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A simple and feasible approach to purify konjac glucomannan from konjac flour – Temperature effect

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ABSTRACT

A simple one-step purification process was provided to extract KGM from KF by phase separation. The results showed that appropriate temperature control was a key factor and the products were inodorous, colourless and of high purity at the optimal temperature 68 °C. In this purification, soluble sugar and starch of extracted KGM were nearly clearly reduced and up to 95%, 80% (T68) of protein and ash were removed, respectively as compared with KF. Odour and transparency were improved 4 ranks and 30%, respectively. Besides, the η_{app} reached 42.30 Pa s and increased by 93.55% as compared, which could stay at a steady level for a week. Furthermore, morphology of extracted KGM displayed regular lamellar and wrinkling distribution for removed impurities. The temperature-controlled method not only enriches the knowledge of KGM purification but also has the potential to broaden the application of KGM.

1. Introduction

Konjac glucomannan (KGM), one of the richest natural polysaccharide, is derived from the tubers of *Amorphophallus konjac* (Fang & Wu, 2004). It is the main constituent in konjac flour (KF) and has been recognized as GRAS since 1994 (Khanna & Tester, 2006). As a kind of non-caloric food, KGM had been applied in many kinds of food as high quality dietary fiber, thickening agent, and fat replacer (Charoenrein, Tatirat, Rengsutthi, & Thongngam, 2011; Jimenez-Colmenero, Cofrades, Herrero, Solas, & Ruiz-Capillas, 2013). Besides, KGM is also an attractive candidate for the preparation of composite materials, biodegradable film and controlled release matrix in medicine, cosmetics and fine chemical fields (Harding, Smith, Lawson, Gahler, & Wood, 2011; Pang et al., 2012). However, commercial KF is usually light-coloured with fish-like smell and slightly harsh taste (Chua, Baldwin, Hocking, & Chan, 2010). Its poor qualities, including low viscosity and poor transparency, usually could not satisfy the demand of food and cosmetic production. Many scholars constantly purified KF before experimentation (Chen, Liu, & Zhuo, 2005; Wu et al., 2012). Therefore, it is practically important to develop a convenient and economical method to obtain KF with high purity, viscosity, and transparency.

As we know, KF is usually separated after the *konjac* tuber washed, sliced, dried and ground. The content of KGM in KF usually ranged from 50% to 70% (Tatirat & Charoenrein, 2011). The main impurities trapped in KGM particles usually derived from the tissue space (Chua et al., 2010). Due to the well processing characteristics of KGM, lots of scholars focus on its application and only limited researchers put their energy in extractive technique. Washing by water, ethanol, and benzene-ethanol solution is often used to remove insoluble impurities (Chen et al., 2005; Zhu, Uhl, Morgan, & Wilkie, 2001). While their disadvantage and inconvenience limit their further applications (Yan, Wang, & Liu, 2006). Recently, high-energy centrifugation (1500*g*) method and combining boiling water with ethanol washing were also exploited to isolate KGM (Jianrong, Donghua, Srzednicki, Kanlayanarat, & Borompichaichartkul, 2008; Tatirat & Charoenrein, 2011). The results testified it was an effective and high yield extraction approach.

Starch is the main impurity that seriously affects the purity and quality of KGM, such as reducing viscosity and increasing turbidity (Yoshimura, Takaya, & Nishinari, 1998). Additionally, starch molecules usually interact with KGM and are difficult to be purified for its low solubility in mild condition. However, gelatinization behaviour efficiently boosts its solubility. KGM molecules possess better swelling ability and become insoluble, whereas the impurities could dissolve in ethanol/water system. Taking all into consideration, phase separation was explored to apply in KF purification.

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During this process, the impurities could be removed by the temperature effect.

To the best of our knowledge, the preparation of refined KGM by ethanol washing with temperature controlled has barely been reported before. In this study, a simple, straightforward extraction route in ethanol/water system was developed. The samples were investigated for compositions, colour, transparency, rheological measurement and morphology to evaluate the refining effects. The results show the method is potential to purify and improve the qualities of KF.

2. Materials and methods

2.1. Materials

KF samples were provided friendly by Hubei Yizhi Konjac Industry Co. Ltd. (Hubei, China). All chemicals used in this study were of analytical grade reagents (Sinopharm Chemical Reagent Co. Ltd., Shanghai, China) and used without further purification.

2.2. Preparation of purified KGM via temperature controlled

KF was dissolved in 40% (v/v) ethanol solution with feed liquid ratio of 1:6. While stirring, the mixture was heated at a series of temperature (28, 38, 48, 58, 68 and 78 °C) and refluxed for 4 h. After purifying, filtering, washing with 40% ethanol solution at the pre-temperature, the precipitates were vacuum dried at 80 °C. By controlling the temperature, a series of KGM products with different characteristic were available and coded as T28, T38, T48, T58, T68, T78, and the control sample was coded as T.

2.3. Composition of KF flour

Different kinds of KF were analyzed for the contents of moisture, ash, starch, protein, and residual sugar according to AOAC methods (Williams, 1984). Moisture content was measured at 105 °C, ash content was performed at 500 °C, starch was measured by acidic hydrolysis method, and protein was measured by the Kjeldhal method, while the residual sugars were measured by the DNS method. KGM and its dry basis content were measured as our previous reported (Li & Xie, 2002; Liu, Wang, Xia, & Li, 2005).

2.4. Transparency and odor evaluation

Transparency of sample solutions were analyzed by the modified method of Kobayashi using 722E visible-infrared spectrometer (Shanghai spectrum instrument Co. Ltd.). Briefly, 1.0% (w/v) KGM solutions were prepared at 25 °C and the transparency was determined at 500 nm against a distilled water blank after viscosity reaching the maximum (Kobayashi, Tsujihata, Hibi, & Tsukamoto, 2002).

Because commercial KF is light-coloured with fish-like smell, osphretic evaluation was carried out by a seven-member panel. Each panel member evaluated each sample using a 0–5 point scale (0 indicates no fish-like smell; 1 imperceptible, 2 light, 3 moderate, 4 great, 5 extreme).

2.5. Rheological measurements

Apparent viscosity and its variation were measured by NDJ-8S Digital display viscometer (Shanghai spectrum instrument co. Ltd.). 1.0% (w/v) KF solution was prepared with stirring for 1 h at 25 °C. Apparent viscosity was determined after each 0.5 h in triple using NO.4 rotor at 12 r min^{-1}. Viscosity stability was also evaluated by determining its variation for 1 week.

Rheological measurements were performed using an AR2000ex rheometer (TA, UK) with a parallel plate (40 mm in diameter, 1.0 mm). In order to avoid the destruction of the structure being formed, a linear viscoelastic regime is needed to determine by measuring steady rheological viscosity. The data was collected with the shear rate ranging from 0–150 s^{-1} at 25 °C. Dynamic viscoelastic parameters, shear storage modulus (G′) and loss modulus (G″) as functions of frequency and temperature were investigated with stress 0.2%. For each measurement, the sample was poured directly onto the lower parallel plate, which had been kept at each measurement temperature without pre-shearing or oscillating. The dynamic temperature sweep measurements were conducted from 25 to 90 °C with heating and cooling rates of 2 °C min^{-1}. While frequent sweep measurements were carried out ranging between 0.01 and 100 rad s^{-1} at constant frequency (0.1 Hz) and strain amplitude (0.2%).

2.6. Morphology observation

Morphology of KF with different temperature disposed was investigated using a scanning electron microscope (JSM-6390LV, Jeol, Japan). The dried samples were coated with gold–palladium before observed under the microscope. The micro-appearance of KF with the magnification of 100, 2000, 8000 could be easily available.

3. Results and discussion

3.1. Composition analysis

From the application aspect, ideal KF should possess high purity properties. KF, produced by the traditional method, generally contains protein and starch, which seriously affect its quality. In this regard, we conducted the study of the component of T, T28, T38, T48, T58, T68 and T78 and the results were displayed in Table 1. The results showed that the KGM content of all refined groups increased companied with impurities decreased as compared with T.

Protein, soluble sugar, starch and ash could be effectively wiped out and shared the same trends during thermal process in ethanol/water system. However, it was interesting that protein and soluble sugar content shared a sharp decrease as the temperature reached 38 °C and then turned to be constant. That indicated protein and soluble sugar were easily dissolved in 40% (v/v) ethanol solution at 38 °C for 4 h. Due to the complex structure and swelling ability, starch solubility was barely affected at low temperature. However, at high temperature (48 °C), starch gelatinization occurred and its solubility enhanced. While the major (77–83%) starch has been removed when the temperature is with 28–38 °C, and increasing temperature above 48 °C could further remove the tiny amount of residual starch. This phenomenon may be cause by the interaction between konjac and starch. The hydrogen bond interaction was the main interaction, besides physical package also existed. Improving temperature contributed to destroy the interaction between konjac and starch. It was noticed that the gelatinization point of starch in KF was lower than other kinds of starch (>60 °C). The phenomenon may be caused by various starch structures and the interactions between starch and KGM. Ash, the total amount of inorganic composition, is a crucial parameter for evaluating quality. In the food production, including KGM, ash is essential to be kept in a controlled range, otherwise the material may be evaluated contaminated or disqualified. Increasing extraction temperature is hopeful to decrease ash content.

As Table 1 showed, soluble sugar and starch of extracted KGM (T68) were nearly removed and protein and ash were reduced 95%, 80%, respectively as compared with KF. It was obvious that