



## Rheological characterization of the hydrocolloid from *Gleditsia amorphoides* seeds

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### ABSTRACT

Espina Corona Gum (ECG) is extracted from *Gleditsia amorphoides* seeds and is used as an additive in Argentinean food industry. Its chemical structure corresponds to a galactomannan. ECG was compared with Guar Gum (GG) as regards several properties, among them those related to rheological behavior, the effect of NaCl concentration, temperature and acidity. Experimental results demonstrated that ECG solutions have a shear-thinning behavior, respond to a power-law model, are influenced by temperature, and show a good stability when heated. The presence of NaCl and acidity did not affect ECG solution viscosity. ECG solutions were less viscous and less shear-thinning than GG ones. The viscoelastic behavior shows that, for low frequencies, the viscous modulus is greater than the elastic one up to the crossover point of the frequency, where this behavior is reverted. The apparent viscosity decreased as the frequency of oscillation increased.

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### 1. Introduction

Hydrocolloids are hydrophilic high molecular weight biopolymers which have the ability to greatly hydrate in contact with water, thus producing colloidal systems of different structures, and which significantly increase the system viscosity even at low concentrations (Vardhanabhuti & Ikeda, 2006). Because of these properties, hydrocolloids are used as food additives to obtain particular textural characteristics (Amina, Ahmad, Yap, Norfariza, & Norhayati, 2007; Glicksman, 1982). They are broadly used in food systems as thickeners, gelling agents, texture modifiers, suspending agents, and stabilizers (Chaubey & Kapoor, 2001; De Man, 1999). Plant hydrocolloids are beneficial to consumers because of their friendly image toward the environment (Vardhanabhuti & Ikeda, 2006). Galactomannans are non-gelling polysaccharides commercially important mainly for their thickening properties. The two galactomannans of utmost commercial importance are Guar Gum (GG), from *Cyamopsis tetragonolobus*, and Locust Bean Gum (LBG), from *Ceratonia siliqua* (Dakia, Blecker, Roberta,

Watheleta, & Paquota, 2008). Espina Corona Gum (ECG) is a galactomannan extracted from the seeds of Espina Corona (*Gleditsia amorphoides*), a leguminous tree native of Latin America that grows in the forests of Northern Argentina. It was grown in Argentina in the 50's and 60's (Riqué & Pardo, 1952; pp. 1–28); then its production was discontinued, although it never ceased completely. Today, due to the increasing cost of similar foreign products, Espina Corona (EC) production is being revitalized through various businesses. However, despite the rich flora biodiversity and the favorable climate for their production, galactomannans from Latin American sources are not well known (Azero & Andrade, 2002).

Galactomannans are neutral polysaccharides consisting of a linear mannose backbone bearing side chains of a single galactose unit (Azero & Andrade, 2002). The chemical composition of ECG was postulated by Cerezo in 1965 as a galactomannan structure composed of 71.4 g/100 g D-mannose and 28.6 g/100 g D-galactose with a mannose-to-galactose (M/G) ratio of 2.5 (Cerezo, 1965). The mannose forms a linear chain of (1 → 4) β-mannopyranose units with one molecule of D-galactopyranose linked at position 6 every three units of mannose. This relation is very similar to that of other galactomannans and already well-known for galactomannans such as Guar Gum, with 1 galactose every 2 units of mannose and 2.0 M/G ratio (Chaubey & Kapoor, 2001; Doublier & Launay, 1981; Whistler & BeMiller, 1997).

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The gum's usual purification processes include its extraction from the seeds using water as a solvent, followed by filtration or centrifugation. The gum can be recovered by precipitation with alcohol, drying in an oven and milling, or freeze-drying or spray-drying (Azero & Andrade, 2002; Ibañez & Ferrero, 2003; Oomah & Mazza, 2001). In general, the purity of ECG commercial products is not too high in the Argentinean market. The gum is not often commercially available in purified forms and contains significant amounts of insoluble fraction. This insoluble fraction reduces the product value since it renders the gum inappropriate to be used in food formulations that need clear shades or light colors. Instead, the product has black spots from the tegumental residues that cover the seed's endosperm. These black spots are generated in the roasting procedure used to remove the tegument.

The composition of ECG plays an important role in its rheological properties, and the rheological behavior also depends on the method used to obtain the gum (Azero & Andrade, 2002).

The objectives of this research were to study a gum purification process applicable in industry, using a dryer to remove impurities and insoluble fraction, as well as to study the chemical composition and the rheological properties of the gum. With this purpose, the effects of concentration, temperature, sodium chloride and glucono- $\delta$ -lactone (GDL) on the flow behavior of polysaccharides were investigated.

## 2. Materials and methods

### 2.1. Materials

The industrial process of ECG carried out by Idea Supply Argentina S.A. (Chaco, Argentina) consists of washing the seeds in chlorinated water, toasting at 120 °C during 1 h, and shelling with grinding equipment. Subsequently, the seeds are sifted to separate the shells and finally they are ground. The ECG flour (ECGF) obtained has light cream thin granules with small dark particles derived from the tegument, and is sold as such.

Guar Gum (GG) is a marketing sample provided by Saporiti S.A. (Buenos Aires, Argentina).

### 2.2. Purification of the Espina Corona Gum flour

ECGF (20 kg m<sup>-3</sup>) was suspended in tap water and stirred at 60 °C for 3 h. Later, it was filtrated with an ASTM 40 (420  $\mu$ m) sieve, thus obtaining a solution from the gum and a retained material composed of tegumental residues and undissolved fractions. Dry solids were determined by weight loss in an oven at 105 °C for both fractions. Subsequently, the gum solution was spray-dried at a pilot plant with a Niro atomizer spray-dryer (Denmark) using an inlet temperature of 200 °C and an outlet temperature of 90 °C.

### 2.3. Chemical analysis

Moisture, crude fat, total protein, crude fiber and ash contents were determined according to the approved AOAC International 934.01, 920.39, 2001.11, 942.05, 962.09 (AOAC, 2006), respectively. All trials were conducted in triplicate.

### 2.4. Color analysis

The color of both powder and solution was evaluated with a Minolta CM-508d colorimeter (Japan) using a D65 illuminant and a 10° observer angle. The samples were compressed for 5 s with a weight of 1 kg. The color was expressed in terms of lightness ( $L^*$ ), redness ( $a^*$ ), and yellowness ( $b^*$ ).

## 2.5. Rheological characterization

### 2.5.1. Determination of intrinsic viscosity

Determination of intrinsic viscosity  $[\eta]$  was carried out by extrapolation of the Huggins and Kramer model, expressed by equations (1) and (2), respectively (Mothé & Rao, 1999), considering the relative viscosities  $[\eta_{rel}]$  within the 1.2–2.0 range.

$$\frac{[\eta_{sp}]}{C} = [\eta] + k_1 \cdot [\eta]^2 \cdot C \quad (1)$$

$$\frac{\ln[\eta_{rel}]}{C} = [\eta] + k_2 \cdot [\eta]^2 \cdot C \quad (2)$$

where  $[\eta_{sp}]$  is specific viscosity.

The viscometric average molecular masses  $\bar{M}_v$  were calculated using the Mark–Houwink equation (3) given by Doublier and Launay (1981) for Guar Gum and modified by Gaisford, Harding, Mitchell, and Bradley (1986) to take into account the different M/G values in galactomannans:

$$[\eta] = 11.55 \cdot 10^{-6} [(1-r)\bar{M}_v]^{0.98} \quad (3)$$

where  $r$  is the galactose/(mannose + galactose) ratio and  $[\eta]$  is the intrinsic viscosity, in dL g<sup>-1</sup>.

### 2.5.2. Rotational rheological analyses

A 20 kg m<sup>-3</sup> sample of an ECG solution was prepared, left overnight at room temperature, and centrifuged at 1800  $\times$  g for 1 h. The supernatant was used for further dilutions. Rheological measurements were carried out with an HAAKE RheoStress RS80 (Haake Mess – Technik GmbH., Germany), with a plate-and-plate geometry of 35 mm diameter and 1 mm of gap between the plates, varying the shear rate ( $\dot{\gamma}$ ) from 0.1 to 1000 s<sup>-1</sup> for rotational trials. For temperature control, an HAAKE N2T bath was used and the data were analyzed with the HAAKE RheoWin 3.50.0011 software. All trials were conducted in triplicate.

The shear-thinning behavior of fluids can be modeled in a limited interval of shear rate ( $\dot{\gamma}$ ) with Ostwald's power law (Rosenthal, 2001; pp. 69–80) equation (4).

$$\eta_{ap} = k\dot{\gamma}^n \quad (4)$$

being  $\eta_{ap}$  the apparent viscosity (Pa s),  $k$  the consistency coefficient (Pa s <sup>$n$</sup> ),  $\dot{\gamma}$  the shear rate (s<sup>-1</sup>) and  $n$  the flow behavior index (dimensionless).

**2.5.2.1. Effect of gum concentration.** The effect of gum concentration on  $\eta_{ap}$  was studied preparing ECG solutions at concentrations of 15 – 10 – 7.5 – 5.0 – 2.5 kg m<sup>-3</sup>. Measurements were carried out at 25  $\pm$  0.1 °C. ECG solutions were compared with GG solutions at 25  $\pm$  0.1 °C at 5.0 and 10 kg m<sup>-3</sup>.

**2.5.2.2. Effect of temperature.** The effect of temperature (10, 25, 40 and 60  $\pm$  0.1 °C) on the  $\eta_{ap}$  of 10 kg m<sup>-3</sup> ECG solution was determined.

The hydrocolloid stability after heating was determined on a 5.0 kg m<sup>-3</sup> ECG solution. First, the  $\eta_{ap}$  of the ECG solution was measured at 25 °C; the solution was then heated for 30 min at different temperatures (60 °C, 75 °C and 90 °C), and finally the temperature was reduced to 25  $\pm$  0.1 °C and  $\eta_{ap}$  was measured again.

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