Analysis of Volatile Components in *Cinnamomum cassia* using Gas Chromatography-Mass Spectrometry and the Effect of Sample Preparation

by

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Master of Science

Institute of Chinese Medical Sciences

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Supervisor

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碩士學位論文

肉桂揮發性成份的氣相色譜分析及樣品製備方法的影響

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University of Macau

Abstract

Analysis of Volatile Components in *Cinnamomum cassia* using Gas Chromatography-Mass Spectrometry and the Effect of Sample Preparation

Lv Guangping

Supervisor: Prof. Li Shaoping

Cortex Cinnamomi (CC), *Rougui* in Chinese, has long been regarded as a medicinal plant and spice in China. *Cinnamomum cassia*, also called Chinese Cinnamon, has been wildly cultivated in China and Southeast Asia with a long history for health care and flavoring spice. Previous studies have proved that it is generally safe and has multiple pharmacological activities, such as antidiabetic and antimicrobial effects. The essential oil is one of the main active fractions in *C. cassia*. Unfortunately, safrole, a toxic compound, was reported in several species, which need to be carefully investigated.

Sample preparation is the crucial first step in analysis of TCMs, which impacts nearly all the later assay steps, including unequivocal identification, confirmation and quantification of analytes. However, the sample preparation step is often not well optimized and frequently considered as an end by a large number of analysts. Pressurized liquid extraction (PLE) is an innovative sample preparation technique which combines elevated temperature and high pressure to achieve fast and efficient extraction of the analytes from the (semi-)solid matrices, which is a promising method

for sample preparation of TCMs.

China is the main producing country, which accounts for more than 80% of the world's production. Therefore, chemical analysis is very important for ensuring the safety and clinical efficacy. In present study, a PLE and GC-MS method was developed for simultaneous determination of seven compounds, including the toxic component safrole in *C. cassia*. The influence of sample preparation, especially the effect of solvent, on the quantification was also investigated in detail.

The dissertation consists of three chapters: **Chapter 1** reviewed the distribution, pharmacological activites and quality control of CC. In **Chapter 2** the effect of sample preparation on the quantification of *C. cassia* was illustrated. Furthermore, alcohol was found incompatible to sample preparation of aldehydes due to the aldol reaction. In **Chapter 3**, a GC-MS method was established for simultaneous determination of seven components in *C. cassia* and successfully applied to quantification of 15 samples collected from different areas of China. The results showed that cinnamaldehyde was the most abundant compound, but safrole was not detected in all samples.

Key words: *Cinnamomum cassia,* Gas chromatography–mass spectrometry, Pressurized liquid extraction, Sample preparation

澳門大學

摘要

肉桂揮發性成份的氣相色譜分析及樣品製備方法的影響

呂廣萍

導師：李紹平

肉桂，作為天然藥物及香料在中國及世界範圍內都有著廣泛的應用。不同的國家及地區，肉桂的來源也不盡相同。樟科植物肉桂*Cinnamomum cassia* Presl.的乾燥樹皮是《中華人民共和國藥典》2005年版收載肉桂的唯一來源,中医常用於治療消化不良，胃炎，血液循環障礙和炎症等疾病。現代藥理研究已證明，肉桂具有抗癌，抗糖尿病，抗氧化,抗菌及抗炎等活性，揮髮油是其主要活性成份。由於肉桂來源混雜，已有研究證明幾種肉桂具有毒副作用，毒性成份為有致畸、致幻、致突變作用的黃樟醚。我國肉桂產量占世界总產量的80％以上，因此為保證肉桂應用的安全有效，對肉桂的化學成分評價顯得尤為重要。

同时，樣品製備是中藥分析過程關鍵的第一步，選擇合適的樣品製備方法對分析結果至關重要。加壓溶劑提取，作為一種新型的樣品製備技術，結合了高溫、高壓實現了快速、高效地從固體、半固體樣品中提取被分析物。本實驗將加壓溶劑提取技術應用於肉桂藥材的提取，詳細的考察了樣品製備過程對於分析結果的影響，特別是溶劑的影響。繼而，運用氣相色譜串聯質譜的方法，對肉桂中的成分進行多指標同時定量分析，有利於提高肉桂質量控制水準。

全文共分三章，第一章綜述了肉桂的資源分佈、主要藥理作用及質量控制現狀。第二章闡釋了樣品製備過程對於肉桂中被分析物定量結果的影響，證明醇類溶劑與肉桂中醛類成份存在縮醛反應，嚴重影響分析結果的准確性。第三章建立了同時測定肉桂中七種成份的 GC-MS 分析方法，運用該方法對收集自全國各地 15 個肉桂樣品進行了分析。結果顯示，肉桂醛為肉桂中的主要成份，在

C. cassia 種中未檢測到毒性成份黃樟醚。

關鍵字：肉桂，加壓溶劑提取， GC-MS，樣品製備

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LIST OF ABBREVIATIONS

Abbreviation	Interpretation
TCM	Traditional Chinese Medicine
HPLC	High Performance Liquid Chromatography
GC	Gas chromatography
TLC	Thin Layer Chromatography
MS	Mass Spectrometry
UE	Ultrasonic Extraction
MAC	Microwave Assisted Extraction
PLE/ASE	Pressure Liquid Extraction
S/N	Signal to Noise Ratio
LOD	Limit of Detection
LOQ	Limit of Quantification
CC	Cortex Cinnamomi
NPSMAE	Non-Polar Solventmicrowave-Assisted Extraction
ISFME	Improved solvent-free microwave extraction
EPA	Environmental Protection Agency
SGPT	Serum glutamic-pyruvic transaminase
SGOT	Serum glutamic-oxaloacetic transaminase
ROS	Reactive oxygen species