



Identification of authentic and adulterated Aquilariae Lignum Resinatum by Fourier transform infrared (FT-IR) spectroscopy and two-dimensional correlation analysis



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ABSTRACT

As a kind of expensive perfume and valuable herb, the commercial Aquilariae Lignum Resinatum (ALR) is often adulterated for economic motivations. In this research, Fourier transform infrared (FT-IR) spectroscopy and two-dimensional (2D) correlation analysis are employed to establish a simple and quick identification method for the authentic and adulterated ALR. In the conventional infrared spectra, the standard ALR has a strong peak at 1658 cm^{-1} referring to the conjugated carbonyl of resin, while this peak is absent in the adulterated samples. The position, intensity, and shape of the auto-peaks and cross-peaks of the authentic and adulterated ALR are much different in the synchronous 2D correlation spectra with thermal perturbation. In the range of $1700\text{--}1500\text{ cm}^{-1}$, the standard ALR has four obvious auto-peaks, while the strongest one is at 1659 cm^{-1} . The adulterated sample w-1 has three obvious auto-peaks and the strongest one is at 1647 cm^{-1} . The adulterated sample w-2 has three obvious auto-peaks and the strongest one is at 1519 cm^{-1} . The adulterated sample w-3 has four obvious auto-peaks and the strongest one is at 1690 cm^{-1} . The above auto-peaks confirm that the standard ALR contains a certain content of resin compounds, while the three counterfeits contain little or different resins. The results show the potential of FT-IR spectroscopy and 2D correlation analysis in the simple and quick identification of authentic and adulterated ALR.

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1. Introduction

Aquilariae Lignum Resinatum (ALR) is the resin-rich wood of *Aquilaria sinensis* (Lour.) Gilg [1]. It is not only a kind of expensive botanical perfume but also a kind of valuable traditional Chinese medicine. The formation of ALR usually takes decades or even hundreds of years. Therefore, the economically motivated adulterations of the expensive ALR are very common in the market. In order to ensure the efficacy and safety of ALR for medical applications, the first thing needed to do is to establish an accurate, quick, and convenient identification method to ensure the authenticity of ALR.

At present, some methods have been reported to identify ALR,

including origin identification, macroscopic identification, microscopic identification, and physicochemical identification. The first three of the above methods are greatly influenced by subjective factors. Physicochemical identification such as thin-layer chromatography (TLC) identification needs the preprocessing procedures, which are complicated and consume organic chemical reagents leading to pollution. So this method is not suitable for rapid analysis of a large number of samples.

The quick, easy, and nondestructive Fourier transform infrared (FT-IR) spectroscopy has been widely applied to the identification and quality control of traditional Chinese medicine [2–4]. This research is going to use the infrared spectroscopic tri-step identification approach to characterize the standard ALR and three counterfeits in order to establish a simple, convenient, and objective quality control method of ALR. At first, the standard ALR and three counterfeits are identified by FT-IR spectroscopy. Next, the second derivative infrared (SD-IR) spectroscopy and two-dimensional correlation infrared (2D-IR) spectroscopy are utilized

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to resolve the overlapped signals and reveal more differences of the authentic and adulterated ALR.

2. Experimental

2.1. Materials

The standard ALR (the resin-rich wood of *Aquilaria sinensis* (Lour.) Gilg) was purchased from National Institutes for Food and Drug Control of China (Batch No. 121222-201102). The three adulterated samples were purchased from the market.

2.2. FT-IR spectroscopy

Spectrum GX FT-IR spectrometer (PerkinElmer, USA) equipped with a deuterated triglycine sulfate (DTGS) detector was used. Each IR spectrum was recorded from an accumulation of 16 scans 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.

Each sample was grounded into powder, blended with KBr powder, then ground again and pressed into a tablet. The IR spectra of all samples were collected at room temperature. The software Spectrum v6.3 (PerkinElmer, MA, USA) was used to auto-correct the spectral baseline and calculate the derivative spectra.

2.3. 2D-IR spectroscopy

IR spectrometer is the same as Section 2.2. A CKW-II programmable temperature controller (Beijing Chaoyang Automatic Instrument Co., China) was used to perform the thermal perturbation. The temperature range is 50–120 $^{\circ}\text{C}$ with a heating rate of 2 $^{\circ}\text{C}/\text{min}$.

Sample preparation is the same as Section 2.2. The sample tablet was put into the sample pool of the temperature controller. The spectra at different temperatures were collected at an interval of 10 $^{\circ}\text{C}$. Each spectrum was recorded from an accumulation of 32

scans 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. 2D-IR spectra were calculated by the 2D-IR software developed by Tsinghua University (Beijing, China).

3. Results and discussions

3.1. FT-IR spectra of the standard and adulterated ALR

Fig. 1 shows the IR spectra of the standard ALR and the adulterated samples. Detailed peak positions and assignments are listed in Table 1. As shown in Fig. 1, the peak shape and peak position are obvious different among the standard and adulterated samples in range of 1800–1500 cm^{-1} . ALR and the three adulterated samples can be distinguished according to the fingerprints of their IR spectra.

The IR spectra of the standard ALR (Fig. 1-a) has strong peak at 1658 cm^{-1} which refers to the conjugated carbonyl of resin, which means that standard ALR is rich in resin compounds. In addition, the peak of aromatic skeletal vibration at 1511 cm^{-1} of the standard ALR (Fig. 1-a) is weak.

The adulterated sample w-1 (Fig. 1-b) has two peaks at 1623 cm^{-1} and 1508 cm^{-1} in range of 1800–1500 cm^{-1} , but the peak at 1658 cm^{-1} does not appear in this sample. This suggests that the sample w-1 contains little resin.

In the range of 1800–1500 cm^{-1} , the strongest peak at 1515 cm^{-1} appears in the IR spectrum of the adulterated sample w-2 (Fig. 1-c), while the peak at 1652 cm^{-1} is middle strong. It is significant that the sample w-2 has more aromatic compounds than the standard ALR.

The abnormal peak at 1695 cm^{-1} appears in the spectrum of the adulterated sample w-3 (Fig. 1-d), while the peak at 1658 cm^{-1} is absent. This indicates that the sample w-3 may be also rich in resin compounds, but the molecular structures of the resin compounds in the sample w-3 are different from those of the standard ALR.

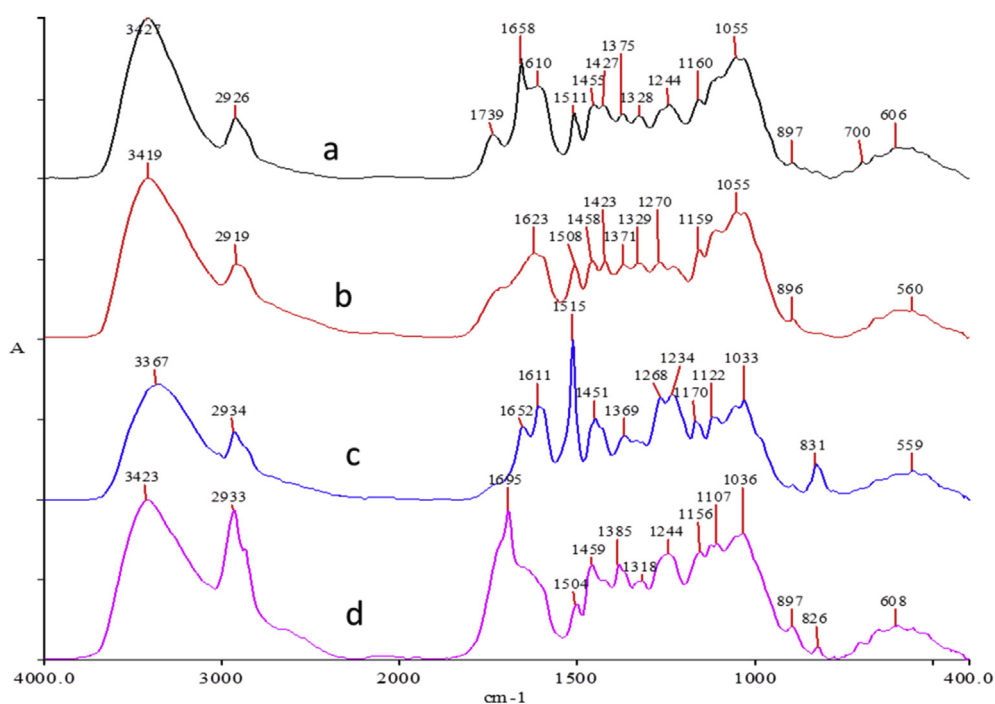


Fig. 1. FT-IR spectra of the standard and adulterated ALR. (a) The standard ALR; (b) the adulterated sample w-1; (c) the adulterated sample w-2; (d) the adulterated sample w-3.

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