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Short Communication: Material Characterisation

Determination of the content of *Eucommia ulmoides* gum by Variable Temperature Fourier Transform Infrared Spectrum

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ABSTRACT

Considering the rapidity and lesser sample amount required, the Fourier Transform Infrared Spectra (FTIR) was often used to quantitatively determine the rubber content of Russian dandelion, guayule, etc. This is because their chemical structure are *cis*-1,4-polyisoprene (CPI) which has a unique and isolate skeleton stretching vibration peak at 835 cm^{-1} band, and is convenient for determining the rubber content. However, this method is not suitable for *Eucommia Ulmoides (EU)* gum which will crystallize easily at room temperature due to the regular chemical structure of *trans*-1,4-polyisoprene (TPI), that will restrict the skeleton stretching vibration of TPI greatly. As a result, its FTIR spectrum only shows a very small peak at 845 cm^{-1} band hiding among a number of crystalline peaks around nearby, thus it cannot be used to determine the rubber content of *Eucommia ulmoides* oliv (*E. ulmoids*) directly. Actually, these crystalline peaks could be eliminated easily by elevating the temperature over 60 °C, and a unique and isolate skeleton stretching vibration characteristic band at 845 cm^{-1} was left alone in the TPI's FTIR spectrum which can be used expediently to determine the content of *EU* gum. At the present paper, the Variable Temperature Fourier Transform Infrared Spectroscopy (VTFTIR) was used to determine the rubber content of *E. ulmoides*. The results indicate that the rubber content of *E. ulmoides* samples (1–40, 5–35, 22–11) provided by Northwest Agriculture and Forestry University (NAFU) are 4.66%, 4.04%, 4.32% respectively, and the errors of average value were less than 5% compared with Soxhlet Extraction.

1. Introduction

With increasing of the people's awareness of environmental protection, oil based synthetic rubber (SR) is considering to be replaced by more renewable materials, such as natural rubber (NR), for carbon fixation [1,2]. However, the NR resources are also becoming scarce owing to the limited tropic rainforest planting condition and the increasing risk of infecting plant diseases and insect pests, especially the South American Leaf Blight (SALB) [3–5]. Therefore, some alternative NR resources, such as Russian dandelion, guayule and a special Chinese hard rubber——*Eucommia Ulmoides (EU)* gum, were paid attention by more and more researchers [6,7].

As a green renewable material, *EU* gum exists in many tissues of *Eucommia ulmoides* oliv (*E. ulmoids*), and the leaves contain about 2–5% [8,9], as shown in Fig. 1. Its chemical structure is *trans*-1,4-polyisoprene (TPI) which is the isomer of NR, whose chemical structure is *cis*-1,4-polyisoprene (CPI) [10,11], as shown in Fig. 2.

In order to meet the requirement of commercialization, people are now working hard to cultivate high-yielding rubber varieties, and the rubber content is a key index to evaluate the germplasm of rubber bearing plant. Therefore, a fast and high throughput determination method of rubber content is necessary for screening the high-yielding strains from millions of rubber bearing plant. Many methods, including soxhlet extraction, FTIR, NMR and NIR match ASE (Accelerated Solvent Extraction), have been set up to determine the rubber content of guayule, Russian dandelion and *E. ulmoides* [12–16]. Due to the soxhlet extraction is time consuming, the NMR and NIR match ASE are very expensive, so FTIR

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Fig. 1. *E. ulmoides* leaves (left) and *Eu* gum silks (right).

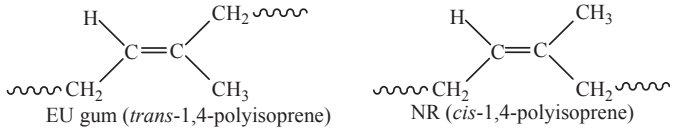

Fig. 2. Macromolecular structures of *EU* gum and NR.

was often used to determine the content of NR by virtue of its rapidity and the lesser sample amount required.

However, the content of *EU* gum cannot be determined by FTIR directly due to the skeleton stretching vibration band, which was used to determine the content of NR, was very weak and overlapped with other peaks caused by crystallization. At present paper, the authors try to establish a modified FTIR method to determine the content of *EU* gum by elevating the temperature over 60 °C, as a result, a unique and isolate skeleton stretching vibration peak at 845 cm^{-1} band is shown in the FTIR spectrum of *EU* gum and the testing results are satisfactory as well.

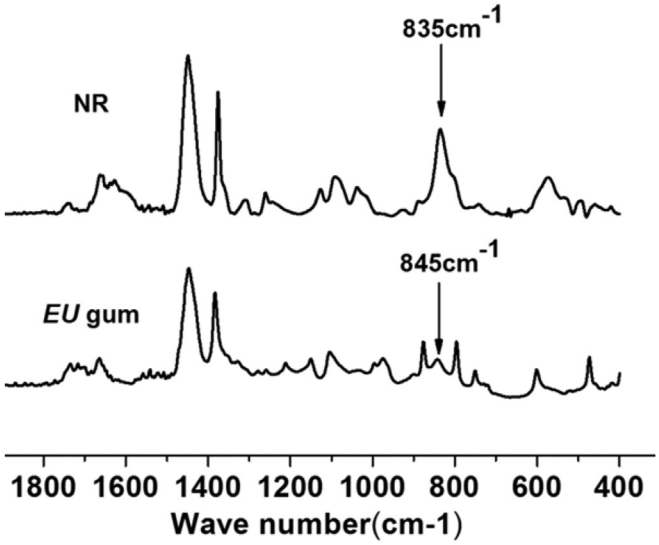

Fig. 3. Infrared spectra of NR and *EU* gum at room temperature.

2. Materials and methods

2.1. Chemical reagents and materials

Toluene, ethyl acetate, acetone and ethanol were purchased from Beijing Chemical Works (China), Polystyrene (PS) standard substance was purchased from China Institute of Metrology, TPI standard substance was purchased from Aldrich Co., Ltd (USA), *E. ulmoides* leaves (1–40, 5-35, 22–11) were provided by Northwest Agriculture and Forestry University (NAFU).

2.2. Sample preparation

Three kinds of *E. ulmoides* leaves were cleaned by deionized water and dried in the vacuum oven at 50 °C to constant weight, then grinded into powder under liquid nitrogen condition by Freeze Grinding Machine (Shanghai Jingxin Technology Co., Ltd Tissuelyser-24, China). Five spots method was adopted to put 100 mg *E. ulmoides* leaves powder into a 2 ml centrifuge tube, adding 1.5 ml acetone and shaking it up and down to remove the resin; putting the tube into the centrifugal machine (Anting Shanghai Scientific Instrument Factory, TGL-20B, China) for centrifuging at 10000 rpm for 3min, then dumping the upper waste liquid. This process was repeated until the liquid was colorless and the sample was dried to constant weight in a vacuum oven at 50 °C. Then *EU* gum was extracted by adding 500 μl toluene into the 2 ml centrifuge tube for centrifuging for 1 min at 10000 rpm, collecting the upper liquid into a 10 ml sample bottle, repeating this process at least five times to ensure all the *EU* gum could be extracted out. The sample concentration was approximately adjusted to 3 mg/ml to obtain a suitable FTIR curves, and 500 μl solution was taken from the sample bottle into a 1.5 ml PE tube by adding 50 μl external standard (PS/toluene solution, 5 mg/ml) for FTIR testing.

2.3. Variable temperature Fourier Transform Infrared Spectra (VTFTIR)

The rubber content of *E. ulmoides* leaves were determined by FTIR (BRUKER TensorII, USA) equipped with a high stability temperature controller (Specac 4000 Series ™, UK) and a variable temperature (VT) cell holder (P/N GS21525, UK), which is designed to allow for the study of solid and liquid samples over a wide temperature range from −190 °C to 250 °C. The FTIR spectra series were collected under transmission mode at a spectral reoslution of 4 cm^{-1} with 32 co-added scans in a wavenumber range 4000-400 cm^{-1}. The sample film was prepared by casting 150 μl sample solution onto a KBr window and volatilizing the solvent by an infrared lamp. The film was heated at a rate of 10 °C/min by using an electric heating element. The variation in temperature is achieved by holding the sample cell in close proximity to the heating device of the VT Cell. To eliminate the temperature difference between sample and the cell, an equilibration time interval of 5 min