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Structural characterization and properties of konjac glucomannan/curdlan blend films

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ABSTRACT

A series of novel edible blend films of konjac glucomannan (KGM) and curdlan were prepared by a solvent-casting technique with different blending ratios of the two polymers. The Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), differential scanning calorimetry (DSC), scanning electron microscopy (SEM), etc. were used to characterize the change of structure and properties of blend films. The results showed that the strong intermolecular hydrogen bonds took place between KGM and curdlan. And the interaction of the blend film was much greater than that of the others when the KGM content in the blend films was around 70 wt% (KC7), resulting in excellent miscibility. The conclusion of the electron tensile testing analysis indicated that the blend film KC7 showed the maximum tensile strength (42.93 ± 1.92 MPa). In addition, the blend films displayed excellent moisture barrier properties, which had a potential application in the food field.

1. Introduction

Recently, biopolymers have addressed much attention in research due to the increasing demand for environment protection, as well as rising costs and supply of fossil fuels. It has been predicted that biopolymers, as one of primary importance resources of renewable energy, will provide the major raw material for the progress of industry in the future (Crini, 2005; Persin et al., 2011). Among natural biopolymers, konjac glucomannan (KGM) is a good candidate for packaging materials. It had attracted great interest since it has excellent film-forming ability, good biocompatibility and biodegradability (Cheng, Abd Karim, & Seow, 2008; Pang, Lin, Zhang, Tian, & Sun, 2003). KGM is one of the high molecular weight water-soluble non-ionic natural polysaccharides isolated from tubers of Amorphophallus konjac K. Koch. It is a β-1,4 linked polysaccharide composed of a D-glucose and D-mannoses backbone lightly branched, with branches through β-1,6-glucosyl units (Chua, Baldwin, Hocking, & Chan, 2010; Jin, Zhang, Yin, & Nishinari, 2006; Jian, Zeng, Xiong, & Pang, 2011; Katsuraya et al., 2003; Li, Kennedy, Peng, Yie, & Xie, 2006). There may be certain short side branches at the C-3 position of the mannoses and acetyl groups randomly present at the C-6 position of a sugar unit. The acetyl groups frequently range from 1 per 9 sugar units to 1 per 20 sugar units (Chua et al., 2010). It has been widely applied in the field of food, pharmaceuticals and chemical engineering (Alonso-Sande, Teijeiro-Osorio, Remunan-Lopez, & Alonso, 2009). However, as a good film-forming material candidate for food and fruit preservation, KGM has the common disadvantages of natural polymers, low mechanical properties and poor antimicrobial activity (Chen, Liu, Chen, Chen, & Chang, 2008). In our earlier work, we found that blending is a convenient and effective method to improve the physicochemical properties and function of the films (Pang et al., 2003). Thus, in this work, the major task is trying to select other natural polymers as the partner with KGM, and investigate the physicochemical properties and the function of blend films.

Curdlan is an extracellular polysaccharide produced by bacteria, such as *Alcaligenes faecalis* var. Myxogenes and its linear structure is composed of (1–3)-β-D-glucosidic linkages (Grandpierre, Janssen, Laroche, Michaud, & Warrand, 2008; Jin et al., 2006; Renn, 1997). Since its first found by Harada in 1966, it has received great attention in food and non-food research due to its unique physicochemical properties (Wong, Ngiam, Kasapis, & Huang, 2010). This neutral polysaccharide is insoluble in water and alcohol, and its aqueous dispersion is capable of forming a gel under heating or alkaline (Funami, Funami, Yada, & Nakao, 2000; Tada, Matsumoto, & Masuda, 1999). It forms two types of gels under heating, named low-set gel and high-set gel. The low-set gel formed when the aqueous dispersion is heated to a certain degree ranging from 55 to 60 °C, which is thermo-reversible. However, it can form high-set gel when the aqueous dispersion is heated above 80 °C, which is

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thermo-irreversible and stable at low temperatures such as freezing and also at high temperatures as in retorting (Nakao et al., 1991; Nishinari & Zhang, 2004). In addition, curdlan also forms a gel when its alkaline solution is neutralized with acids under a static condition (Tada et al., 1999). Due to its unique functions including its thermal gelling properties, curdlan has been widely used in various foods such as bean curd, noodles, jellies and low-fat meat products (Nakao et al., 1991). Similar to other microbial polysaccharides, it has been reported that curdlan has bioactivity such as anti-tumor activity and anti-HIV activity, etc. (Gao et al., 2010; Lee et al., 2001).

So far, KGM has been reported to interact synergistically with individual chitosan, xanthan, gellan, k-carrageenan and starch (Li, Kennedy, Peng, et al., 2006; Xu, Li, Kennedy, Xie, & Huang, 2007; Yu, Jiang, Zou, Duan, & Xiong, 2009). However, no paper has been published on the preparation and application of KGM and curdlan as edible films. The objective of the present work was to prepare KGM/curdlan blend films with potential applications as edible films and biodegradable food packaging materials. The structure, miscibility and mechanical properties of blend films were investigated by FT-IR, XRD, DSC, SEM and electron tensile test etc. Furthermore, the relationship between the structure and the properties of the KGM/curdlan films was discussed as well.

2. Materials and methods

2.1. Materials

Konjac glucomannan was purchased from San Ai Konjac Food Co. Ltd. (Sichuan, China), and purified by the method described in our previous work (Jian, Yao, Wang, Guan, & Pang, 2010). Curdlan was provided by Takeda-Kirin Foods Corporation (Tokyo, Japan), which was used without further purification. Other analytical grade chemical reagents were purchased from Sinopharm Group Chemical Reagent Co. Ltd. (China).

2.2. Film preparation

Konjac flour was first dissolved in distilled water to prepare a 1% (w/w) solution, and curdlan power was dissolved in pH = 8 aqueous solution to ensure the final concentration of 1% (w/w). Then two types of solutions were conditioned in a water bath at 90 °C with a constant stirring for 3 h, respectively. Two types of solutions were mixed together at different weight ratios, and the final volumes of each were adjusted to 400 mL by the addition of 1.5 mL glycerin. The mixtures were continuously stirred at 90 °C for 3 h, poured onto glass plates with a 19 cm × 19 cm film-forming area, and then evaporated in an oven at 60 °C for 8 h to obtain dry films.

The films of different ratios above were coded as KGM, KC1, KC2, KC3, KC4, KC5, KC6, KC7, KC8, KC9 and curdlan, respectively. Before various characterizations, the resulting films were kept in a conditioning desiccator of 50 ± 5% relative humidity (RH) for more than one week at room temperature to ensure the equilibrium of the water in the films.

2.3. Fourier transform infrared spectroscopy

Fourier transform infrared spectroscopy (FT-IR) spectra of films in the mode of attenuated total reflection were recorded with a Nicolet (Madison, WI, USA) 170SX Fourier transform infrared spectrometer in the wavelength range of 4000–400 cm^{-1}.

2.4. X-ray diffractometry

X-ray diffraction (XRD) patterns of the films were carried out with Cu Kα radiation using a diffractometer (Rigaku, Corp., Tokyo, Japan). The diffractometer was operated at a voltage of 36 kV and current of 20 mA. The scanning rate was 4°/min in the angular range of 10–60° (2 h). The degree of crystallinity of the films was calculated by the equation as follows:

$$\text{Crystallinity}\quad(\%) = \frac{A_c}{A_c + A_a} \times 100\%$$

where A_c and A_a are the areas of crystalline and non-crystalline regions, respectively. To avoid the influence of relative humidity on relative crystallinity, blend films were placed in desiccators and conditioned in the atmosphere of 95% RH for one week before XRD testing.

2.5. Scanning electron microscopy

The blend films were fixed on a cylindrical microscope stub covered with carbon strip and coated with 100–200 Å thickness of gold, followed by observation under a scanning electron microscope (XL30, Philips, Holand).

2.6. Differential scanning calorimetry analyses

Differential scanning calorimetry (DSC) analyses of films were carried under a nitrogen atmosphere with a rate of 25 mL/min by a DSC Q10 (TA, America) from 20 to 400 °C at a heating rate of 20 °C/min.

2.7. Water vapor permeability (WVP) determination

The WVP of films was determined gravimetrically at 25 ± 1 °C according to China National Standard GB1037-88 (1998). The WVP ($\text{g m}^{-1}\,\text{s}^{-1}\,\text{Pa}^{-1}$) was calculated according to the following equation:

$$\text{WVP} = \frac{\Delta md}{At\,\Delta P}$$

where Δm (g) is the amount of water vapor movement across the film (weight gain); d (mm) is the film thickness; A (mm^2) is the area of the exposed film; ΔP (Pa) is the actual difference in partial water vapor pressure between the two sides of the film specimen; t (s) is the time during which a stable weight gain occurred. At least three replicates of each film type were tested for WVP.

2.8. Moisture uptake measurement

The moisture uptake of the films studies was measured at 30 °C according to the procedure described by Chen et al. (2008). The films used were thin rectangular strips with dimension of 50 mm × 10 mm. They were vacuum-dried at 80 °C overnight to ensure the equilibrium of the moisture before measurement. The moisture uptake (MU) of the samples was calculated according to the following equation:

$$\text{MU} = \frac{W_1 - W_0}{W_0} \times 100\%$$

where W_0 and W_1 were the weight of the sample before exposure to 98% RH and after equilibrium, respectively. At least three replicates of each film type were taken for MU.

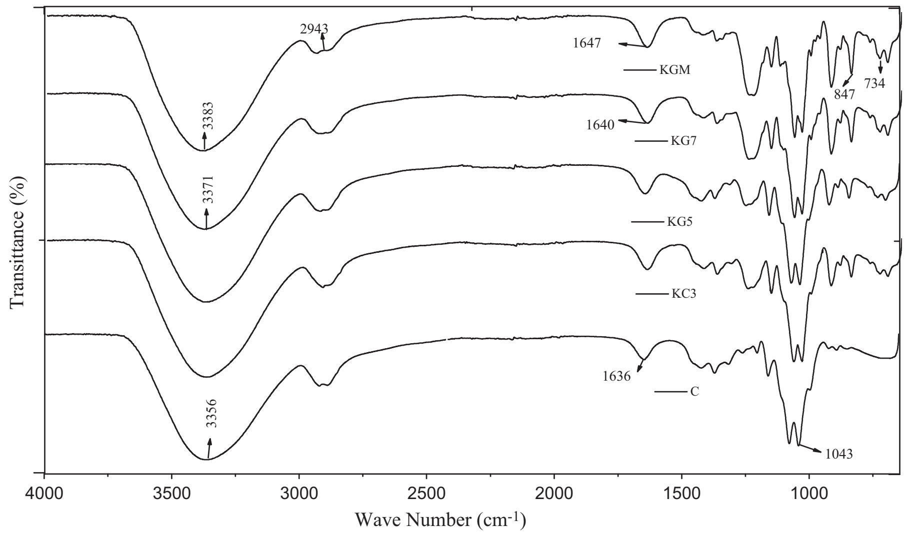

Fig. 1. FTIR spectra of blend films.

2.9. *Mechanical properties*

The tensile strength and elongation at break of the films were determined by using a Micro-electronics Universal Testing Instrument Model Sans 6500 (Shenzhen Sans Test Machine Co. Ltd., Shenzhen, China) according to the Chinese standard method (GB 13022-91). The tensile rate was 250 mm/min, and at least three replicates of each film type were taken.

3. Results and discussion

3.1. *Structure and miscibility*

3.1.1. *FT-IR analysis*

The FT-IR spectra on pure konjac glucomannan, pure curdlan, and the blend films in a wave number range of 4000–400^{-1} were shown in Fig. 1. As what was shown in the figure, the main chain elements of konjac glucomannan and curdlan were similar to each other. In the neat KGM film, the stretching and bending vibration of the hydrogen bonding groups and C—H of methyl of KGM occurred at 3383 cm^{-1} and 2943 cm^{-1}, respectively. The broad peak at 1647 cm^{-1} in the spectrum of native KGM was assigned to the existence of water, which seemed to be unable to be completely eliminated from the film. A similar phenomenon was found in some other polysaccharides, such as curdlan and chitosan (Jin et al., 2006; Sun et al., 2011). And the characteristic peaks bands of mannose in KGM appeared at 847 cm^{-1} and 734 cm^{-1}. In the FT-IR spectrum of the neat curdlan film, the absorption peaks of hydrogen bonding hydroxyl groups of curdlan, due to the complex stretch vibration associated with free inter- and intra-molecular bonding hydroxyl groups, occurred at 3356 cm^{-1} and 1636 cm^{-1}, respectively (Jin et al., 2006). The absorption band at 1043 cm^{-1} was due to the stretching vibration of C—O.

As far as the position and intensity of the various bands of the blend films were concerned, the major bands centered at 3400–3200 cm^{-1} showed significant changes. They broadened and shifted to a higher wave number as the KGM content increased. For the blend films, the strong absorption band broadened at 3383 cm^{-1} and shifted to 3376 cm^{-1}. It indicated the increase of intermolecular hydrogen bonds between KGM and curdlan, especially the KGM content at 70 wt%. Furthermore, the peaks at 1646 cm^{-1} shifted to a lower wave number with the decrease of KGM content, suggesting that new hydrogen bonds between KGM and curdlan molecules were formed in the blend films. Based on the results of FT-IR, it can be concluded that the miscibility between KGM and curdlan was due to the formation of synergistic interaction and intermolecular hydrogen bonds during the blending and film-forming process.

3.1.2. *X-ray diffraction analysis*

The X-ray diffraction (XRD) patterns of blend films were shown in Fig. 2. The patterns of pure KGM film displayed a broad peak at $2\theta = 21.4°$ with a small and weak peak appeared at 31.65°, which indicated that the neat KGM is in the amorphous phase, a result consistent with what was found by Li, who had investigated the fine structure of KGM (Li et al., 2011). The pattern of curdlan showed a broad peak and a stronger peak at $2\theta = 20.4°$ with a small and weak peak appeared at 28.08°. If KGM and other polymers had good compatibility, the crystalline of each polymer would be larger than in the blend films (Xiao, Liu, Lu, & Zhang, 2001). The crystallinities of KGM, KC-7, KC-5, KC-3 and curdlan based on X-ray diffraction were 31.42%, 29.72%, 32.68%, 45.88%, 35.88%, respectively. Compared the patterns of KC7, KC5, KC3 with that of curdlan, it was obvious that the diffraction peak became weaker with the reduction of curdlan down to 30 wt%. It can be expected that the intermolecular interactions between KGM and curdlan made the curdlan molecules

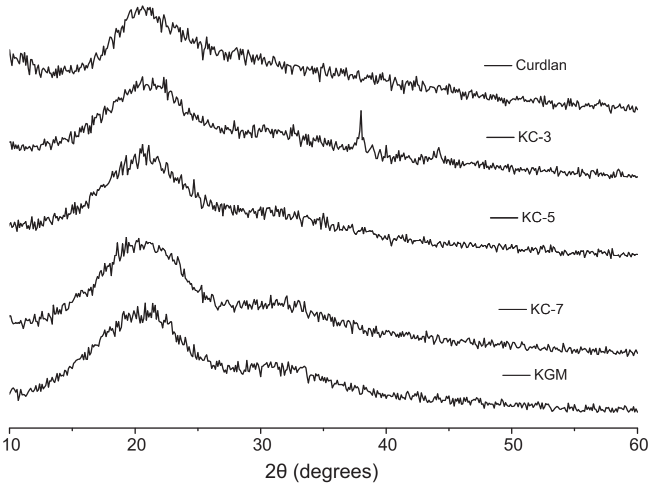

Fig. 2. XRD curves of blend films.

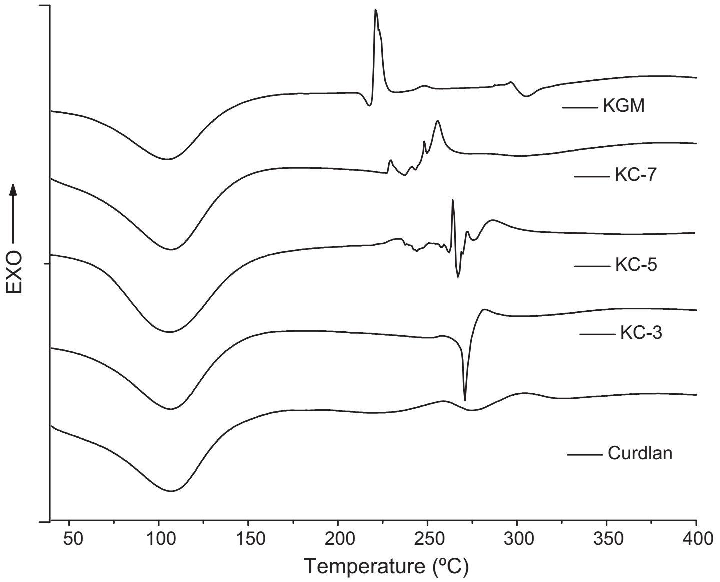

Fig. 3. DSC curves of blend films.

disperse into the KGM matrix and destroyed the original crystalline domains of KGM and curdlan, which indicated this two polymers have excellent blend miscibility. The result supported the FI-IR analysis conclusion that excellent miscibility existed between KGM and curdlan when the KGM content was more than 30 wt% and the best miscibility appeared when the KGM content was 70 wt%.

3.1.3. DSC analysis

The thermal behavior of the pure and blend films was investigated by means of DSC measurements (Fig. 3). As for the DSC curve of the pure KGM film, the endothermic peaks around 58.5–140 °C was due to the loss of small amount moisture. An exothermic peak appearing at about 220.9 °C was attributed to the thermal degradation of KGM. The DSC curve of curdlan showed two endothermic peaks at about 107.0 and 311.1 °C respectively, which was attributed to the disordering of the structure or swelling of curdlan particles accompanying the breakup of hydrogen bonds (Jin et al., 2006). The DSC curves of the blend films of KC7 and KC5 showed an exothermic peak at 275.7 °C and 255.3 °C. It indicated that KC7 has the highest therm-stability of the blend films, which was caused by the good miscibility. It also can be seen that with the decrease of the KGM content in the blend, the thermal degradation peak shifted to a lower temperature and the enthalpy decreased at the same time, which indicated that their crystalline domains and hydrogen bonding interaction enhanced the thermal stability of the films (Xu et al., 2007). It was also found the DSC curves of KC5 and that KC3 displayed obvious endothermic peaks around 260.3–280.2 °C, which were attributable to the melting of the crystal phase in the blend films. These results of DSC were in agreement with results of FI-IR and X-ray.

3.1.4. Morphology analysis

Morphological properties of native and blend films were observed by scanning electron microscopy (Fig. 4). It displayed that the blend film KC7 exhibited a much smoother and more homogeneous surface morphology than other films, suggesting high miscibility and blend homogeneity between KGM and curdlan at the composite ratio of KGM to curdlan to 7:3 by weight. This result supports the conclusion of miscibility between KGM and curdlan in the films and was in good agreement with those from FT-IR, XRD, and DSC.

3.2. Physical properties

3.2.1. Mechanical properties of blend films

The study of mechanical properties was of primary importance for determining the various kinds of performance of materials, especially that of film materials. And it is known that the interaction among polymers will influence the mechanical properties of the blended polymer (Fan, Zhu, Zheng, Xu, & Zhang, 2007).

The dependence of the tensile strength and breaking elongation on the KGM content for blend films was shown in Fig. 5. The tensile strength of the neat KGM film was 40.09 ± 1.92 MPa and much higher than that of curdlan film (4.53 ± 1.89 MPa). For the blend films, the tensile strength of the blend films improved with the increase of KGM content and reached a maximum point at around 70 wt% KGM content, achieving 42.93 ± 1.75 MPa. The breaking elongation of the blend films increased with the increase of the curdlan content and the pure curdlan film had the maximum point 48.33 ± 1.71%. These results indicated that the curdlan does not have a good film-forming ability, even when curdlan formed 'high-set' thermo-irreversible gels at temperature 90 °C (Sun et al., 2011). The considerable enhancement in the tensile strength of the blend films might be attributed to such factors as the hydrogen-bonding interactions between KGM and curdlan molecule, the glycerin as a bridging or cross-linking agent to improve the intermolecular interactions between two polymers, the increase of crystalline domains suppressing the slipping behavior of polymer molecular chains (Li, Kennedy, Jiang, & Xie, 2006). It can be seen that the addition of the KGM to curdlan was an effective method to enhance the mechanical properties films.

3.2.2. Water vapor permeability (WVP) of blend films

KGM films have been reported to be ineffective moisture barriers due to their hydrophilic nature (Xiao et al., 2001). Therefore, to improve the water-resistance properties of a KGM film, water-insoluble curdlan polysaccharide, which has the potential to improve material's water barrier capability, was implemented in this work. However, the result, as shown in Fig. 6, was dissimilar to the expected one, the addition of curdlan to KGM. The WVP of blend films showed a decreasing trend and got to its minimum value when the curdlan content was 30–40 wt%, then with the ulterior increase of curdlan content the WVR increased slightly, and the

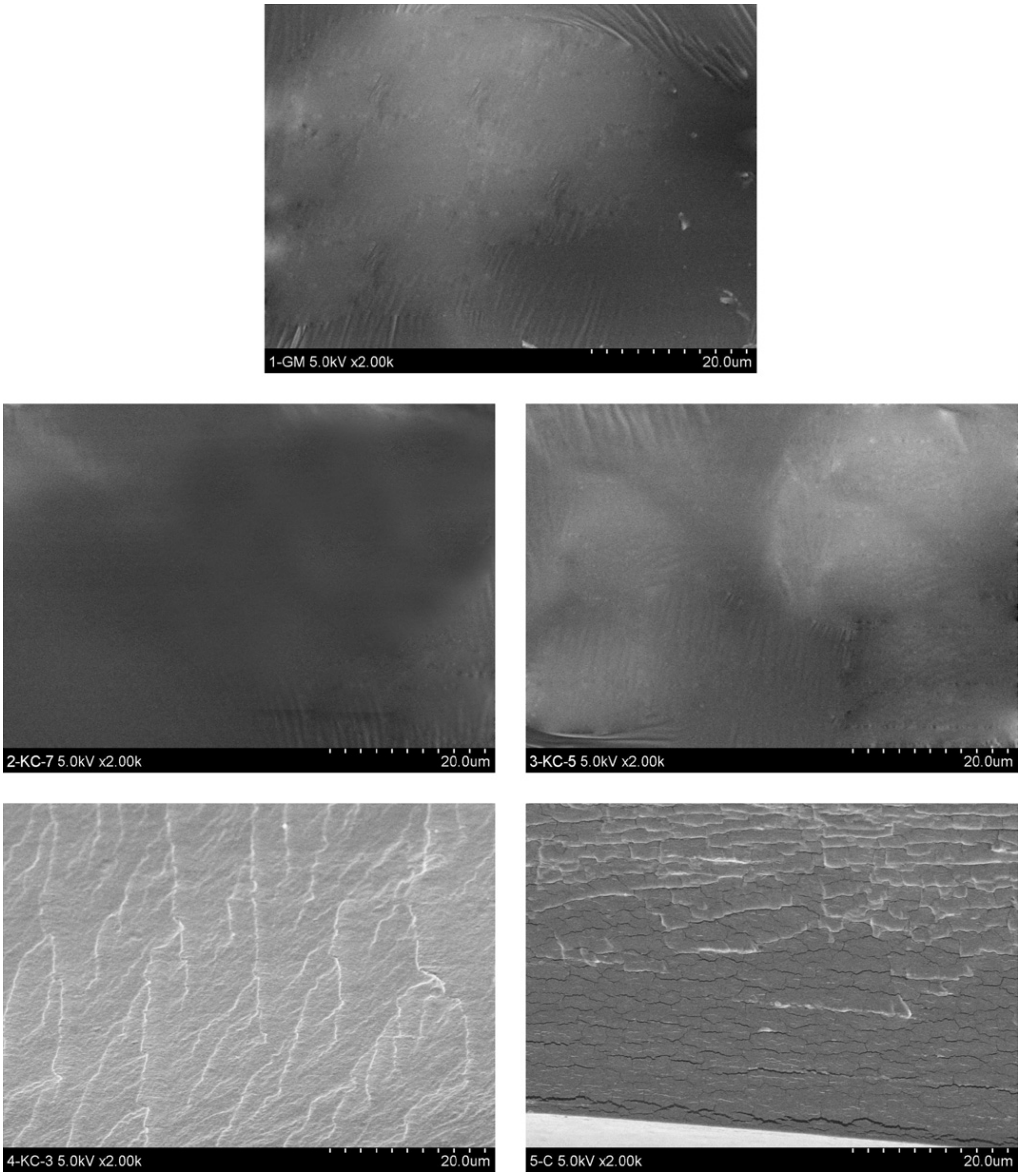

Fig. 4. SEM photographs of the blend films.

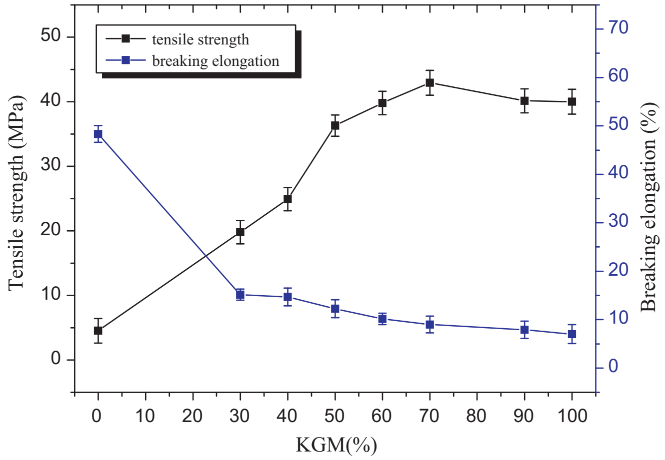

Fig. 5. The dependence of the tensile strength and breaking elongation on the KGM content for blend films.

WVP of the blend films was still lower than that of pure KGM films which has the maximum value. The improvement in moisture barriers is due to the excellent moisture barrier's ability of curdlan. It also indicated the increase of intermolecular interactions and the decrease of the mobility of the macromolecules of both KGM and curdlan when the mixed ratio was 7:3.

3.2.3. *Moisture uptake of blend films*

The dependence of the moisture uptake on the KGM content for the blend films was displayed in Fig. 7. The moisture uptake at equilibrium increased as the KGM content increased. This maybe related to the higher hydrophilic property of KGM. The KGM might facilitate the permeation of moisture into curdlan when submitted to highly moisture atmosphere. In general, the higher the moisture uptake of the film is, the poorer the tensile strength will be (Chen et al., 2008). However, in this work, with an increase of KGM content in the blend films, both the moisture uptake and tensile strength of the films increased at the same time. This indicated that the blend films exhibit higher tensile strength in addition to the greater

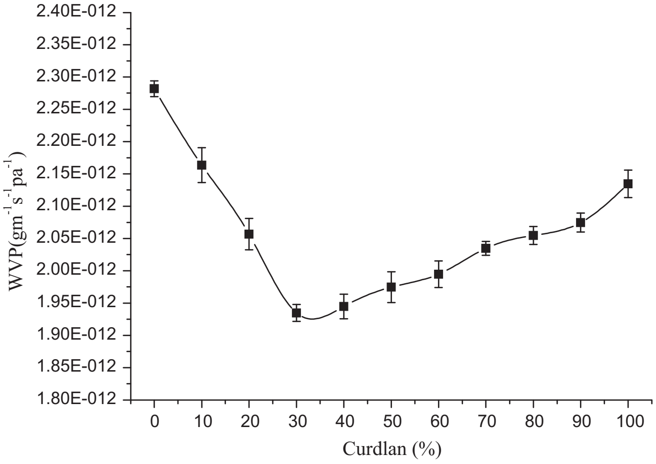

Fig. 6. The dependence of the WVP on the curdlan content for blend films.

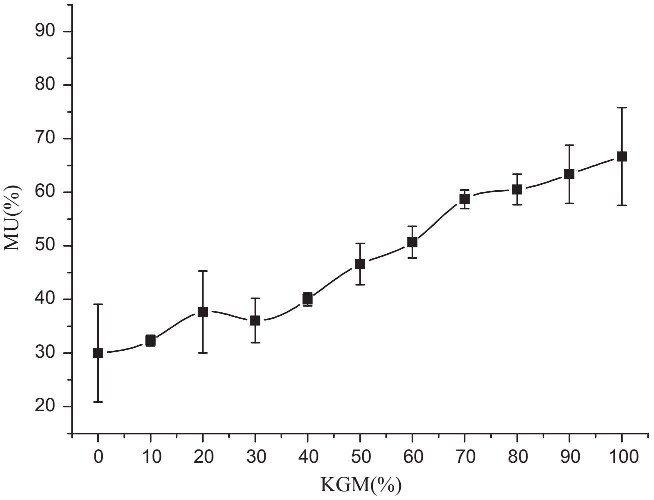

Fig. 7. The dependence of the moisture uptake on the KGM content for the blend films.

moisture retention than that of the neat curdlan film, which might have a potential valuable application in the food field.

4. Conclusions

A series of edible films were prepared from KGM and curdlan. The FT-IR, XRD, SEM and DSC data indicated that strong intermolecular hydrogen bonding occurred between KGM and curdlan. The interaction of the blend film was much greater than that of others when the KGM content in the blend films was around 70 wt% (KC7), resulting in excellent miscibility. Compared with the neat curdlan film, the tensile strength of the blend films increased from 4.54 ± 1.89 MPa to 42.93 ± 1.92 MPa as KGM content increased from 0 to 70 wt%. Conversely, breaking at elongation of the blend films exhibited an obviously opposite trend. And the neat curdlan film reached a maximum value of 48.33 ± 1.71%. What's more, the presence of curdlan improved the moisture barrier properties of the KGM/curdlan films. From this work, the blend film KC7 has a potential application in the food field such as edible food films and coatings.

Acknowledgments

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