



High-pressure induced thermo-viscoelasticity and dynamic rheology of gum Arabic and chitosan aqueous dispersions



Hamed Vatankhah, Ali R. Taherian, Hosahalli S. Ramaswamy*

Department of Food Science and Agricultural Chemistry, McGill University, 21,111 Lakeshore, Ste. Anne-de-Bellevue H9X 3V9, QC, Canada

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ABSTRACT

Effect of high pressure treatment (200 MPa, 400 MPa, 600 MPa) on rheological properties of aqueous dispersions of gum Arabic (6 g/100 g, 15 g/100 g, and 20 g/100 g) and chitosan (2 g/100 g, 4 g/100 g) was studied. Dynamic properties were assessed using an angular frequency range of 1–25 rad s⁻¹. Flow properties were measured by applying a programmed shear rate of 0.1–100 s⁻¹ for 10 min (upward) and 100–0.1 s⁻¹ for the next 10 min (downward). A power law shift function was used to describe the deviations from the Cox-Merz theory in all samples. Thermo-viscoelasticity was evaluated using a programmed heat/hold/cool cycle (22 °C/70 °C/20 °C). Two term-exponential and a Gaussian model well fitted the thermo-viscoelastic behavior of both gums. The thermal compatibility, regarding a logarithmic phase shift of loss tangent ratios, was also studied. Results showed higher incompatibilities at lower temperatures, while the shift phases tended to zero at higher temperatures. Overall, HP treatment intensified the existence of a stronger and temperature sensitive hydrocolloid network for 4 g/100 g chitosan and 20 g/100 g GA. The highest incompatibility was seen in 20 g/100 g gum Arabic which was a 1–1.5 order of magnitude.

1. Introduction

Food and pharmaceutical products are complex systems containing different fractions of major constituents such as moisture, carbohydrates, proteins, fats, and several minor components like vitamins and minerals. During various stages of processing, the components transform & /or relocate either individually & /or through interaction with other components and ultimately influence the physical, functional, nutritional and sensory characteristics of the final product. In this regard, significant structural alterations of macromolecules such as proteins, carbohydrates, and fatty acids have been recognized during thermal processing techniques. Recently, attention has been focused on non-thermal methods of food processing techniques to minimize these changes (Hussain, Singh, Vatankhah, & Ramaswamy, 2017; Ramaswamy, Chen, & Rattan, 2015). High pressure processing (HPP) has been widely explored and has demonstrated a great potential for several food processing applications in recent years. The effect of HPP on different components of the food matrix is variable and need detailed studies to exploit their full potential (Hussain, Vatankhah, Singh, & Ramaswamy, 2016; Knorr, Heinz, & Buckow, 2006).

Among several aspects of physical and chemical properties, rheological/flow behavior of liquid food systems are critical due to their direct effect on processing conditions such as processing efficiency,

design, and evaluation of the process equipment as well as consumer acceptance and physical stability of the finished product. Our recent studies (Hussain et al., 2017, 2016) focussed on the interaction of locust bean gum and resistant starch revealed an enhancement of rheological properties such as apparent viscosity as well as storage and viscous moduli. Several previous studies have highlighted the effects of HPP on rheological, thermal and morphological properties of essential macromolecules associated with different food matrices (Hussain et al., 2016; Ramaswamy et al., 2015). The current study is directed in a similar fashion but to further explore the pressure-induced rheological changes on selected aqueous hydrocolloid gum dispersions at relatively much high concentration levels as used in specific applications such as film formation, food thickening for gel production, drug delivery vehicle, cosmetology, artificial skin, etc. (Muzzarelli et al., 2007; Patel & Goyal, 2015).

Chitosan (CH), an important derivative of crustacean chitins containing partially deacetylated N-acetyl glucosamine (Wang, 2015), is one of the most abundant polysaccharides and is recognized as a promising non-toxic biocompatible and bioactive compound. Regarding the molecular structure, CH has amino groups at C-2. Moreover, there are primary and secondary hydroxyl groups located at C-3 and C-6, respectively (Chen, Xie, Zhuang, Chen, & Jing, 2008). CH is known as an improving agent of other weak, and brittle polymers like gelatin and

* Corresponding author.

E-mail address: Hosahalli.ramaswamy@mcgill.ca (H.S. Ramaswamy).

this functional character of CH reveals the importance of its inter- and intra-molecular interactions (Lin, Lv, & Li, 2012). Moreover, dynamic low diffusion characters of CH have attracted many researchers and industry in areas such as surgical dressings, drug encapsulations, tissue engineering, 3D printing, water filtration, wastewater treatment, cosmetics and fruit coating (Rayner, Östbring, & Purhagen, 2016). Furthermore, being the only naturally cationic polysaccharide, CH has shown a high capability to bind to anionic surfaces. It is a copolymer of a random composition of two monosaccharides, N-acetyl- β -D-(1,4)-glucosamine and β -D-(1,4)-glucosamine generally in a 1:4 ratios in commercial materials. CH is commonly derived by deacetylation of chitin in aqueous sodium hydroxide solution. The complete deacetylation yields up to 98 percent product. The commercially available CH has a molecular weight between 3800 and 20,000 Da (Thomas, Susanne, & Maya, 2005).

Another high demand hydrocolloid gum which is widely accepted as a stabilizing agent in soft drinks formulae is gum Arabic (GA), (Lopez-Torrez, Nigen, Williams, Doco, & Sanchez, 2015). It is a natural exudate of Acacia tree which consists about 2 g/100 g proteinaceous weight fraction. Basically, this gum made of three groups of components including arabinogalactans (AG), arabinogalactan proteins (AGP), and glycoproteins (GP). The effect of HPP at remarkably high concentrations of GA dispersions was studied by Panteloglou, Bell, and Ma (2010) who reported that significant changes in both viscous (G'') and elastic (G') moduli occurred in 40 g/100 g GA dispersions after treatment at 800 MPa for 10 min. However, usage of such high concentrations in food processing is not common, and further studies are therefore required at lower concentrations.

Considering the need for robust research on introduction of pressure resistant thickening agents, as well as assessment of their rheological behaviors, the objective of this study was to assess the effect of industrially relevant HPP treatments on selected of GA and CH dispersions which are more in line with their applications, in mid-range concentrations, in food, drug, biomedical, and biomaterial industries. In this article, a novel methodology for evaluating thermo-viscoelasticity was established for elucidating the changes in rheology after HPP. Also, a comprehensive set of systematic steady flow and dynamic shear rheology experiment were carried out and compared to obtain an applied view of possible rheological behavior alterations.

2. Materials and methods

2.1. Sample preparation

Food grade purified and instant gum Arabic (GA) (Nexira, Rouen Cedex, France) powder was added to double distilled water (conductance: 18 V, Milli-Q, Millipore, Bedford, Massachusetts, USA) to make 6 g/100 g (GA-I), 15 g/100 g (GA-II), and 20 g/100 g (GA-III) dispersions. Aqueous CH dispersions of 2 g/100 g (CH-I) and 4 g/100 g (CH-II) were made by adding CH powder (Nex-xus, Montreal, Quebec, Canada) to 0.2 mol equi. L^{-1} aqueous solution of hydrochloric acid (Fisher Scientific, Fair Lawn, New Jersey, USA). All aqueous dispersions were prepared at room temperature (22 °C) and kept for 24 h to obtain full hydration of hydrocolloids. Sodium azide (0.02 g/100 g) aqueous solution was used as an antimicrobial agent to prevent spoilage during the hydration process. As the last step of preparation, the dispersions were centrifuged at 5000 \times g for 30 min, and the supernatant was used for further experiments.

2.2. High pressure processing (HPP)

HP treatment was given in an isostatic pressure unit with a cylindrical pressure chamber of 5 L volume (ACIP 6500/5/12VB-ACB Pressure Systems, Nantes, France) using water as the pressure transfer medium. A 30-mL aliquot of each sample was filled and sealed in polyethylene bags (Whirl Pak, Nasco, Fort Atkinson, Wisconsin, USA).

The samples were HP-treated individually for 30 min at 200, 400 and 600 MPa. The HP treatment operation included three stages: compression (pressure build-up), pressure hold and decompression (pressure release). Pressure build up time was approximately 1, 2 and 3 min, respectively with a pressurization rate of 200 MPa min^{-1} . The treatments were terminated after a rapid decompression (~ 5 s) to atmospheric pressure level.

The samples were introduced into the high pressure vessel at 15–18 °C so that temperatures of the medium and sample were around 25 °C after the pressure treatment. During the pressurization, it is expected that there will be an increase in sample temperature of about 3 °C/100 MPa due to the adiabatic heating. Because the vessel was jacketed and held at room temperature, an approximate average process temperature around the room temperature conditions was achieved by taking the sample and liquid at selected predetermined lower temperatures according to the targeted pressure level (Ramaswamy, Shao, & Zhu, 2010).

2.3. Rheological measurements

The rheology measurements were made out using a cone/plate AR2000 Rheometer (TA Instrument, New Castle, Delaware, USA) equipped with 60 mm, 1.59° solvent trap cone and attached computer and run by the software supplied by the company (Rheology Advantage Data Analysis Program, TA Instrument). A 2-min rest phase was designed for all the rheological tests. All measurements were done in duplicate.

2.3.1. Dynamic oscillation tests

The viscoelastic properties of samples were measured in the same rheometer in the angular frequency range of 1–25 ($rad\ s^{-1}$). An oscillation stress of 1 Pa (obtained from the linear region of stress sweep 0.1–100 Pa) was used to conduct the experiments at the constant temperature of 22 °C.

2.3.2. Flow rheology tests

The flow tests were carried out using the same TA instrument based on application of an upward-downward one cycle shear ramping in which the shear rate was increased linearly from 0.1 s^{-1} to 100 s^{-1} in 7 min and then decreased from 100 down to 0.1 s^{-1} in the next 7 min for a total cycle time of 14 min.

2.3.3. Applicability of Cox–Merz rule between steady shear and oscillation data

The Cox–Merz rule was used to investigate the relationship between the flow and oscillation results (Cox & Merz, 1958). The rule states that the steady shear viscosity (η) and magnitude of the complex viscosity (η^*) have a linear superimposition at the same magnitude of steady shear rate (ω) and oscillatory shear rate ($\dot{\gamma}$), (Eq. (1)).

$$|\eta^*|(\omega) = \eta(\dot{\gamma})|_{\omega=\dot{\gamma}} \quad (1)$$

The magnitude of complex viscosity was obtained using Eq. (2):

$$|\eta^*| = \sqrt{(\eta')^2 + (\eta'')^2} \quad (2)$$

where η' and η'' where viscose and elastic components of dynamic viscosity (Pa s).

2.3.4. Temperature sweep tests

The temperature sweep tests were performed to study the change in viscoelastic properties of test samples as a function of temperature. The complete sweep cycle included a heating stage to increase the temperature at a linear rate from room temperature (22 °C) to 70 °C in 10 min followed by a hold at this temperature for 10 min and finally a cooling stage from 70 °C to 22 °C in the next 10 min. So, each programming step lasted 10 min with a linear increase, steady hold, and a

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