Terahertz Spectra Study on Chemical Constituents from *Amalocalyx Yunnanesis*

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***Abstract*— In this work, we measured the absorption spectra of seven compounds isolated from *Amalocalyx yunnanensis* Tsiang (Apocynaceous) in the frequency range from 0.2 to 2.0 THz by terahertz time-domain spectroscopy (THz-TDS). From the observed characteristic absorption peaks, it is found that the luteolin (a), luteolin-7-*O*-*β*-D-glucopyranoside (b), amalogenin A (c) and amaloside B (d) show remarkable similarities and differences. This is because that (b) is a glycoside with aglycone (a), as well as (c) is the aglycone of (d). Accordingly, THz-TDS is an efficient method for identifying chemical constituents of natural products.**

I. INTRODUCTION

Terahertz is electromagnetic wave ranging from 0.1 to 10 THz, whose wavelength is between that of infrared and microwave. Terahertz time-domain spectroscopy (THz-TDS) is an effective means for detecting material spectra information in the THz region by employing ultrafast laser technology. It has a comparative high signal-to-noise ratio in a wider frequency band and can be used to detect material composition and subtle structural changes. The main advantage of THz spectroscopy is the ability to access directly information related to crystal lattices, which provides an effective method for molecular structure identification [1]. However, there is little research on chemical constituents of natural products by THz-TDS, which is of great significance for natural medicines.

Amalocalyx yunnanensis Tsiang (Apocynaceous) is a liana of genus *Amalocalyx* which is commonly used in Yunnan traditional medicine to promote milk secretiveness [2-4]. However, research on the bioactivity of this plant has not been reported up to now. In our previous study, we isolated ten compounds from *A. yunnanensis*, two compounds showed significant cytotoxic effects against MDA-MB-231 cells and A549 cells. To characterize and quantify the active pharmaceutical ingredients, we measured the absorption spectra of seven compounds isolated from *A. yunnanensis* in the frequency range from 0.2 to 2.0 THz by THz-TDS. In this paper, we give the results of two flavones and two steroidal constituents.

II. RESULTS

The leaves and twigs of *A. yunnanensis* were collected from Xishuangbanna Tropical Botanical Garden (XTBG), Chinese Academy of Sciences (CAS), Yuannan Province, P. R. China. Air-dried and powdered twigs and leaves were extracted with 95% aqueous EtOH (4 × 10 L) at room temperature and evaporated under vacuum. The crude extract was partitioned with H_2O and extracted with EtOAc. The EtOAc extract was isolated and purified by repeated column chromatography (CC), thin-layer chromatography (TLC) and high-performance liquid chromatography (HPLC) using various materials including positive phase silica gel, reverse phase silica gel, polyamide and Sephadex LH-20, to afford pure compounds.

All the compounds isolated from *A. yunnanensis* were put into a vacuum drying oven with a temperature of 50 °C, and dried about two hours until they were completely dry. Then the powder-form samples were pressed into a thin round slice with a tablet press under a pressure of about 4t. The diameter of a sample slice was about 13 mm and the thickness was between 1.0 mm and 1.2 mm. To reduce the influences of multiple reflections, the slice surface was kept smooth and without cracks. The THz spectra were acquired on the Teraview TPS3000 THz–TDS system at room temperature.

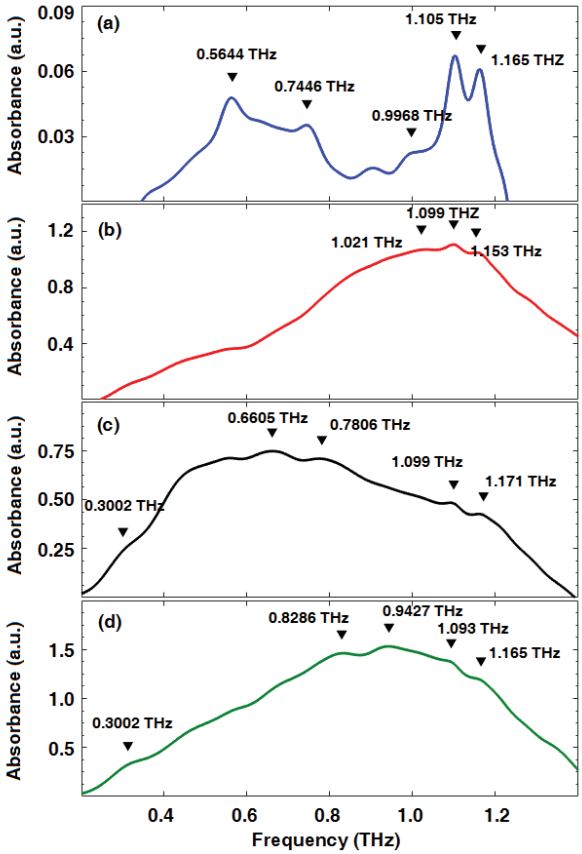

Figure 1. Absorbance spectra of compounds (a), (b), (c) and (d). The low signalto-noise ratio may be caused by the low amount of the isolated compounds (< 30 mg). And the peaks around 1 THz may be caused by water.

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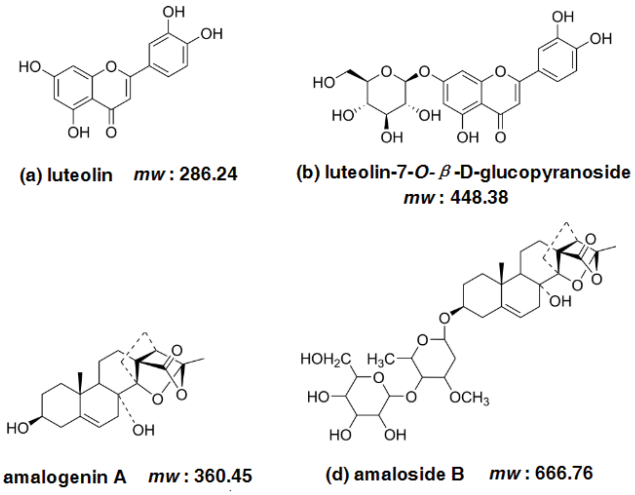

Figure 2. Structures of compounds (a), (b), (c) and (d).

For the complex hydrogen bond networks in the crystals, resulting from a large number of hydroxyl groups in the molecules, the absorbance spectra can be observed by THz-TDS. As shown in Fig. 1, some of these absorption peaks of compound (a) and (b) are in accordance with each other, for the same skeleton structures (Fig. 2). Meanwhile, compound (b) is composed of a D-glucose and compound (a), by the sugar linked to the hydroxyl at C-7 of the aglycone with a β-glycosidic bond. And then compound (b) is available to form an intramolecular hydrogen bond, while there are only van der Waals interaction and intermolecular hydrogen bond in compound (a). Accordingly, there appear different peaks between the compound (a) and (b). For the same reason, compound (c) and (d) present similar results.

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