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Comparative study of microwave-vacuum and vacuum drying on the physicochemical properties and antioxidant capacity of licorice extract powder

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ABSTRACT

Licorice is the dried root of *Glycyrrhiza* plants, which have long been used as flavourings, sweeteners, demulcents and expectorants in Western countries; as anti-allergic and anti-inflammatory drugs in China and Japan; and as therapeutics for diseases, such as cancers, heart disease and diabetes, in Russia and Eastern Europe. Licorice extract powder is the aqueous extract of licorice. Lycyrrhizic acid and flavonoids are the main bioactive constituents of Licorice extract powder that have health functions. Two drying methods, vacuum drying and microwave-vacuum drying, were comparatively studied to determine their impact on the physicochemical and antioxidant capacity of licorice extract powder, including its water-solubility, hygroscopicity, flowability, compression moldability, disintegration time, GA and total flavonoids compound contents and antioxidant capacity. The results showed that the physicochemical and antioxidant capacity of licorice extract powder were affected by the drying method. Microwave-vacuum drying maintained the GA and flavonoids components contents as well as the antioxidant capacity and reduced hygroscopicity. However, microwave-vacuum dried powders had a lower water-dissolution and a longer disintegration time than powders tablets.

1. Introduction

Glycyrrhiza species (Leguminosae family) are ligneous perennial shrubs that grow approximately 1.5 m tall and have subterranean stems (rhizomes) as well as highly branched roots that grow underground horizontally. The plant lives for multiple years has blue and violet flowers [1].*Glycyrrhiza* plants grow best in subtropical and warm zones with access to the sun as well as in nitrogen-rich drylands. *Glycyrrhiza* plants are distributed from Western Europe to Russia, with particular abundance in China and Mongolia [2].

Licorice is the dried root of *Glycyrrhiza* plants, which is an herb that is widely used in many countries and regions of the world and has been consumed for approximately 6000 years. The roots are traditionally used as flavourings, sweeteners, demulcents and expectorants in Western countries as well as anti-allergic and anti-inflammatory drugs in China and Japan [3]. Particularly in China, licorice is officially recorded in all editions of Chinese Pharmacopoeia, is characterized as gentle and sweet, and plays a role in spleen strengthening, lung nourishing, heat-clearing, and detoxifying, urgency, pain relieving and drug reconciling, as well as acts as an anti-tussive and expectorant [4]. In clinical prescriptions of TCM, licorice is known as a "guide drug" or "ambassador drug" and is commonly used according to a classic proverb "Ten prescriptions, nine licorices" for the treatment of gastric or duodenal ulcers, sore-throats, hepatitis, coughs, bronchitis, allergies, arthritis, and cardiovascular diseases [5]. Approximately 500 compounds have been identified in licorice [2], mainly flavonoids, polysaccharides and triterpene saponins [6]. Among them, glycyrrhizin or glycyrrhizic acid (GA), a triterpinoid saponin, and licorice flavonoids are considered to be the main biologically active components in licorice with known pharmacological effects, including antioxidant, antiviral and anti-inflammatory properties [5,7–9]. Additionally, GA is a natural sweeteners and is approximately 50 times sweeter than cane sugar [10].

Licorice extract, the aqueous extract of licorice, takes the form of a brown powder with special and lasting sweetness and is included in Chinese Pharmacopoeia. Licorice extract retains and concentrates the active ingredients, including GA (controlled at a concentration of >7.0% in Chinese Pharmacopoeia) and flavonoids, contained in licorice. Therefore, licorice extract can not only be used as a sweetener but can also be directly diluted with water for various pharmacological activities. In this regard, the quality indicators of licorice extract powder as a final product should be considered, including the moisture content, water-solubility, hygroscopicity, flowability, chemical components and pharmacological properties. Moreover, in China, licorice extract is an important raw material for many TCM preparations, such as Compound

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Licorice Tablets and Licorice Formula Granule. Thus, in addition to the considered quality indicators of licorice extract powder used as a final product, the formability and disintegration time should also be included its quality indicators when used as raw material.

Drying is an indispensable step to obtain licorice extract while extending the storage period by evaporating the moisture in the extract up to a certain value [11]. Currently, different drying methods have been adopted to dry licorice extract, and these methods have their own unique features. Previously, vacuum drying (VD) has been the main method for dehydrating licorice extract as heat in this method is provided by conduction and the temperature can be controlled low levels [12]. However, VD is an inefficient process, takes a long time and may degrade biologically active components in herb extracts. By contrast, microwave-vacuum drying (MVD) ensures the rapid and efficient distribution of heat within the material, which largely reduces the drying time; saves energy consumption; and contributes to produce high quality dry products [13]. Hence, MVD meets the industrial requirements and has gradually become a common drying method in the drying of licorice extract.

Recent studies have revealed that the different drying methods have their own drying characteristics and have an effect on the estimation of the physicochemical properties and bioactive activities in a sample [14–16]. However, there is no information about the influences of the drying method on licorice extract. Considering the above, the aims of the present study were to investigate the effects of the selected drying methods (VD and MVD) and parameters on water-solubility, hygroscopicity, flowability, compression moldability, disintegration time, GA and total flavonoids compound contents. Moreover, taking into account the antioxidant activity of licorice extract determined by the GA and flavonoid contents and that almost all chronic diseases are associated with the danger of free radicals [17], the antioxidant capacity (via DPPH assays) of licorice extracts was estimated.

2. Materials and methods

2.1. Materials and chemicals

Licorice decoction pieces were purchased from the Sichuan Neautus Traditional Chinese Medicine Co., Ltd. (Chengdu, China). HPLC grade ethanol and acetonitrile were purchased from Merck (Darmstat, Germany). KOH was purchased from Kelong Chemical Reagent Co., Inc. (Chengdu, China). GA for content determination was purchased from the National Institutes for Food and Drug Control (Beijing, China). Distilled water was used in all experiments.

2.2. Preparation of licorice concentrated solution for drying

According to Chinese Pharmacopoeia [4], licorice decoction pieces were extracted 3 times by adding water at a ratio of 1:8 (w/v) for 2 h each under atmospheric pressure. Then, the extract was collected, filtered with crude filter paper, concentrated to a density of 1.28 g/cm^3 by rotary evaporator (RE-3000, Shanghai Yarong biochemistry instrument factory, China) at 60 °C, and stored at room temperature.

2.3. Drying methods

In all of the drying tests, approximately 100 g of licorice extract was loaded onto a plastic plate with dimensions of 20 × 10 cm with a thickness of 3.9 mm. Vacuum drying (VD) was then performed in a vacuum dryer (DZF-6050, Shanghai Boxun Industry & Commerce Co., Ltd., China) at 60, 70 and 80 °C under a pressure of −95 KPa. Microwave-vacuum drying (MVD) was performed in a microwave oven (WZ-1, Changzhou, Zhenhua drying equipment Co. Ltd., China) at a microwave power of 100, 200, 300 and 400 W under a pressure of −95 KPa.

During the drying period, the moisture loss was recorded at 30 s intervals in the microwave oven dryer and every 10 min in the vacuum oven dryer by removing the sample and weighing it on a digital balance (Matou YP10002, Shanghai, China) with a 0.01 g accuracy. All drying experiments were stopped as they reached a final moisture content of approximately 5%.

2.4. Physicochemical properties

2.4.1. Sample preparation

The dried extracts were crushed to a powder by a laboratory-scale grinder (XY-200,Zhejiang Yongkang Songqing Hardware Factory, China), sifted using an 80 mesh sieve (Pharmacopoeia sieve, Ejiang, Shanyn, China), transferred to a glass desiccator (1351-01,Yangcheng Cordial Co., Ltd., China), and stored at room temperature.

2.4.2. Water solubility

Water solubility was measured following the Cano-Chauca et al. method [18] with some modifications. 100 ml of distilled water and 1 g of powder sample were carefully transferred to a blender (MS-S BlueSpin, Zhengzhou Nanbei Instrument Equipment Co., Ltd., China). After running the blender at 500 rpm for 5 min, the solution was placed in a tube and centrifuged (TDL5M, Xian MoJiNa instrument manufacturing Co., Ltd., China) at 2000 rpm for 5 min. 20 ml of the supernatant was placed in weighing bottle that was pre-dried to a constant weight and immediately oven-dried at 105 °C to a constant weight. The water solubility (%) was calculated by the weight difference.

2.4.3. Hygroscopicity

Hygroscopicity was determined following the method of Chinese Pharmacopoeia [4] by using an artificial climate box (MGC-800HP-2, Shanghai Yiheng Scientific Instrument Co., Ltd., China). Approximately 2 g of powdered sample was placed in a dried weighing bottle, and the experiment was performed at a temperature of 20°C and relative humidity of 80%. The weight gain due to moisture absorption was recorded daily until reaching a constant weight, and the weight percent gain (%) was calculated.

2.4.4. Flowability

According to the method of Caliskan and Dirim [19], the Carr index (CI) and Hausner ratio (HR) were used to evaluate the flowability values of the samples. Both CI and HR were calculated from the tapped (ρ_{tapped}) and bulk (ρ_{bulk}) densities of the samples using the following Eqs. (1) and (2).

$$CI = \frac{\rho_{tapped} - \rho_{bulk}}{\rho_{tapped}} \times 100\% \quad (1)$$

$$HR = \frac{\rho_{tapped}}{\rho_{bulk}} \quad (2)$$

2.4.5. Compression moldability

The tensile strength (σ_T) recommended by Tye et al. [20] was used to assess the compression moldability of samples with some modification. The direct compression method was adopted to prepare tablets that were 0.35 g in weight, 10 mm in diameter (d) and approximately 3 mm in thickness (h). After being placed in a desiccator at room temperature for 24 h, the hardness (f) of the tablets were measured by using a Hardness meter (TBH300, ERWEKA Company, Germany). The tensile strength was calculated according to Eq. (3).

$$\sigma_T = \frac{2f}{\pi hd} \quad (3)$$

2.4.6. Disintegration time

The disintegration time was determined using a dissolution tester (YZHZB-lE, Beijing Centrwin Technology Co., Ltd., China) following the