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A feasibility study on the non-invasive analysis of bottled Compound E Jiao oral liquid using near infrared spectroscopy

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ABSTRACT

The aim of this study was to explore the feasibility of near infrared (NIR) spectroscopy for the quantitative analysis of Compound E Jiao oral liquid, a popular oral TCM preparation, through intact glass vials. Transmission spectra were acquired with a sample holder tailored to the instrument's sample compartment. Total flavonoids, total saponins and soluble solid contents were considered as quality indicators of this oral preparation and calibration models were built using partial least-squares regression (PLSR) algorithm. The sample set was comprised of production samples as well as laboratory samples prepared by a concentrating-diluting method so as to extend the concentration range beyond the production variation. The developed models gave root mean square error of prediction (RMSEP) of 0.084 mg mL^{-1} for total flavonoids, 0.014 mg mL^{-1} for total saponins and 0.21% for soluble solid contents. To better evaluate the prediction performance, an independent set of ten production samples was introduced for the external validation. The models are compared with those built with the transflectance spectra collected in the destructive way. The results of paired *t*-test and *F*-test at a significance level of 0.05 revealed that the two sets of models exhibited comparable model performance. The overall results demonstrate that the proposed non-destructive method can be used as an advantageous alternative in the quality inspection of Compound E Jiao oral liquid production.

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1. Introduction

In the pharmaceutical industry, final products are commonly sealed in containers such as blisters, vials or bottles made of various materials [1]. Currently, well-established chromatographic techniques are widely adopted for quantitative analysis of these products. However, chromatographic analysis methods inevitably imply heavy chemical reagent consumption and prolonged analysis. Most importantly, these methods bear a destructive nature, which means that the samples employed for analysis are generally wasted afterwards. In light of this, a method enabling non-invasive analysis of the final products shall be indeed beneficial to pharmaceutical production and quality assurance operations.

Near-infrared (NIR) spectroscopy has gained wide acceptance in the pharmaceutical fields as it affords minimum sample preparation and rapid, simultaneous determination of several analytes from a single spectrum [2]. NIR spectroscopy assesses chemical structures by recording multi-frequency and co-frequency information of C–H, N–H, C–O and O–H bonds [3], while materials such as glass are fairly transparent or have a suitable window for the NIR radiation [4]. This particular feature of NIR spectroscopy opens up the possibility of carrying out non-invasive analysis of samples in unopened containers. Over the last several years, a few research groups have reported on the non-destructive measurement of pharmaceutical products in varied containers using NIR spectroscopy. Ali et al. [5] explored the capability of NIR spectroscopy to identify two benzodiazepine derivatives non-invasively through USP vials. Sanches et al. [6] proposed a method for fast near-infrared determination of dipyrone in injectable formulations without violation of the ampoule. Rodionova et al. [7] applied the NIR/chemometrics approach to the counterfeit detection of injectable dexamethasone formulations, which are aqueous solutions with low concentration of active ingredients, directly in the closed ampoules. Other researchers in the food industry also investigated the possibility of making non-destructive measurement of wine composition through containers such as bottles [8–11].

Compound E Jiao oral liquid, with its recognized curative effect of invigorating vital energy and nourishing blood, is a widely embraced Chinese patent drug. It is made from Shudihuang (prepared rhizome of Rehmannia), Shanzha (fruit of Crataegus

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pinnatifida), Dangshen (Radix Codonopsis) and Hongshen (Radix Ginseng Rubra) along with E Jiao (donkey-hide gelatin). Main active compounds in Compound E Jiao oral liquid include flavonoids and triterpenoid saponins. Due to its complex composition, quality control of this oral preparation remains an arduous task.

In the present work, NIR spectroscopy was applied for the determination of three quality indicators (*viz.* total flavonoids, total saponins and soluble solid contents) in Compound E Jiao oral liquid. A sample holder was manufactured, which is tailored to the 20 mL glass vial of the product and the instrument's sample compartment. The spectra were collected through the intact glass vials and the method was hence non-destructive. To the best of our knowledge, the NIR spectroscopy has not been applied to the non-destructive analysis of oral TCM preparations before.

2. Experimental

2.1. Samples preparation

A total of 200 Compound E Jiao oral liquid samples with an age range of three years (between August 2010 and March 2013) were kindly provided by the TCM manufacturer, Shandong Dong-e E-jiao Co., Ltd. (Shandong, China).

As known, calibrations based exclusively on production samples might suffer from insufficient variations in the present production plant environment. Here a set of prepared samples was introduced into the sample population to expand calibration range [12].

The 200 samples were first split into two groups. The first group with a total of 40 samples went directly to reference assays and spectral measurements, while the second group with the remaining 160 production samples were randomly divided into eight subgroups. Samples from the same subgroup were mixed together and concentrated to approximately half of their original volume with a Laborota 4000 rotary evaporator (Heidolph, Germany). The concentrated solutions were then diluted with ultrapure water several times, and after each dilution, a sample was collected. The dilution factors (final volume/water aliquot) were set at random in a bid to minimize inter-correlation [13]. In this way, a total of 78 samples were prepared. The total number of calibration samples, $N = 118$, of which 40 were production samples and 78 were prepared ones, offers sufficient concentration variations for subsequent modelling.

2.2. NIR spectra acquisition

The sample spectra were acquired in both transmission and transflectance mode using an Antaris II FT-NIR Analyzer (Thermo Fisher Scientific, USA).

The transflectance spectra were collected with the SabIR fiber optic probe and a transflectance adapter. Since the optic probe and its adapter can not be put directly into the glass vial, the vials had to be opened and the contents transferred to beakers for spectra acquisition, which makes this sampling technique destructive. On the other hand, the transmission spectra were collected using a sample holder. The sample holder (Fig. 1) was used to support the glass vial in the instrument's sample compartment during spectral acquisition. With this accessory, unopened Compound E Jiao oral liquid samples were mounted in the sampling module, and the optical pathlength equals the diameter of the glass vial. It is worth noting that apart from the sample holder, no additional hardware modifications to the original instrument configuration were attempted.

Spectra were measured at 4 cm^{-1} interval over the spectral region 4000–10,000 cm^{-1} for both the transflectance and the transmission mode. Each spectrum was an average of 128 scans with air as the background. All samples were equilibrated to room temperature (25 °C) prior to spectral acquisition. Instrument control and spectral acquisition were performed with the Result Integration software (Version 8.5, Thermo Fisher Scientific, USA).

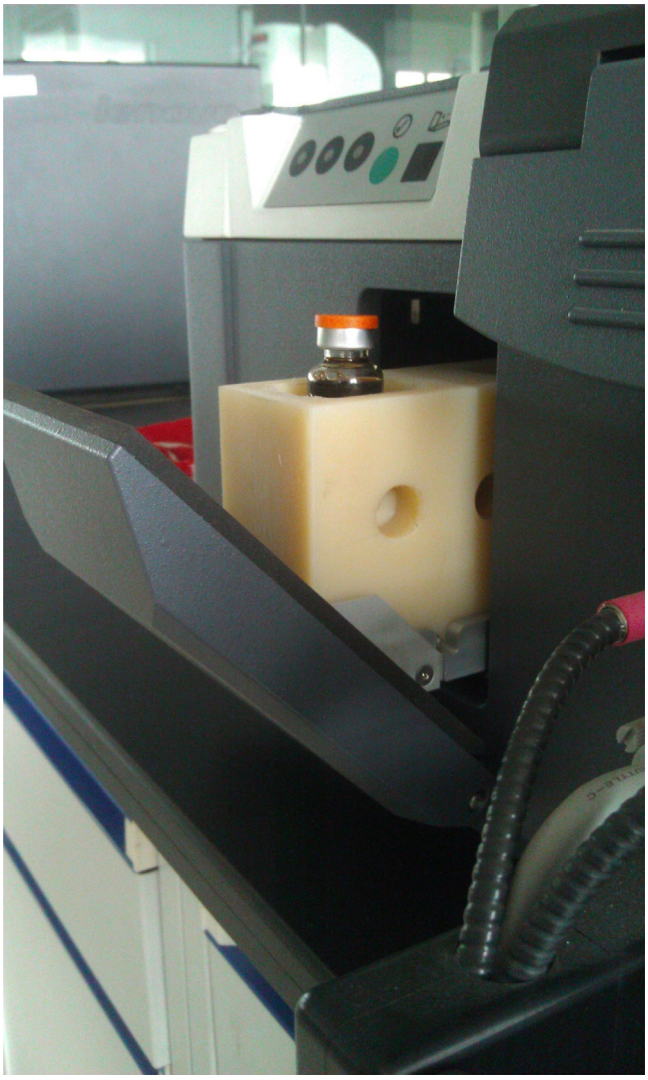

Fig. 1. NIR analyzer with the sample holder used to support the glass vial in the instrument sample compartment.

2.3. Reference assays

Immediately after spectral acquisition, total flavonoids, total saponins and soluble solid contents were determined by their corresponding reference methods. The quantification of total flavonoids and total saponins was performed using sodium nitrite-aluminium nitrate and vanillin-perchloric acid colorimetric method, respectively. Details of the colorimetric methods can be found in Refs. [14,15]. Soluble solid contents were determined using the loss-on-drying method [2] on a HG63 halogen moisture analyzer balance (Mettler Toledo, Swiss Confederation). Samples were heated at 90 °C to constant weight, and the results were expressed in wt.%. Results of reference assays are detailed in Table 1.

2.4. Division of sample set

Among the 118 samples, 10 production samples were used as an independent external validation set to assess the prediction ability of the established models. And the left 108 samples were split into

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