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Research paper

Determination of the concentration of major active anti-emetic constituents within commercial ginger food products and dietary supplements

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ABSTRACT

Introduction: Studies suggest that the bioactive compounds contained within the rhizome of ginger (*Zingiber officinale*) could exert a beneficial effect on the symptoms of several chronic conditions (e.g. diabetes, arthritis) and in the reduction of nausea. However, it is unknown if ginger supplements and food products contain sufficient quantities of the necessary active ingredients to achieve a therapeutic effect. This study analyzed twenty commercially available ginger products including ginger dietary supplements, ginger spices (ground dried ginger), and ginger-containing drinks and food products and determined the concentration of [6]-, [8]- and [10]- gingerol and [6]- and [10]-shogaol.

Methods: The samples were extracted prior to separation by Reversed Phase-High Performance Liquid Chromatography (RP-HPLC) and detected by ultraviolet (UV) spectrophotometry.

Results: Considerable variation between individual items within each food type was observed. Per gram, ginger supplements, particularly the standardized extracts, contained the greatest concentration of measured compounds (10.08 ± 7.92 mg, mean ± standard deviation), while the concentration of compounds within spices (9.29 ± 6.73 mg), beverages (1.77 ± 1.06 mg), confectionery (0.43 ± 0.32 mg), and teas (0.13 ± 0.00 mg) was considerably lower. When the concentration of compounds was measured per standardized serve, four ginger confectionery and beverage products contained total gingerol and shogaol concentrations that were similar to the analyzed dietary supplement.

Conclusion: Of the twenty commercially available ginger products examined, those with the highest content of active, antiemetic constituents were the standardized ginger extracts and supplements although ginger spices also showed high levels of active constituents per serve. In addition, standard deviation reveals a large variation within each product type.

1. Introduction

The rhizome of ginger (*Zingiber officinale*) contains many bioactive compounds. The gingerol class of compounds is the primary bioactive agent within the non-volatile, pungent component of ginger. Shogaols are the degradation product of gingerols. These are present in low concentrations in fresh ginger but increase when gingerols are exposed to heat, acid or other conditions which allow the alkene to form (an elimination product) [2]. The shogaols and gingerols are differentiated by the presence of either an aliphatic hydroxyl group, beta to the carbonyl (gingerols) or elimination of the hydroxyl group to form a double bond (shogaols) on the alpha carbon to the aliphatic carbonyl.

These compounds have been studied in clinical and pre-clinical studies for their effect on several chronic conditions such as diabetes and arthritis [1,10,13]. The potentially beneficial effect of ginger on nausea has also been an area of significant research interest. A growing body of clinical trials has provided preliminary support for its use in multiple types of nausea such as motion sickness, morning sickness and chemotherapy-induced nausea and vomiting [11,12,19,21]. Studies that have investigated the antioxidant, anti-inflammatory, and chemo-preventive effect of individual compounds contained in ginger have reported different levels of activity depending on the chain length and presence of an alpha, beta-unsaturated ketone group. For example, when several gingerol and shogaol compounds were compared, Dugasini et al. reported [6]-shogaol to be the most potent inhibitor of

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inflammation and reactive oxygen species production and [10]-gingerol to be the most potent gingerol [5].

Because of purported medicinal effects, ginger products are often used by the general population as complementary medicines and are sometimes recommended by healthcare professionals as adjuvants to standard therapy [6,9,14]. However, there are currently few studies that have investigated the concentration of active compounds in a wide-variety of commercially available products, and thereby providing a reliable guide to appropriate use of these products [16,17]. Due to the increasing public use of complementary treatments such as dietary supplements, information regarding the potency of available ginger preparations will also be of interest to healthcare professionals seeking these products for their adjuvant medicinal properties to determine their potential to produce side effects and interactions. Due to the differing biological activity of the gingerol and shogaol compounds, it is also prudent to measure the concentration of each of these individual compounds within commonly consumed ginger products.

The aim of this study was to determine the concentration of the primary bioactive compounds within an expanded range of commercially available ginger products, including dietary supplements and ginger-containing drinks and food products using Reverse Phase-High Performance Liquid Chromatography (RP-HPLC). Using the results of this analysis, the clinical and research implications of the therapeutic use of ginger products will be discussed.

2. Methods

2.1. Chemicals and materials

HPLC-grade water, methanol, ethyl acetate, and acetonitrile were purchased from ThermoFisher (Massachusetts, USA) and Sigma Aldrich (Missouri, USA). [6]-, [8]- and [10]- gingerol and [6]- and [10]-shogaol standards were purchased from Chromadex (Irvine, CA, U.S.A). Ginger products were purchased from one local supermarket (Gold Coast, Australia) and one online store (based in New Zealand) in April 2014. In addition, one supplement was supplied by the respective manufacturer for use in this study. In total, 20 products were purchased, including dietary supplements, beverages, spices (ground ginger), teas, and confectionery.

2.2. Sample preparation

Due to the variety of types of ginger products analyzed, two extraction protocols were required. All samples were prepared in triplicate. Percentage yield was determined by conducting each extraction protocol with a 0.08 mg/mL standard mix.

2.3. Ethyl acetate extraction

In order to analyze the ginger supplements, biscuit, and spices, ethyl acetate (10 mL) was added to 500 mg samples of spices, pierced supplements or crushed biscuit.

Samples were vortexed then sonicated for 30 min using a CamLab TransSonic T310 sonicator. Samples were centrifuged (20 min at 2500 rpm and 25 °C) using a Beckman Coulter Allegra X-15R centrifuge. The supernatant was subjected to second pass extraction using an additional ethyl acetate (10 mL). The supernatants of both extractions from each product were combined and evaporated to dryness. The samples were reconstituted in methanol (1.5 mL) and stored at 4 °C.

Serving sizes for the supplements and biscuit were in accordance with suggested serving sizes by the manufacturer. The serving size for the spices was set at 0.2 g.

2.4. Liquid/liquid extraction

Beverage samples (50 mL, degassed) and confectionery samples (500 mg) were diluted in HPLC water (15 mL). For the tea products, three tea bags were infused in HPLC-grade water (50 mL, room temperature) for three minutes. All samples were left overnight and then extracted in ethyl acetate (10 mL). A second pass extraction was conducted. The supernatants of both extractions from each product were combined and evaporated to dryness. The samples were reconstituted in methanol (1.5 mL) and stored at 4 °C.

The serving size of the beverages was set at 250 mL and confectionery serving size was defined as 5 g, as this was found to be the approximate weight of a single piece of confectionery.

2.5. Standard preparation

Stock solutions (10 mL) of each standard were prepared from the 5 mg material supplied by the manufacturer Chromadex (Irvine, CA, U.S.A). A dilution series of the standard mix were prepared as needed between 0.50 ug/mL to 200 ug/mL. Working standards were prepared in the range of 0.0005–0.2 mg/mL and stored at 4 °C.

2.6. HPLC analysis

Ginger samples were separated on a Waters Alliance e-2695 Separations System RP-HPLC and detected with a 2489 Dual-Beam UV detector. A 150 × 4.6 mm C-18 reversed phase column (Luna C18 5 μM; Phenomenex, USA) was fitted with a guard column.

The mobile phase consisted of HPLC-grade water (A) and acetonitrile (B) at starting conditions of 90% A. Analytical conditions included an injection volume of 10 μL, flow rate of 1.5 mL per minute and a column temperature of 27 °C. A binary gradient elution system was applied as follows: 0.0–1.85 min, 10–50% B; 1.86–7.88 min, 55% B; 7.89–11.59 min, 66% B; 12–17.6 min, 100% B. 17.61–25 min 10% B. The UV absorbance was measured at 205 nm. Peak identification was based on the retention time of the standards.

3. Results

Calibration curves for [6]-gingerol, [6]-shogaol, [8]-gingerol, [10]-shogaol, and [10]-gingerol were linear between 0.5 to

Table 1
Correlation coefficient and Limits of Detection/Quantitation for each component.

Ginger component	Coefficient of determination (R[2])	Limit of Detection (LoD)	Limit of Quantitation (LoQ)
[6]-Gingerol	0.9995	7.05×10^{-3} ug/mL	21.4 ug/mL
[8]-Gingerol	0.9996	6.77×10^{-3} ug/mL	20.5 ug/mL
[10]-Gingerol	0.9994	8.68×10^{-3} ug/mL	26.2 ug/mL
[6]-Shogaol	0.9993	7.71×10^{-3} ug/mL	23.4 ug/mL
[10]-Shogaol	0.9992	8.47×10^{-3} ug/mL	25.7 ug/mL