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尾叶香茶菜不同部位挥发性成分比较分析

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摘　要:本文采用气流吹扫微萃取法对尾叶香茶菜根、茎、叶挥发性成分进行分析比较。采用 GC-MS 对挥发性成分进行分离检测,与标准谱库对比进行定性,采用面积归一化法计算各化合物的相对含量。尾叶香茶菜共检测到 149 个峰,与标准谱库比对,根、茎、叶分别鉴定出 80、85、79 种挥发性成分,占各部位挥发油总量的 93.66%、88.05%和 72.62%。采用化学方法结合主成分分析法对尾叶香茶菜根、茎、叶的挥发性成分进行分析,结果显示根、茎、叶的挥发性成分在组成和含量上均存在一定差异,本研究为尾叶香茶菜的进一步开发利用提供参考依据。

关键词:尾叶香茶菜;挥发性成分;不同部位;气流吹扫微萃取法;主成分分析

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Comparative Analysis of the Volatile Components from Different Parts of *Rabdosia excise*

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Abstract:In this study the volatile compositions in root,stem and leaf of Rabdosia excise (Maxim.) Hera were determined by gas purge micro-extraction techniques. The contents were analyzed by gas chromatography-mas spectrum (GC-MS). The qualitative of volatile components was completed by comparison with the standard spectrum library,and the relative content of each compound was calculated using the area normalization. The 149 peaks were detected in Rabdosia excise,comparison with the standard spectrum library,80,85 and 79 components were identified in root,stem and leaf, which accounted for 93.66%,88.05% and 72.62% of the total peaks in each part. The volatile components of root, stem and leaf for Rabdosia excise were compared by chemical method and principal component analysis. The results showed that the chemical compositions and relative contents were different to some extent for the volatile components in root,stem and leaf. This study provides reference basis for development and utilization of *Rabdosia excise*.

Key words:*Rabdosia excisa*;volatile components;different parts;gas purge microextraction method (GP-ME);principal component analysis (PCA)

尾叶香茶菜 *Rabdosia excisa* (Maxim.) Hera 为唇形科香茶菜属植物,属于多年生草本植物,广泛分布黑龙江、吉林及辽宁等省区[1,2]。主要生长于草丛、林中、林缘等阴生地带。尾叶香茶菜全草入药,性味苦凉,具有清热解毒、健脾、活血等功效[3]。在民间,尾叶香茶菜被用于治疗风湿感冒、喉咙肿痛、关节疼痛、蛇虫咬伤等[4]。

目前,关于尾叶香茶菜的研究重点聚焦于水或醇提取物化学成分分离及药理活性研究,且多为全草[5]。在中草药开发中,尾叶香茶菜作为药用植物的一员,在抗癌、抗肿瘤等研究中的价值越来越受到科研人员的关注。目前关于尾叶香茶菜挥发性成分的研究报道很少,且重点聚焦于尾叶香茶菜挥发性成分的定性分析[6-8],及活性探究,并发现了挥发性成分具有降低血液中胆固醇浓度[7]、抑菌、抗氧化活性[8]等作用。关于尾叶香茶菜不同部位挥发性成分比较的相关研究未见报道,且尾叶香茶菜中已定性挥发性成分非常有限。为此,本研究采用气流吹扫微萃取方法与 GC-MS 联用对尾叶香茶菜挥发性成分开展系统性研究,利用化学方法和统计学方

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法(主成分分析)比较其不同部位(根、茎、叶)挥发性成分的差异,为尾叶香茶菜的进一步开发提供参考依据。

1　仪器与材料

1.1　仪器与试剂

气相色谱-质谱联用仪(岛津,日本,GC-MS plus 2010),100 μL 衬管(安捷伦,美国),AS20500AH 型超声波清洗器(天津 AU-TOSCIENCE 公司),FW-100 高速万能粉碎机(天津泰斯特),进样垫(岛津,日本),聚四氟乙烯管等。色谱纯正己烷(Fisher,美国)。

1.2　材料

尾叶香茶菜于 2016 年 8 月采自吉林省长白山,将根、茎、叶分别剪切、洗净后置于阴凉通风处在自然条件下阴干。干燥的尾叶香茶菜用粉碎机粉碎,过 200 目筛,置于棕色样品瓶中,-20 °C 贮存备用。

2　实验方法

2.1　挥发性成分的萃取

依据先前报道[9-11],操作步骤如下:①准确称取粉碎的尾叶香茶菜植物样品 5.0 mg,放入样品槽中,并安装仪器;②取 50 μL 正己烷置于 100 μL 衬管(接收器)中;③打开开关开始萃取;④萃取后,利用正己烷定容至 100 μL,随后取 2 μL 进样分析。气流吹扫微萃取条件:氮气流速为 2　mL/min,萃取温度为 250 °C,萃取时间为 2 min,冷凝温度为-4 °C。

2.2　挥发性成分的 GC-MS 分析

DB-5 MS 毛细石英柱(30 m × 0.25 mm × 0.25 μm),载气 He (纯度≥99.99%),进样口温度 280 °C,进样量 2 μL,分流比 50∶1,载气流速为 0.98 mL/min,升温程序:45 °C 保持 1 min,4 °C/min 升至 250 °C, 6 °C/min 升至 280 °C,保持 5 min。离子源 EI,离子源温度 200 °C,电子能量 70 eV。

利用岛津化学工作站中 NIST 10 标准质谱图库进行检索,并结合相关文献图谱解析对挥发性成分进行定性分析。同时,应用手动积分方法对信噪比大于 10 的所有色谱峰进行积分,并按面积归一化法计算出各化学成分的相对含量,具体见附表 1。

表 1　尾叶香茶菜不同部位挥发性成分比较

Table 1　Comparative of the volatile components from different parts of *Rabdosia excise*

序号 No.	时间 Time	化合物名称 Compound	不同部位含量 Content (%)		
			根 Root	茎 Stem	叶 Leaf
1	5.349	3-Hexanone	0.50	0.33	0.27
2	5.434	Unknown-1	2.44	1.68	0.81
3	5.699	3-Hexanol	0.56	0.35	0.17
4	5.806	3-Methyl-1-pentanol	0.48	0.16	0.07
5	5.966	(Z)-2-Octene	–	0.57	0.54
6	6.383	Unknown-2	–	0.30	–
7	6.475	2-Methylpyrazine	–	0.29	0.31
8	6.737	Furaldehyde	12.58	5.73	1.94
9	7.433	. alpha. -Furfuryl alcohol	3.54	6.20	1.91
10	7.535	(3E)-3-Hexen-1-ol	–	–	0.28
11	7.942	1-Methoxy-2-propyl acetate	0.86	1.42	0.75
12	8.036	o-Xylene	–	–	2.28
13	8.384	2-Cyclopentene-1,4-dione	1.41	1.10	0.57
14	8.668	Pivalic acid vinyl ester	0.13	0.07	0.01
15	8.804	Cinnamene	0.11	0.22	0.14
16	8.958	4-Hydroxy-3-hexanone	–	0.20	–
17	9.228	Unknown-3	0.35	0.86	0.18

续表 1(Continued Tab. 1)

序号 No.	时间 Time	化合物名称 Compound	不同部位含量 Content (%)		
			根 Root	茎 Stem	叶 Leaf
18	9.338	2-Oxo-2,5-dihydrofuran(2-[5H]-furanone)	1.1	1.32	0.42
19	9.433	Unknown-4	0.66	1.13	0.59
20	9.557	2,5-Dimethylpyrazine	0.20	0.07	0.07
21	9.671	Unknown-5	0.32	0.37	0.28
22	9.91	1,2-Cyclopentanedione	0.71	3.49	0.67
23	10.206	Unknown-6	0.05	0.10	–
24	10.281	Unknown-7	0.07	0.10	0.06
25	10.388	5-Methyl-2(5H)-Furanone	0.11	0.22	0.15
26	10.886	3-Hydroxy-4,4-dimethyldihydro-2(3H)-furanone	0.26	0.11	0.1
27	11.099	5-Methyl-2-Furanmethanol	0.25	0.18	0.08
28	11.22	2,3-Dimethyl-2-cyclopenten-1-one	0.11	0.21	0.06
29	11.438	5-Methyl-2-furaldehyde	3.75	0.60	0.3
30	11.507	Benzaldehyde	0.53	0.42	0.34
31	11.625	Propanoic acid, 1-methylpropyl ester	–	0.41	0.11
32	11.742	(3E)-3-Methyl-3-hepten-2-one	–	0.12	0.11
33	11.85	2-Methyl-2-butenolide	–	0.23	0.12
34	11.876	5-Hydroxymethyldihydrofuran-2-one	0.16	–	–
35	12.132	Phenol	0.31	1.38	1.67
36	12.279	.alpha.-Methylstyrene	0.15	–	–
37	12.292	1-Octen-3-ol	–	5.75	3.13
38	12.566	2H-Pyran-2,6(5H)-dione	0.15	0.45	0.17
39	12.664	2-n-Pentylfuran	0.06	–	–
40	12.759	Acetic acid furfurylester	0.06	0.50	–
41	12.9	Unknown-8	–	0.62	–
42	12.954	2,2-Diethyl-3-methyl-1,3-oxazolidine	3.79	–	–
43	13.398	2-Formylpyrrole	1.1	0.47	0.44
44	13.5	(*E*,*E*)-2,4-Heptadienal	–	0.17	0.2
45	13.542	Unknown-9	–	0.07	–
46	13.725	Unknown-10	–	0.16	0.26
47	13.829	3-Methyl-1,2-cyclopentanedione	0.24	1.49	0.8
48	14.175	Unknown-11	0.2	0.28	0.14
49	14.342	Benzenemethanol	–	0.42	0.54
50	14.716	2-Methoxy-5-methylthiophene	1.13	–	–
51	14.75	Benzeneacetaldehyde	–	0.17	0.24
52	14.975	Unknown-12	–	0.21	–
53	15.05	Unknown-13	–	0.76	–
54	15.09	4-Hydroxy-2,5-dimethyl-3(2H)furanone	0.44	–	–
55	15.429	2-Acetylpyrrole	0.53	0.35	0.16

续表 1(Continued Tab. 1)

序号 No.	时间 Time	化合物名称 Compound	不同部位含量 Content (%)		
			根 Root	茎 Stem	叶 Leaf
56	15.588	Hypnone	0.56	0.36	0.19
57	15.692	3-Methyl-2,5-Furandione	4.45	–	–
58	15.783	2-(4-Methylcyclohexyl) ethanol	–	0.17	0.06
59	15.9	3-Methylcyclohexanone	–	0.33	0.25
60	16.006	2,5-Furandicarboxaldehyde	0.30	–	–
61	16.105	2-Furyl hydroxymethyl ketone	0.37	0.62	0.17
62	16.342	2-Methoxy-Phenol	2.01	3.25	0.97
63	16.52	3-Ethyl-2-hydroxy-2-cyclopenten-1-one	0.22	2.14	0.89
64	16.776	2-Methyl-1,3-cyclohexanedione	0.23	–	0.11
65	16.833	3-Ethenyl-3-methylcyclopentanone	–	2.84	0.46
66	17.017	Linalool	–	1.19	0.43
67	17.051	2,6-Pyridinediol	0.23	–	–
68	17.202	Maltol	1.27	0.73	0.52
69	17.333	4,5-Dimethyl-2-cyclohexen-1-one	–	0.18	–
70	17.417	Benzeneethanol	–	0.55	0.91
71	17.779	N-methyl-2-formylpyrrole	0.13	–	–
72	17.882	Pentanoic acid, pentyl ester	–	–	0.32
73	17.892	Unknown-14	–	0.31	–
74	18.196	Unknown-15	0.19	–	–
75	18.274	4-Vinylcyclohexanone	–	0.34	0.18
76	18.767	1,3-Dimethyltetrahydro-2(1H)-pyrimidinone	–	0.44	0.54
77	18.917	Unknown-16	–	0.22	–
78	20.125	Unknown-17	–	0.64	0.3
79	20.333	1,2-Benzenediol	0.67	2.51	1.3
80	21.35	Dihydrobenzofuran	–	0.52	1.6
81	21.574	5-Hydrxoymethylfurfural	1.85	0.39	–
82	21.705	Unknown-18	0.07	0.16	–
83	22.534	Picolinamide	0.11	–	–
84	22.666	2-Hydroxy-4-methylphenol	0.12	1.10	0.22
85	23.346	2,6-Dihydroxyacetophenone	0.30	1.31	0.47
86	23.853	1,7,7-Trimethylbicyclo[2.2.1]hept-2-yl acetate	0.27	–	–
87	24.042	Indole	–	–	0.48
88	24.678	4-Hydroxy-2-methylacetophenone	3.52	3.43	3.31
89	25.895	2,6-Dimethoxy phenol	1.13	5.78	2.28
90	26.112	3-Allyl-2-methoxyphenol	0.55	0.24	0.63
91	26.242	Unknown-19	–	–	0.33
92	26.9	4-Ethyl-1,2-benzenediol	–	1.24	1.37
93	27.512	4-hydroxy-3-methoxy-Benzaldehyde	3.45	2.41	0.31

续表 1(Continued Tab. 1)

序号 No.	时间 Time	化合物名称 Compound	不同部位含量 Content (%)		
			根 Root	茎 Stem	叶 Leaf
94	28.767	Unknown-20	–	–	0.90
95	29.231	cis-Isoeugenol	3.74	1.15	0.20
96	29.583	Unknown-21	–	–	0.17
97	29.733	Unknown-22	–	–	0.19
98	30.124	6-Methoxy-3-methylbenzofuran	1.01	–	–
99	30.25	(3*E*)-4-(2,6,6-Trimethyl-1-cyclohexen-1-yl)-3-buten-2-one	–	–	0.57
100	30.278	1-(4-hydroxy-3-methoxyphenyl)-ethanone	0.76	0.47	0.12
101	30.358	Unknown-23	–	–	0.29
102	31.258	4a-Methyl-4,4a,5,6,7,8-hexahydro-2(3H)-naphthalenone	–	–	0.07
103		Unknown-24	–	–	0.1
104	31.565	Guaiacylacetone	0.65	1.74	0.44
105	31.758	4,5,7,7a-Tetrahydro-4,4,7a-trimethyl-2(6H)benzofuranone	–	–	0.39
106	32.669	3,5-Dimethoxyacetophenone	0.92	1.66	0.14
107	32.925	Unknown-25	–	–	0.29
108	33.256	Di(sec-butyl) 2-methylsuccinate	0.54	0.43	0.14
109	33.334	2-Amino-1-(3-hydroxy-4-methoxyphenyl)ethanone	0.66	0.11	–
110	33.526	Diethyl Phthalate	0.35	0.31	0.32
111	33.731	Unknown-26	0.29	0.28	0.13
112	33.882	Unknown-27	0.23	–	–
113	33.971	2,6,10,15-Tetramethylheptadecane	0.21	0.26	0.13
114	34.561	2,4a,5,8a-Tetramethyl-1,2,3,4,4a,7,8,8a-octahydro-1-naphthalenyl acetate	3.12	–	0.26
115	35.05	Unknown-28	–	–	0.09
116	35.377	Syringaldehyde	1.04	0.58	–
117	35.558	Unknown-29	–	–	0.18
118	35.8	Unknown-30	–	–	0.25
119	35.942	Unknown-31	–	–	0.22
120	36.308	Unknown-32	–	–	0.25
121	36.648	2,6-Dimethoxy-4-allylphenol	1.68	1.44	0.17
122	37.533	3-(4-Hydroxy-3-methoxyphenyl)-2-Propenal	12.61	3.34	0.25
123	37.622	4-((1*E*)-3-Hydroxy-1-propenyl)-2-methoxy-phenol	0.61	1.11	–
124	37.993	Unknown-33	0.31	–	–
125	38.339	Unknown-34	0.15	0.24	0.04
126	38.525	Unknown-35	–	0.21	0.28
127	40.447	(2*E*)-3,7,11,15-Tetramethyl-2-hexadecen-1-ol	2.75	2.81	17.81
128	40.592	Unknown-36	–	0.34	0.93
129	40.982	Diisobutyl phthalate	0.76	0.52	0.41
130	41.083	Unknown-37	–	0.43	3.18
131	41.541	Unknown-38	1.01	1.00	6.94

续表 1(Continued Tab. 1)

序号 No.	时间 Time	化合物名称 Compound	不同部位含量 Content (%)		
			根 Root	茎 Stem	叶 Leaf
132	43.217	Unknown-39	–	–	0.26
133	43.361	Dibutyl phthalate	0.65	0.48	0.21
134	43.6	Palmitic acid	–	0.25	0.77
135	43.824	3,5-Dimethoxy-4-hydroxycinnamaldehyde	1.68	1.04	–
136	46.525	Nonadecanol	–	–	0.96
137	46.825	Methyl linolenate	–	–	0.81
138	47.065	Phytol	1.16	1.69	7.03
139	47.8	Unknown-40	–	–	7.36
140	48.367	Unknown-41	–	–	0.68
141	51.067	Behenic alcohol	–	–	0.35
142	51.6	Unknown-42	–	0.58	0.7
143	52.45	Unknown-43	–	–	0.41
144	54.533	Unknown-44	–	–	0.24
145	55.508	Unknown-45	–	0.49	–
146	57.85	Unknown-46	–	0.41	–
147	58.825	Unknown-47	–	–	0.35
148	60.437	Spinacene	1.11	0.54	2.4
149	61.83	n-Hexatriacontane	0.6	0.88	1.58

注:–未检出。
Note: – not deteated.

2.3　统计分析

统计分析采用主成分分析(PCA)方法,利用 SPSS Statistics 统计软件完成。

3　结果与讨论

3.1　尾叶香茶菜中挥发性和半挥发性成分分析

尾叶香茶菜中共检测出 149 个色谱峰,根、茎、叶分别检测到 86、97、106 个。与标准谱库比对,已定性成分各占总成分的 93.66%、88.05%、72.62%,尾叶香茶菜中共有 102 个峰被定性。各部位挥发性成分的 GC-MS 分析的总离子流程图结果见图 1。采用数据处理中的手动积分法对每个峰进行积分处理,以峰面积归一化法测得其中各组分相对百分含量。具体信息见附表 1。

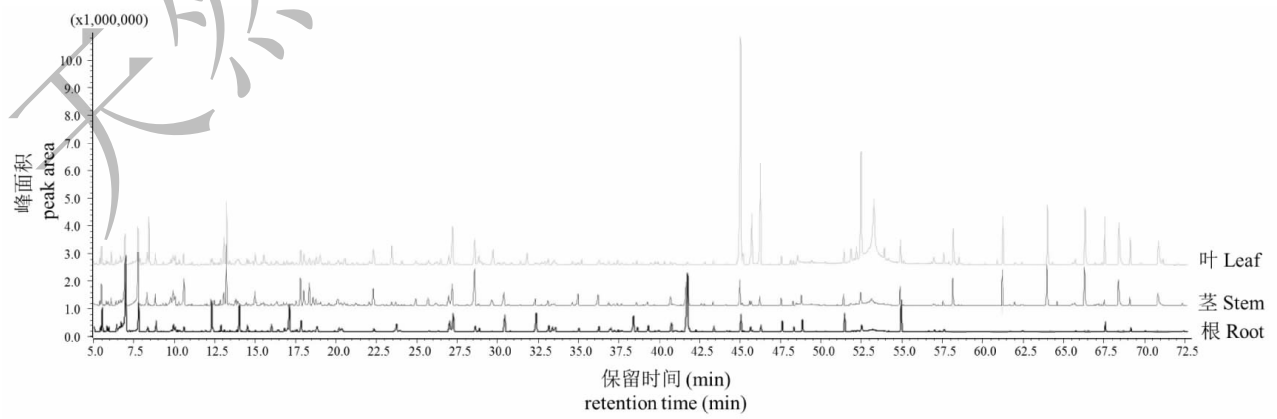

图 1　尾叶香茶菜不同部位挥发性成分总离子流程图

Fig. 1　Total ion current chromatogram of volatile components in different parts of *Rabdosia excise* (Maxim.) Hera

文献报道,尾叶香茶菜挥发性成分提取方法有 CO_2 超临界萃取法[6,8]、水蒸气蒸馏法[7],两种方法定性挥发性成分分别为 15 和 34 种,本文采用气流吹扫微萃取法定性挥发性成分 102 种。从样品消耗量方面考虑以往方法一般需 200 g 左右[8],气流吹扫微萃取法仅需 5.0 mg。另外,以往方法至少需要 60 min 萃取时间[8],而气流吹扫微萃取法仅需 2 min。综合上所述,中草药挥发性成分定性分析推荐采用气流吹扫微萃取法。

3.2 不同部位化学组成比较研究

从色谱的总离子流图可知,尾叶香茶菜的各部分均检测到多种挥发性成分,但从总离子流图中很难直观区分三者的差异。另外,即使经检索、解析和文献比对,确认每种成分,但仍难以从成分和含量信息中直观区分三者。为此,本文分别采用化学和统计学方法进行比较分析。

3.2.1 按不同官能团比较分析

解析已定性成分的结构,发现所有挥发性成分都是 C4 ~ C36 的化合物,涉及化合物类型包含醇类、酮类、醛类、酯类、酚类、酸类等。为此,将所有成分依据化合物所含官能团共分 12 类。从图 2 可知,根中含量最多的成分是醛类(37.5%),其次是酮类(12.9%)、其他含 N-、O-类(12.4%)及酚类(10.8%);叶中最多成分为醇类(33.9%),其次是酮类(10.5%)和酚类(8.81%);茎中酮类(23.3%)、醇类(19.7%)、酚类(19.2%)三类组分相差不多,其次是醛类(14.9%)。分析结果显示尾叶香茶菜根、茎、叶中挥发性物质的组成类型有明显差异。

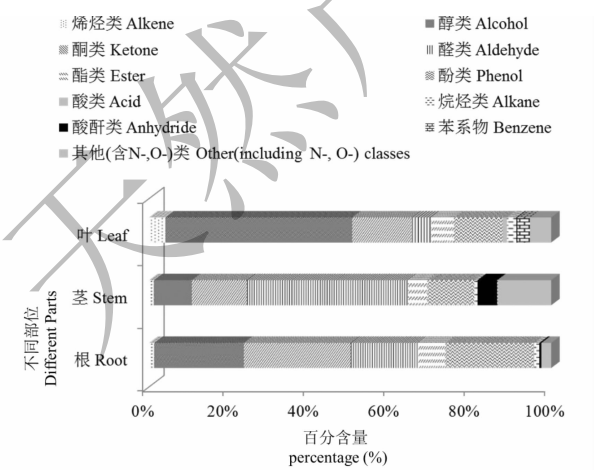

图 2 尾叶香茶菜不同部位化学成分官能团分类比较

Fig. 2 Comparison of functional groups of chemical components in different parts of *Rabdosia excise* (Maxim.) Hera

3.2.2 主成分(PCA)分析

为了更直观的辨别尾叶香茶菜不同部位之间是否存在实质性差异,本文采用 PCA 统计方法进行比较,分别以 58 个共用成分及全部成分的官能团分类数据作为变量,具体结果见图 3 和图 4。在图 3 中第一个主成分(X-axis:PC1)占 57.6%,第二个主成分(Y-axis:PC2)占 29.8%。在图4 中第一个主成分(X-axis:PC1)占 64.0%,第二个主成分(Y-axis:PC2)占 29.6%。在两种作图方式中,所选择的两个主成分之和占总变量的比例均大于 85%。两种变量的统计分析结果均清晰显示尾叶香茶菜的三个部位挥发性成分有明显差异。通过分析可知每一部位的主要贡献成分,根中主要贡献成分是醛类、酮类、其他含 N-、O-类及酚类;茎中主要贡献成分是酮类、醇类和酚类;叶中主要贡献成分则是醇类、酮类和酚类。该结果显示,利用统计学方法可以将复杂的药用植物挥发性成分数据简单化,可直观显示部位之间的异同,这说明统计学方法有望应用于香茶菜属植物种类之间的鉴别,下一步将开展相关研究,希望能够为药用植物间的鉴别提供一种科学、直观的方法。

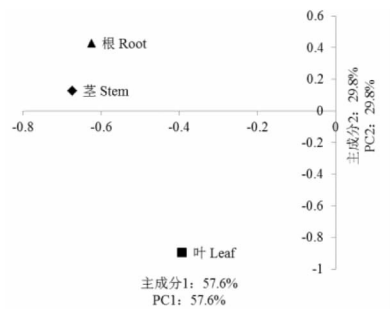

图 3 共有成分主成分分析图

Fig. 3 PCA diagram of common component

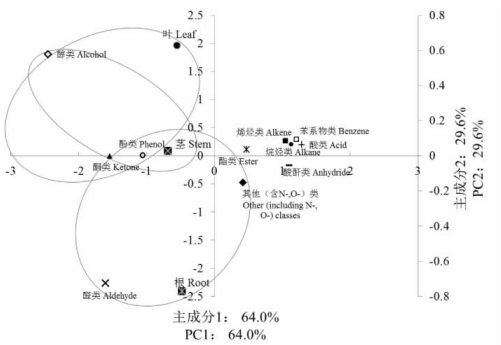

图 4 全部成分的官能团分类主成分分析图

Fig. 4 PCA diagram of functional group classification of all components

4　结论

本文研究结果显示采用气流吹扫微萃取技术可从尾叶香茶菜中萃取到更多的挥发性成分,而更多的成分信息可为后续尾叶香茶菜的深入研究提供基础。研究结果说明统计方法可应用于植物挥发性成分的比较中,并将其结果与化学方法相结合,能够更加直观准确的说明尾叶香茶菜不同部位中挥发性成分存在差异及各部位主要贡献成分。本文对尾叶香茶菜不同部位挥发性成分的比较研究将为尾叶香茶菜的进一步开发利用提供基础数据支撑。

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