



# Characterisation of a new biodegradable edible film based on sage seed gum: Influence of plasticiser type and concentration



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## ABSTRACT

The present work dealt with investigating physicochemical, barrier, mechanical and surface properties of sage seed gum (SSG) edible films as a function of plasticiser type (glycerol and sorbitol) and concentration (20, 40, 60, 80 & 100, w/w%). Based on the results, it was revealed that glycerol and sorbitol effectively plasticise the films of SSG while glycerol efficiency was higher than sorbitol. Although the films plasticised by glycerol were of high moisture content (27–49%), moisture uptake (110–140%) and solubility in water (~80%), but they exhibited low permeability to water vapour, acceptable mechanical properties, surface hydrophilicity, and transparency. In contrast, the films plasticised by different concentrations of sorbitol exhibited a low moisture content (~14%), water vapour permeability (lower than  $4 \times 10^{-11}$  g/m.s.Pa), and moisture uptake (below 90%) with high surface hydrophobicity but less desirable mechanical properties. The probable mechanisms of plasticising are discussed in details regarding the observed characteristics of SSG edible films. Concluding, the films of SSG in present work showed a substantial potential to be incorporated in food packaging applications.

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## 1. Introduction

Nowadays, due to the non-degradability of synthetic packaging materials, there have been increasing research interests toward natural resources to produce biodegradable edible films. These studies mainly focused on developing edible films and improving their characteristics to exert the main desired features of conventional synthetic materials, such as high mechanical strength, softness, lightness, transparency, water resistance, and so on.

The major potential alternatives to plastics, which have been studied extensively, are biopolymers including polysaccharides, proteins, and lipids. Based on the diversity, availability, and unique characteristics, most effort has been put on studying the film forming and characterisation of edible films from hydrocolloids of different sources with diverse chemical structures, e.g. kefiran, *Opuntia nopal* mucilage, psyllium mucilage, starches of different sources, gellan, cress seed gum, guar, locust bean gum (LBG), hydroxypropyl methylcellulose (HPMC), alginate, chitosan, agar, and gelatin (Ahmadi, Kalbasi-Ashtari, Oromiehie, Yarmand, & Jahandideh, 2012; Bergo et al., 2008; Dias, Muller, Larotonda, &

Laurindo, 2010; Espino-Diaz et al., 2010; Ghasemlou, Khodaiyan, & Oromiehie, 2011; Ghasemlou, Khodaiyan, Oromiehie, & Yarmand, 2011; Imran, El-Fahmy, Revol-Junelles, & Desobry, 2010; Jouki, Khazaei, Ghasemlou, & HadiNezhad, 2013; Laohakunjit & Noomhorm, 2004; Mikkonen et al., 2007; Olivas & Barbosa-Canovas, 2008; Phan, Debeaufort, Luu, & Voilley, 2005; Piermaria et al., 2011; Piermaria, Pinotti, Garcia, & Abraham, 2009; Rivero, Garcia, & Pinotti, 2010; Seyedi, Koocheki, Mohebbi, & Zahedi, 2014; Thakhiew, Devahastin, & Soponronnarit, 2010; Yang & Paulson, 2000).

Although edible films fabricated from hydrocolloids showed many appropriate characteristics (e.g. good gas, aroma, and lipid barriers) but their industrial application is limited because of the inherent brittleness originated from their branched primary structure and weak intermolecular forces. By definition, plasticisers are high boiling point low molecular substances that increase the flexibility, workability, or distensibility of matrix biopolymer via reducing its electrostatic charge, at the same time increasing the chain flexibility, resistance to fracture and dielectric constant (Vieira, da Silva, dos Santos, & Beppu, 2011). Compatibility and durability are important factors for selecting a plasticiser to achieve favourite functional properties in edible films of biopolymers. Polyols, mono-, di- and oligosaccharides are the most commonly used plasticisers, mainly glycerol, sorbitol, and polyethylene glycol (PEG), which impart greater plasticising effect to edible films of

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hydrocolloids (Mali, Grossmann, Garcia, Martino, & Zartitzky, 2004; Piermaria et al., 2011; Pongjanyakul & Puttipatkhachorn, 2007; da Silva, Krause Bierhalz, & Kieckbusch, 2009; Yang & Paulson, 2000).

Sage (*Salvia macrosiphon*) is an endemic plant belonging to the genus *Salvia*. It has been reported that the gum extracted from sage seeds, regarded as SSG hereinafter, is a galactomannan (mannose to galactose ratio of 1.78–1.93:1) containing 28.2–32.2% uronic acids reflecting polyelectrolyte nature with a rigid rod-like conformation of  $4 \times 10^5$  Da average molecular weight, which exhibited a stronger shear viscosity, pseudoplasticity, and elasticity compared with other galactomannans based on rheological characterisations (Razavi, Cui, Guo, & Ding, 2014; Razavi, Taheri, & Quinchia, 2011; Razavi, Taheri, & Sanchez, 2013). Recent studies indicated that SSG has great potential to exert stabilising, thickening, and emulsifying properties in food applications, comparable with commercial hydrocolloids (NikNia, Razavi, Koocheki, & NayebZadeh, 2010; Razavi, Mohammadi Moghaddam, Emadzadeh, & Salehi, 2012; Razmkhah, Razavi, Behzad, & Mazaheri Tehrani, 2010; Razavi et al., 2011, 2013).

To the best of our knowledge, there is no published study regarding characterisation of edible films made from SSG. Therefore, the aims of the present study were to investigate the effect of different proportions (20–100% w/w) of plasticisers of different types, i.e. glycerol, sorbitol, and PEG-400 on physicochemical, mechanical, permeability, surface and thermal properties of biodegradable SSG films.

## 2. Materials and methods

### 2.1. Materials

Glycerol, sorbitol, PEG-400, anhydrous calcium chloride, sodium chloride, and ethylene glycol (EG) were purchased from Merck (Germany). Potassium sulphate and sodium azide were from AppliChem (Germany), and calcium nitrate tetrahydrate was from Panreac (Spain). At all preparations and analyses, deionised water was used, unless otherwise specified. For those experiments that required sample conditioning, the relative humidity (RH) inside the chamber were monitored by AM-4205A hygrometer (Lutron Electronic Enterprise, Taiwan) to ensure the appropriate humidity has been provided.

The sage seed gum (SSG) was extracted at optimised conditions according to the method described by Bostan, Razavi, and Farhoosh (2010), dried by a ventilated oven at 45 °C until dryness, then grinded and kept in air-tight containers prior to use.

### 2.2. Film preparation

The film forming solution was prepared by the following sequential procedure. First of all, the aqueous hydrocolloid solution (1.5% w/w) was prepared by adding the appropriate amount (20, 40, 60, 80, and 100%) of glycerol, sorbitol, or PEG-400 to water, stirred and warmed up to 80 °C using a hot-plate magnetic stirrer at 150 rpm. After adjusting the temperature, the powdered SSG gum was added and stirred at 1200 rpm using magnetic stirrer at 80 °C for 10 min. This sequential procedure was used to ensure that SSG polymers completely react with plasticiser molecules because of the fast hydration of SSG powder which otherwise did not allow complete mixing of the two constituents together. To homogenate the film forming solution, T25 Ultra-Turrax (Ika, Germany) was used for one minute at 3200 rpm speed. After homogenisation, the solution was placed on rotating roller mixer for 24 h at room temperature to allow complete hydration of hydrocolloid and also providing enough time to react with plasticiser molecules for any

possible configurationally changes. The film forming solution was casted on polyethylene casts ( $\phi = 15$  cm), placed at ambient conditions (~53% RH, 23 °C) to complete dryness, then the films were peeled off from the cast and stored at cool and dry place before performing the experiments. Each film preparation was replicated thrice per sample. It should be mentioned that the same procedure was performed to prepare control sample without plasticiser addition.

### 2.3. Film thickness ( $\delta$ )

The thickness of films was measured using a digital QLR-Digit micrometre (Qinghai Measuring & Cutting Tools Group Co., China) at ten random points of films. Measurements were replicated three times with independent film samples and the mean values were reported. The  $\delta$  of films required for water vapour permeability, density, and mechanical properties experiments was characterised independently.

### 2.4. Moisture content

The moisture content (MC, w.b.%) of films was determined gravimetrically using a ventilated oven at 105 °C until constant weight was achieved. The test specimens were cut to 2 cm squares, preconditioned at 75% RH at  $23 \pm 2$  °C by a chamber containing saturated NaCl solution for 48 h, and then shredded immediately before performing the experiments. Nine replicates were performed for each sample.

### 2.5. Film density

The film pieces of  $4 \times 4$  cm dimensions were conditioned according to the previous section. The thickness was measured at 9 points for each replicate and it was repeated three times, and averaged. The film density was calculated as the ratio of film mass to its volume (the product of area and thickness).

### 2.6. Moisture uptake

With minor modifications, the procedure described by Angles and Dufrense (2000) was used to determine moisture uptake by the samples. Briefly, a 2 cm square cut of sample was conditioned at 0% RH using anhydrous calcium chloride for 5 days at  $23 \pm 2$  °C, weighed, conditioned at 25 °C using saturated potassium sulphate (97% RH), weighed consequently until constant weight was achieved. The initial and equilibrium weights of sample ( $m_1$  and  $m_2$ , respectively) were used to calculate moisture uptake through Eq. (1).

$$\text{Moisture uptake(\%)} = \left( \frac{m_2 - m_1}{m_1} \right) \times 100 \quad (1)$$

### 2.7. Water solubility ( $S_w$ )

The method of Irissin-Mangata, Bauduin, Boutevin, and Gontard (2001) was used to measure the  $S_w$  of films. Before measurements, the samples were conditioned at 75% RH for 48 h at room temperature. In this respect, 2 cm square pieces of samples were weighed ( $m_1$ ), dried at  $75 \pm 2$  °C until constant weight ( $m_2$ ) to calculate the moisture mass fraction ( $\omega$ ) as the ratio of weight loss ( $m_1 - m_2$ ) to  $m_1$  of sample. Then, the same dried pieces of films were immersed in 45 ml of 0.02% w/v sodium azide solution in screw capped containers, maintained at room temperature under mild agitation for 24 h using roller-mixer, and filtered under vacuum through MN-640 m filter papers (Macherey-Nagel, Germany). The

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