



Comparative Analysis of Volatile Flavor Compounds of *Poppy* Seed Oil Extracted by Two Different Methods via Gas Chromatography/Mass Spectrometry

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Abstract

The improved simultaneous distillation extraction (SDE) was used to separate and extract volatile aroma components in these two kinds of *poppy* seed oil (full aroma and flat aroma) and gas chromatography/mass spectrometry (GC-MS) was used to identify them. From full aroma *poppy* seed oil 15 kinds of volatile components were isolated and identified mainly including 2-methylbutyraldehyde, Hexanal, 2-heptanone, 2,5-dimethylpyrazine, 2-pentylfuran, 2-ethyl-3-methylpyrazine, 5-ethyl-1-acetaldehyde-1-cyclopenten-2-ethyl-3,6-dimethyl pyrazine. From flat aroma *poppy* seed oil 14 kinds of volatile components were separated and identified mainly including 2-pentanone, toluene, Hexanal, (Z)-2-heptenal, 1-octen-3-ol, 2-pentylfuran, 2-decenal, 2,4-decadienal. Pyrazine compounds play an important role for the formation of unique aroma of full aroma *poppy* seed oil and enal compounds were major components causing sour flavor of flat aroma *poppy* seed oil. This simple method could effectively identify the authenticity of different *poppy* seed oil, which was of far-reaching significance for the depth development, brand building and technical protection of *poppy* seed oil resources.

Keywords: gas chromatography/mass spectrometry GC/MS, volatile components, *poppy* seed oil, SDE

1. Introduction

Poppy seed oil is the oil formed after pressing seeds of fresh *poppy* and it has no morphine [1]. *Poppy* seeds are rich in amino acids to enhance the flavor such as glycine, alanine, tyrosine, phenylalanine, aspartic acid and so on, therefore they can be used as spicy seasonings to add delicious flavor to food. The *poppy* seed oil can be used for cooking various dishes or processing into health-care food. On June 6th 2006 *poppy* seed oil was formally recognized by our country and in line with "Food Sanitation Law of People's Republic of China" and "New Resources Management of Food Hygiene Approach". From then on it was approved as China's new food resource.

Currently there are two types of *poppy* seed oil for sale. One is pressed using traditional technology and smells flat while the other is the natural full aroma *poppy* seed oil pressed using mature process. In the past, there was little literature reporting about the *poppy* seed oil for the object for research was difficult to obtain. Li Zhaolin et al. [2] grinded *poppy* seeds and extracted volatile oil. Then 55 components were isolated by GC and 36 components were determined by MS. Seen from the identification results, *poppy* seed oil contains hydrocarbons, aldehydes, ketones, oxygenated derivatives of aromatic compounds and furan compounds, as well as some organic acids and esters compounds. Yongkuan Chen et al. [1] extracted one kind of volatile oil from *poppy* seed using steam distillation and qualitatively analyzed main volatile components of *poppy* seed oil using GC-MS, identifying 55 kinds of chemical compositions mainly including aldehydes and higher fatty acids. Sabine Krist et al. [3] used solid-phase microextraction for GC-MS analysis of several *poppy* seed oil samples, finding that n-pentanol, n-hexanal, hexylalcohol, 2-pentylfuran and hexanoic acid were major volatile compounds in this sample. Xiao hongli et al. [4] extracted the volatile chemical composition from *poppy* seed using steam distillation and adopted GC-MS to qualitatively analyze, identifying 72 kinds of compounds.

With the legal development of domestic *poppy* seed oil resources it is necessary to scientifically and effectively identify and quantify some characteristics of these special resources and to distinguish subtle differences of *poppy* seeds oil squeezed by different technology. In order to determine the nature of unique flavor of *poppy* seed oil and

seek scientific bases to identify authenticity of *poppy* seed oil two processes producing *poppy* seed oil were adopted to analyze the aroma components of *poppy* seed oil.

Since yield of aroma components obtained from *poppy* seed oil was low and these ingredients were easy to volatilize. It would inevitably result in the loss of volatile aroma components by steam distillation followed by organic solvent extraction and concentration. "Simultaneous distillation extraction" (SDE) is a method of volatile component extraction for plants developed by Likens and Nickerson in 1966 [5] making steam distillation and solvent extraction into one step. At the same time, the volatile oil obtained by SDE method is the solution in the organic solvent and the volume is small and it is easy to operate, avoiding the adsorption loss on the wall when extracting oil and the difficult operation when transferring trace oils. In recent years, this method has been used widely in analysis of volatile components of food [6-9]. Therefore, in this paper SDE for volatile flavor compounds was used to enrich and GC-MS was used to analyze and identify them.

2. Materials and Methods

2.1. Materials

Full aroma *poppy* seed oil (sample A) was purchased from Ken Gan Wuwei New Resources Food Science and Technology Co., Ltd. Flat aroma *poppy* seed oil (sample B) was purchased from Gansu Province Agribusiness Medicine Herbs Station Vegetable Products Factory. SDE was purchased from American Thermolectricity Ultra. The gas chromatograph/mass spectrometer was Trace GC DSQ. Dichloromethane and anhydrous sodium sulfate were both analytical grade reagents.

2.2. Experimental conditions

The column used was a DB-5MS capillary column (30 m×0.25 mm, 0.25µm film thickness.). The column temperature was programmed with an initial temperature of 50°C keeping for 5 min, then an increase at the rate of 10°C/min to 200°C. Helium gas was used as a carrier gas with a flow rate of 1ml/min. The injection split ratio was 10:1. Inlet temperature was 240°C and interface temperature was 250°C.

2.3. Extraction of volatile aroma components

Sample A of 500ml was added into a round bottom flask and connected to one end of SDE device, which was leaded to water vapor and heated by electric sets to keep the solution boiling. The steam was condensed in the SDE and water solution stratified then inflowed the flask through the upper elbow. The other end of SDE was connected with a 100ml round bottom flask containing 50ml dichloromethane and was heated in water bath of 45°C and kept the solvent boiling. The steam was condensed in the SDE device and layered when encountering with the aqueous solution then inflowed the flask through the bottom elbow. SDE was connected with condensed water and continued distillation and extraction for 2h and volatile aroma components in the sample was extracted into dichloromethane. After extraction, dichloromethane was dried with anhydrous sodium sulfate

and analyzed directly by GC-MS. Sample B was processed with the same method.

3. Results and Discussion

3.1. Analyses of volatile components

According to the above conditions, total ion current graph about volatile aroma components of the two kinds of *poppy* seed oil were got as shown in Fig. 1 and Fig. 2.

The relative percentage was calculated using the area normalization method. Volatile chemical compositions of the two kinds of *poppy* seed oil and their significant differences were confirmed by NIST (2002 edition) mass spectrometry database computer retrieving the peaks of MS datas, in conjunction with artificial spectrum analysis. The results were shown in Table 1.

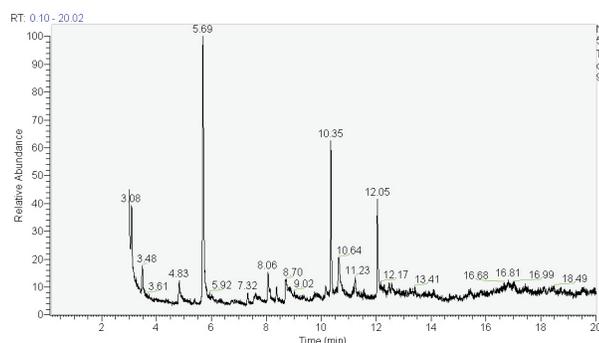


Fig. 1: Total ion chromatogram of volatile flavor compounds from full aroma *poppy* seed oil

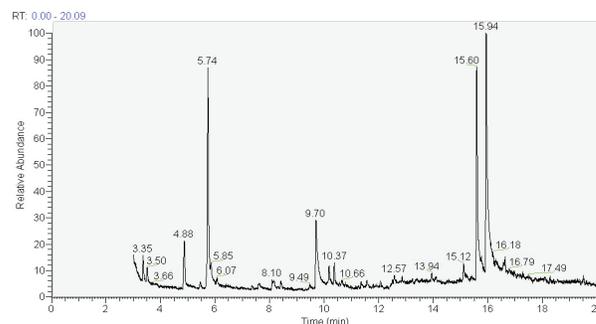


Fig. 2: Total ion chromatogram of volatile flavor compounds from flat aroma *poppy* seed oil

Table 1. The volatile flavor compounds and their relative contents in two kinds of *poppy* seed oil

No	RT (min)	Name of compound	Relative content (%)	
			full aroma poppy seed oil	flat aroma poppy seed oil
1	3.08	2-methylbutyraldehyde	3.56	-
2	3.35	2-pentanone	-	1.91
3	3.48-3.50	Pentanal	2.65	1.40
4	4.83-4.88	Toluene	3.33	5.68
5	5.69-5.74	Hexanal	26.32	16.61
6	5.81-5.85	(E)-3-octene	0.65	0.56
7	7.32	Ethylbenzene	1.15	-
8	8.06-8.10	2-heptanone	4.98	0.66
9	8.38-8.41	Heptanal	2.15	0.71
10	8.70	2,5-dimethylpyrazine	4.06	-
11	9.70	(Z)-2-heptenal	-	8.87
12	10.16-10.18	1-octen-3-ol	1.95	1.88
13	10.35-10.37	2-pentylfuran	11.22	1.75
14	10.64	2-ethyl-3-methylpyrazine	5.75	-
15	11.23	5-ethyl-1-acetaldehyde-1-cyclopentene	3.91	-
16	12.05	2-ethyl-3,6-dimethylpyrazine	8.98	-
17	12.56-12.57	Undecanal	1.73	0.91
18	15.12	(E)-2-decenal	-	1.47
19	15.60	(E,Z)-2,4-decadienal	-	20.17
20	15.94	(E,E)-2,4-decadienal	-	22.46
21	-	Other	17.61	14.96

As seen in Table 1, 15 kinds of volatile aromatic components were identified from full aroma *poppy* seed oil and the main ingredients were 2-methylbutyraldehyde (3.56%), Hexanal (26.32%), 2-heptanone (4.98%), 2,5-dimethylpyrazine (4.06%), 2-pentylfuran (11.22%), 2-ethyl-3-methylpyrazine (5.75%), 5-ethyl-1-acetaldehyde-1-cyclopentene (3.91%), 2-ethyl-3,6-dimethyl pyrazine (8.98%). And 14 kinds of volatile aroma components were identified from flat aroma *poppy* seed oil and the main ingredients were 2-pentanone (1.91%), toluene (5.68%), hexanal (16.61%), 2-heptenal (8.87%), 1-octen-3-ol (1.88%), 2-pentylfuran (1.75%), 2-decenal (1.47%), (E,Z)-2,4-decadienal (20.17%), (E,E)-2,4-decadienal (22.46%).

3.2. Comparison of volatile aroma components of the two samples

There is a significant difference in taste of the two *poppy* seed oil produced using different technology and analysis in this paper provides a rational basis. Full aroma *poppy* seed oil produced by Ken Gan Wuwei new resources Food Science and Technology Co., Ltd using the mature process contains a variety of specific pyrazines compounds and are firstly discovered in *Poppy* seed oil. Pyrazines compounds are commonly found in nuts and have a strong aroma. In maturation process of *poppy* seed protein denaturation and aminocarbonyl reaction [9] occurred to the kernel of *poppy* seed, forming pyrazine compounds, which gave the *poppy* seed oil strong flavor like sesame oil. Flat aroma *poppy* seed oil produced using virgin birth was easy to change the taste and cause rancidity during actual usage and storage, however, the cause of taste changing had not yet been analyzed. In paper studying the volatile components in *aristichthys nobilis* fish Zhao *et al.* [10] stated that 2,4-decadienal is an ingredient in forming oyster flavor and in this experiment we found that there were large 2-decenal and 2,4-decadienal in *poppy* seed oil produced by virgin birth. Studies had reported that there were great relationship between the smell ingredients of food and lipoxygenases [11-12]. Therefore, in this article we speculated that a higher degree of protein denaturation did not occur to protein in *poppy* seeds in virgin birth and enzymes still had some activity, making such *poppy* seed oil easy to be oxidated and become rancid during storage.

3.3. Evaluation of sample extraction conditions

This method provided good selectivity and it was adapted to extract low-boiling volatile components. Compared with the literatures [1-2, 4], higher boiling compounds such as hydrocarbon and organic acids were not detected. The reason was that no liquid water was added during extraction in this experiment, avoiding oil-water boiling together for a long time, and that the short distillation time (only 2h) greatly eliminated sample degradation and hydrolysis reactions, resulting in advanced fatty acids and their esters being not detectable. Besides, SDE was a sealing system and this experiment could be directly analyzed after the extract was dried, avoiding losses of low-boiling compounds during concentration. The effect of this method is similar to the purge-trap method, namely purging volatile substances using steam and condensing by dichloromethane.

4. Conclusion

The experimental result showed that this device was simple and quick and it could be more effective in separating and identifying main

aroma components of *poppy* seed oil, which had far-reaching significance for the depth development, brand building and technical protection of *poppy* seed oil resources. In the initial recognition, unique volatile aroma components of full aroma *poppy* seed oil produced using mature process contained 2-methylbutyraldehyde, hexanal, 2-heptanone, 2,5-dimethylpyrazine, 2-pentylfuran, 2-ethyl-3-methylpyrazine, 5-ethyl-1-acetaldehyde-1-cyclopentene, 2-ethyl-3,6-dimethylpyrazine. Unique volatile aroma components of flat aroma *poppy* seed oil produced using virgin birth contained 2-pentanone, toluene, Hexanal, 2-heptenal, 1-octen-3-ol, 2-pentylfuran, 2-decenal, 2,4-decadienal. In this article the connection between pyrazine compounds and mature process and connection between enals and virgin birth were discussed and it could provide a theoretical guidance for development of *poppy* seed oil resource to a certain extent. At the same time, an effective method to identify different *poppy* seed oil and distinguish its truth was found. It was a very complex process for production of the volatile aroma components of *poppy* seed oil and it had close relationship with the variety, origin and process conditions. Results obtained in this study were only preliminary and more research to uncover its mysterious veil was still needed.

Conflicts of Interest

The author declared no competing interests.

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