Journal of Food Engineering xxx (2017) 1–8

Contents lists available at ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

Effect of high speed shear on the non-linear rheological properties of SPI/κ-carrageenan hybrid dispersion and fractal analysis

Chong-hao Bi [a, 1], Lu-tong Li [b, 1], Ying-dan Zhu [c], Yu-de Liu [a], Min Wu [c], Gang Li [a], Zhi-gang Huang [a, *]

[a] School of Materials Science and Mechanical Engineering, Beijing Technology and Business University, No.11 Fu Cheng Road Haidian District, Beijing 100048, China

[b] School of Mechanical, Aerospace and Civil Engineering, The University of Manchester, Pariser Building, Sackville Street, Manchester, Greater Manchester M13 9PL, UK

[c] College of Engineering, China Agricultural University, P.O. Box 50, 17 Qinghua Donglu, Beijing 100083, China

A R T I C L E I N F O

Article history:
Received 25 May 2017
Received in revised form
7 July 2017
Accepted 9 September 2017
Available online xxx

Keywords:
Soy protein isolate
κ-carrageenan
High speed shear
Rheological properties
Fractal analysis

A B S T R A C T

In this research, the effect of shear rate (rotation speed) of high speed shear treatment on the linear and non-linear rheological properties and microstructure of soy protein isolate (SPI)/κ-carrageenan (κ-KARA) hybrid dispersion was studied. It is found that high-speed shear treatment makes the hybrid system more homogeneous in a certain shear rate range (around 6000N) within which the mixing effect is stronger than the shearing effect on the mixed system. However, the elastic (G') and viscous properties (F') of the hybrid system increase when the shear rate is above a certain range (over 8000N). The effect of the destruction of the hybrid system structure becomes gradually greater (G' decrease) than the effect of the mixing, resulting in a decrease of the storage modulus and apparent viscosity. The result of the LAOS test shows that the shear rate only affects the magnitude of the $I_{3/1}$ peak and does not have a significant effect ($p < 0.05$) on the peak strain value. In other words, the shear rate only has a significant effect on the influence of higher harmonics. The trend of the Lissajous curve shows that the elasticity of the hybrid system dispersion increases with increasing shear rate. The microstructure and fractal analysis shows that with the increase in the high speed shear rate, the fractal dimension of the mixed system solution increases significantly ($p < 0.05$), indicating that the degree of chaos of the hybrid system dispersion increases with increasing shear rate.

1. Introduction

The interaction between protein and polysaccharide has always been a hot topic in food research (Franco, 1997; Baeza, and Carp, 2002) because the interaction between these two types of molecules has an important impact on food quality (Corredig et al., 2011; Perez et al., 2009). Therefore, the interaction between the protein and polysaccharide system has important significance and application value for the guidance of the design and improvement of food industry processes (Foegeding et al., 2010).

The functional properties of food proteins such as solubility, gelation, emulsification, foaming, interfacial activity and conformational stability are related to the interaction of proteins with polysaccharides and other substances such as water, fats, emulsifiers and metal ions in the system. The competitive interaction of these substances determines the relationship between the performance and structure of the food. Tolstoguzov team found the thermodynamic incompatibility of the 100 types of studied protein-polysaccharide-water three-phase systems (Vya and Tolstoguzov, 1997; Tolstoguzov, 1993, 2002). It was also concluded that the thermocompatibility of the protein-polysaccharide mixed system solution was prevalent (Doublier et al., 2000). Miquelim et al. (2010) found an interfacial interaction between ovalbumin and nonadsorbed polysaccharides at pH 7.5. The results showed that carrageenan had no significant effect on the interfacial tension, while xanthan gum and guar gum increased the surface tension of the system. However, the increase in the modulus of elasticity of the guar gum under different

* Corresponding author.
E-mail address: huangzg@btbu.edu.cn (Z.-g. Huang).

[1] These authors contributed equally to this work and should be considered co-first authors.

http://dx.doi.org/10.1016/j.jfoodeng.2017.09.007
0260-8774/© 2017 Published by Elsevier Ltd.

conditions also showed a reduced effect.

Previous studies have shown that high speed shear treatment with a variety of appropriate surfactants can effectively control or reduce the size of food and cosmetic emulsion particles (Salager et al., 2004). Different homogeneous treatment methods and different surfactants have a certain effect on the size and emulsion stability of the emulsion particles (Fradette et al., 2007). Floury et al. (2000) introduced the effect of high-pressure homogenization on the physical properties of dairy products. It was found that there is a certain functional relationship between the protein and the polysaccharide, indicating that the particle size decreases with the increase of the homogeneous pressure; the higher fineness in turn leads to a more homogeneous distribution (Pandolfe, 1981). Hernandez and Harte (2008) elucidated the effect of high-pressure homogenization and high-pressure homogenization-heat treatment on acid-induced gelation of skim milk, and found that the homogenized acid-induced gel shows better stability. To the best of our knowledge, there has been no research specifically focusing on the effect of high-speed shear treatment on the rheological properties and microstructure of the SPI/κ-carrageenan hybrid dispersion. In this paper, the effects of the shear rate on the rheological properties and microstructure of SPI/κ-KARA hybrid system dispersion were studied.

2. Materials and method

2.1. Materials

Commercial soy protein isolate (protein content >90%) was obtained from Messenger Biotechnology Co., Ltd. as a gift. κ-carrageenan (BR grade) was bought from Source Leaf Biotechnology Co., Ltd. Rhodamine B (AR grade) was purchased from Beijing Yinghai Fine Chemical Co., Ltd.

2.2. Equipment

High-speed shear homogenizer (Ultra-Turrax T25) from *IKA Group (Germany).*

Rheometer (AR2000ex) from *American TA Instruments (American).*

Confocal laser scanning microscopy (LEICA TCS SP5 II) from *LEICA Instruments (Germany).*

2.3. Method

2.3.1. Sample preparation

Soy protein stock dispersion: 56 g of soy protein was added to 700 g of deionized water. The solution was stirred with an electric stirrer at 20 °C for 3 h. A soy protein stock solution of 8% concentration was obtained.

Carrageenan stock solution: 0.32 g of carrageenan powder was added to 200 g of deionized water. The mixture was then stirred at 20 °C for 2 h with a magnetic stirrer for even stirring. A carrageenan stock solution with the concentration of 0.16% was obtained.

In the final mixed solution, the mass ratio of the protein solution to the carrageenan solution was 3: 1, so that the concentration of the soy protein solution in the final mixed solution was 6% and the concentration of the carrageenan solution was 0.04%.

Six aliquots (15 g each) were taken from 8% soy protein stock dispersion and 6 aliquots (5 g each) were removed from 0.16% carrageenan solution. These soy protein and carrageenan solutions were divided into 5 experimental samples and 1 control sample. The five experimental samples were subjected to 6000 N, 8000 N, 10000 N, 12000 N and 14000 N shear rate of high speed shear, respectively, and the remaining sample that was not subject to the high speed shear treatment was used as the control sample.

Before experiment, for complete hydration, the 6 samples of the mixed solution were allowed to stand in a refrigerator at 4 °C for 12 h.

Prior to performing a confocal laser scanning microscopy experiment (CLSM), the samples for CLSM observation were prepared as follows:

The SPI/κ-KARA hybrid system dispersion was stained with Rhodamine B aqueous solution; 1 ml of the Rhodamine B solution with a mass fraction of 0.01% was added to 10 ml of the sample. The dyed samples were then placed in a single-sided recessed slide and sealed with nail polish around the coverslip. The stained and sealed samples were covered with aluminium foil to maintain a dark environment in order to avoid fluorescence quenching.

2.3.2. Rheological test

All tests for rheological properties were performed using a AR2000ex rheometer. The AR2000ex instrument is a stress-controlled rheometer equipped with a sensor balanced control that can measure the torque in the range from 0.03 μN mm to 200 mNmm. At the same time, the high-resolution motor of the rheometer can generate angular frequencies from 7.5×10^{-7} rad/s to 628 rad/s. The Peltier system connected to the water bath at the bottom of the rheometer controls the temperature of the sample.

2.3.2.1. Frequency sweep test. The frequency sweep tests were conducted at 0.1% oscillating strain, temperature of 20 °C and the applied frequency is from 0.1 Hz to 1 Hz. The storage (G') and loss (G'') moduli (data not shown) of the SPI/κ-carrageenan hybrid system were measured and recorded.

The frequency dependence of the storage (G') and loss (G'') moduli can be fitted by the power law model:

$$G' = K' \cdot f^{n'} \tag{1}$$

$$G'' = K'' \cdot f^{n'} \tag{2}$$

The parameter K', K'' are power law constants in units of Pa • s^n/rad, and n', n'' are the dimensionless frequency indexes. These two parameters reflect the viscoelasticity of the sample (Hagiwara et al., 1997).

2.3.2.2. Steady-state shear test. The steady-state shear test is a static rheological experiment. The experimental temperature is controlled at 20 °C, and the shear rate rises from 0.1 to 100 s^{-1}, and the data recording frequency is 10 points per logarithmic decade. This experiment was used to determine the effect of the shear rate on the apparent viscosity of the SPI/κ-KARA hybrid dispersion. The curve can be fitted by the model function:

$$\eta = F' \cdot \gamma^{m'} \tag{3}$$

The parameter F' is viscosity index, and m' is shear thinning/thickening index, which indicates the extent of non-Newton viscosity phenomenon.

2.3.3. LAOS test

The strain sweep is applied to the oscillating model where the strain varies from 0.01% to 1000% and the fixed angular frequency is 6.283 rad/s. Logarithmic points with each logarithmic interval of 10 log points were used and the test temperature was set at 20 °C. The changes of the storage modulus (G') and the loss modulus (G'') curves of the SPI/κ-KARA hybrid system dispersion in the case of increasing strain were examined.

FT-Rheology is the most efficient method for the conversion of a

Please cite this article in press as: Bi, C.-h., et al., Effect of high speed shear on the non-linear rheological properties of SPI/κ-carrageenan hybrid dispersion and fractal analysis, Journal of Food Engineering (2017), http://dx.doi.org/10.1016/j.jfoodeng.2017.09.007