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Effect of different drying methods on chemical composition and bioactivity of finger citron polysaccharides

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ABSTRACT

Traditionally, people like to take dried finger citron fruits (FC) as adjuvant herbal medicines to treat a diversity of chronic diseases like asthma, hypertension and respiratory tract infections. Many healing properties are attributed to FC polysaccharides (FCPs), one of the main active ingredients of FC. Three drying methods, freeze drying (FDM), hot air drying (HDM) and vacuum drying methods (VDM) were comparatively studied on the physicochemical and antioxidant properties of FCPs. The results showed these FCPs were similar in UV and FT-IR spectrum. However, they showed significant differences ($p < 0.05$) in yields of crude polysaccharides and contents of protein and ash. Compared with VDM and HDM, FDM resulted in the properties of FCPs with lower molecular weight distribution, higher reducing power and scavenging abilities on DPPH•, OH•, and $O_2^{\bullet -}$. Available data obtained *in vitro* models suggested that FDM was an appropriate and effective treatment for obtaining crude polysaccharides from FC fruits. Hence, drying methods used for preparation of FCPs can affect physicochemical and associated functional properties.

1. Introduction

The finger citron [*Citrus medica* L. var. *sarcodactylis* (Noot.) Swingle] fruits (FC), have been long used as traditional Chinese medicinal material and functional vegetables, and preserved as sweetmeats [1]. FC fruits belonging to the family *Rutaceae*, commonly known as "Five finger orange" in commercial vegetable markets, is an important functional food source in a specific diet [2]. Earlier studies reported that FC fruits possessed antioxidant, insulin secretagogue, anti-inflammatory, anti-microbial and anthelmintic activities [3,4].

Rapid drying process of Chinese herbal pieces is crucial to its quality control and pharmaceutical effect [5,6]. The active ingredients and pharmacological efficacy of Chinese herbal pieces are greatly influenced by different processing technologies. Drying is a very common preservation method used in Chinese herbal pieces and the quality of the final products is strongly dependent on the technique and the process variables used [7]. The World Health Organization estimates that 80% of the world's population uses medicinal plants medicines and related extracts in some way [8]. According to this growing demand for medicinal extracts, artificial drying has been one of the most important needs of the pharmaceutical industry, which does not have structure to use fresh plants in the quantities required for industrial production. Also, increasing usage of drying procedures within various technological lines in the food industry and biotechnology has made studies of the drying process of important practical interest.

In recent years, diets rich in selected natural antioxidants and various extracts from FC fruits are related to reduced risk of incidence of hyperlipidemia, obese and other chronic diseases has lead to the revival of interest in plant-based foods [9,10]. To date, the studies have been mainly focused on the influence of the drying methods on the essential oils or volatile fractions of *Citrus* herbs in order to optimize the drying [11,12]. Polysaccharides extracted from FC fruits (FCPs) are of particular interest because of their effective bioactivities, such as antioxidant [3]. In general, physicochemical properties and effective bioactivities of crude polysaccharides extracted from fruits, vegetables and medicinal plants was greatly influenced by different drying process including freeze drying (FDM), hot air drying (HDM) and vacuum drying (VDM) [13,14]. Various drying methods have been developed to preserve herb plants and have exhibited their own characteristics [15]. However, there was no information about the effects of drying methods on composition and antioxidant activities of polysaccharides from FC fruits. Additionally, the ability to determine suitable drying methods would increase the grower's control on both FC fruit yield and its quality. A recommendable drying method should tend to avoid undesirable changes and maintain the good quality

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of dried product. In spite of our earlier work in this area [3], the situation regarding the influence of different drying methods on the physicochemical properties and antioxidant activities of FCPs is far from clear. The aims of the present study were, therefore, to determine physicochemical properties and antioxidant activities of FCPs as affected by the different drying methods (traditional hot-air, vacuum, and freeze drying).

2. Materials and methods

2.1. Materials

The FC fruits [*C. medica* L. var. *sarcodactylis* (Noot.) Swingle], were harvested from different trees in Liangping county (coordinates: lat. 30°40′ N, long. 107°48′ E), Chongqing, China, at an altitude of 450 m, and authenticated by the Application and Development Institute of Chongqing Academy of Chinese Materia Medica (Chongqing, China). All the collected FC fruits were cut into 5 mm strips before drying at 60 °C for 5 h. All chemicals used in the experiment were of analytical grade.

2.2. Extract of crude polysaccharides

The dried FC fruits were refluxed with 95% ethanol at 70 °C in a water bath for 2 h to deactivate the endogenous enzymes and remove some soluble materials, including free sugars, amino acids and some polyphenols. The combined extract was vacuum dried at 60 °C for 12 h, and it was suspended in the water and sonicated at the temperature of 50 °C and actual sonic power of 45 W for 40 min. After rapid cooling to room temperature using ice water, the supernatant was concentrated in a rotary evaporator under reduced pressure (Buchi R-124, Flavil, Sweden). The extract was filtered, and then precipitated using 150 mL of 95% ethanol, 100% ethanol and acetone, respectively. After being left overnight at 4 °C, the precipitates were collected by centrifugation at 3000 rpm for 20 min, redissolved in deionized water, deproteinated by the method of Sevag et al. [16], and dialyzed in a dialysis bag (MWCO 1400 Da, Union Carbide). The precipitates were then hot air dried, vacuum dried or freeze dried, respectively.

2.3. Drying procedure

The precipitates were hot air dried in an electric thermostatic drying oven (DHG-9240A, Yiheng Scientific Instruments, Shanghai, China) at 50 °C. Vacuum drying was performed at 50 °C, at 95 kPa of vacuum degree for 24 h in a vacuum oven (ZF-6020, Yiheng Scientific Instruments, Shanghai, China). For freeze drying, the precipitates was first frozen for 24 h at −18 °C. Frozen samples were then freeze-dried (Christ ALPHA 1-2 LD plus) at −50 °C. The samples were dried until the moisture contents below the 8%. FCPs drying by hot air drying, vacuum drying and freeze drying methods were named FCPs-H, FCPs-V and FCPs-F, respectively. All experiments were performed at least in duplicate. The flow chart was shown in Fig. 1.

2.4. Compositional and relative viscosity analysis

The percentage FCPs extraction yield (%) was calculated with the formula of y (%) = $c/w \times 100$, where c was the polysaccharides content of extraction, and w represented dried sample weight (1 g). The sugar content was determined by the reaction of sugars with phenol in the presence of sulfuric acid using glucose as a standard [17]. Ash were determined according to AOAC (1990) method [18], while the protein content in the solid polysaccharide was determined using the Kjeldahl method with a conversion factor of 6.25 [19]. Relative viscosity (to deionized water) of LMPs was measured in NDJ-1

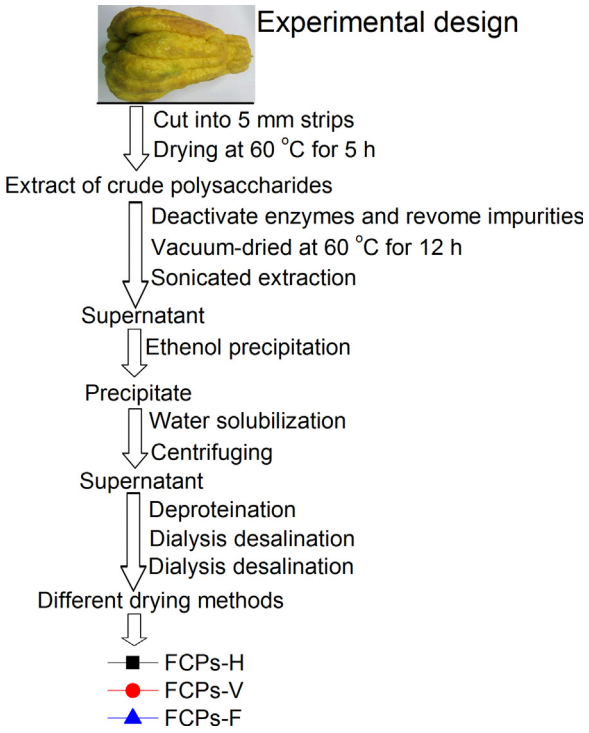

Fig. 1. Scheme of extraction and fractionation of polysaccharides from finger citron fruits. The polysaccharides obtained from finger citron fruits by the hot air drying, vacuum drying and freeze drying methods were named FCPs-H (■), FCPs-V (●) and FCPs-F (▲), respectively.

Rotation Viscometer (Jinghai Technology Co. Ltd., Shanghai, China) at a concentration of 10 mg/mL and 25 °C [3,20].

2.5. Average molecular weight (AMW) determination of FCPs

The AMW of FCPs was determined by gel-permeation chromatography (GPC) on a Sephadex G-100 column (1.6 cm × 100 cm) using distilled water as eluent at a flow rate of 24 mL/h [21,22]. The eluate (5 mL/tube) was collected and monitored for carbohydrate content using phenol–sulfuric acid method at 490 nm [17]. The average molecular weight of FCPs was calculated as the following equation [23]:

$$\mathrm{AMW} = \frac{\sum M_i \times C_i}{\sum C_i} \quad (1)$$

where M_i was the molecular weight of each fraction, obtained from the regression line of the Dextran T-series standard of known molecular weight (T-2000, T-70, T-40, T-20, and T-10) *versus* elution volume plot. C_i was the total carbohydrate concentration of each fraction, analyzed by phenol–sulfuric method [17].

2.6. Ultraviolet–vis and Fourier transform infrared spectroscopy analysis

The ultraviolet–vis (UV) absorption spectroscopy of the above-mentioned FCPs samples was recorded using an UV-2600 spectrophotometer (Shimadzu, Japan) in the wavelength range from 200 and 700 nm. Sample solution was prepared by dissolving 5 mg of sample in distilled water to a concentration of 1.0 mg/mL, and distilled water was taken for the blank. Fourier transform infrared spectrometry (FT-IR) was obtained using a Spectrum 100 FT-IR spectrophotometer (Perkin Elmer, USA). The dried FCPs were grinded with potassium bromide power and pressed into pellet for