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Application of mid-infrared spectroscopy in analyzing different segmented production of Angelica by AB-8 macroporous resin

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ABSTRACT

As complicated mixture systems, chemical components of Angelica are very difficult to identify and discriminate, so as not to control its quality effectively. In recent years, Mid-infrared spectroscopy has been innovatively employed to identify and assess the quality of Traditional Chinese medicine (TCM) products. In this paper, the macroscopic IR fingerprint method including Fourier transform infrared spectroscopy (FT-IR), the second derivative infrared spectroscopy (SD-IR) and two-dimensional correlation infrared spectroscopy (2D-IR), are applied to study and identify Angelica raw material, the decoction and different segmented production of AB-8 macroporous resin. FT-IR spectrum indicates that Angelica raw material is rich in sucrose and the correlation coefficient is 0.8465. The decoction of Angelica contains varieties of polysaccharides components and the content is gradually decreased with increasing concentration of ethanol. In addition, the decoction of Angelica contains a certain amount of protein components and 50% ethanol eluate has more protein than other eluates. Their second derivative spectra amplify the differences and reveal the potentially characteristic IR absorption bands, then we conclude that the decoction of Angelica contains a certain amount of ferulic acid and ligustilide. And 30% ethanol eluate, 50% ethanol eluate and 70% ethanol eluate are similar to ligustilide. Further, 2D-IR spectra enhance the spectral resolution and obtain much new information for discriminating the similar complicated samples. It is demonstrated that the above three-step infrared spectroscopy could be applicable for effective, visual and accurate analysis and identification of very complicated and similar mixture systems of traditional Chinese medicines.

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1. Introduction

Traditional Chinese medicine (TCM) is one of the oldest and most frequently used medicines worldwide and has influenced various other Asian medical systems [1,2]. Comprehensive quality control is critical to ensure the efficacy and safety in clinical use. Currently, many methods [3–5] have been applied to control the quality of medicine, which only involve the qualitative and quantitative assay of one or several chemical markers, such as HPTLC, HPLC, LC–MS, GC–MS, etc. Since the medicinal effects of TCM are determined by the holistic function of various components, the contents of several so-called index components cannot accurately represent the quality of each TCM sample. Besides, as mentioned before, because of quality control methods depending only on a few index components leave the other components uncontrolled, natural medicines could be adulterated by chemical industrial products. Furthermore, these techniques are time-intensive, labour-intensive and expensive [6] and most regulatory bodies concerned with the quality of medicine do not routinely test products [7]. Therefore, it is highly desirable to find a quick and effective identification and discrimination method to entirely monitor and

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capture the whole constituents of TCM and extract products.

Compared to other analytical techniques, infrared spectroscopic techniques are rapid, nondestructive, effective and low-cost. The IR instruments are commonly configured for mid-infrared (MIR; 4000–400 cm^{-1}) or near-infrared (NIR; 12500–4000 cm^{-1}) analysis. In recent years, both NIR and MIR have been successfully employed to identify TCM. For example, using NIR method to determinate main components in Rhizoma Atractylodis Macrocephalae [8], analysis Honeysuckle extracts [9], discriminate Rhizoma Corydalis from different sources [10]. Applying MIR method to discriminate genuine Glycyrrhizae Radix et Rhizoma and counterfeit Glycyrrhiza pallidiflora Maxim [11], identify wild and cultivated Ginseng [12,13], differentiate Paeonia lactiflora Pall from different areas [14].

The root of *Angelica sinensis* (Oliv.) Diels (Umbellifeae), known as Danggui in China, is a famous traditional Chinese medicine in common use [15]. It was first cited in the Shennong Bencao Jing (200–300 A.D., Han Dynasty) [16]. The described functions of Angelica in the Shennong Bencao Jing were to replenish blood, invigorate blood, stop pain, and moisten the intestines. It was reported that the active constituents in Angelica included essential oil and water-soluble substances [17]. In this article, tri-level infrared macro-fingerprint method, Fourier transform infrared spectroscopy (FT-IR) associated with second derivative infrared spectroscopy and two-dimensional correlation infrared spectroscopy (2D-IR) were applied to study the holistic variation rules of chemical constituents in Angelica and its different segmented production of HPD-100 macroporous resin. The aim of this study is to develop an effective analysis method for studying integrally the main constituents in the medicinal materials and their corresponding extracts, comparing the categories of chemical constituents in the different extracts and monitoring the qualities of medicinal materials.

2. Experimental

2.1. Apparatus

Spectrum GX Fourier-transformer infrared spectrometer (Perkin Elmer, USA), equipped with a deuterated triglycine sulfate (DTGS) detector was used. All IR spectra were recorded from an accumulation of 32 scans, and 0.2 cm/s of optical path difference (OPD) speed in the range of 4000–400 cm^{-1} with a resolution of 4 cm^{-1}. The interferences of H_2O and CO_2 were subtracted when scanning. The CKW-II programmable temperature controller (Beijing Chaoyang Automatic Instrument Co., China) was used to perform the thermal perturbation.

2.2. Materials

Angelica were obtained from AnGuo Company for traditional Chinese herbs (Anguo, China) and authenticated by associate professor Jingjuan Wang (Beijing University of TCM).

Ferulic acid and ligustilide (Purity > 98%) were provided by the National Institute on Drug Abuse of China (Beijing, China); Sucrose and protein (Purity > 98%) were purchased from Chengdu Pufeide Biotech Co., Ltd. of China (Chengdu, China).

Ethanol (AR grade) was purchased from Beijing Chemical Works (Beijing, China). AB-8 macroporous resin was supplied by Dingguo Changsheng Biotechnology Co. Ltd. (Beijing, China).

2.3. Procedure

224 g of Angelica was put into a round bottom flask containing 10 volumes of deionized water and dipping for 60 min, then refluxed for 90 min. The extract was filtered, and the residue was extracted once more for 60 min. The filtrates were combined and concentrated to 800 mL under reduced pressure at 60 °C. The decoction of Angelica was made for follow-up experiment.

AB-8 macroporous resin was immersed in 95% ethanol until they swelled and then washed with ethanol until there was no white cloudiness, even when some water was added to the eluate. Subsequently, the resins were washed with deionized water until the ethanol was thoroughly replaced with deionized water.

AB-8 macroporous resin was wet-packed in a glass column (7 cm × 50 cm). The decoction of Angelica was loaded into the column and the leak flow rate was 3 mL/min. Then gradient desorption procedures were carried out at different ratio (10%, 20%, 30%, 50%, 70%) of ethanol in water respectively until each gradient effluent was colorless. Each effluent of different ratio was combined. Ethanol in effluent was then evaporated yielding different concentrations of ethanol eluate. The specific procedure is shown in Fig. 1.

Each sample was ground into powder and was over 200 mesh. About 2–4 mg of the obtained powder was blended with 200 mg KBr powder and then was ground again and pressed into a tablet. Likewise, about 2–4 mg of sucrose, protein, ferulic acid and ligustilide were blended with 200 mg KBr powder and then pressed them into tablets, respectively. After that, the IR spectra of all samples were collected. Second derivative spectra were obtained by using Savitzky Golay polynomial fitting method. For collecting the dynamic FT-IR spectra at different temperatures from 50 to 120 °C, the tablet was put into the pool of the temperature controller to record the IR spectra at interval of 10 °C. The sample pool was continuously heated with an increasing rate of 2 °C/min. 2D-IR correlation spectra were obtained by the treatment of the series of dynamic spectra with 2D-IR correlation analysis software developed by Tsinghua University, Beijing, China.

2.4. Data processing

The raw FT-IR data was processed with PE spectrum software of Perkin–Elmer FT-IR spectrometer (Version 6.3.5). The SD-IR spectra were obtained after 13-point smoothing of the original IR spectra. 2D-IR correlation spectra were obtained by analyzing the series of temperature-dependent dynamic spectra, which were obtained after baseline correction and 13-point smoothing of the dynamic original spectra, with 2D-IR correlation analysis software programmed by Department of Chemistry of Tsinghua University (Beijing, China).

3. Results and discussion

Both near-infrared (NIR) and mid-infrared (MIR) spectroscopy have been considered as an alternative analytical method, easy to perform, avoiding problems associated with sample preparation, as well as possessing the advantage of determining several substances with a single measurement. Generally, the MIR reflects the nature of the molecular fundamental vibrations and the associated rotational–vibrational structure involving, while the NIR provides more complex structural information because it reflects the overtone or harmonic vibrations of bonds [18]. So NIR could only provide limited structural information and has poor fingerprint, especially when studying complicated system, such as TCM and their extract products. Since TCM contains hundreds of components and produces curative effects through synergic reaction, the specific markers identified by chromatography cannot fully reflect the real qualities of TCM. Therefore, MIR spectra are more suitable for the identification of TCM than NIR spectra.

In this article, the conventional transmission method is applied to analyze all the samples, which are prepared into tablets with KBr.

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