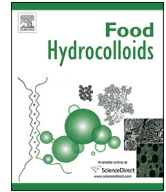




Contents lists available at ScienceDirect

## Food Hydrocolloids

journal homepage: [www.elsevier.com/locate/foodhyd](http://www.elsevier.com/locate/foodhyd)

## Rheological characterization of hydrocolloids interaction: A case study on sage seed gum-xanthan blends

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### ARTICLE INFO

#### Article history:

Received 18 July 2016

Received in revised form

14 November 2016

Accepted 18 November 2016

Available online xxx

#### Keywords:

Gum  
Recovery  
Rheology  
Synergy  
Thixotropy

### ABSTRACT

In this paper, a first robust scheme for in-depth rheological study on the interaction of two attractive hydrocolloids (sage seed gum (SSG) and xanthan gum (XG) at five blending levels (1-0, 3-1, 1-1, 1-3, and 0-1)) was defined by utilizing flow behavior, thixotropy and viscoelasticity measurements. Shear thinning as the important rheological behavior of biopolymers were found to be broadly different in steady and dynamic shear tests. We attributed this difference to the longer timescale of segment-segment interaction in SSG chains than that of XG. A well-developed three-parametric exponential model satisfactorily fitted the recovery phase data of in-shear structural recovery test. There was a negative correlation between time effects in SAOS (small amplitude oscillatory shear) and thixotropy with  $R^2$  equal to  $-0.90$ . The highest recovery parameter (83.25%) and the lowest extent of time dependency (determined in SAOS) were obtained for XG. Excellent correlation existed between the extent of time dependency and the difference between static and dynamic yield stresses ( $R^2 = 94-96\%$ ). The highest and lowest departure values from *Cox-Merz* rule were found for SSG (83.87%) and XG (46.18%), respectively. The strongest interaction between SSG and XG among all ratios tested, occurred for 3-1, which was confirmed by the *Cole-Cole* plot.

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### 1. Introduction

*Sage* (*Salvia macrosiphon*) is a pharmaceutical endemic plant in Iran belonging to the genus *Salvia*. The extracted sage seed gum (SSG) contained 6.72% moisture, 0.85% lipid, 8.17% ash, 2.84% protein, 1.67% crude fiber and 79.75% carbohydrate. SSG is a galactomannan (mannose to galactose ratio of 1.78–1.93:1), containing 28.2–32.2% uronic acids donate typical polyelectrolyte behavior, with an average molecular weight of  $4 \times 10^5$  Da. Also, it exhibited a high zero-shear viscosity, pseudoplasticity, and elasticity compared with other galactomannans (Razavi, Cui, Guo, & Ding, 2014; Razavi, Taheri, & Quinchia, 2011; Razavi, Taheri, & Sanchez, 2013a). SSG adopted a rigid rod conformation in aqueous solution with more chain stiffness than xanthan. Mechanical spectra and deviation from the *Cox-Merz* rule suggested SSG has a self-structured and ordered conformation and exhibited weak gel behavior like xanthan (Razavi, HasanAbadi, Ghadiri, & Salehi, 2013b; Razavi et al., 2013a; Razavi et al. 2014). SSG

became a potential additive in food and non-food industries as stabiliser, thickener, and gelling agent (NikNia, Razavi, Koocheki, & Nayebeh Zadeh, 2010; Razavi, Mohammadi Moghaddam, Emadzadeh, & Salehi, 2012; Razavi et al., 2011, 2013a; Razmkhah, Razavi, Behzad, & Mazaheri Tehrani, 2010). Xanthan gum (XG) is an exocellular polysaccharide produced by the bacterium *Xanthomonas campestris* consist of a main chain of (1,4)- $\beta$ -D-glucose residues with a trisaccharide side chain attached to every glucose (Fitzpatrick, Meadows, Ratcliffe, & Williams, 2013). It can interact with galactomannans to form a thermoreversible gel (Dea et al., 1977; Fernandes, Gonçalves, & Doublier, 1991; Casas, Mohamedano, & García-Ochoa, 2000). XG solutions exhibited high zero-shear viscosity and shear thinning behavior, therefore widely used to stabilise dispersions and emulsions because of its ability to inhibit particle sedimentation and droplet creaming (Fitzpatrick et al., 2013).

There has been a widespread interest in investigation of the properties of gum blends, particularly those consisting of xanthan gum and a galactomannan from new source (Azero & Andrade, 2006). Polysaccharides possess benefits more than synthetic ones because of their sustainable, biodegradable and bio-safe

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characteristics (Han et al., 2015). Texture of gum mixtures highly depends on the nature of the components and their ratios (Fernandes, 1995). Rheological properties of polysaccharide gums are important to understand their structure and potential functionalities in a wide range of applications. Depending on the nature of the association, various rheological techniques could be used to quantitatively characterize the interchain associations in gum blends. As SAOS tests allow tenuous intermolecular associations to be preserved, they could monitor the structure in weak gels or viscoelastic fluids; while steady shear measurements may be employed to follow the breakdown of structure at different rates of shear (Dea et al., 1977; Laity, Gilks, & Holland, 2015; Lin, Shangguan, Zuo, Harkin-Jones, & Zheng, 2012). Despite the aforementioned progresses on SSG, as the variety of hydrocolloids are still limited within only several types, it is necessary to achieve more complete understanding of the physical properties and structure of them, individually and in combination with other gums, to meet the world demands (Albuquerque et al., 2014). Therefore, our framework included:

- (1) Investigating the effect of two kinds of molecular motions involved in the dynamic and steady shear measurements, at various ranges, on the interaction behavior of biopolymer blends.
- (2) Studying the effect of frequency at various logarithmic cycles on the power-law index parameters of frequency dependency of  $G'$  (storage modulus),  $G''$  (loss modulus) and the extent of difference between these two parameters in gum blends.
- (3) Investigating the effect of gums ratios on their recovery responses under low shear condition after high shear treatment with developing a three-parametric exponential model (rate of recovery ( $k_1$ ), instantaneous recovery of the structure ( $\tau_0$ ), and the structural stability parameter ( $\tau_\infty$ )), for gaining a deeper understanding on the microstructure of systems.
- (4) Comparing the extent of difference between static and dynamic yield stresses with the recovery parameter (extent of thixotropy).
- (5) Evaluating the synergistic/non-synergistic effects of several steady and dynamic rheological parameters for various blend ratios, using interaction coefficient and Cole–Cole plots.
- (6) Studying the applicability of the Cox–Merz relationship between steady-shear and oscillatory properties.

This new insight to flow behavior, thixotropy and viscoelasticity measurements can be useful to characterize the interaction behavior of any biopolymer blends and is expected to be the base of the physical characteristics of SSG and its blends with XG, and thus will aid in the eventual utilization of the selected gum blends.

## 2. Materials and methods

### 2.1. Sample preparation

The extraction of sage seed gum (SSG) was performed using the method described by Bostan, Razavi, & Farhoosh, (2010). Xanthan gum was purchased from Sigma Com, Spain. In this work, three SSG–XG ratios (1–3, 1–1, & 3–1%w/w) were employed and compared with individual SSG and XG, while the total polysaccharide concentration was kept constant at 1%w/w. Samples were prepared by thoroughly dispersing the desired amount of gum powders in de-ionized water, stirring at 300 rpm and at room temperature (25 °C) overnight and kept for further 24 h at the same temperature to complete the hydration. In addition, sodium azide (0.02%w/w) was used for all dispersions to prevent the

microbial putrescence.

### 2.2. Rheological tests

All rheological measurements were carried out using a controlled stress/strain rheometer (Gemini 150, Bohlin Instruments Ltd., Malvern, UK) equipped with a cone-plate geometry (40 mm of diameter, 4° cone angle, and 1 mm gap). The temperature was fixed, using a Peltier system, at 30 °C and then each sample was equilibrated, at least for 5 min before the rheological test and was coated around their periphery with light silicone oil, to minimize loss of water. All rheological tests were performed, at least, in triplicate.

#### 2.2.1. Steady shear measurements

Flow behavior of prepared gum dispersions was evaluated at controlled strain mode to obtain shear stress-shear rate data. The shear stress was measured at a logarithmically increasing scale of shear rates from 0.01 to 300 s<sup>-1</sup>. Also, the apparent viscosity at each shear rate was determined as the ratio of shear stress to shear rate (Steffe, 1996). Apparent viscosity-shear rate data was fitted using a well-known power-law equation:

$$\eta_a = k\dot{\gamma}^{(n-1)} \quad (1)$$

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

#### 2.2.2. In-shear structural recovery measurements

In-shear structural recovery of the samples was determined according to the procedure of Mezger (2002). The samples were loaded into the rheometer, then a three stepped shear flow test was performed as follows: (I) a constant shear rate of 1 s<sup>-1</sup> was applied for 120 s (with pre-shear at 1 s<sup>-1</sup> for 30 s) (II) a constant shear rate of 300 s<sup>-1</sup> was applied for 60 s (III) a constant shear rate of 1 s<sup>-1</sup> was applied for 120 s. The percentage ratio of average apparent viscosity during the first 120 s in step III to average apparent viscosity in step I was expressed as extent of recovery ( $R$ , %). It also represents the time effect in large deformation.

Furthermore, in order to provide more structural features from time-dependent behavior of samples; herein, we proposed a three-parametric exponential model (Eqn. (2)), which was fitted on shear stress ( $\tau$ ) data of step III vs. time ( $t$ ) as follows:

$$\tau(t) = \tau_\infty + (\tau_0 - \tau_\infty)\exp(-k_1 t) \quad (2)$$

where  $\tau_0$ (Pa) indicates instantaneous recovered structure,  $k_1$  (1/s) represents the rate of recovery, and  $\tau_\infty$ (Pa) shows the structural stability of recovered material. This equation can cope with built up in step-up tests.

#### 2.2.3. Yield stress measurements

To directly determine the yield stress, the stress was ramped linearly from 0.1 to 15 Pa over 120 s. The instantaneous viscosity was plotted versus the stress. It results in an increase in the viscosity as the structure is maintained (elastic behavior), followed by a rapid decline in the viscosity as the structure collapses and the material starts to flow (fluid behavior). The corresponding stress at the peak of viscosity curve was introduced as the yield stress (Steffe, 1996; Bohlin Instruments, 2004). An alternative procedure to directly obtain the value of yield stress is to determine a zone where the slope of the strain-stress curve rapidly decreased, or in other words, the yield stress can be determined by extending the two straight lines corresponding to solid-like and

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