



Analysis of deacetylated konjac glucomannan and xanthan gum phase separation by film forming



Weiping Jin ^{a,b,1}, Rukun Song ^{a,1}, Wei Xu ^{a,b}, Yuntao Wang ^{a,b}, Jing Li ^{a,b},
Bakht Ramin Shah ^{a,b}, Yan Li ^{a,b}, Bin Li ^{a,b,*}

^a College of Food Science and Technology, Huazhong Agricultural University, Wuhan, Hubei, China

^b Key Laboratory of Environment Correlative Dietology (Huazhong Agricultural University), Ministry of Education, China

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ABSTRACT

In this study, the influences of deacetylation degree (DD) on phase separation of konjac glucomannan/xanthan gum blended systems were investigated through film forming properties. The transparency, mechanical properties and structure were characterized by UV–Vis, electronic tensile tester and FTIR (Fourier Transform Infrared) spectroscopy respectively. When DD of deacetylated konjac glucomannan (DKGM) was 52.34%, the mechanical properties, transparency, moisture absorption of blended films were the highest. Subsequently, the hydrogen bonds between DKGM and xanthan gum were enhanced and the surface of the resultant film was smooth and flat. Besides, the phase separation particles were analyzed by X-Ray diffraction, which were similar size with DKGM self-aggregations. Thermogravimetry (TG) indicates high DKGM blended film possessed higher stability arising from DKGM self-aggregations. In conclusion, the results provided a method of studying phase separation between two macromolecules by film forming.

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1. Introduction

Biodegradable and edible films have been extensively studied and applied in the food packaging industry (Deng, Li, et al., 2011; Deng, Wang, et al., 2011; B Li, Kennedy, Jiang, & Xie, 2006; Ye, Kennedy, Li, & Xie, 2006). There is becoming a green and eco-friendly trend for composited films made by biopolymers and their blends, such as polysaccharides (Du, Yang, Ye, & Li, 2013). Therefore, the miscibility and interactions between polysaccharides play a leading role in exploiting the new sources of biodegradable and edible films.

Konjac glucomannan (KGM), due to its high water-absorbing capacity and good film forming ability, has been widely used in food drug release matrices and biodegradable films (Chua, Baldwin, Hocking, & Chan, 2010; Liang, Huang, Liu, & Yam, 2009; Xiangdong & Lixin, 2003). KGM is one of the neutral polysaccharides composed of a linear chain of β -1, 4-linked D-glucose and D-

mannose residues in a molar ratio of 1:1.6 (Takigami, 2000). The present evidences have demonstrated that KGM and its derivatives have potential applications in personal healthcare such as in controlling obesity, improving intestinal activity and lowering blood cholesterol (Bin Li, Xia, Wang, & Xie, 2005; Yeh, Lin, & Chen, 2010). Xanthan gum, an exocellular polysaccharide produced by bacterium, has been widely used in food and pharmaceutical industries (Kim, Takemasa, & Nishinari, 2006). Xanthan molecules are comprised of a main chain of β -1, 4-linked D-glucose residues, which is the same as cellulose, with a trisaccharidic branch (Fitzpatrick, Meadows, Ratcliffe, & Williams, 2013). The side branches, which consist of α -D-mannose, β -D-glucuronic acid, and β -D-mannose, attach to every second glucose residue of the main chain. The secondary structure of xanthan is a 5-fold helix which is established as a rigid, ordered structure (Agoub, Smith, Giannouli, Richardson, & Morris, 2007). A synergistic interaction could happen between KGM and xanthan, inducing the formation of a physical hydrogel, which is attributed to the originating from the interaction between them (Fitzsimons, Tobin, & Morris, 2008; Kim et al., 2006; Paradossi, Chiessi, Barbiroli, & Fessas, 2002). The detailed mechanism and the main driving force are still unclear, although similar classes of synergistic gels have been studied intensively (Fitzsimons et al., 2008; Goycoolea, Richardson, Morris, & Gidley, 1995; Paradossi et al., 2002).

* Corresponding author. College of Food Science and technology, Huazhong Agricultural University, Wuhan, Hubei, China. Tel.: +86 27 63730040; fax: +86 27 87282966.

E-mail address: libinfood@mail.hzau.edu.cn (B. Li).

¹ Co-first author of this paper.

When the total concentration of the macromolecules exceeds a critical value, phase separation will generally take place among them. The limited thermodynamic compatibility among macromolecules is a fundamental phenomenon that has been demonstrated in food systems (Ako, Durand, & Nicolai, 2011; Zhang, Royall, Faers, & Bartlett, 2013). It is acknowledged that excluded volume determines the phase separation, explained by a type of stereospecific blockade resulting from self-aggregation or the weakness of interaction between different molecules (Lorén et al., 2001; Tuinier, Dhont, & De Kruif, 2000). In our previous studies, the deacetylation of KGM induced by alkaline treatment has been demonstrated to increase self-aggregation among KGM molecules, caused by transforming molecular chains from semi-flexible linear to strong self-crimping elastic microspheres (Chen, Li, & Li, 2011). Therefore, it is crucial to take the effect of deacetylation into consideration for the formation of KGM and xanthan blended films.

In the present study, the blended films were formed by deacetylated KGM (DKGM) and xanthan. And meanwhile, the phase separation behavior between them was investigated. The influence of degree of deacetylation (DD) on the films' formation, mechanical properties and interactions was evaluated by transparency, tensile strength, breaking elongation and FTIR. The microstructure of films was observed by SEM and XRD. This study provides facile and detailed information on using a film formation method to evaluate phase separation.

2. Materials and methods

2.1. Materials

Konjac glucomannan (KGM) was kindly provided by Hubei Konson Konjac Gum Co., Ltd (Wuhan, China). Xanthan was purchased from Shijimei Food additives Co., Ltd (Henan, China). Sodium carbonate (Na_2CO_3) and glycerol were purchased from Sinopharm Chemical Reagent Company. They were all of A.R. grade and used without further purification.

2.2. Preparation of blended films

0.5% (w/w) xanthan solution was prepared by dissolving xanthan powder into distilled water and stirring for 2 h. DKGM solution was obtained by dissolving KGM powder into Na_2CO_3 solution for 2 h and heating at 80 °C for 30 min. Then, xanthan solution was mixed with the resultant DKGM solution with equal volume (final volume 1500 mL), accompanied with 3.5 mL glycerol. The mixture was heated at 80 °C for 30 min. Before casting into films, the mixture was stored for 3 h. The casting film was dried at 50 °C for 20 h. The films made of xanthan gum (XG) with DKGM was marked as a*, b*, c*, d* and e*, successively representing different DKGM deacetylation degree. Besides, the sole DKGM films were marked accordingly by a, b, c, d and e, respectively.

2.3. Determination of deacetylation degree (DD)

1.00 g of DKGM powder and 50 mL of 50% aqueous alcohol were mixed and kept in a 50 °C water bath for 1 h prior to the determination of DD. Then, 1 mL of 0.5 mol/L KOH solutions was added and the mixture was stirred for 48 h for the reaction. Finally, phenolphthalein was added as the indicator and the excess KOH was back titrated with 0.02 mol/L HCl. The titration process for each sample was repeated in triplicate within 2 h. The acetyl content in KGM (N_{acetyl}) was obtained by repeating the procedure described above with KGM powder. The degree of

deacetylation (%) in the sample was calculated by the following formula.

$$\text{DD}(\%) = \left[N_{\text{acetyl}} - (V_a - V_b)C_{\text{HCl}} \times 10^{-3} \right] / N_{\text{acetyl}},$$

where V_a and V_b are the volume of HCl consumed for the blank and samples in liters, respectively. And C_{HCl} is the normality of the HCl, N_{acetyl} is the acetyl content of KGM (Liu et al., 2010). The deacetylation degrees of DKGM were illustrated in Table 1.

2.4. Characterization of blended films

2.4.1. Transparency

Films were divided into narrow strips (size: 10 × 20 mm) and fixed onto the side of cuvette. The transparency (T) of the films was measured at 490 nm using a UV-1100 spectrophotometer (MAPADA, Shanghai, China).

2.4.2. Moisture absorption capability

The samples used were thin square strips with dimension of 40 mm × 40 mm × 0.1 mm. They were vacuum-dried at 50 °C overnight and then kept at 0% relative humidity (RH) (P_2O_5) for one week. After weighing, they were conditioned at 25 °C in a desiccator of 98% RH ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ saturated solution) for one week to ensure the equilibrium of the moisture before testing. The moisture absorption of the samples was calculated as follows:

$$W = [(W_1 - W_0)/W_0] \times 100\%$$

where W is the moisture absorption percentage, and W_0 and W_1 are the mass of the films before exposure to 98% RH and after equilibrium, respectively. An average value of five replicates for each sample was taken.

2.4.3. Mechanical properties

The tensile strength (TS) and breaking elongation (ELO) of the films were measured on an electronic tensile tester CMT-6104 (Shenzhen Sans Test Machine Co. Ltd., China) with a tensile rate of 250 mm/min according to the Chinese standard method (GB/T4456-96).

2.4.4. Fourier Transform Infrared spectroscopy (FT-IR) analysis

Films were cut into small pieces, blended with potassium bromide (KBr) and then laminated. The IR spectra were recorded with a Nicolet Nexus 470 FT-IR spectrometer (Thermo Nicolet, USA).

2.4.5. Scanning electron microscopy (SEM) observation

Morphology of blended films surface was investigated by SEM (JSM-6390LV, JEOL, Japan). Before observation, the dried samples were coated with about 20 nm gold–palladium under argon atmosphere using a gold sputter module in a high vacuum evaporator. The sputtered time was about 30 s and the accelerating voltage was 15 kV. Images with magnification of 5000 were recorded.

Table 1
The nomination and DD of the films.

Sample	DD (%)
a (a*)	0.00
b (b*)	38.45
c (c*)	52.34
d (d*)	70.86
e (e*)	86.57

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