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Development, physiochemical characterization and forming mechanism of *Flammulina velutipes* polysaccharide-based edible films

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ABSTRACT

Edible films of *Flammulina velutipes* polysaccharide were prepared and characterized in terms of rheological, optical, morphologic, mechanical and barrier properties to evaluate their potential application in food packaging. Results suggested that FVP film prepared by the solution of 1:150 (w/v) had the optimal mechanical property, smooth and uniform surface, and good barrier property to water (37.92 ± 2.00 g mm/m^2 h kPa) and oxygen (37.92 ± 2.01 meq/kg). The capacity of film-formation might be related to inter-molecular and intra-molecular hydrogen bonds of FVP and formation of β-glycosidic bonds during the process of film-formation. These findings will contribute to a theoretical basis for the development of FVP film in food packaging.

1. Introduction

Edible films, as a kind of novel biodegradable natural materials, are mainly prepared from polysaccharides, proteins, and lipids. (Cerqueira et al., 2011; Galus & Kadzińska, 2015; Salarbashi et al., 2013; Valenzuela, Abugoch, & Tapia, 2013). These films can be applied to the packaging of meat, seafood, vegetables, fruits, and candies for better barrier, antibacterial and antioxidant properties (Forato, de Britto, de Rizzo, Gastaldi, & Assis, 2015; Tavassoli-Kafrani, Shekarchizadeh, & Masoudpour-Behabadi, 2016; Volpe et al., 2015). Growing attention has been driven to search novel edible film materials from cereals, vegetables and fruits (Gutiérrez, Morales, Pérez, Tapia, & Famá, 2015; Moreno et al., 2014; Salgado, Ortiz, Musso, Di Giorgio, & Mauri, 2015). However, most of the films possess undesirable characteristics such as rough surface, poor mechanical or barrier properties. Therefore, it is further interest to seek new biomacromolecules with excellent film-forming abilities.

Polysaccharides, mainly starch, cellulose, chitosan, alginate, and pullulan, are one of the most frequently used edible materials for development of edible film (Elsabee & Abdou, 2013; Gutiérrez et al., 2015; Wu et al., 2012; Xiao, Tong, & Lim, 2012). However, these polysaccharides exhibit poor film-forming properties and by adding such additives, such as glycerol (Ghasemlou, Khodaiyan, & Oromiehie, 2011), sorbitol (Talja, Helén, Roos, & Jouppila, 2008), and polyethylene glycol (Cao, Yang, & Fu, 2009), the film performance can be improved (Debeaufort, Quezada-Gallo, & Voilley, 1998), which may bring bad flavor, taste into the film and ultimately affect edibility of films. *Flammulina velutipes* is a widely cultivated and commercially available mushroom in the world due to its desirable taste and high nutritional components including polysaccharides, fungal immunomodulatory proteins, and flavonoids. Our previous studies have proved bioactivity of *F. velutipes* polysaccharides (FVP) such as anti-proliferation activity and learning and memory improvement in rats (Yang, Fang, Liang, & Hu, 2011; Yang et al., 2012, 2015). Another interesting finding is that the FVP showed excellent film-forming during the preparation process, which indicated the potential of FVP as a material of edible film. Moreover, no use of additives for the preparation of edible films makes FVP more attractive in terms of edibility safety. Up to today, no studies are available on the application of FVP in edible films.

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In this study, FVP was prepared and the rheological properties of film-forming solutions (FFS) were studied. Subsequently, edible films were prepared from FFS and the physical, mechanical and microstructure properties of films were investigated. Additionally, the mechanism for film-formation of FVP was analyzed using Fourier transform infrared spectroscopy.

2. Materials and methods

2.1. Preparation of FVP

Fresh *F. velutipes* mushrooms were purchased from a local market (Nanjing, China). After washing, the mushrooms were dehydrated at an air temperature of 60 °C for about 4.5 h and a constant relative humidity of RH 20% in an electric thermostatic drying oven (DNF610, YAMATO Scientific Co. Ltd, Japan). Dried *F. velutipes* was powdered and sieved through a No.100 mesh. A 200 g powder was extracted by 10 L deionized water with stirring for 5 h at 80 °C. The extraction was collected by centrifugation (5000 rpm, 10 min, 4 °C). The supernatant was pooled and concentrated into one-tenth of the original volume under reduced pressure. Then, FVP extract was deproteinized four times with Sevag reagent (chloroform: butanol, 4:1) before precipitating with 4-fold volume anhydrous ethanol at 4 °C for 12 h. After centrifugation at 10,000 rpm for 20 min, the precipitate was dialyzed in distilled water which was renewed every 2 h for 24 h at 4 °C, and lyophilized as FVP.

2.2. Film preparation

Preliminary experiments (data not shown) had demonstrated that the solid-to-liquid ratio higher than 1:300 (w/v) was not suitable for the preparation of films and the ratio lower than 1:300 (w/v) was used in this study. FVP was dispersed in distilled water following the solid-to-liquid ratio of 1:100, 1:150, 1:200, 1:250, 1:300 (w/v), stirred for 24 h at room temperature to achieve complete hydration, and degased for 15 min as FFS ($S_{1:100}$, $S_{1:150}$, $S_{1:200}$, $S_{1:250}$, $S_{1:300}$). In order to speed up the dissolution of FVP, the distilled water was heated to 60 °C. Then a 10 mL of solution was poured into disposable petri dish and left to dry in a biological cabinet at 25 °C and RH 64% for 12 h. The layers of five kinds of films prepared at ratio of 1:100, 1:150, 1:200, 1:250 and 1:300 ($F_{1:100}$, $F_{1:150}$, $F_{1:200}$, $F_{1:250}$, $F_{1:300}$) were obtained for further experiments. Three replicates were prepared based at each solid-to-liquid ratio.

2.3. Rheological properties of FFS

The rheological properties of FFS were measured by a rheometer (MCR302, Anton Paar, Austria) equipped with cone-and-plate geometry of CP50-1 (diameter: 50 mm, cone angle: 1°, gap between cone and plate: 0.105 mm) at 25 °C. All samples were sheared continuously at the shear rate ranging from 0.1 to 500 s^{-1}. Three replicates were performed for each sample. Flow behaviors of the solutions were measured as a function of shear rate. Power law model as an equation was employed to examine flow properties of FFS (Eq. (1)).

$$\sigma = K \times \gamma^{n} \quad (1)$$

where σ is the shear stress (Pa), γ is the shear rate (s^{-1}), K (Pa s) is consistency coefficient and n is flow behavior index. K and n were determined at increasing shear rate (0.1–500 s^{-1}) and calculated by Eq. (1).

The apparent viscosity (η, Pa s) values of FFS were calculated according to Eq. (2).

$$\eta = K \times \gamma^{n-1} \quad (2)$$

2.4. Fourier transform infrared spectroscopy

A Bruker Tensor27 (Ettlingen, Germany) equipped with an attenuated total reflectance (ATR) accessory was used to record Fourier transform infrared spectroscopy (FT-IR) of FFS, which could suggest the change of chemical structure of film during film formation. FFS was dripped on the accessory, and scans were done at intervals of 5 min during 2 h at 25 °C and 64% RH. The spectra in the range of 4000–700 cm^{-1} were rationed and signal averages were collected for 50 scans at a resolution of 4 cm^{-1}.

2.5. Film thickness

An electronic digital micrometer (Guilin Guanglu Measuring Instrument Co. Ltd., China) was used to determine film thickness to the nearest 0.0001 mm. Ten measurements were made at random positions on each testing sample, and the mean values were calculated to analyze water vapor permeability (WVP) and tensile strength (TS).

2.6. Barrier properties

2.6.1. Water vapor permeability measurements

The WVP was measured according to ASTM method E96 (1996) (Rezvani, Schleining, Sümen, & Taherian, 2013) with some modifications. Briefly, polystyrene jars (ID: 3 cm, height: 5.5 cm) were filled with deionized water up to 1 cm from the jar mouth. The containers were then covered with FVP films without pores or any defects and further sealed with paraffin wax. The jars were placed in a constant temperature and humidity biological cabinet at 25 °C and 50% RH. A fan was operated within the cabinet to remove permeating water vapor from the surface of containers. The jars were weighted after 2 h, ensuring that the equilibrium has been reached and then weighed at intervals of 1 h for 10 h. The weight changes of the jars were recorded and the relation between the weight and time was plotted. Water vapor transmission rate (WVTR) was obtained from the slope of each straight line divided by fill area (m^2), which was calculated by linear regression ($R^2 > 0.99$). WVP (g mm/m^2 h kPa) was calculated by Eq. (3).

$$WVP = \frac{WVTR}{|S(R_1 - R_2)|}D \quad (3)$$

where WVTR is the measured water vapor transmission rate (g h^{-1} m^{-2}) through the film specimen; S is the saturation vapor pressure of water (Pa) at test temperature (25 °C); R_1 is the RH of test environment (50%) and R_2 is the RH in the jars (100%); D is the film thickness (mm). Determinations were made in triplicate.

2.6.2. Oxygen barrier

Oxygen barrier property of FVP film was measured indirectly according to the method reported by Kurt and Kahyaoglu (2014). Fresh corn oil (50 mL) was poured into a 100 mL jar, which was covered with different FVP films, sealed with paraffin wax, and stored at 60 °C for 10 d. Besides, in the same storage condition, two jars were filled with the same volume corn oil. One of them was sealed with a rubber plug for no oxygen penetration test (NOPT), and the other was kept open for no barrier test (NBT). The peroxide value (PV) of the corn oil was determined by sodium thiosulfate titration method. Three replications of all tests were measured.

2.7. Mechanical properties

Tensile strength (TS) and the elongation at break (EAB), two typical measurements of mechanical properties, were determined using a texture analyzer (TA.XT2i Texture Analyzer, Stable Micro