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Journal of Pharmaceutical and Biomedical Analysis 43 (2007) 1270–1276
JOURNAL OF PHARMACEUTICAL AND BIOMEDICAL ANALYSIS
www.elsevier.com/locate/jpba

Quantitative and qualitative determination of six xanthones in *Garcinia mangostana* L. by LC–PDA and LC–ESI-MS

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Received 1 May 2006; received in revised form 4 October 2006; accepted 17 October 2006
Available online 28 November 2006

Abstract

A new method was developed for the simultaneous analysis of six naturally occurring xanthones (3-isomangostin, 8-desoxygartanin, gartanin, α-mangostin, 9-hydroxycalabaxanthone and β-mangostin). The quantitative determination was conducted by reversed phase high performance liquid chromatography with photodiode array detector (LC–PDA). Separation was performed on a Phenomenex Luna C18(2) (150 mm × 3.00 mm, 5 μm) column. The xanthones were identified by retention time, ultraviolet (UV) spectra and quantified by LC–PDA at 320 nm. The precision of the method was confirmed by the relative standard deviation (R.S.D.), which was ≤4.6%. The recovery was in the range from 96.58% to 113.45%. A good linear relationship was established in over two orders of magnitude range. The limits of detection (LOD) for six xanthone compounds were ≤0.248 μg/mL. The identity of the peaks was further confirmed by high performance liquid chromatography with time-of-flight mass spectrometry (LC–TOF MS) system coupled with electrospray ionization (ESI) interface. The developed methods were applied to the determination of six xanthones in *Garcinia mangostana* products. The satisfactory results showed that the methods are effective for the analysis of real samples.

Keywords: Xanthone; Mangostin; *Garcinia mangostana*; Mangosteen; LC–PDA; LC–ESI-MS

1. Introduction

The mangosteen (*Garcinia mangostana* L.), belonging to the family Guttiferae, is a tropical evergreen tree. Its origin is in Southeast Asia. It can now be found in Northern Australia, Brazil, Central America, Hawaii, Southern India, Indonesia, Malaysia, Thailand, and other tropical countries. The edible fruit is deep reddish purple when ripe. In Asia, it is known as the "Queen of Fruits" due to its pleasant flavor [1,2].

The fruit hull of mangosteen has been used for hundreds of years in Southeast Asia as a medicine for skin infection, wounds, dysentery and diarrhea [1–3]. Recently other interesting properties of mangosteen are slowly being revealed. Its rind is about the fourth of an inch in thickness. It contains high amounts of xanthones, a class of polyphenolic compounds. Xanthones have antioxidant [4–7], antibacterial [8–10], antifungal [11], antiinflammatory [3,12,13] antitumor [5,14–21], antiplatelet aggregation [22], antithrombotic [23] and vasorelaxant activities [24], prevent oxidative damage of LDL [4], histamine and serotonin receptor blockers [25,26], and also inhibit HIV [27]. However, there is no data from clinical trials to verify these effects in humans. The health benefits of mangosteen need further to be proven scientifically.

Mangosteen has been used as an ingredient in several popular commercially available nutritional supplements, including Vemma and Xango, now. It can be purchased online and in herbal shops. For safety and efficiency, it is important to set up the method to control the quality. There is only one paper published using gas chromatography method to detect trimethylsilyl ethers of xanthones from *G. mangostana* [28]. The purpose of this study is to set up a new method for quality and quantity determination of xanthones in hull of mangosteen.

Herein a simple high performance liquid chromatography with photodiode array detector (LC–PDA) method is reported

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doi:10.1016/j.jpba.2006.10.018

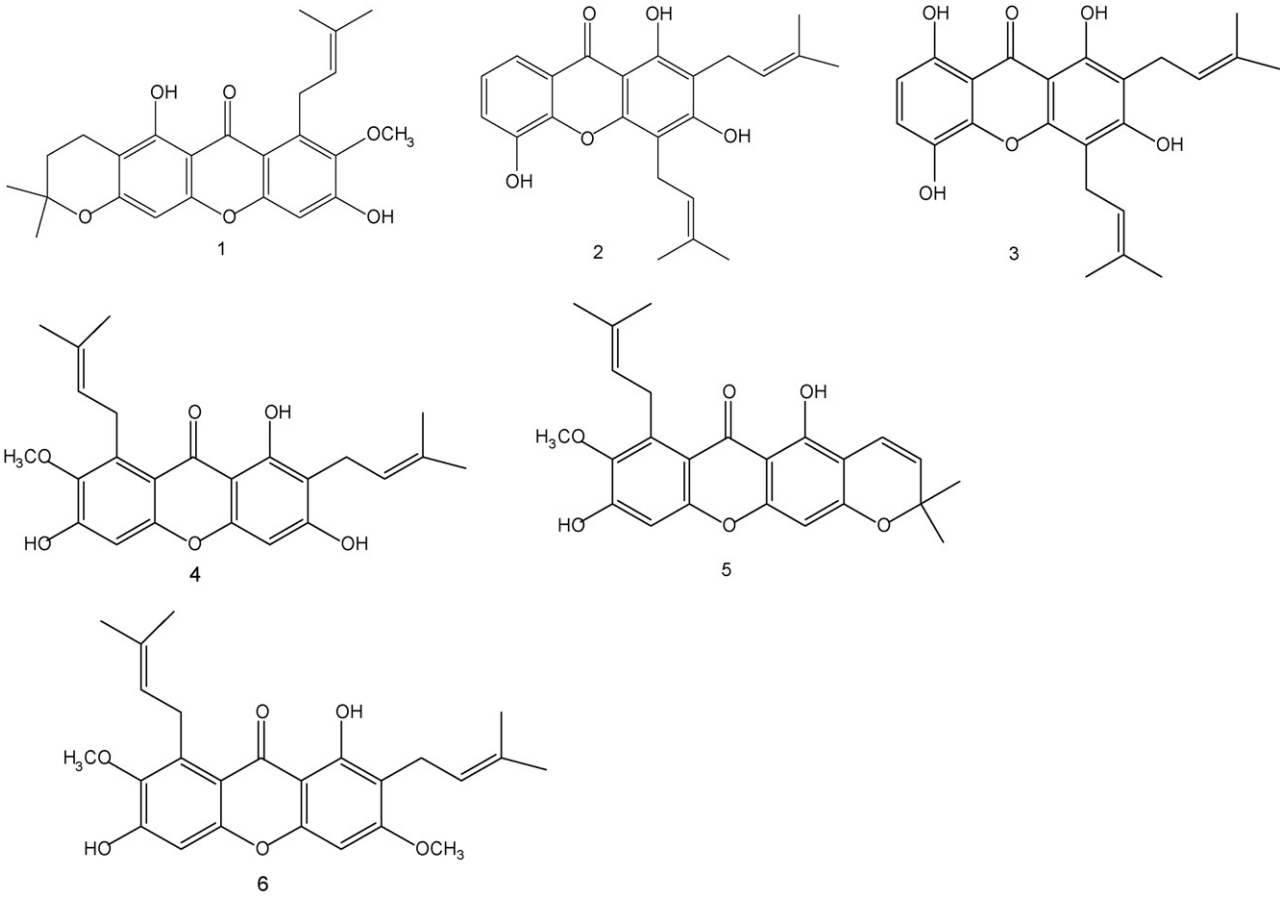

Fig. 1. Structures of xanthones: (**1**) 3-isomangostin; (**2**) 8-desoxygartanin; (**3**) gartanin; (**4**) α-mangostin; (**5**) 9-hydroxycalabaxanthone; (**6**) β-mangostin.

which detects and quantifies six xanthones (3-isomangostin (**1**), 8-desoxygartanin (**2**), gartanin (**3**), α-mangostin (**4**), 9-hydroxycalabaxanthone (**5**) and β-mangostin (**6**)) (Fig. 1). Xanthone peaks were further confirmed by high performance liquid chromatography with mass spectrometry coupled with electrospray ionization interface (LC–ESI-MS). The methods were applied for four products with satisfactory results obtained.

2. Experiment

2.1. Reagents and materials

Methanol, acetonitrile, isopropanol, reagent alcohol, acetone, ethyl acetate, water and acetic acid are HPLC grade (Fisher Scientific, Fairlawn, NJ). Formic acid and trifluoroacetic acid (TFA) are reagent grade from Sigma–Aldrich (Steinheim, Germany).

XTerra RP18 (150 mm × 4.6 mm, 5 μm, Waters), Gemini C18 (150 mm × 3.00 mm, 5 μm, Phenomenex), Luna 18(2) (150 mm × 3.0 mm, 5 μm and 250 mm × 4.6 mm, 5 μm, Phenomenex), and Luna C(8) (150 mm × 3.00 mm, 3 μm, Phenomenex) were used.

The standard compounds **1**–**6** were purchased from ChromaDex Inc. (Santa Ana, CA). The purity of these standard compounds was calculated (88.08%, 99.31%, 99.98%, 97.03%, 87.94%, 98.96%, respectively) by relative area percentage with chromatogram at Max plot using the same LC condition as sample detection. Four products were obtained from commercial source, which claim to be dried powder extract of pericarp of *G. mangostana* with different content of α-mangostin.

2.2. Sample preparations

In order to perform the determinations, about 0.1–0.2 g powdered products were weighed separately and then transferred to the separate centrifuge tubes, filled by 3 mL of acetone as extraction solvent. All the samples were vortexed and then sonicated for 20 min at room temperature. After centrifugation of the sonicated sample, the supernatant was transferred to a 10-mL volumetric flask. This procedure was repeated twice with the corresponding supernatants transferred to the corresponding 10-mL volumetric flask. The samples were then diluted to the final volume with acetone. Prior to injection, each sample was filtered through a 0.45 μm nylon membrane filter.

2.3. LC–PDA analysis

A Waters 2695 Alliance Separations Module equipped with a 996 PDA detector (Waters, Milford, MA) was used. Separation was achieved on a 150 mm × 3.0 mm, 5 μm Luna C18(2) column (Phenomenex, Torrance, CA). The mobile phase consisted of water with 0.1% TFA (A), methanol with 0.1% TFA (B) and isopropanol (C), which were applied in the following gradient