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Studies on Compositions of Ginger Oleoresin by Supercritical CO_2 Extraction Compare with Ultrasonic Solvent Extraction

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Abstract: *The chemical compositions of ginger oleoresin by supercritical CO_2 extraction compare with ultrasonic solvent extraction were studied in order to identify the superiority of supercritical extraction in the extract of ginger oleoresin and determine the possibility of making these oleoresins into finished product for other further applications. The various components of the ginger oleoresins were separated and identified using Gas Chromatography/Mass Spectrometry, and these components were then classified according to structure and function. The experimental results show that the fragrant and pungency compounds are main compositions in the Supercritical CO2 extract, and the relative contents are 66.31% and 24.832%. For the ultrasonic solvent extract, the pungency compound is the main composition, and the relative contents of fragrant and pungency compounds are 14.143% and 57.855%. It is concluded that ginger oleoresin extracted by supercritical CO2 contains more functional compositions than that of ultrasonic solvent extraction.*

Keywords: *Ginger Oleoresin, Supercritical Extraction, Ultrasonic Solvent Extraction, Gas Chromatography /Mass Spectrometry*

1. Introduction

Ginger is the fresh rhizome of *zingiber officinale*, a perennial herb that is widespread in China. Ginger has been used both as a flavoring additive in food and also widely used in Chinese traditional medicine throughout China's long history. Ginger has been studied and believed to be beneficial in the effect of antioxidation and diminishing certain types of inflammations [1].

Ginger oleoresin is a mixture extracted from dried ginger by either SC-CO_2 or solvent that contains both volatile fragrant and fixed pungent components. Ginger oleoresin is currently used widely in the food industry [2-4] and believed has many pharmacological functions as well[5-7]. At present, the oleoresins are mainly used as additives and adjuvants in food and medicine and the finished products in medicine and health protection are in relative low proportions.

The ginger selected for this study was from Shandong (China) and is famous for its sound quality, rich nutrition, full-bodied pungency, and high content of volatile and pungent compounds. Research [8] has also shown that the contents of volatile oil, starch, protein and vitamin C are higher than some of the others. The ginger oleoresins' compositions were studied in this paper in order to investigate the possibility of making them into finished product for other further applications.

Ginger oleoresins were extracted by two methods for comparative purposes, SC-CO_2 extraction and ultrasonic solvent extraction. Gas Chromatography/Mass Spectrometry was used to quantify and qualify the components of the two extracts.

2. Materials and Methods

Low temperature should be used in the drying of raw materials, for the active components in ginger are water-fast, thermosensitive and volatilizable. In this experiment, materials were dried in the sun, and the extraction process was maintained and performed at low temperature as well.

2.1. Materials

Ginger rhizomes used in this study were obtained from market at the autumn harvest season. CO_2

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(food grade) was used in the supercritical fluid extraction. Ethanol was used in the ultrasonic solvent extraction.

2.2. Preparation of Ginger Powders

The gingers were cut into pieces, and predrying treatment using solar energy helped to dehydrate the gingers until constant mass was achieved with the moisture content of approximately 10% (dry weight basis). The dried gingers were ground into powders using a muller. Powders were separated into size fractions using sieves, and ginger powders used in ultrasonic solvent extraction were with a granularity of 150μm, while ginger powders used in supercritical CO_2 extraction were with a granularity of 3 mm.

2.3. Extraction of Ginger Oleoresin by Supercritical CO_2

Ginger oleoresin was extracted in Supercritical Fluid Extraction equipment. One hundred and fifty grams of ginger powders were put into the extractor. The CO_2 was frozen in the store by a refrigerating machine in advance, and then purified and frozen CO_2 was compressed by a reciprocating pump and sent into the extractor. The extractor was controlled at 40～50℃ by a constant temperatures controller. Extraction process happened in the condition of 40～50℃. During the extraction, supercritical CO_2 took ginger oleoresin into segregator. In the segregator, the pressure dropped to a lower level, which was controlled at less than 5Mpa. The ginger oleoresin separated with CO_2 and subsided in the bottom of the segregator because of the solvency of CO_2 declined. Separated CO_2 returned to store through a clarifier, which was frozen and compressed again, then sent into the extractor for circulative usage.

After completion of the extraction process, CO_2 in the circulation was depressurized through a valve and went into the store. The CO_2 in the extractor was vented to atmosphere. Turned on the switch under the segregator and obtained the oleoresin. The oleoresin extracted by SCF-CO_2 is a bisque and ropy mixture.

2.4. Extraction of Ginger Oleoresin by Ultrasonic Solvent

Some accurately quantified ginger powders were mixed with food grade ethanol. The mixture was placed in a canister with a small orifice in the cover and extracted by ultrasonic firstly at 40～50℃ for 30 minutes, and then kept in a constant temperature heated water bath and shaken at 40～50℃ for 8 hours. The filtrate and solid were separated by vacuum filtered, and the filtrate was distilled at 40～50℃ by vacuum distillatory until all the ethanol was separated. Ginger oleoresin extracted by this method is ropy and brown red mixture.

2.5. Analysis of Ginger Oleoresin by Gas Chromatography-Mass Spectrometry

Ginger oleoresins were separated and identified by Gas Chromatography-Mass Spectrometry apparatus obtained from Agilent fitted with a HP-5MS capillary column. Helium was the carrier gas. Oven temperatures increased with a rate of 3-5 ℃/min from 60℃ to 300 ℃, and then maintained for 20 min. Ginger oleoresin was dissolved in ethanol to a concentration of 100ppm. One microlitre of sample was injected with a split ratio of 100:1. The ionization voltage was 70eV. Ionization source temperature was 230℃. Mass scan ranged from 29 to 500. Under these conditions the compounds in oleoresins were isolated and identified.

The percentages of the components in the extracts were computed from summation of the peak areas of total compositions. NST database was used to search the spectrum data of the components. Then checked the mass spectrum and identified the name of each compound. The GC/MS total ion chromatographs of the two oleoresins were shown in figur1and figur2 respectively. The components were classified by structure and function, and the content of each category was shown in Table1.

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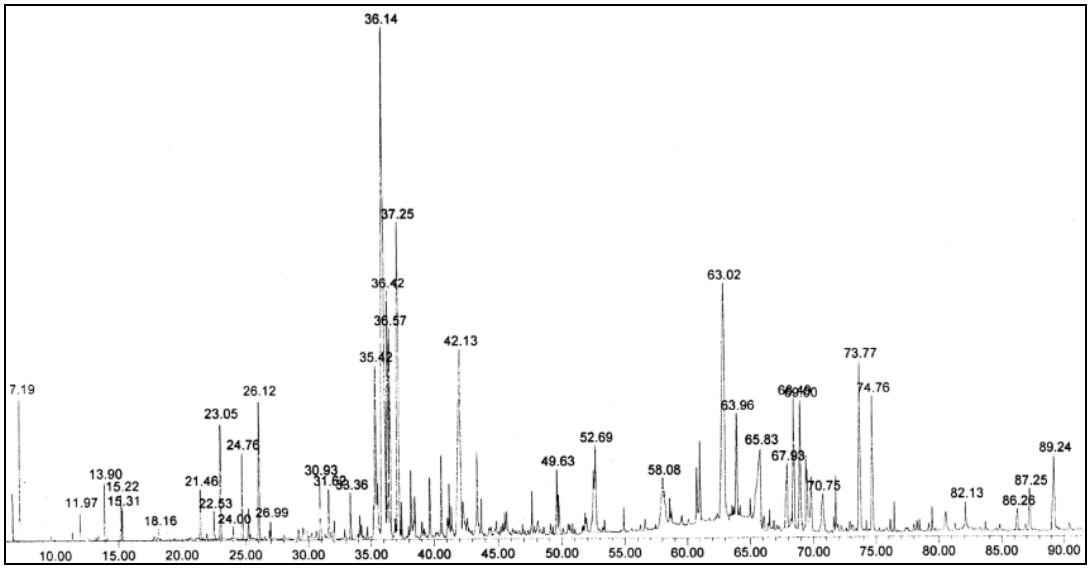

Figure1 GC/MS total ion chromatography of oleoresin extracted by SC-CO_2.

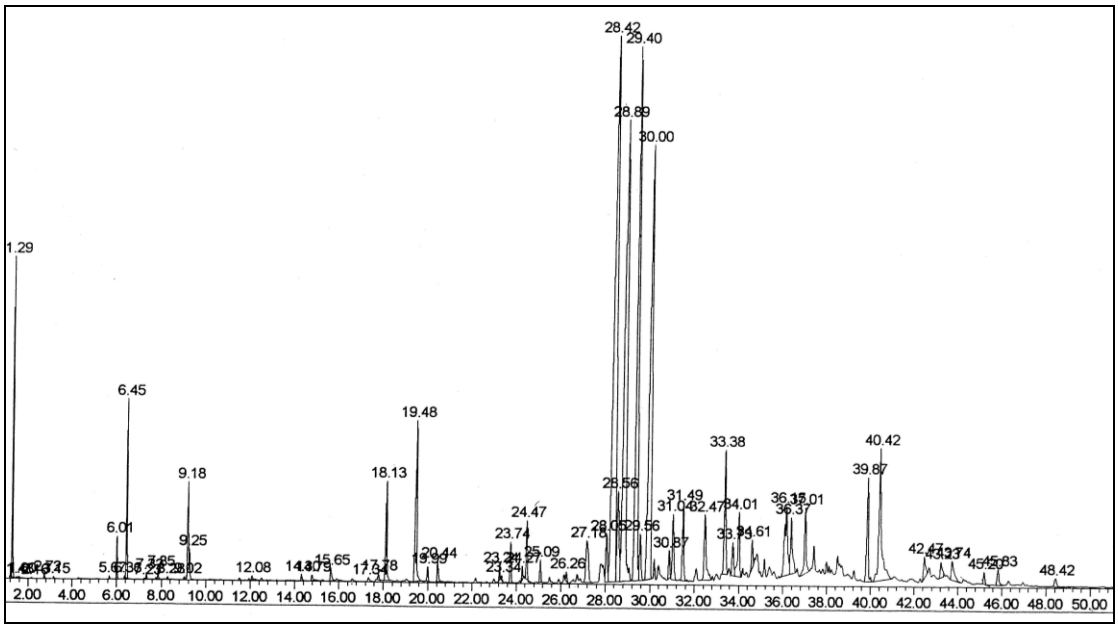

Figure 2 GC/MS total ion chromatography of oleoresin extracted by ultrasonic solvent.

3. Results and Discussion

3.1. Comparison of the Main Compounds in the Two Ginger Oleoresins

From the total ion chromatographs of the two extracts in figure1 and figure2, the qualitative analysis result of the oleoresins could be observed easily. The chemical compositions of the oleoresins obtained by the two methods differed significantly. As can be seen that there are more compounds have short retention time in figure1 than in figure2. Compounds have short retention time are mainly fragrant components including monoterpene, oxygenous monoterpene, sesquiterpene and oxygenous sesquiterpene, whose molecular weight are lower than pungency compounds that have long retention time.

From the quantitative analysis of GC-MS, the componential difference of the extracts can be clearly observed too. In the ginger oleoresin extracted by supercritical CO_2, the content of sesquiterpenes such as betta-bisabolene, alpha-farnesene, betta-sesquiphellandrene and zingiberene is 56.627%. The content of pungent components mainly including methoxy-phenol compounds such as 6-shogaol, zingerone and gingerol is24.832%.

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In the oleoresin extracted by ultrasonic solvent, the content of pungency compound is 57.855%. While, the content of sesquiterpenes is 10.360%. The compositive comparison of the two oleoresins is shown in Table 1.

The oleoresin obtained by SC-CO_2 fluid extraction contains less impurity and more small molecular substances than the one by ultrasonic solvent extraction. In the ultrasonic solvent extracted oleoresin, pungency compounds are the main components, which have pharmacological effect [9], but monoterpene and sesquiterpene account for low proportion in it, which are the main compositions of fragrant compounds. While, in the SC-CO_2 extracted oleoresin, the contents of sesquiterpene and pungency compounds are all high. Generally, ginger oleoresin extracted by SCF-CO_2 contains more functional compounds than ultrasonic solvent extraction oleoresin. SCF-CO_2 extraction can not only extract the pharmacological compounds but the fragrant components. Componential differences also influence the comparative densities of the oleoresins.

Table 1 Comparison of production extracted by SCF-CO2 and ultrasonic solvent.

Category	Oleoresin extracted by supercritical CO_2	Oleoresin extracted by ultrasonic solvent
Monoterpene	1.323%	0.824%
Oxygenous monoterpene	7.026%	1.285%
Sesquiterpene	56.627%	10.360%
Oxygenous sesquiterpene	1.334%	1.440%
Diterpene	——	1.637%
Pungency compound	24.832%	57.855%
Triterpene	——	4.080%
Oxygenous triterpene	3.056%	10.833
Straight	4.322%	5.374%
Rest	1.226%	6.036%

4. Conclusions

Conclusions can be drawn from both the qualitative and quantitative analysis by GC/MS. The SC-CO_2 extract contains more useful components than ultrasonic solvent extract. The oleoresin extracted by SCF-CO_2 made into further products directly without further process. Although they all can be used as medicament and food additive, the oleoresin extracted by ultrasonic solvent is difficult in the remove of disultrasonic solvent, and there is no nocuous solvent remained in SCF-CO_2 extract that can be used directly.

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