



The effect of degradation on κ -carrageenan/locust bean gum/konjac glucomannan gels at acidic pH



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ABSTRACT

The feasibility of textural and rheological modification of gels containing κ -carrageenan (KC) and locust bean gum (LBG) by addition of konjac glucomannan (KGM) was investigated. Special attention was paid to the effect of polysaccharide degradation during heating at acidic pH. The general effect of polysaccharide degradation was to decrease the Young's modulus, while the fracture strain in extension was scarcely affected unless the degradation was very severe.

Differential scanning calorimetry showed that the melting peak corresponding to dissociation of KC–KGM bonds decreased faster than the melting peak of KC-only bonds with increasing degree of polysaccharide degradation. The implication is that as degradation proceeds, fewer KGM molecules can interact with KC to form elastic bonds, and the excess of KGM which reinforces the existing elastic network and increases the fracture strain actually increases. For this reason, the fracture strain remains nearly unchanged with increasing degradation levels. A decrease in fracture strain is thus observed only at very severe degradations, where KC no longer forms a self-supporting gel by itself.

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1. Introduction

κ -Carrageenan (KC) is a non-branched sulphated polysaccharide extracted from marine red algae, with the repeat unit β -D(1→3)-galactopyranose and 3,6-anhydro- α -D(1→4)-galactopyranose. Along with other marine red algae polysaccharides, it forms the second most used polysaccharide group in the food, chemical, pharmaceutical and cosmetic industries (Piculell, 2006), the first being starch and its derivatives. One popular way to modify the properties of KC-based gels in the food industry is to add non-gelling galactomannans, such as locust bean gum (Morris, 1990), with resulting gels showing higher fracture strains and less syneresis. The addition of konjac glucomannan, while less common, was shown to affect both the melting and rheological properties of KC-gels more strongly than LBG (Williams, Clegg, Langdon, Nishinari, & Phillips, 1992; Williams, Clegg, Langdon, Nishinari, & Piculell, 1993). Recent investigations of the nature of interactions and the resulting change in texture of ternary gels containing KC, LBG and KGM as a function of mixing ratio have revealed that two types of interactions are present between KC and LBG and also between KC and KGM (Brenner, Achayuthakan, & Nishinari, 2013;

Brenner, Wang, Achayuthakan, Nakajima, & Nishinari, 2013). One interaction leads to formation of elastic bonds that are stronger than bonds between KC chains, with a weight ratio of about 1–5 (LBG to KC) or 1:7 (KGM to KC). Any excess KGM or LBG present beyond this concentration can interact with the elastic network in a different way, and this excess galactomannan or glucomannan increases the yield strain of the gels without increasing the elastic modulus. Thus, by changing the mixing ratios of KC, LBG and KGM, the Young's modulus may be fine-tuned, and the elasticity may be increased, yielding gels that can be extended to 6 times their original length (Brenner, Wang, et al., 2013).

The effect of polysaccharide thermal degradation on food quality has been long recognized (Fagerson, 1969). For the gels under study, acidification of the mixture post thermal treatment led to substantially higher Young's moduli than were obtained after heating the acidified solution (Brenner, Achayuthakan, et al., 2013). In the present study, we elucidate the effect of this thermal degradation on the non-linear rheology of the gels and compare the results with DSC endotherms.

2. Materials and methods

2.1. Gelling powders

The following gelling agent powders were a gift from San-Ei Gen FFI Co. (Osaka, Japan): Vistop D-2134 (KGM), Gel up J-4535

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(KC) and Vistop D-2050 (LBG). The sulphate group, cationic content and molecular weights of these gelling agents have been reported (Brenner, Achayuthakan, et al., 2013). For simplicity, we refer to each gelling agent by its polysaccharide species, i.e., we refer to LBG, KC and KGM powders. The total gelling powder content is henceforth referred to as the total polysaccharide content.

2.2. Preparation of the gels

The total polysaccharide (gelling powder) content was 1.2 wt%, and the sucrose content was 25 wt%. The concentrations of KC and LBG powders were kept equal, while the concentration of the KGM powder was varied. The ratio LBG/KC=1 was chosen because according to Chen, Liao, Boger, and Dunstan (2001), the rupture stress in compression of LBG/KC gels in the presence of low KCl concentrations showed a peak around this ratio, while the shear modulus of the gels decreased at higher LBG:KC ratios. Similar results were obtained in a previous investigation of our group (Brenner, Wang, et al., 2013). We will refer to the KGM powder content by its relative weight fraction of the total polysaccharide (gelling powder) content, χ_{KGM} ($\chi_{KGM} = C_{KGM}/1.2\%$). Values of χ_{KGM} in the range 0–0.5 were tested, allowing for a systematic study of the effect of KGM. No salt was added to the solution, so that apart from counter ions, the only salt present is KCl from the KC powder at concentrations between 5.5 and 11 mM. This series of gels imitates commercially available dessert jellies, in which both citric acid trisodium salt and anhydrous citric acid are common ingredients. The citric acid trisodium salt and anhydrous citric acid concentrations were fixed at 0.15 and 0.22 wt%, respectively. The resulting pH of the mixtures was 3.4–3.7, and increased slightly with increasing χ_{KGM} .

Four different dissolution methods were tested, referred to as M1–M4. For M1, First, the sucrose (25 wt%) and citric acid trisodium salt (0.15 wt%) were dissolved in MQ-grade water. Then, LBG, KC and KGM powders were added, in this order, under mechanical stirring at 70 °C. The solution was further heated at 80 °C for 40 min, followed by addition of anhydrous citric acid (0.22 wt%). The solution was poured into moulds and allowed to cure at 5 °C quickly after the anhydrous citric acid dissolution. For methods M2–M4, anhydrous citric acid (0.22 wt%) was added with the sucrose and citric acid trisodium salt, and the polysaccharides were added slowly and gradually under stirring at 45–50 °C. The solutions were further heated as follows: 65 °C for 30 min and 75 °C for 30 min (M2); 75 °C for 30 min and 85 °C for 30 min (M3); 45–50 °C for 90 min, 75 °C for 30 min and 85 °C for 30 min (M4). Following heating, the hot solutions were poured into moulds of different dimensions and then kept in a refrigerator (5 °C) for 16–20 h. Gels were equilibrated to room temperature (25 ± 2 °C) (about 60–120 min) before measurement.

2.3. Ring extension

Ring extension was performed on an XT.T2 Texture Analyser (Stable Micro Systems, Surrey, UK). Rings ($h = 11$ mm, outer $\varnothing = 51$ mm, inner $\varnothing = 21$ mm) were held with 2 metal bars ($\varnothing = 8$ mm). The lower bar was fixed and the upper bar was raised to ring rupture (Kohyama, Iida, & Nishinari, 1993). An engineering stress is obtained in this case by dividing the measured force with the initial cross section of the ring, 330 mm^2 . An estimate of the average engineering strain that neglects strain due to body forces was suggested in the literature (Tschoegl, Rinde, & Smith, 1970). The body forces, i.e., gravity, cause vertical elongation of the hung ring. We have followed the newly suggested analysis, where the total engineering strain at its maximum, i.e., at the inner edge of

the ring, is estimated (Brenner, Wang, et al., 2013). In this case the engineering extensional strain is estimated with:

$$\varepsilon = \frac{D - \delta}{\pi R_i} \quad (1)$$

where D is the distance the upper bar is raised and R_i is the initial inner radius of the ring (10.5 mm), while δ is a constant chosen so that the error in estimating the extensional strain is minimized ($\delta = 8.5$ mm). Such an estimate is justified because the rupture was always observed from the inner edge toward the outer edge of the ring (Brenner, Wang, et al., 2013). Within our approximation, the raising speed used, 5 mm/s, corresponds to an extensional deformation rate $\dot{\varepsilon} = 0.15 \text{ s}^{-1}$. The test was started with a distance of 13 mm between bar centers, because the initial inner diameter is 21 mm and the bar diameter is 8 mm.

2.4. Complex Young's modulus

Values of the complex Young's modulus were obtained on a Rheograph Gel (Toyo Seiki Seisakusho, Tokyo, Japan), as described by Nishinari et al. (1980). The Rheograph measures the longitudinal vibrations of the specimen at $F = 3$ Hz and amplitude = 100 μm (strain = 0.32%) and yields E' and E'' values (accuracy 0.1 kPa).

2.5. Intrinsic viscosity measurements

A KRK Ubbelohde viscometer (Vidrex, Fukuoka, Japan) was equilibrated at 30 ± 0.2 °C using a BR-61 uni-thermo bath (Yamato Scientific Co.). While the exact temperature was not determined to better accuracy than ± 0.2 °C using a simple thermometer, the total fluctuation during each individual measurement, as well as between different measurements, was ensured to be within ± 0.02 °C using a Beckmann thermometer (Nishinari et al., 1991). LBG solution in 0.05 M NaCl was prepared using protocols described in the text, with a resulting pH of 3.5. The specific viscosity, η_{sp} , was determined at different concentrations c . The intrinsic viscosity $[\eta]$ and Huggins coefficient k_H were determined using a linear expansion (Young, 1983):

$$\frac{\eta_{sp}}{c} = [\eta] + k_H c [\eta]^2 \quad (2)$$

2.6. Shear viscosity measurements

The shear viscosity of concentrated solutions was determined using a Rheostress 600 (Thermo Scientific) rheometer with a cone (2°) and plate geometry. Solutions were loaded at room temperature and measured at 25 °C.

2.7. Extrusion test

The extrusion test was performed as previously described (Brenner, Hayakawa, et al., 2013; Brenner, Wang, et al., 2013). The liquid solutions were drawn into 10 ml syringes (Terumo syringe SS10SZ, Terumo Co., Tokyo, Japan), followed by removal of air-bubbles by tapping on the inverted syringe. The gels were cured in the same way as other molds. The measurement of the blank force, as well as recalculation of sensory scores from the extrusion data was as described elsewhere (Brenner, Hayakawa, et al., 2013; Brenner, Wang, et al., 2013).

2.8. Differential scanning calorimetry

Differential scanning calorimetry (DSC) was performed on a Micro-DSC III (Setaram, Caluire, France). Samples (0.8 ml) were loaded into stainless steel cells, heated to 85 °C to erase the thermal

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