Brama australis gel obtention and rheological characterization

Abstract

Gelatine from marine sources has been looked upon as a possible alternative to bovine and porcine gelatine. Common problems connected with fish gelatine from cold water species, such as those from Chilean coast, are low gelling and melting temperature and low gel modulus. Squeezing flow technique was applied to obtain rheological parameters as function of moisture content, temperature dependence of extensional viscosity, etc. This work studied effects of pH and concentration on the extensional viscosity of \textit{brama australis} fish gelatine gels using lubricated squeezing flow method and obtained that biaxial extensional viscosity increases with increasing Brama australis fish gelatine concentration.

Keywords: squeezing flow; brama australis; fish gelatin; extensional viscosity

1. Introduction

Gelatine from marine sources (fish skin, bone and fins) has been looked upon as a possible alternative to bovine and porcine gelatine, especially since the outbreak of the BSE (mad cow disease) in the 80s. The commercial interest in fish gelatine has this far, however, been relatively low. This is due to different physical properties compared to mammalian gelatine. Common problems connected with fish gelatine from cold water species, such as those from Chilean coast, are low gelling and melting temperature and low gel modulus.

Several authors have used the squeezing flow technique to study rheological properties of foods [1]. This technique has been applied to products such as cheese, mayonnaise, tomato paste, wheat dough, yogurt and mustard, among others [2]. During the formation of foams, bubble boundaries expand biaxially and shrink in thickness in a manner similar to squeezing films; some phenomena occurring
during food intake can be modelled using squeeze flow: chewing between teeth and/or gums resembles a compression between (irregular) plates [3]. The squeezing flow technique has been applied to obtain rheological parameters such as power law model parameters, extensional viscosity, extensional viscosity as function of the moisture content, temperature dependence of extensional viscosity, etc. [2].

The lubricated squeezing flow viscometry technique is one of the basic types of biaxial extensional flow. The usual geometry to carry out the test consists of a bottom fixed plate where the sample is placed, and an upper plate operated at a constant downward speed. Samples are lubricated with a low viscosity fluid [4]. The following assumptions are made in lubricated squeezing flow viscometry [2]: a) perfect slip at the surface walls; b) negligible end effects; c) incompressible material; and d) constant temperature. The objective of this work was to study effects of pH and concentration on the extensional viscosity of *brama australis* fish gelatine gels using lubricated squeezing flow method.

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2. Materials & Methods

*Brama australis* fish was obtained from the local market. The procedure by Nagai & Suzuki [5] was applied to *Brama australis* (skin) to prepare collagen. Moisture, proteins and ash analyses were performed to samples obtained in the laboratory according to the AOAC [6]. Lipids analyses were performed according Bligh & Dyer method [7].

Samples of *Brama australis* fish gelatine powder, produced in our laboratory, were prepared at 7; 9 and 11 % (w/v) at different pH values (4; 5.5; 7; 8.5 and 10). *Brama australis* fish gelatine powder was added to distilled water at room temperature and dissolved by increasing the controlled water bath temperature to 40°C while stirring. When the sample reached 40°C either a NaOH solution (0.5M) or HCl solution (0.5M) was added drop by drop until the desired pH, controlled by a pH-meter (Cole Parmer,
USA), was reached. The sample was removed from the controlled water bath and left to cool at room temperature and afterwards stored at 10°C for approximately 24 hrs. Formed gels (120 samples) were cut into 21 mm diameter cylinders with 18 mm height.

2.1 Rheological testing

*Brama australis* gelatine gel samples were placed between two parallel plates lubricated with petroleum jelly to minimize friction effects and to guarantee only extensional deformation [8]. Once the sample was loaded, a resting time of 5 min prior to testing was allowed to stabilize internal tensions. Samples were subjected to uniaxial deformation at a constant deformation rate (10; 20; 30 and 40 mm/min) and compressed 30% of their initial height using a Material Testing Machine (Zwick, Model DO – FBO. 5TS, Germany) interfaced with a Texture data processor (Zwick®). Force - distance data was automatically recorded. Each test was done in triplicate and average values were used for data analysis. Analysis of Variance was applied (α=0.05) to determine differences between treatments. Rheological tests were performed at 7 - 9°C in an acclimatised laboratory room.

Results were analyzed with Microsoft® Excel 2000 software (Microsoft Corporation, 1983-1999). Treatment averages were considered significantly different at P<0.05.

2.2 Data Treatment

The following scheme illustrates data treatment [2] to obtain the extensional viscosity (Figure 1).

\[ \eta_b = \frac{\sigma}{\varepsilon_b} \]

where: \( h_0 \) is the Initial sample height, \( D_0 \) is the Sample Diameter, \( A_0 \) is the Sample Area, \( V \) is the Compression speed, \( D \) is the Distance traveled by plunger into the sample, \( t \) is the Time of compression, \( F \) is the Force of compression, \( h \) is the Sample height at time \( t \), \( \varepsilon_b \) is the Shear rate, \( \sigma \) is the Biaxial Stress and \( \eta_b \) is the Extensional Viscosity.

3. Results & Discussion

Squeezing flow technique results are shown in Figure 2, where the effects of concentration of *Brama australis* fish gelatine gel, from 7, 9 and 11% w/v, are studied using an increasing compression rate (10, 20 mm/min, 30 and 40 mm/min) to reach a 30% gel compression, when controlling pH at a value of 8.5.

From Fig. 2 it can be seen that at a constant shear rate, when concentration increases the biaxial stress increases; also it can be seen that although the compression rate is kept constant, the shear rate values...
vary due to concentration effects. This behavior can be explained because if a gel has higher concentration, it has less water content and therefore is more rigid due to a higher number of inter and intra molecular bonds, which leads to a more stable tridimensional network [9].

![Graphs showing concentration effects on biaxial stress versus shear rate](image)

Fig. 2. Concentration (7 % w/v, 9% w/v and 11% w/v) fish gelatine gel effects on biaxial stress versus shear rate for increasing compression rate (top left 10 mm/min, top right 20 mm/min, bottom left 30 mm/min and bottom right 40 mm/min) for pH 8.5 using squeezing flow technique.

pH effects on extensional viscosity versus shear rate are shown in Figure 3, for *Brama australis* fish gelatine concentration, from 7, 9 and 11% w/v, for 20 mm/min compression rate using the squeezing flow technique.

The explanation for this behavior could be due to the gel isoelectric point, since for pH values higher or lower than that corresponding to the gel isoelectric point, the protein charge is negative or positive, respectively, and therefore, water molecules react with these charges facilitating solubility; with the minimum solubility value being at the isoelectric point [9]. Protein solubility is higher at alkaline than acid pH.

*Brama australis* fish gelatine isoelectric point is around pH of 5.5; and the maximum biaxial stress values are found in the alkaline range (pH = 5.5 and pH = 10), whereas the minimum biaxial stress values are found in the acid region (pH = 4). These findings are in agreement with Fennema [10] which states that as the pH is away from the isoelectric point the gel should present a stronger structure, whereas when it is near to the isoelectric point its structure is weaker.
From Figure 3 it is observed that for all concentration and compression rate values applied in this study the higher extensional viscosity values correspond to those obtained when pH reaches a value of 8.5.

4. Conclusion

Biaxial stress is a textural parameter influenced by concentration and pH; when Brama australis fish gelatine concentration increases biaxial stress values increases. Biaxial extensional viscosity increases with increasing Brama australis fish gelatine concentration. Biaxial extensional viscosity is higher in the alkaline pH range.

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References


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