Food Chemistry 171 (2015) 405–411

Contents lists available at ScienceDirect

Food Chemistry

journal homepage: www.elsevier.com/locate/foodchem

Analytical Methods

# Chemical fingerprint analysis for quality control and identification of Ziyang green tea by HPLC

Xiaoye He [a], Jianke Li [a,*], Wei Zhao [a], Run Liu [a], Lin Zhang [a], Xianghong Kong [b]

[a] College of Food Engineering and Nutritional Science, Shaanxi Normal University, Xi'an, Shaanxi 710062, PR China
[b] Entry-Exit Inspection and Quarantine Bureau of Shaanxi, Xi'an, Shaanxi 710068, PR China

ARTICLE INFO

*Article history:*
Received 28 March 2014
Received in revised form 1 September 2014
Accepted 6 September 2014
Available online 16 September 2014

*Chemical compounds studied in this article:*
Epigallocatechin gallate (PubChem CID: 65064)
Epigallocatechin (PubChem CID: 72277)
Epicatechin gallate (PubChem CID: 107905)
Epicatechin (PubChem CID: 72276)
Gallocatechin gallate (PubChem CID: 199472)
Gallocatechin (PubChem CID: 9882981)
Catechin (PubChem CID: 9064)
Gallic acid (PubChem CID: 370)
Chlorogenic acid (PubChem CID: 1794427)
Caffeine (PubChem CID: 2519)

*Keywords:*
Ziyang green tea
Chemical fingerprint
HPLC
Similarity analysis
Quantitative determination

ABSTRACT

A simple and reliable HPLC fingerprint method was developed and validated for the quality control and identification of Ziyang green tea. Ten batches of Ziyang green tea collected from different plantations in Shaanxi Ziyang of China were used to establish the fingerprint. The feasibility and advantages of the used HPLC fingerprint were verified for its similarity evaluation by systematically comparing chromatograms with professional analytical software recommended by State Food and Drug Administration (SFDA) of China. The similarities of the fingerprints of 10 batches of tea samples were all more than 0.981. Additionally, simultaneous quantification of 10 major bioactive ingredients in the tea samples was conducted to interpret the consistency of the quality test. The results indicated that the HPLC fingerprint as a characteristic distinguishing method combining similarity evaluation and quantification analysis can be successfully used to assess the quality and to identify the authenticity of Ziyang green tea.

© 2014 Elsevier Ltd. All rights reserved.

## 1. Introduction

Tea is the most widely consumed beverage in the world which ranks after water (Wang et al., 2013) and is especially popular in Asian countries (Lee et al., 2006). The chemical composition of tea is complex and includes polyphenols, alkaloids (caffeine, theophylline and theobromine), amino acids, carbohydrates, proteins, chlorophyll, volatile compounds, minerals, trace elements and other unidentified compounds (Karori, Wachira, Wanyoko, & Ngure, 2007). Among these compositions, polyphenols constitute the most interesting group and are the main bioactive molecules found in tea (Cabrera, Giménez, & López, 2003). The major polyphenolic compounds in tea are flavan-3-ols (i.e. catechins) which include: (+)-catechin (C), (−)-epicatechin (EC), (−)-epigallocatechin (EGC), (−)-epigallocatechin gallate (EGCG), (−)-gallocatechins (GC), (−)-epicatechin gallate (ECG), and (−)-gallocatechin gallate (GCG) (Kerio, Wachira, Wanyoko, & Rotich, 2013). The tea beverage has been continuously considered as a medicine since the ancient times because of its polyphenols. Numerous studies have recorded the beneficial effects of tea, which include antioxidant (Vinson & Dabbagh, 1998), anti-carcinoma (Sadzuka, Sugiyama, & Sonobe, 2000), anti-inflammation (Karori, Ngure, Wachira, Wanyoko, & Mwangi, 2008), and antimicrobial properties (Vaquero, Alberto, & Maca de Nandra, 2007). Therefore, tea appears to be an effective chemopreventive agent for toxic chemicals and carcinogens.

Over the last decade, consumers have shown strong interest in foods with identified place of origin (Luykx & van Ruth, 2008). This has brought more and more products with geographical

---

* Corresponding author. Tel.: +86 29 85310519; fax: +86 29 85310517.
*E-mail address:* jiankel@snnu.edu.cn (J. Li).

http://dx.doi.org/10.1016/j.foodchem.2014.09.026
0308-8146/© 2014 Elsevier Ltd. All rights reserved.

indications to the market. Therefore, the determination of geographical origin has become increasingly essential (Cordella, Moussa, Martel, Sbirrazzuoli, & Louisette, 2002; Luykx & van Ruth, 2008). One geographically specific selenium-enriched Ziyang tea (*C. sinensis* L.) is widely distributed in the seleniferous region, in Ziyang County, Shaanxi province, China (Wang, Zhao, Sun, & Yang, 2014). The selenium-enriched tea is currently being explored as one promising dietary selenium supplement (Fang, Wu, & Hu, 2003). Interestingly, selenium-enriched green tea is shown to exhibit significantly higher antioxidant activity than regular green tea via *in vitro* experiments (Li et al., 2008).

In the event of frauds or commercial disputes, it is necessary to introduce sensitive, accurate and fast detection methods as the supplement of the food traceability system to prevent and verify the fake information and products (Zhang, Zhang, Dediu, & Victor, 2011). Chromatographic fingerprint, a comprehensive and quantifiable identification method, is able to reveal chemical information of botanical products, and therefore could be applied in food adulteration detection (Ma et al., 2011). It has the potential to characterise both the marker components and the unknown components in a complex system (Li et al., 2010). Both U.S. Food and Drug Administration (FDA) (FDA, 2004) and European Medicines Agency (EMEA) (EMEA, 2006) have recommended this strategy to assess the quality and consistency of botanical products. State Food and Drug Administration of China (SFDA) has also required that all the injections made from herbal medicines be standardised by chromatographic fingerprinting (SFDA, 2000). Moreover, SFDA has also suggested that all of herbal chromatograms should be evaluated by their similarities, a commonly employed approach based on calculating the correlative coefficient of original data (Gong, Liang, Xie, & Chau, 2003). Among the chromatographic fingerprinting applied to the authentication and qualitative evaluation of botanical products over the past decade (Chen, Qi, & Shi, 2013; Ding et al., 2011; Liu, Wu, Yang, Ding, & Wu, 2013; Zhang, Cui, He, Yu, & Guo, 2005), high performance liquid chromatography (HPLC) fingerprint emerges to be the most widely used method attributed to its convenience and efficiency. Also HPLC is by far the most acceptable method for the analysis of tea catechins, gallic acid, purine alkaloids, theanine, etc. (Peng, Song, Shi, Li, & Ye, 2008).

According to the best of our knowledge, there are no reports regarding the fingerprint analysis of Ziyang green tea. The objective of this study was to establish an effective HPLC fingerprint method for the identification and quality evaluation of Ziyang green tea. The chromatograms of the extracted samples from different tea plantations of Shaanxi Ziyang were compared visually and analysed by similarity evaluation. Moreover, ten components in ten batches of Ziyang green tea were simultaneously quantitated by HPLC method.

## 2. Materials and methods

### 2.1. Chemicals and solutions

HPLC grade methanol and trifluoroacetic acid (TFA, purity $\geqslant 99\%$) were obtained from Fisher Scientific International Inc. (Fair Lawn, New Jersey, USA) and Tokyo Chemical Industry Co., Ltd. (Shanghai, China), respectively. Analytical-reagent grade anhydrous ethanol was obtained from Tianjin Fuyu chemical Co., Ltd. (Tianjin, China). Deionised water used for all the solutions and dilutions was prepared by Ultrapure Water Polishing System of Shanghai Moller scientific instrument Co., Ltd. (Shanghai, China). Ten chemical standards were purchased from Sigma–Aldrich Co. (St. Louis, Missouri, USA), which are (−)-epigallocatechin gallate (EGCG), (−)-epigallocatechin (EGC), (−)-epicatechin gallate (ECG), (−)-epicatechin (EC), (−)-gallocatechin gallate (GCG), (−)-gallocatechin (GC), (+)-catechin (C), gallic acid (GA), chlorogenic acid (CA), and caffeine (CAF). The purity of these standards was all above 98%.

Stock solutions (2 mg/mL) of the ten marker compounds were prepared in methanol and stored at 4 °C in darkness for further analysis. Standard solutions were prepared by serial dilution of the stock solution to the mobile phase working range of each substance. The concentration of mixed standard solution was selected according to the level of the components that are expected in the tea samples. The standard solutions were filtered through 0.45 μm filters prior to HPLC analysis.

### 2.2. Apparatus and chromatographic conditions

Waters 1525 HPLC system (Waters Corp., Milford, Massachusetts, USA) consisted of a binary pump, an ultraviolet detector (Waters 2487) and a column temperature controller. System control and data analysis were processed with Waters Empower 2 software. The chromatographic separation was performed on an Agilent Zorbax SB-C18 column (5 μm, 4.6 mm × 250 mm) using methanol-0.1% TFA solution (solvent A; 5:95, v/v; pH 2.28) and methanol (solvent B) as mobile phase at a flow rate of 1 mL/min. The gradient programme was set as follows: 0–10 min, 0–19% B; 10–20 min, 19–22% B; 20–30 min, 22–28% B; 30–35 min, 28–35% B; 35–40 min, 35–35% B; 40–50 min, 35–65% B; 50–60 min, 65–0% B. The chromatogram was monitored at a wavelength of 278 nm during the experiment. The column temperature was maintained at 30 °C and the injection volume of each sample and standard solution was 20 μL. The HPLC mobile phase was prepared fresh daily, filtered through a 0.45 μm membrane filter and then degassed before injecting.

### 2.3. Plant materials and sample preparation

Ten batches of fresh tea leaves were collected from different areas of Ziyang in Shaanxi province in May, 2013. Among these ten batches of fresh tea leaves, Samples 1–4 were collected from four tea plantations in Hongchun Town, Sample 7 and 8 were obtained from two tea plantations in Dongmu Town, Sample 5, 6, 9 and 10 were picked from tea plantations in Gaoqiao Town, Haoping Town, Shuanghe Town, and Huan'gu Town, respectively. The breed of all the tea trees was Ziyang population, which was a large group distributed most widely in Shaanxi province.

Through basic production process of making green tea (including de-enzyming, rolling, and drying), the dried green tea bud leaves were prepared. Each of the dried samples was pulverised, and then sifted through a 250 μm sieve. 1.0 g dried power was precisely weighed and then put into a 100 mL conical flask with cover. Approximately 25 mL of 50% ethanol solution was added to the flask. Followed by heating in 80 °C water bath for 20 min, the extracted solution was cooled to room temperature and was filtrated through an analytical filter paper. The filtered solution was then vacuum-dried at 40 °C and the dried extract was dissolved in 100 mL 35% methanol solution. The sample was filtrated through a 0.45 μm membrane filter prior to HPLC analysis.

### 2.4. Standard curves, limits of detection and recovery rates of quantitative analysis

The standard curves were obtained by plotting the peak area against nominal concentration of each compound and were fitted to a linear function of type $y = ax + b$. In this equation, $y$ and $x$ represent peak area and nominal concentration in mg/L, respectively. The limit of detection (LOD) was estimated as the minimum concentration of the compounds needed to produce signals that were

Download English Version:

https://daneshyari.com/en/article/7594782

Download Persian Version:

https://daneshyari.com/article/7594782

Daneshyari.com