

# 气相色谱-三重四极杆质谱分析人参炮制品中的挥发性成分

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**摘要** 人参炮制的化学成分变化研究主要集中在皂苷和糖类, 本文首次从挥发性成分角度阐释了人参不同炮制品的物质基础。利用气相色谱-质谱联用(GC-MS/MS)方法, 对鲜参、生晒参和红参中挥发性成分及其衍生规律进行研究。采用TG-5SILMS非极性气相色谱柱, 以He为载气, 通过NIST MS Spectral Database对挥发性成分进行检测并鉴定。鲜参、生晒参、红参中分别检出30、33和34种挥发性成分, 其中生晒参中(-)-斯巴醇含量为鲜参含量的31.98倍, 辛醛等8种挥发性成分为鲜参中含量的3倍以上, 红参中有环癸等10种挥发性成分为鲜参中含量的3倍以上。生晒参和红参中各有4种挥发性成分在鲜参中未检出。

**关键词** 气相色谱-三重四极杆质谱; 鲜参; 生晒参; 红参; 挥发性成分

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人参是多年生五加科草本植物人参(*Panax Ginseng* C. A. Mey.)的干燥根和根茎, 具有生津养血、安神益智和抑制肿瘤等功效<sup>[1]</sup>。人参中活性成分主要包括人参皂苷、人参多糖、氨基酸、挥发性成分等<sup>[2]</sup>, 其中人参皂苷与人参糖类的研究较多<sup>[3-5]</sup>。人参挥发性成分气味独特, 抗疲劳等功能性产品已上市。人参中的挥发性成分质量分数仅为0.1%~0.5%, 但其消炎、镇咳、抗疲劳、使神经中枢兴奋等作用明显, 还能够通过抑制癌细胞的核酸代谢、糖代谢达到抑制癌细胞生长的作用<sup>[6]</sup>, 其中 $\beta$ -榄香烯在抗癌方面得到了较多的研究和关注<sup>[7-8]</sup>。人参有生晒参和红参等不同炮制品, 功效不同, 气味也不同, 生晒参中炔醇类物质较蒸制过的炮制品中含量高, 其对革兰阳性金黄色葡萄球菌、枯草芽孢杆菌、肺炎双球菌等抑菌作用更强<sup>[9]</sup>, 同时其抗肿瘤和神经保护等活性更强<sup>[10-11]</sup>。气相色谱-质谱联用(GC-MS/MS)技术与NIST MS Spectral Database结合使用, 常用作脂肪酸和挥发性成分的测定<sup>[12-13]</sup>。本研究采用水蒸气回流提取法结合气相色谱-三重四极杆质谱联用检测技术<sup>[14-15]</sup>, 建立了GC-MS/MS测定人参挥发性成分的方法, 应用于对鲜参、生晒参和红参中挥发性成分的比较, 明确鲜参及其炮制品挥发性成分的组成, 推断生晒参和红参中的挥发性成分衍生路径, 为人参炮制机理的研究提供挥发性成分的化学数据。

## 1 实验部分

### 1.1 仪器和试剂、药材

TRACE 1310 GC-Triple Quadrupole MS型气相色谱串联质谱仪(美国Thermo Science公司)。

正己烷(色谱纯, TEDIA公司), 蒸馏水(实验室自制)。鲜参(5年生); 生晒参(由同批次鲜参人参挖出洗净放置通风阴凉处阴干后的炮制品); 红参(由同批次鲜参经过清洗、分选、蒸制5 h后、50℃烘干等工序加工而成)。

### 1.2 实验方法

1.2.1 样品提取 精密称取粉碎后过150  $\mu\text{m}$  筛的样品100 g, 加入600 mL水, 水蒸气回流提取8 h, 将提取到的挥发性转移至离心管中, 并用2 mL正己烷冲洗容器内壁, 涡旋3 min, 取正己烷层, 从中准确吸取20  $\mu\text{L}$ 于1 mL容量瓶中, 用正己烷稀释并定容, 即得供试品溶液。生晒参和红参按照炮制前后重量比例提取制备。

1.2.2 GC-MS操作条件 GC条件: TG-5SILMS非极性色谱柱(30 m  $\times$  0.25 mm  $\times$  0.25  $\mu\text{m}$ )。进样口

温度为 250 ℃, 初始温度 50 ℃, 保持 3 min; 升温程序, 从 50 ℃ 开始, 先以 5 ℃/min 升至 120 ℃, 再以 1 ℃/min 升至 125 ℃, 保持 3 min, 再以 1 ℃/min 升至 130 ℃, 保持 3 min, 再以 1 ℃/min 升至 135 ℃, 保持 3 min, 再以 1 ℃/min 升至 140 ℃, 保持 3 min, 再以 1 ℃/min 升至 145 ℃, 保持 3 min, 再以 1 ℃/min 升至 150 ℃, 保持 3 min; 载气为 He, 载气流量为 1.2 mL/min; 分流比为 35, 进样量为 1 μL。

MS 条件: EI 离子源, 进样口温度 270 ℃, 离子源温度 250 ℃, 传输线温度 250 ℃, 扫描范围 50 ~ 500  $m/z$ 。

## 2 结果与讨论

### 2.1 挥发性成分的定性分析

从图 1 可以看出, 鲜参、生晒参和红参中的挥发性成分差异较大, 在化合物数量和含量方面, 炮制前后及不同方法炮制均对挥发性成分有显著的影响。

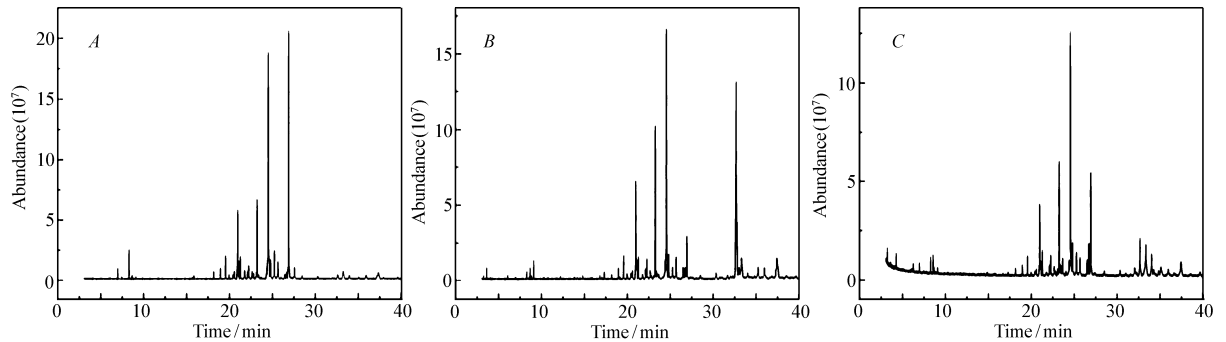


图 1 鲜参总离子色谱图(A)、生晒参总离子色谱图(B)和红参总离子色谱图(C)

Fig. 1 The total ion chromatography of the fresh ginseng(A), the sun-dry(B) and the red ginseng(C)

GC-MS 由于具有恒定的 70 eV 电离能和标准数据库, 因此能够不使用标准对照品, 鉴定挥发性成分<sup>[16-17]</sup>。本研究通过 GC-MS/MS 一级质谱图分子量和二级串联质谱子离子信息, 与 NIST MS Spectral Database 数据库比对, 鉴定了 40 个人参中的挥发性化合物, 具体如表 1 所示。

表 1 鲜参、生晒参、红参挥发性成分定性分析

Table 1 The volatile components in qualitative analysis of fresh ginseng, sun-dry ginseng and red ginseng

No.	Retention time/min	Compounds	Formula	Molecular mass	Relative peak area		
					Fresh ginseng	Sun-Dry ginseng	Red ginseng
1	6.96	$\alpha$ -Pinene	C <sub>10</sub> H <sub>16</sub>	136	1	0.24	0.77
2	7.43	Camphene	C <sub>10</sub> H <sub>16</sub>	136	1	0.17	0.53
3	8.27	$\beta$ -Pinene	C <sub>10</sub> H <sub>16</sub>	136	1	0.24	0.38
4	8.66	Ningene	C <sub>10</sub> H <sub>16</sub>	136	1	1.98	1.05
5	9.07	Octanal	C <sub>8</sub> H <sub>16</sub> O	128	1	4.90	1.70
6	15.85	Benzene, 2-methoxy-4-methyl-1-(1-methyl)-	C <sub>11</sub> H <sub>16</sub> O	164	1	0.48	-
7	16.78	Cyclodecanone	C <sub>10</sub> H <sub>18</sub> O	154	-	+	+
8	17.31	6-(5-Methyl-furan-2-yl)-hexan-2-one	C <sub>11</sub> H <sub>16</sub> O <sub>2</sub>	180	-	++	+
9	18.13	Cedrene	C <sub>15</sub> H <sub>24</sub>	204	1	0.51	0.76
10	18.93	$\beta$ -Elemene	C <sub>15</sub> H <sub>24</sub>	204	1	0.71	0.61
11	19.14	4,4,6-Trimethyl-cyclohex-2-en-1-ol	C <sub>9</sub> H <sub>16</sub> O	140	-	+	-
12	19.52		C <sub>14</sub> H <sub>20</sub> O	204	1	0.83	0.56
13	19.93	2-(3-Isopropyl-4-methyl-pent-3-en-1-ynyl)-2-methyl-cyclobutanone	C <sub>14</sub> H <sub>20</sub> O	204	1	1.11	0.72
14	20.95	Ginsengene	C <sub>15</sub> H <sub>24</sub>	204	1	1.29	0.67
15	21.24	Caryophyllene	C <sub>15</sub> H <sub>24</sub>	204	1	0.77	0.70
16	21.76	Neoclovene	C <sub>15</sub> H <sub>24</sub>	204	1	-	0.40

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No.	Retention time/min	Compounds	Formula	Molecular mass	Relative peak area		
					Fresh ginseng	Sun-Dry ginseng	Red ginseng
17	22.22	Aromadendrene	C <sub>15</sub> H <sub>24</sub>	204	1	1.15	0.89
18	22.64	Aristolene	C <sub>15</sub> H <sub>24</sub>	204	1	0.90	0.77
19	23.21	α-Elemene	C <sub>15</sub> H <sub>24</sub>	204	1	1.52	0.89
20	23.65	Farnesene	C <sub>15</sub> H <sub>24</sub>	204	-	-	++
21	24.50	Transcaryophyllene	C <sub>15</sub> H <sub>24</sub>	204	1	0.88	0.68
22	24.76	Ai Li Mo phenol	C <sub>15</sub> H <sub>24</sub>	204	1	1.09	1.04
23	25.22	β-Selinene	C <sub>15</sub> H <sub>24</sub>	204	1	0.32	0.49
24	25.63	δ-Cadinene	C <sub>15</sub> H <sub>24</sub>	204	1	1.05	0.58
25	26.47	α-Guaiene	C <sub>15</sub> H <sub>24</sub>	204	-	++	-
26	26.63	1 <i>H</i> -Cycloprop[ <i>e</i> ] azulene, 1 <i>a</i> , 2, 3, 5, 6, 7, 7 <i>a</i> , 7 <i>b</i> -octahydro-1, 1, 4, 7-tetramethyl-, [ 1 <i>aR</i> -( 1 <i>a</i> ù, 7 <i>a</i> ù, 7 <i>a</i> ù, 7 <i>b</i> ù) ]-	C <sub>15</sub> H <sub>24</sub>	204	1	1.21	2.97
27	26.87	Azulene, 1, 2, 3, 3 <i>a</i> , 4, 5, 6, 7-octahydro-1, 4, dimethyl-7-(1-methylethenyl)-, [ 1 <i>R</i> , ( 1 <i>a</i> , 3 <i>a</i> ù, 4 <i>a</i> , 7 <i>a</i> ) ]-	C <sub>15</sub> H <sub>24</sub>	204	1	0.16	0.27
28	27.56	Naphthalene, 1, 2, 3, 5, 6, 7, 8, 8 <i>a</i> -octahydro-1, 8 <i>a</i> -dimethyl-7-(1-methylthethyl)-, [ 1 <i>R</i> -( 1 <i>a</i> , 7 <i>a</i> ù, 8 <i>a</i> ù) ]-	C <sub>15</sub> H <sub>24</sub>	204	++	-	-
29	28.44	1-(1, 5-dimethylhexyl)-4-methylbenzene	C <sub>15</sub> H <sub>24</sub>	204	1	0.93	1.10
30	30.26		C <sub>15</sub> H <sub>26</sub> O	222	1	1.86	1.66
31	32.04	New ginseng terpene alcohols	C <sub>15</sub> H <sub>26</sub> O	222	-	-	++
32	32.61	( - )-Spathulenol	C <sub>15</sub> H <sub>24</sub> O	220	1	31.98	5.47
33	33.24	β-Piperonyl butylene	C <sub>15</sub> H <sub>24</sub>	204	1	1.86	-
34	33.35	Globulol	C <sub>15</sub> H <sub>26</sub> O	222	-	-	++
35	33.94	ξ-EIMACHALENE	C <sub>15</sub> H <sub>26</sub> O	222	1	1.42	4.32
35	34.83	7- <i>epi</i> - <i>cis</i> -Sesquisabinene	C <sub>15</sub> H <sub>26</sub> O	222	-	-	++
37	35.02	à-Acorenol	C <sub>15</sub> H <sub>26</sub> O	222	1	-	3.74
38	35.22	Caryophyllene oxide	C <sub>15</sub> H <sub>24</sub> O	220	-	++	-
39	35.97	4- <i>epi</i> -Cubedol	C <sub>15</sub> H <sub>26</sub> O	222	-	++	+
40	37.42	2 <i>H</i> -3, 9 <i>a</i> -Methanol-1-benzoxepin, octahydro-2, 2, 5 <i>a</i> , 9-tetramethyl-, [ 3 <i>R</i> -( 3 <i>a</i> ù, 5 <i>a</i> ù, 9 <i>a</i> ù, 9 <i>a</i> ù) ]-	C <sub>15</sub> H <sub>26</sub> O	222	1	2.10	1.35

In the table; the peak area of compound in fresh ginseng was 1 and the relationship area of processed ginseng was calculated by ratio between the amount of processed ginseng and fresh ginseng. “ - ”: none of the components, “ + ”: indicates the peak area of 10<sup>6</sup>, “ ++ ”: peak area is 10<sup>7</sup>, “ +++ ”: peak area of 10<sup>8</sup>.

由表 1 和图 2 可知,鲜参、生晒参和红参中共检测出 40 种挥发性成分,其中烯类 22 种,醛类 1 种,酮类 3 种,醇类 8 种,杂环类 6 种。鲜参中共检测出烯类 20 种挥发性成分,醛类 1 种,酮类 1 种,醇类 4 种,杂环类 4 种,共 30 种挥发性成分;生晒参中检测出烯类 20 种,醛类 1 种,酮类 3 种,醇类 4 种,杂环类 5 种,共 33 种挥发性成分;红参中检测出烯类 19 种,醛类 1 种,酮类 3 种,醇类 8 种,杂环类 3 种,共

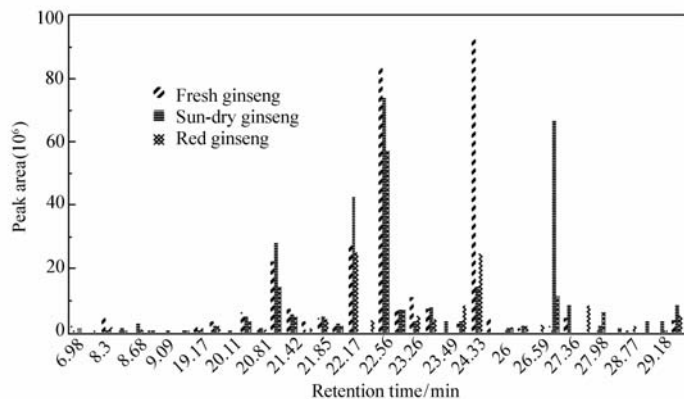


图 2 鲜参、生晒参、红参挥发性成分定性分析比较

Fig. 2 The comparison of the volatile compounds in fresh ginseng, sun-dry ginseng and red ginseng

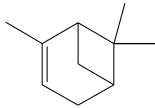
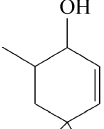
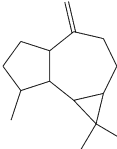
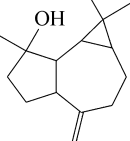
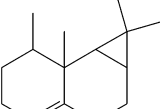
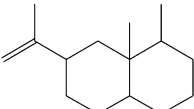
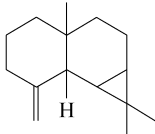
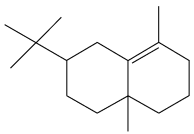
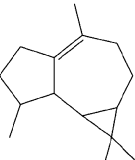
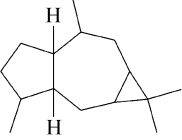
34 种挥发性成分。

## 2.2 炮制过程中挥发性成分的转化

由图 2 中挥发性成分研究结果可知,生晒参在阴干或晾干过程中挥发性成分有部分损失,使含量降低或挥发性成分种类减少,有少数新挥发性成分生成;红参由于经过高温蒸制及低温烘干等热处理后,一部分挥发性成分含量明显增高,推测是由与其结构相似的挥发性成分转化而来,使其含量增加。推断炮制后部分发现的新挥发性成分的衍生途径如表 2。

表 2 挥发性成分存在的转化关系

Table 2 Transformation relationship of volatile components

No.	Structural formula	Raw material		Transformation products	
		Compounds	Structural formula	Compounds	
1		$\alpha$ -Pinene		4,4,6-Trimethyl-cyclohex-2-en-1-ol	
2		Aromandrene		(-)-Spathulenol	
3		$\alpha$ -Elemene		Farnesene	
4		Ginsengene		New ginseng terpene alcohols	
5		1 <i>H</i> -Cycloprop[ <i>e</i> ]azulene, 1 <i>a</i> ,2,3,5,6,7,7 <i>a</i> ,7 <i>b</i> - octahydro-1,1,4,7-tetramethyl-, [1 <i>a</i> R-(1 <i>a</i> ü,7ü,7 <i>a</i> ü,7 <i>b</i> ü)]-		Globulol	

炮制过程中,鲜参中具有不饱和双键的挥发性成分,加热条件下,易发生加成反应,生成饱和的或部分饱和的加成产物。人参中化学成分含量较多且相互作用复杂,红参的炮制过程中化学成分之间相互作用,有大量酶参与反应,故一些含量较高的成分不能确定是由结构相似的化合物转化而得,其生成过程有待进一步研究和讨论。

生晒参中(-)-斯巴醇含量为鲜参含量的 31.98 倍,为红参含量的 5.84 倍,是生晒参中检测出最多的挥发性成分。研究表明,(-)-斯巴醇属聚炔醇类化合物,是五加科植物中较常见的成分,具有很强的抗肿瘤活性,可抑制血管紧张素 II 诱导的血管收缩,降低自发性高血压大鼠平均动脉压,同时还具有抗炎及抗血小板凝集活性。人参脂溶性成分(含聚醇类)具明显促进神经细胞生长的活性,可降低血压、降血脂及对冠心病患者有食疗作用<sup>[18-21]</sup>。由于鲜参、生晒参和红参中艾里莫酚烯含量均相对较高,且可能转化为(-)-斯巴醇,由此可见鲜参在晾晒成生晒参时,一部分艾里莫酚烯转化为(-)-斯巴醇,使生晒参在抗肿瘤活性、降血压、降血脂方向的作用更加明显。

红参中金合欢烯是鲜参和生晒参中均不含有的特有成分,有报道<sup>[22]</sup>称金合欢醇具有消炎健胃作用,民间将金合欢醇用于治疗风湿扭伤等病,符合无梗五加在《中华本草》中传统药理记载。由于鲜参中不含该成分,不具有该功效,但由于鲜参中含有的 $\alpha$ -榄香烯含量较红参高,且在鲜参炮制成红参的过程中可能转化为金合欢烯,故红参较鲜参增加了消炎健胃的作用。

### 3 结 论

本文通过气相色谱-质谱联用方法,检测了鲜参、生晒参、红参中的挥发性成分,并对炮制前后差异较大的化合物进行了衍生规律推测,结合已报道的化合物生物活性,从挥发性成分的角度阐释了人参炮制品的物质基础,为人参的临床应用和规范化炮制质量控制提供了化学数据支持。

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## Analysis of Volatile Components in Processed Ginseng by GC-MS/MS

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**Abstract** The research of chemical changes in ginseng processing has been focusing on saponins and sugars. In this paper, volatile components in ginseng were studied for the first time, which provide a substance basis for different processed ginseng. The volatile components and their derivative paths of fresh ginseng, sun-dry ginseng and red ginseng were studied by gas chromatography-mass spectrometry (GC-MS/MS). The volatile components were detected and analyzed using TG-5SILMS nonpolar GC column, with He as the carrier gas through the NIST MS Spectral Database. Thirty, thirty-three and thirty-four species of volatile compounds in fresh ginseng, sun-dry ginseng and red ginseng were detected, respectively. The content of (-)-spartak alcohol in sun-dry ginseng is 31.98 times higher than that in the fresh ginseng, while the levels of eight volatile components such as octanal in sun-dry ginseng is three times higher than those in the fresh ginseng. The contents of ten volatile components such as cyclodecanone in red ginseng are 3 times higher than those in the fresh ginseng. Four volatile compounds in sun-dry ginseng and in red ginseng are not detected in fresh ginseng.

**Keywords** gas chromatography-mass spectrometry; fresh ginseng; sun-dry ginseng; red ginseng; volatile compound

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