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Analysis of Ginger Oleoresin Extracted from Vacuum Freeze-dried Ginger Compare with Sun-dried Ginger by Supercritical CO_2 with GC-MS

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Abstract: *In spite of mature processing techniques and the large availability of raw materials having sufficient quality, there is no study about the relation between the chemical components and material pretreatment method of ginger oleoresin extracted by supercritical fluid CO_2. In order to obtain high content of effectual compositions in ginger oleoresin, Ginger was dehydrated by vacuum freeze–drying method and extracted by Supercritical CO_2. The components in the extracts were quantified and qualified using Gas Chromatography-Mass Spectrometry method. Same studies were also done with the sun-dried ginger. The results show that the main components are different in the two samples. In the freeze-dried sample, zingerone is the highest substance at 19.472%, pungency compounds are the main components at53.920 %, and the content of sesquiterpene is 20.041%. By comparison, in the sun-dried sample, the content of bisabolene is 26.328% that is the highest, the pungency components are 24.832%, and the main components are sesquiterpene at 56.627%.*

Keywords: *vacuum freeze-drying, supercritical CO_2 fluid extraction, ginger oleoresin, gas chromatography -mass spectrometry*

1. Introduction

Ginger is the fresh rhizome of *Zingiber officinale* that has been grown in china for long histories [1], which can be used as both food additives and traditional Chinese medicine. The compositions have pharmacological action are pungency compounds. As additives used in food industry, ginger pungency compounds are main compositions that have pungent flavor, and volatile compounds have fragrant flavor[2, 3].

The fresh ginger rhizomes are not prone to storage for long time, so they could be made into further products for deep applications. And the further products are mainly ginger oleoresin and ginger essential oil[4, 5]. The contents of pungency and violate fragrant compounds are important guideline of their process character. Supercritical fluid CO_2 is a new method to extract ginger oleoresin that is rising in recent years.

Ginger needs to be dehydrated before SCF-CO_2 extraction. Ginger oil is stored in the interspace of cornuted cells in ginger roscoe[6], and the volatile substances are easy to losing during the dehydrating process. It differs grately in the quality and compositions with different dehydrating method. Besides dehydrating method, the contents of functional compositions are also influenced by other factors, such as extraction methods, the variety of ginger and so on. Ginger is an excellent plant in China, which is famous for its fleshy rhizome and less fiber, rich nutrition, full-bodied pungency and high content of functional compounds. The extraction conditions were confirmed in the last study in which the yields are highest. Gingers dehydrated by vacuum freeze-drying and sun-drying were extracted by SC-CO_2, and Gas Chromatography-Mass Spectrometry was used to quantify and qualify the components of the two extracts.

2. Experimental method

2.1. Equipment and reagent

Gas Chromatography-Mass Spectrometry apparatus (Agilent), freeze-drying machine (Beijing), low temperature refrigerator；supercritical fluid extraction equipment（Jiangsu）,CO_2 （food grade）

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2.2. The freeze-drying of fresh ginger

Gingers were washed clean and cut into pieces of 1-2mm thickness, then put them on the trays of and froze to 4 hours in the low temperature refrigerator, which was controlled at -40℃. Then they were put in the shelf of vacuum freeze-drying machine, which was vacuumized to 10 Pa later. The drying process would last for 24 hours. Predrying processes using the vacuum freeze-drying helped to dehydrate the ginger until constant mass was achieved. Dried gingers were ground into particles using a muller. The particles were separated into size fractions using sieves, and ginger particles used in supercritical fluid CO_2 extraction were with a granularity of 1-3mm.

2.3. The sun-drying of fresh ginger

Gingers were cut into pieces of 1-2 mm thickness and dried in the sun, and all the other procedures were same as above.

2.4. The extraction of ginger oleoresin by supercritical CO_2

Ginger oleoresin was extracted by Supercritical CO_2, and 150.00 grams of ginger particles were put into the extractor, then the purified, frozen and compressed CO_2 was sent into the extractor, and ginger oleoresin was extracted in the condition of 50℃ and $30*10^6$ Pa. 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 $10*10^6$ Pa. The ginger oleoresin separated from CO_2 and gathered at the bottom of the segregator. The separated CO_2 returned to circulator, which was purified, frozen and compressed again, then sent into the extractor for circulative usage. The extraction time would last 5 hours.

After 5 hours of extraction, turned on the switch of the segregator and obtained the oleoresin. The oleoresin extracted by SCF-CO_2 is a deep yellow and viscous mixture.

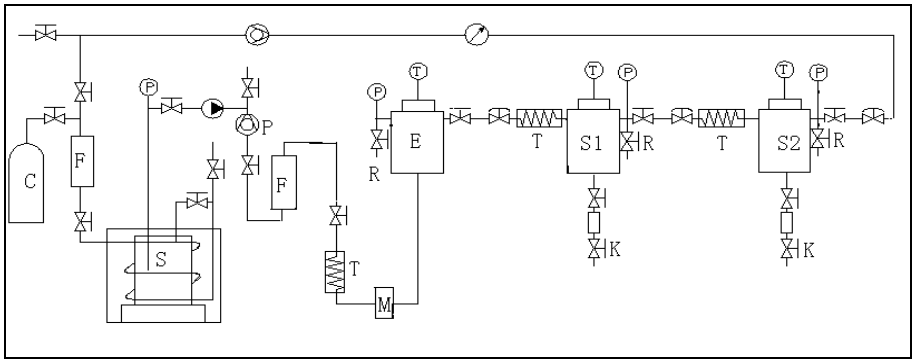

Figure 1 Flow sheet of Supercritical CO_2 Fluid Extraction.

C-CO_2, E-extractor, F-purifier, K-outlet port, M-flow meter, P-reciprocating pump, R-air relief valve, S-cooling box, S1-seperatorI, S2-seperatorII, T-constant temperature water bath controller

2.5. Analysis of ginger oleoresin with Gas Chromatography-Mass Spectrometry

Ginger oleoresins were separated and identified by Gas Chromatography-Mass Spectrometry apparatus, fitted with a HP-5MS capillary column, (60m×0. 25mm i.d, film thickness 1.00μm, Agilent). Helium was the carrier gas as a flow rate of 1 ml /min. Oven temperatures increased with a rate of 3-5 ℃/min from 60℃ to 300 ℃, and then maintained for 20 min. The temperature of injector port was 300℃. Ginger oleoresin was dissolved by ethanol to a concentration of 100ppm. One mic-rolitre of sample was injected with a split ratio of 100:1. Mass spectra were obtained at the ionization voltages of 70eV. Ionization source temperature was 230℃, transfer line temperature was 280℃, and interface temperature was 280℃. Mass scan ranged from 29 to 500. The compounds of ginger oleoresins were isolated and identified in these conditions.

The percentages of the components in the extracts were computed by peak areas normalization method. NIST 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 ginger oleoresins are shown in figure2 and figure3 respectively.

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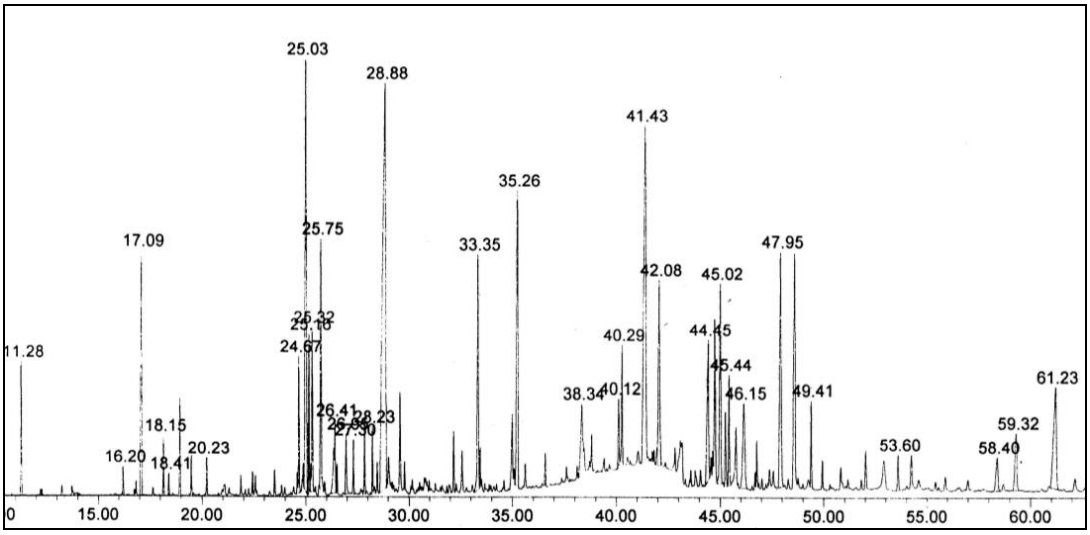

Figure 2 Total ion chromatogram of ginger oleoresin extracted from freeze-dried ginger

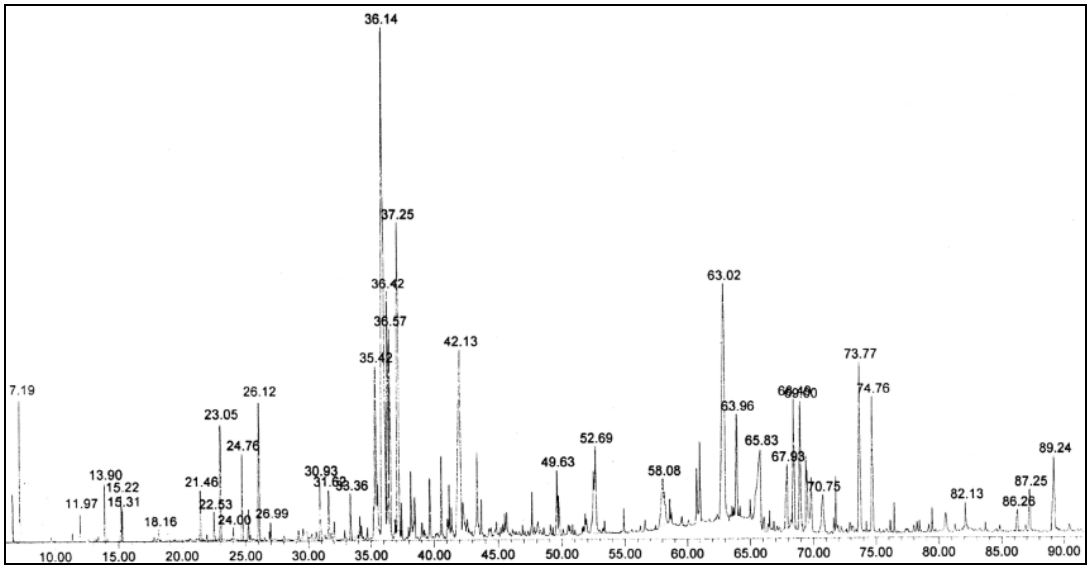

Figure 3 Total ion chromatogram of ginger oleoresin extracted from sun-dried ginger

The components were classified by structure and function. The contents of the categories are shown in table 1.

Table 1 Composition comparison of the two ginger oleoresins

Category	oleoresin extracted from freeze-dried ginger	oleoresin extracted from sun-dried ginger
Monoterpene	1.019%	1.323%
Oxygenous monoterpene	1.031%	7.026%
Sesquiterpene	20.042%	56.627%
Oxygenous sesquiterpene	1.117%	1.334%
Pungency compound	53.920%	24.832%
Triterpene	1.330%	——
Oxygenous triterpene	9.239%	3.056%
Straight	10.150%	4.322%
Rest	2.033%	1.226%

3. Result and discussions

The total ion chromatographies of two ginger oleoresins are shown in Fig2 and Fig3. For the

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analyze conditions of gas chromatography-mass spectrometry were different, the retention time of same substance is different. As can be seen that there are more compounds have short retention time in Figure2 than in Figure3. 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 analysis of GC-MS, the componential difference of the extracts can be clearly observed too. In the oleoresin extract from freeze-dried ginger, the richest compound is zingerone with a content of19.427%. The content of pungency components mainly including methoxy-phenol compounds such as 6-shogaol, zingerone and gingerol is 53.920%. The content of sesquiterpenes such as betta-bisabolene, alpha-farnesene, betta-sesquiphellandrene and zingiberene is 20.042%, and the content of volatile balmy components is 21.372%.

In the oleoresin extracted from sun-dried ginger, the content of bisabolene (including isomer) is26.328%, which is the highest. The content of sesquiterpenes is 56.627%, and the volatile fragrant component is 63.574%. While the content of pungency compound is 24.832%. The compositive comparison of the two oleoresins is shown in Table 1.

The masses of the material before and after the extraction were accurately quantified. Calculate the extraction ratio according to mass losing. The extraction ratios of the two processes are both about 5.5%. The oleoresin obtained from freeze-dried ginger by SC-CO_2 contains more pungency compounds and less volatile fragrant compounds than sun-dried ginger. In the oleoresin extracted from freeze-dried ginger, pungency compounds are the main components, which have pharmacological effect, but monoterpene and sesquiterpene account for low proportion in it, which are the main compositions of ginger essential oil. While, in the oleoresin extracted from sun-dried ginger, the content of sesquiterpene is higher than that of pungency compounds.

For the impetus of vacuum freeze-drying is higher than that of sun-drying process, and along with the transfer of water the losing of low molecular and volatile components happens too. The spongy structure of ginger by vacuum freeze-drying also makes the losing of low molecular weight, and during the SC-CO_2 extraction the pungency compounds are easily extracted too. While, the sun- dried ginger would be hard and wizened during the dehydrate process, and the apertures are blocked up too. So fixed oil is not propitious to transfer and the content of volatile oil is higher than pungency compounds.

The vacuum freeze-drying is a drying method controlled in high vacuum and low temperature, which is controlled in anoxic and aphotic, and it is in favor of keep the aroma, odor, colour and lustre and shape, and can keep all the vitamin, cellulose, protein and mineral. The freeze-dried ginger is superior to the sun-dried ginger in colour, luster and shape, and is alike with fresh ginger. There is no report about the relationship between the drying method and the chemical compositions of ginger oleoresin. S.Balachandran, S.E.Kentish, R.Mawson[7] modeling to study the influence of season and preparation method on the extraction efficiency from mass transfer aspect. Z.D. Guo, X.N. Zhang, J.C. Zhang [8] compared the chemical compositions of ginger oil extracted by SC-CO_2 and steam distillation. And the study about freeze-drying ginger powder's superiority is merely in physical property. Z. Zhang, X.M. Liu [9] studied the influences of dehydrating method on ginger powder's physical properties, and the results showed that sensory characteristic of ginger powers dehydrating by vacuum freeze-drying is superior to other dehydrating method, and shape, color and luster, flavour and odor keep nicer.

In this Article, the analysis of ginger oleoresins extracted by Supercritical CO_2 of different drying method show that the content of pungency compounds from freeze-dried ginger is higher than that of sun-dried ginger, and the content is more than 50%. Pungency is the pharmacological compounds of ginger. So freeze-drying is an effect method to get ginger pungency compounds.

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