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ARTICLE

Study on the Influence of Different Processing Methods on the Volatile Components of Alisma Purpurei

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ABSTRACT

Objective: To establish a gas chromatography-mass spectrometry (GC-MS) method for the determination of volatile components in different prepared products of Alisma purpurea. **Methods:** The volatile components were determined by GC-MS, and the types and relative contents of volatile components were compared in 4 kinds of processed products. **Result:** The main volatile components of 30 kinds of different products from Zelica were analyzed and identified, which were mainly sesquiterpenes, ketones and alcohols, mainly Pogostol, olive, guaiacene, caryophyllene and so on. **Conclusion:** There are differences in the types and relative contents of volatile components in different products of Alisma platyphylla, in order to provide a basis for improving the quality standards of different products of Alisma platyphylla.

1. Introduction

Rhizoma alismatis was originally published in Shennong Herbal Classics and listed as top quality [1], and now published in the 2020 edition of Chinese Pharmacopoeia. Rhizoma alismatis are traditional Chinese medicinal herbs derived from the dry tuber of *Alisma orientale*（Sam.）Juzep. or *Alisma plantago-aquatica* Linn. of alisma family. It has the effect of clearing moisture, draining heat, turbidizing and lowering lipid. Clinically, it is mainly used for the treatment of adverse urination, swollen water, oliguria, dizziness from phlegm and fluid, astringent pain from heat and hyperlipidemia [2]. It has been reported[3] that the chemical components of Purpura mainly include triterpenoids, diterpenoids and sesquiterpenoids, in addition to alkaloids, flavonoids, nitrogenous compounds, phenylpropanoids, steroids and sugars, among which volatile components also play an important pharmacological role.

Rhizoma alismatis has a long history and many processing methods, the earliest records of processing technology can be traced back to the Southern and Northern Dynasties Liu Song period in the *Lei Gong Pao Zhi Lun* [4], the methods used today are net preparation, cutting, salt preparation and frying. At present, there are 5 kinds of purgatory products recorded in the processing standards of various provinces and cities in China: raw purgatory, stir-fried purgatory, bran purgatory, salt

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purgatory and soil purgatory. Because of the different processing methods, the chemical composition and pharmacological action of the prepared products are also different. On the basis of the research results of chemical composition and pharmacological action, the volatile components of radix purpurea, stir-fried radix purpurea, stir-fried with bran and salted radix purpurea were analyzed and compared by gas chromatogram-tandem mass spectrometry (GC-MS) in order to provide reference for the quality evaluation of radix purpurea.

2. Instruments and drugs

Experimental instruments are shown in Table 1.

Reagent: n-hexane is analytically pure, the manufacturer is Fuchen (Tianjin) Chemical Reagent Co., LTD.

Raw alisma decoction pieces (Z01) were purchased from Anguo Medicinal Materials Market, and prepared in accordance with the provisions of *Chinese Pharmacopoeia* 2020 Edition of the fourth General Rule 0213 processing general rule [2], stir-fried alisma decoction pieces (CZ01), bran fried alisma decoction pieces (FZ01) and salt-alisma decoction pieces (YZ01).

Table 1. Information of experimental instruments and equipment

Name	Model number	Manufacturer
Gas chromatography-mass spectrometry	GC2030/TQ8040	Shimadzu Technology Co., LTD
Electronic analytical balance	XS105DU	Mettler Toledo LTD
CNC ultrasonic cleaner	KQ-500DB	Kunshan Ultrasonic Instrument Co., LTD

3. Methods and results

3.1 Instrument operating condition

3.1.1 Gas chromatographic conditions

Column: HP-5MS capillary column (30 m×0.25 mm, 0.25 μm, Shimadzu Company, Japan); The carrier gas was high purity helium, and the column flow rate was 1.0 mL/min. Heating procedure: the initial temperature was 50 ℃, maintained for 1 min, increased to 135 ℃ at 5 ℃ /min, and then increased to 250℃ at 8℃ /min for 10 min; Inlet temperature: 250 ℃; Injection volume: 1μL; Injection method: shunt injection, shunt ratio set at 20:1.

3.1.2 Mass spectrometer

Ionization mode: electron bombardment ion source (EI); Monitoring mode: Q3 Scan; Bombardment energy: 70 eV; Ion source temperature: 200℃; Transmission line temperature: 250℃; Solvent delay time: 3 min; Scanning range: 45 ~ 450 m/z.

3.2 Preparation of sample solution

Take the appropriate amount of decoction pieces, crush them, pass through the second screen, accurately weigh 2g, place them in a tapered bottle with a plug, accurately add 10ml n-hexane, weight them, soak them for 2 hours, ultrasonic treatment for 10 minutes, cool them to room temperature, then weigh them, make up the weight lost with n-hexane, and filter them with 0.45μm microporous filter membrane to obtain.

3.3 Result

According to the operating conditions of the instrument "2.1", sample preparation was conducted by "2.2" method, and the volatile components in the four batches of decoction pieces of Purpura, stir-fried purpura, stir-fried purpura with bran and salt-purpura were analyzed by GC-MS. The total ion flow diagram is shown in Figure 1. The results were imported into the spectrum library of the National Institute of Standards and Technology (NIST20) for retrieval, and the relative contents of each component of the volatile oil were calculated by peak area normalization method. The results are shown in Table 2.

4. Conclusion

In this study, GC-MS technology was used to analyze and identify 30 main volatile components in different products of Alisma. As is shown in Table 2, there were differences in the types and contents of volatile components of alisma sativa before and after processing, among which gamma-serpentene, trans-caryophyllene, (-)-spatol and lignin alcohol were produced after processing. The content of elemene was different in different processed products. The content of elemene increased in bran stir-fried alisma sativa and stir-fried alisma sativa, but decreased in salt alisma sativa, or it was related to the excipients. In addition, the structure of the compound was changed during processing, such as γ-elemene was produced after processing. The above differences may be related to the changes in the efficacy of salt-fried purgatory, bran fried purgatory and stir-fried purgatory, which can provide a basis for the quality evaluation of purgatory products and the in-depth study of "drug-effect consistency".

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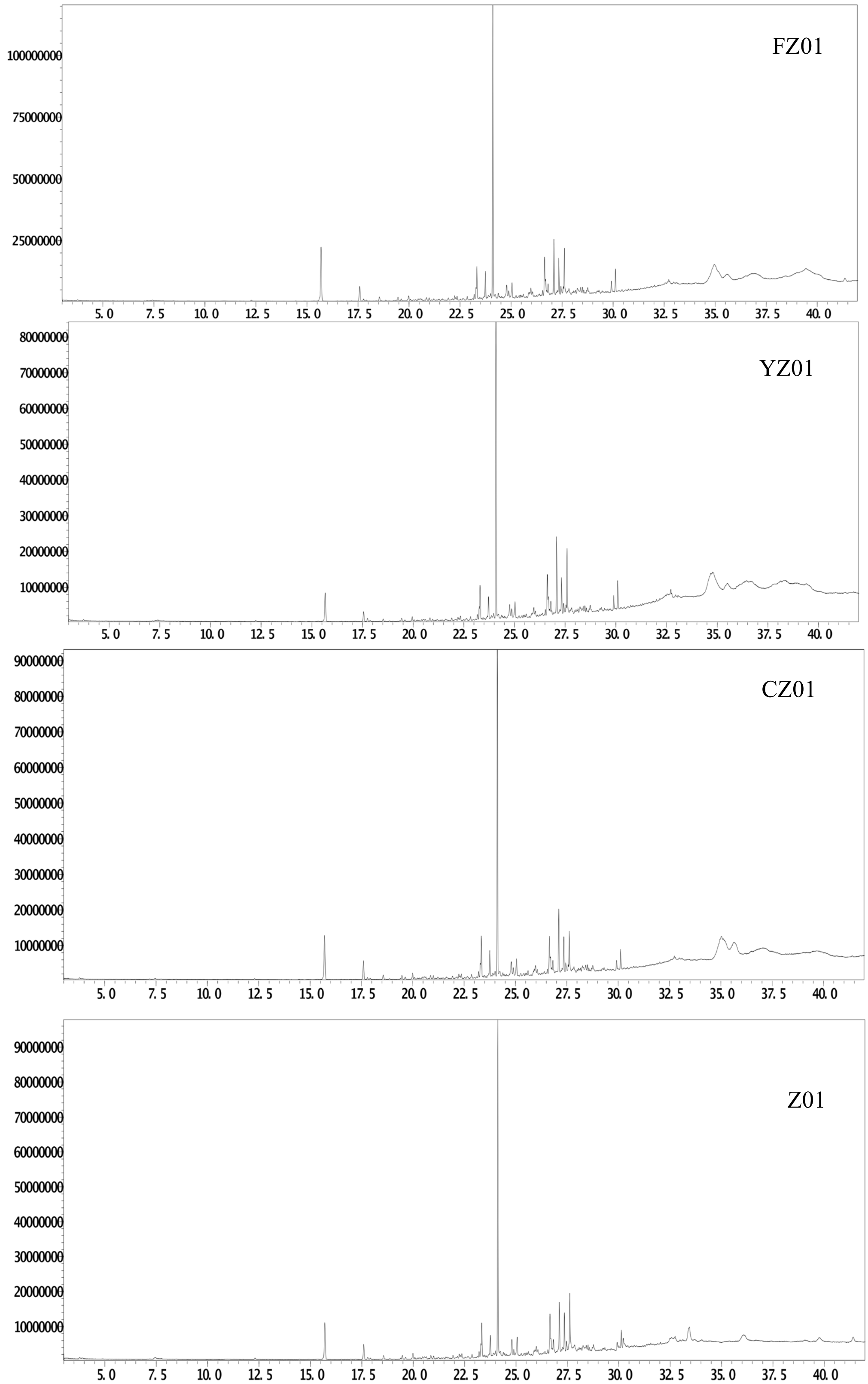

Figure 1. Total ion flow chromatogram of volatile components of 4 different products of Alisma purpura
The horizontal axis is time and the vertical axis is abundance

Table 2. Results of identification and relative content determination of main volatile components of 4 batches of prepared products from Alisma alisma

Serial number	RT（min）	Compounds	Relative content (%)			
			Z01	CZ01	FC01	YZ01
1	15.6	δ-elemene[a]	4.45	5.83	7.26	3.63
2	17.5	β-elemene[a]	1.59	2.20	1.74	1.07
3	17.7	2-butyl-2-octenal [a]	0.20	0.21	0.19	0.30
4	17.9	Caryophyllene	0.06	0.14	0.10	0.08
5	18.5	Trans-caryophyllene	-	0.52	0.47	0.24
6	18.8	γ-elemene	-	-	0.12	0.06
7	19.4	α-amorpholene[a]	0.37	0.39	0.35	0.30
8	19.9	Bicyclogermacrene [a]	0.62	0.57	0.57	0.46
9	20.0	γ- selinene	-	0.13	0.10	0.10
10	20.3	Germacrene D	0.10	-	0.07	-
11	20.4	α- guaiene	0.14	0.19	0.18	-
12	20.5	α- selinene	0.29	0.36	0.33	-
13	20.6	β- patchoulene	0.2	0.20	0.04	-
14	20.8	(-)-α-gurjunene[a]	0.34	0.36	0.30	0.30
15	21.3	β- selinene	0.05	-	0.07	-
16	22.1	Cinetol	0.13	-	0.23	-
17	22.2	α- acoradiene	-	0.40	-	0.27
18	22.2	γ-ylangolene [b]	-	-	0.41	-
19	22.3	Melaleucanol [a]	0.36	0.37	0.37	0.37
20	22.8	Globulol[a]	0.32	0.41	0.38	0.38
21	23.1	Epicuruginol[a]	0.52	0.60	0.51	0.47
22	23.2	Caryophyllin[a]	1.02	1.12	0.90	0.94
23	23.4	Retinal	0.25	-	0.24	-
24	23.7	Phoratene	0.47	-	2.43	1.91
25	23.8	Epoxidized humuleneII [a]	0.21	0.56	0.36	0.53
26	24.1	Pogostol [a]	24.56	25.66	24.01	22.60
27	24.2	1,6--dimethyl-4 -(1-methylethyl) naphthalene	0.79	0.78	-	0.82
28	24.3	（-）- spathyl alcohol	-	0.43	0.61	0.44
29	25.5	Berkheyaradulene [a]	0.27	0.26	0.23	0.24
30	25.6	Coxyl alcohol	-	0.54	0.45	0.43

Note:(1) “-” indicates that it is not detected; (2) “a” represents the common component; (3) “b” stands for characteristic ingredient.

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