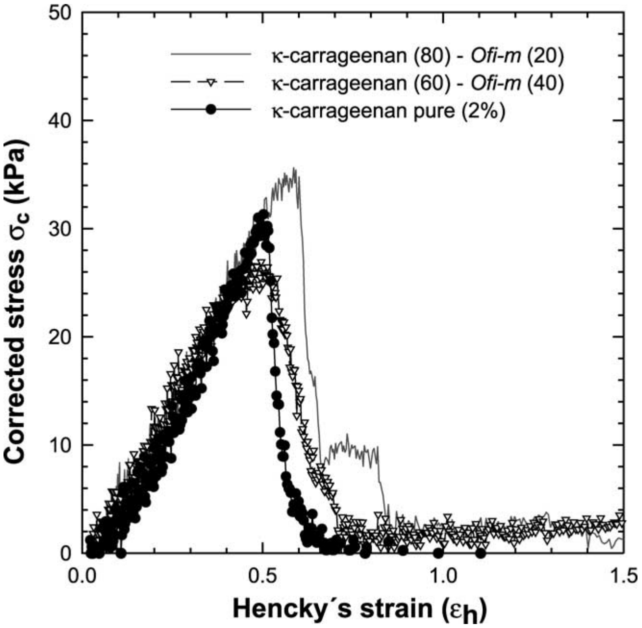

Fig. 7. Failure properties as a function of κ-carrageenan fraction in the mixed gel with $I = 12$ mM and total polysaccharide concentration was 2% (w/w).

Table 1
Mean values of failure properties and Young's modulus for the binary system kappa-carrageenan/mucilage gum and selected gels from literature

% gum substitution	Failure strain ε_f	Failure stress σ_f (kPa)	Young's modulus (kPa)	Reference
κ-carrageenan (100/0) at 2% and $I = 12$ mM KCl	0.49	31.01	67.15	This work
Mixtures				
κ-c/*Ofi* (80/20)	0.59	35.67	65.85	
κ-c/*Ofi* (60/40)	0.49	26.93	59.54	
κ-c/*Ofi* (50/50)	0.37	17.58	47.07	
Carrageenan gel, 1.75%, polymer	0.50	40.5	–	Mirza and Lelievre (1992)
Carrageenan (CA) and locust bean gum (LBG) mixed gels, 1.75% total polymer, CA/LBG = 60/40	1.09	60.3	–	Mirza and Lelievre (1992)
Gellan low acyl, 1% polymer with 5 mM Ca^{++}	0.38	61.0	–	Tang et al. (1996)
Gellan low acyl, 1% polymer with 20 mM Ca^{++}	0.28	54.0	–	
Mixed gellan gel, 1.0% polymer, high (H)/low acyl (L), H/L = 50/50				
30 mM Ca^{++}	1.03	63.5		Mao et al. (2000)
60 mM Ca^{++}	1.06	40.1		
Mixed gellan gel, 1.0% polymer, high (H)/low acyl (L), H/L = 25/75				
30 mM Ca^{++}	0.65	56.4		
60 mM Ca^{++}	0.70	40.4		

κ-c, kappa-carrageenan; *Ofi*, mucilage gum from *Opuntia ficus indica*.

of selected food gels reported in the literature. The Young's modulus for the gels of this work is also included in Table 1. When compared the results of this work with previous reports (Mao, Tang, & Swanson, 2000; Mirza & Lelievre, 1992), it is clear that depending on type of mixtures, concentration and ionic strength, there are several gels with comparable strength and deformability to the gels analyzed in this work. A strong effect of the ionic strength on the failure properties is also noted.

The rupture stress and strain data as a function of κ-carrageenan/mucilage ratios and KCl concentration is shown in Figs. 8 and 9, respectively. The rupture stress gradually increases with potassium concentration, but it decreases as the mucilage concentration increases, except for the 80/20 ratios at 12 and 60 mM KCl. The increase in failure stress for the 80/20 ratios is significant and this again suggests synergy between these two hydrocolloids. This behavior is similar to the one observed by Chen et al. (2001) for κ-carrageenan/locust bean gum mixtures. On the other hand, at all potassium concentrations, the failure strain increased for the pure κ-carrageenan gels (Fig. 9). A similar trend for mixed gellan gels with calcium concentration was reported by Mao et al. (2000). However, as the mucilage concentration increases, failure strain decreases and the behavior is dependent on the ionic strength. At 12 mM KCl, the rupture strain behavior follows a similar trend as the failure stress, this means, for the 80/20 ratio, a significant increase in both failure properties is observed. At these last conditions, the increase in rupture stress and strain with the incorporation of the mucilage gum indicates that the gel becomes harder to fracture.

Young's modulus of pure κ-carrageenan gels at 2% total polymer concentration showed a weak dependence on potassium concentration (see points at 100% κ-carrageenan in Fig. 10). However, as the mucilage gum concentration increases in the binary mixture, Young's modulus increased with increasing potassium

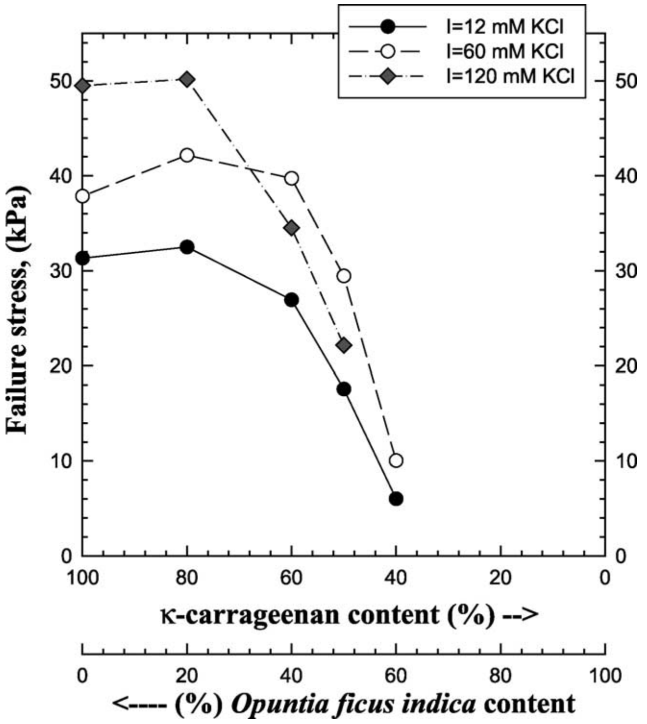

Fig. 8. Failure stress of κ-carrageenan and mucilage gum (*Ofi*) as a function of KCl concentration.

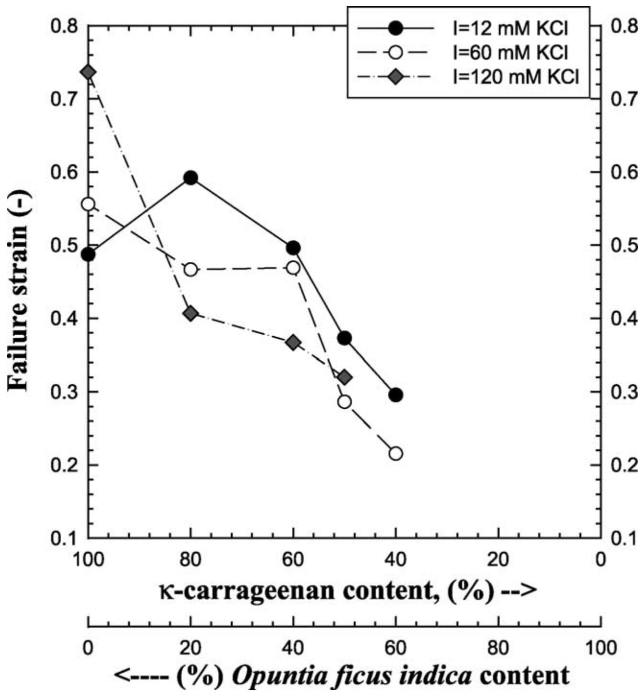

Fig. 9. Failure strain of κ-carrageenan and mucilage gum from *Ofi* as a function of KCl concentration.

concentration up to a maximum value and then decreased with a further increase in mucilage gum. Mao et al. (2000) as well as Tang et al. (1996) reported a similar behavior for mixed gellan gels. On the other hand, Chen et al. (2001) as well as several authors cited in their work did not found any peak when measuring the Young's modulus as a function of κ-carrageenan/locust bean gum ratios. The authors suggested differences in the molecular weight of the κ-carrageenan used as a possible reason for their findings. In our study, it should be noted that the maximum value corresponds to 80/20 ratio at all potassium concentrations.

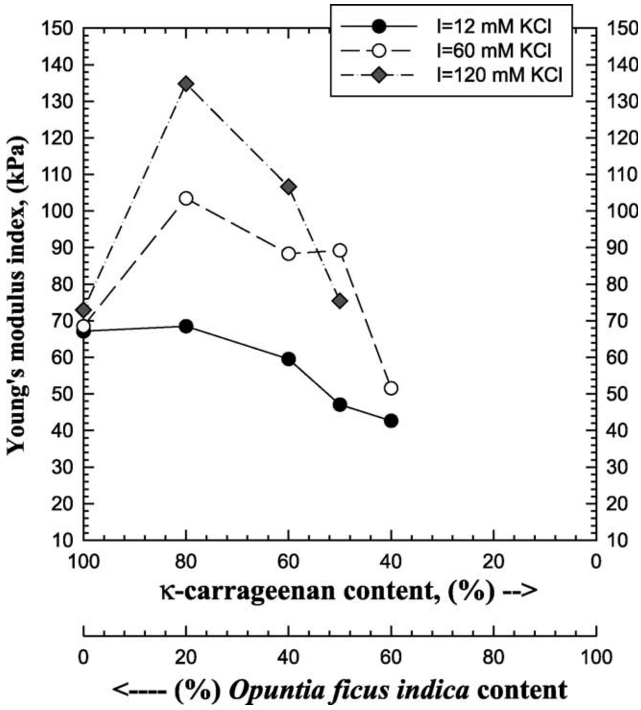

Fig. 10. Young's modulus of mixed κ-carrageenan–mucilage gum from *Ofi* as a function of KCl concentration.

4. Conclusions

The mechanical spectra in the linear viscoelastic region, as a function of mucilage gum concentration and κ-carrageenan or i-carrageenan have been examined. For the 2% total binary system of i-carrageenan and mucilage gum, no enhancement of the mechanical properties with respect to the pure i-carrageenan was observed. On the other hand, for the κ-carrageenan/mucilage gum system, at 80/20 and 60/40 ratios, higher gel rigidity [G'] than the pure κ-carrageenan gels was observed.

Failure properties (strain and stress) were also studied here. The rupture stress increases as the ionic strength (potassium concentration) increases, but it decreases as the mucilage gum increases, except for the binary system κ-carrageenan/mucilage gum at an 80/20 ratio where an increase in this property was observed. Regarding the rupture strain, it decreases as mucilage gum concentration increases and this is a function of the ionic strength, except for the blend κ-carrageenan/mucilage gum at an 80/20 ratio where it increases.

Under the specified experimental conditions, the results of this study suggest that for the binary system of κ-carrageenan and mucilage gum, at an 80/20 ratio, there is a synergistic effect between these two hydrocolloids.

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