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Integrative Medicine Research

journal homepage: www.imr-journal.com

Original Article

# A comparison of volatile fractions obtained from *Lonicera macranthoides* via different extraction processes: ultrasound, microwave, Soxhlet extraction, hydrodistillation, and cold maceration

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ARTICLE INFO

*Article history:*
Received 20 March 2015
Received in revised form
29 May 2015
Accepted 3 June 2015
Available online 12 June 2015

*Keywords:*
*Lonicera macranthoides*
volatile fraction
extraction method
hydrodistillation
traditional Chinese medicine

ABSTRACT

*Background:* Hydrodistillation has been traditionally used to extract volatile fraction in traditional Chinese medicine. However, with the development of Soxhlet extraction (SE), microwave (MW), ultrasound (US), and cold maceration (CM), hydrodistillation (HD) is being replaced to meet some practical requirements. In this study, we investigated the effect of the five methods on the volatile fraction extract of *Lonicera macranthoides*.
*Methods:* Volatile fraction from the flower buds of *Lonicera macranthoides* was obtained by using different extraction methods, HD, SE, MW, US, and CM. The compositions of volatile fraction were analyzed by gas chromatography–mass spectrometric and further compared among extraction methods.
*Results:* Extracts obtained by the five methods reveal the qualitative and quantitative diversity in their compositions, especially for the low-content compositions. According to the results, SE shows the great value in the research where the high molecular-mass compound is of primary interest, and MW offers a way for the isolation of specific compound like octadecadienoic acid and hexadecanoic acid. HD, US, and CM have the advantage over SE and MW for the integrity of the constituents, whereas the phenomenon of compound degradation seems not so serious in solvent extraction methods such as US or CM as HD. Additionally, US and CM show superiority over time or material saving and diversity of the constituent.
*Conclusion:* HD is still the best choice for the pure volatile fraction without organic solvent pollution. However, when it comes to some specifically actual demands, it can be replaced

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http://dx.doi.org/10.1016/j.imr.2015.06.001

by the four methods for the volatile fraction extraction process, especially for production of certain compound groups.



## 1. Introduction

*Lonicera macranthoides*, one of the most important varieties in the *Lonicera* family, is widely cultivated and applied as a substitute for *Lonicerae japonicae* in China for its similar chemical compositions, pharmacological effects but relatively higher yield and lower cost.[1–3] It has been reported that the effective components in *L. macranthoides* bud possess diverse biological activities, such as antibacterial,[4] antipyretic,[5] antioxidant,[6] and hepatoprotective ability.[7] It has been reported that volatile fractions together with chlorogenic acid are the main active ingredients in *L. macranthoides*.[8] Other than the normal medical value, volatile fractions in *L. macranthoides* also attract intensive attention in fields such as cosmetics, shampoos, beverages, flower tea, and baked goods, all of which are owing to its properties of thirst-quenching, heat-clearing, and detoxifying, and render *L. macranthoides* highly appreciated in Chinese herbal medicine.

It is generally known that the budding flower of flower-employed Chinese medicine, rather than leaves or other parts of plants, is conventionally selected as the material for volatile fraction extraction and always ranks first in volatile fraction yields. Previous research also indicates that genetic and environment factors may influence the content and composition of volatile fraction in plants,[9] as do the development stages.[10] In addition, the volatile fraction also varies with extraction methods.[11]

With increasing energy consumption and the drive to improve efficiency, industries and research institutions are challenged to find ways which can simplify operation procedure, meet low cost requirements and achieve good quality. Apart from conventional techniques such as hydrodistillation (HD),[12] Soxhlet extraction (SE),[13] ultrasound (US),[14] microwave (MW),[15] and cold-maceration (CM),[16,17] some relatively new methods such as supercritical $CO_2$ extraction[18] and head space analysis[19] have been employed for volatile fraction extraction research in laboratories. However, both supercritical $CO_2$ extraction and head-space analysis need special equipment, thus making them too expensive for large-scale volatile fraction extraction when compared with traditional methods. For example, HD is widely treated as the conventional application of the volatile fraction in food and Chinese medicine[20] and usually takes about 6–8 hours with distilled water for the whole extraction progress. The fraction obtained by this way is completed and pure without organic solvent pollution when compared with ultrasound and microwave. Likewise, SE progress is similar to HD but with organic extraction reagent and mainly used in the volatile fraction extraction of the material that is rich in fat, such as seed or spice.[14,21] Similar research for the volatile fraction extraction on flower materials has also been reported by Guan et al.[22] Unlike HD and SE, both MW and US extractions offer a faster and simpler procedure and require less plant material.[23] Thus, the two methods are widely applied for fast extraction at the cost of compounds integrity.

Additionally, CM always results in an odor similar to that in the original plant material without causing degradation of the thermo labile compounds present in the fraction due to the low extraction temperature similar to cold pressing,[24] rather than heating in HD or SE, which makes heating a factor investigated for extraction of volatile fractions from aromatic flowers. However, the exact differences about the volatile fractions extracted via the five methods mentioned above have not been reported yet.

Over the years, procedures such as US and MW extraction have replaced some of the conventional processes such as HD and SE that have been used in industries and laboratories for decades. We wonder if the relatively new methods are exactly fit for the volatile fraction extraction in Chinese medicine, because it is the entirety of constituent rather than a single compound that cures diseases. In this paper, we present a comparative study of the content and composition of volatile fraction extracted by different methods from *L. macranthoides* in order to find the differences in terms of their quantity and quality.

## 2. Methods

### 2.1. Chemicals and plant material

Analytical grade anhydrous sodium sulfate and ethyl acetate were purchased from Kelong Chemical (Chengdu, China). The buds of *L. macranthoides* were authenticated by Professor Xingfu Chen in Sichuan Agricultural University (Sichuan, China) and collected in July 2012 from Suining in Sichuan province of China. The materials were dried and pulverized to a fine powder using a mechanical grinder.

### 2.2. Extraction procedures

The extraction of volatile fraction from *L. macranthoides* was performed using five different methods, and each test was carried out in triplicate.

#### 2.2.1. HD

A 20 g sample of *L. macranthoides* was subjected to hydrodistillation, according to the China Pharmacopeia,[21] and extracted with 200 mL of distilled water for 6 hours (until no more volatile fraction was obtained). The volatile fraction was collected, dried with anhydrous sodium sulfate and stored at 4 °C until used.

#### 2.2.2. SE

The samples (20 g) were weighed and transferred into the Soxhlet apparatus (SOX500, Haineng, China, with 6 individual