赤松与长白赤松松针挥发油成分的 GC – MS 分析*

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摘要 **目的:** 利用 GC – MS 分析赤松与长白赤松松针挥发油的成分。**方法:** 以水蒸气蒸馏法分别提取赤松与长白赤松挥发油,利用气相色谱 – 质谱联用技术分别对所提取的挥发油成分进行分析。进样口温度 230 ℃,色谱柱为 HP – 5(0.25 mm × 30 m,0.25 μm) 石英毛细管柱,柱流速为 1 mL·min^{-1},进样量 0.5 μL,分流比 40∶1,程序升温 [50 ℃(5 ℃·min^{-1}),120 ℃(8 ℃·min^{-1}),180 ℃(5 ℃·min^{-1}),200 ℃]。质谱条件: 离子源为 EI 源,电子能量为 70 eV,离子源温度 230 ℃,接口温度 280 ℃,质量扫描范围为 20 ~ 500 amu。**结果:** 从赤松松针挥发油共分离出 36 个成分,经谱图检索确认了其中的 30 个成分,已鉴定的化合物占色谱总馏出峰面积的 98.37%,其中相对含量较高的为 β – 水芹烯(20.964%)、α – 蒎烯(16.081%)、莰烯(11.529%)、檀香三烯(16.471%);从长白赤松松针挥发油中共分离出 34 个成分,经谱图检索确认了其中的 31 个成分,已鉴定的化合物占色谱总馏出峰面积的 99.24%,其中相对含量较高的为 1 – *R* – α – 蒎烯(17.592%)、石竹烯(15.398%)、β – 水芹烯(14.813%)、[*S* – (*E*,*E*)] – 1 – 甲基 – 5 – 亚甲基 – 8 – 异丙基 – 1,6 – 环癸二烯(7.573%)、β – 蒎烯(5.929%)、异松油烯(4.663%)。**结论:** 赤松与长白赤松松针挥发油成分大体一致,均富含具有较强药理活性的 α – 蒎烯、β – 水芹烯、莰烯、檀香三烯等成分。

关键词: 赤松; 长白赤松; 松针; 挥发油; 化学成分; 比较研究; 气相色谱 – 质谱

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GC – MS analysis of volatile oil from the pine needles of *Pinus densiflora* and *Pinus sylvestris* var. *sylvtriforms**

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Abstract **Objective:** To analyze volatile oil from the pine needles of *Pinus densiflora* Sieb. et Zucc. and *Pinus sylvestris* L. var. *sylvtriforms* Cheng et C. D. Chu by GC – MS. **Methods:** The volatile oil was extracted by steam distillation from the pine needles of *P. densiflora* and *P. sylvestris* var. *sylvtriforms*, respectively, and analyzed by gas chromatography – mass spectrometry(GC – MS) technology. injector temperature was 230 ℃. The GC – MS analysis with a HP – 5 fused silica capillary column(0.25 mm × 30 m, 0.25 μm). Flow rate was 1 mL·min^{-1}. The split ratio was 40∶1. The injection volume was 0.5 μL. The temperature programming was begun at 50 ℃, then programmed from 50 to 120 ℃ at 5 ℃·min^{-1}, then programmed from 120 to 180 ℃ at 8 ℃·min^{-1} and finally, programmed from 180 to 200 ℃ at 5 ℃·min^{-1}. Mass spectrometer conditions were: ionization mode, EI; electron energy 70 eV; interface temperature 280 ℃; ion source temperature 230 ℃. The mass selective detector was operated in the TIC mode, mass scan range, 20 – 500 amu. **Results:** From volatile oil of the pine needles of *P. densiflora*, 36 compounds were separated, 30 of them were identified; These 30 compounds amounted totally to about 98.37% of the total peak area; The major components in the volatile oil were β – phellandrene(20.96%), α – pinene(16.081%), camphene(11.529%), and santolina triene(16.471%). From volatile oil of the pine needles of *P. sylvestris* var. *sylvtriforms*, 34 compounds were separated, 31 of them were identified; These 31 compounds amounted totally to about 99.24% of the total peak area; The major components of the volatile oil were 1 – *R* – α – pinene(17.592%), caryophyllene(15.398%), β – phellandrene(14.813%), [*S* – (*E*,*E*)] – 1 – methyl – 5 – methylene – 8 – (1 – methylethyl) – 1,6 – cyclodecadiene(7.573%), β – pinene(5.929%), 1 – methyl – 4 – (1 –

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methylethylidene) - cyclohexene(4.663%). **Conclusions:** Components of volatile oil of the pine needles of *P. densiflora and P. sylvestris* var. *sylvtriforms* are similar, and both rich with a strong pharmacological activities such as α-pinene, β-phellandrene, camphene and santolina triene.

Key words: *Pinus densiflora* Sieb; *Pinus sylvestris sylvtriforms*; pine needles; volatile oil; chemical composition; comparative study GC-MS

松针药食同源,据《本草纲目》记载,松针长期服用,能治百病、安五脏、生毛发、耐寒暑、耐风吹雨打、轻身益气、守中而辟谷延年。现代研究表明松针具有抗炎、镇咳、抗突变、降血脂、祛风活血、明目安神、解毒、止痒、降低总胆固醇和低密度脂蛋白胆固醇等药理作用。用于治疗流行性感冒、风湿关节痛、跌打肿痛、夜盲症、高血压病、神经衰弱,外用治冻疮[1]。松针含有大量蛋白质、脂类物质和胡萝卜素、丰富的叶绿素、维生素和微量元素等,可用于研究开发营养保健品和营养饲料[2]。赤松(*Pinus densiflora* Sieb. et Zucc.)主要分布在华东及北部沿海地区,北亚热带落叶常绿阔叶混交林区东部[3]。长白赤松(*Pinus sylvestris* L. var. *sylvtriforms* Cheng et C. D. Chu)又称为长白松、美人松,是长白山特有品种,被定为我国第1、第2批珍稀濒危植物资源。天然长白赤松生于长白山北坡海拔800~1600 m的高海拔地区,目前哈尔滨、吉林白城、沈阳等地均已引种栽培[4]。本研究首次对吉林通化地区赤松与长白赤松松针挥发油化学成分进行研究报道,为后期研究提供相关的理论基础。

1 仪器与材料

气相色谱-质谱联用仪(HP6890/HP5973 美国惠普公司)。

赤松与长白赤松松针,均采自吉林省通化市,经于俊林教授鉴定为赤松(*Pinus densiflora* Sieb. et Zucc.)与长白赤松(*Pinus sylvestris* L. var. *sylvtriforms* Cheng et C. D. Chu)的松针,将其阴干备用。

2 方法与结果

2.1 样品前处理 将松针剪成1 cm左右的小段,分别称取200 g用蒸馏水浸泡2 h后放入挥发油提取器中,采用水蒸气蒸馏法在循环蒸馏器中进行蒸馏,蒸馏时间为4 h,得具特殊气味的乳白色油状液体。用乙醚溶解并稀释约0.01 mg·mL^{-1}的挥发油溶液,0.45 μm的微孔滤膜过滤后分别进行GC-MS联用测定。

2.2 色谱条件 气相色谱条件:进样口温度230 ℃,载气为高纯氦,HP-5(0.25 mm×30 m,0.25 μm)石英毛细管柱,柱流速为1 mL·min^{-1},进样量0.5 μL,分流比40∶1,程序升温[50 ℃(5 ℃·min^{-1}),120 ℃(8 ℃·min^{-1}),180 ℃(5 ℃·min^{-1}),200 ℃]。质谱条件:离子源为EI源,电子能量为70 eV,离子源温度230 ℃,倍增器电压1.482 kV,四极杆温度150 ℃,发射电流34.6 μA,接口温度280 ℃,质量扫描范围为20~500 amu,溶剂延迟时间3 min[5]。

2.3 实验结果 用毛细管气相色谱法分别对赤松与长白赤松的松针挥发油化学成分进行了分析,经气相色谱机用面积归一化法测定了各组分的百分含量,并用GC/MS联用技术对总离子流各色谱峰进行检测(见图1和图2),所得质谱图经计算机NIST质谱数据库检索,并按各峰的质谱裂片图与文献资料核对,确定了松针的挥发油成分,鉴定结果见表1和表2。

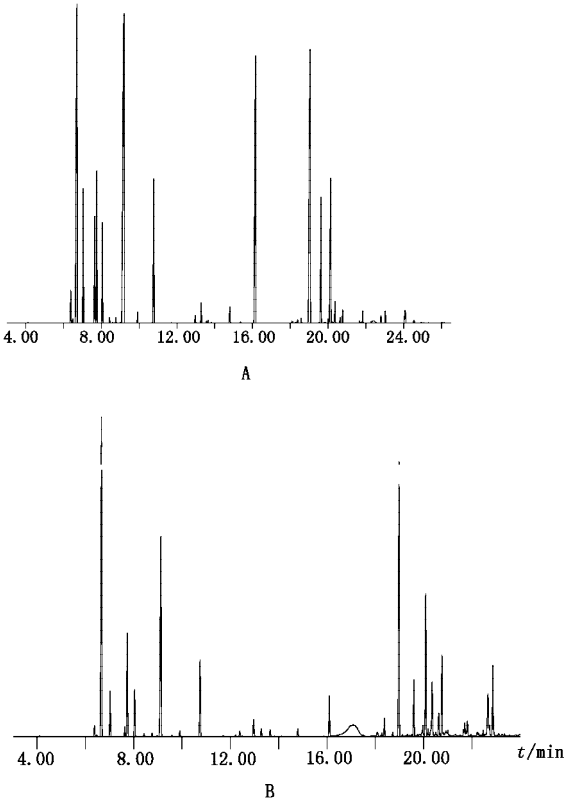

图1 赤松松针(A)及长白赤松松针(B)挥发油总离子流图

Fig 1 The total ions chromatograms of volatile oil from the pine needles of *P. densiflora*(A) and *P. sylvestris* var. *sylvtriforms*(B)

表1 赤松松针挥发油成分的 GC-MS 分析结果

Tab 1 Analytical results of chemical components of volatile oil from the pine needles of *P. densiflora*

峰号 (peak No.)	t_R/min	化合物 (compound)	分子式 (Formula)	M_r	相对含量(relative content)/%
1	6.386	1,7,7-三甲基-三环[2.2.1.02,6]-庚烷 (1,7,7-trimethyl-tricyclo[2.2.1.0(2,6)]heptane)	$C_{10}H_{16}$	136	0.874
2	6.486	2-甲基-5-(1-甲基乙基)-二环[3.1.0]己-2-烯 (2-methyl-5-(1-methylethyl)-bicyclo[3.1.0]hex-2-ene)	$C_{10}H_{16}$	136	0.113
3	6.715	α-蒎烯(alpha-pinene)	$C_{10}H_{16}$	136	16.081
4	7.046	莰烯(camphene)	$C_{10}H_{16}$	136	3.703
5	7.655	4-甲基-1-(1-甲基乙基)-1,4-环己二烯 (4-methyl-1-(1-methylethyl)-bicyclo[3.1.0]hex-2-ene)	$C_{10}H_{16}$	136	3.431
6	7.762	B-蒎烯(beta-pinene)	$C_{10}H_{16}$	136	4.600
7	8.064	β-月桂烯(beta-myrcene)	$C_{10}H_{16}$	136	2.892
8	8.442	α-水芹烯(alpha-phellandrene)	$C_{10}H_{16}$	136	0.132
9	8.772	1-甲基-4-(1-甲基乙基)-环己烯 (1-methyl-4-(1-methylethyl)-1,3-cyclohexadiene)	$C_{10}H_{16}$	136	0.125
10	9.204	β-水芹烯(beta-phellandrene)	$C_{10}H_{16}$	136	20.964
11	9.921	松油烯(1-methyl-4-(1-methylethyl)-1,4-cyclohexadiene)	$C_{10}H_{16}$	136	0.254
12	10.780	异松油烯(1-methyl-4-(1-methylethylidene)-cyclohexene)	$C_{10}H_{16}$	136	4.956
13	12.969	龙脑((1S-endo)-1,7,7-trimethyl-bicyclo[2.2.1]heptan-2-ol)	$C_{10}H_{18}O$	154	0.210
14	13.287	4-甲基-1-(1-甲基乙基)-3-环己基-1-醇 (4-methyl-1-(1-methylethyl)-3-cyclohexen-1-ol)	$C_{10}H_{18}O$	154	0.584
15	13.571	α,α,4-三甲基-3-环己二烯-1-醇 (alpha,alpha,4-trimethyl-3-cyclohexene-1-methanol)	$C_{10}H_{18}O$	154	0.057
16	13.654	α-萜品醇(2-methoxy-4-methyl-1-(1-methylethyl)-benzene)	$C_{10}H_{18}O$	154	0.070
17	14.798	乙酸冰片酯(bornyl acetate)	$C_{12}H_{20}O_2$	196	0.450
18	16.156	3-蒈烯(3-carene)	$C_{10}H_{16}$	136	11.529
19	18.086	未知物(unknow)			0.024
20	18.389	1-十四炔(1,2-dimethoxy-4-(2-propenyl)-benzene)	$C_{13}H_{22}$	178	0.076
21	18.577	3,7-二甲基-1,3,7-辛三烯((1R)-2,2-dimethyl-3-methylene-bicyclo[2.2.1]heptane)	$C_{10}H_{16}$	136	0.108
22	19.053	檀香三烯(santolina triene)	$C_{10}H_{16}$	136	16.471
23	19.622	7,7-二甲基-2-亚甲基-二环[2,2,1]-庚烷 (7,7-dimethyl-2-methylene-bicyclo[2.2.1]heptane)	$C_{10}H_{16}$	136	3.612
24	20.007	1,1-双亚乙烯基-环丙烷 (1,4-bis(methylene)-cyclohexane)	C_8H_{12}	108	0.103
25	20.139	2,5,5-三甲基-1,3,6-庚三烯 (2,5,5-trimethyl-1,3,6-heptatriene)	$C_{10}H_{16}$	136	5.990
26	20.364	未知物(unknow)			0.569
27	20.632	3,7,11-三甲基-1,6,10-十二三烯-3-醇 (1,1′-ethenylidenebis-cyclopropane)	C_8H_{12}	108	0.122
28	20.767	未知物(unknow)			0.278
29	21.663	香树烯氧化物(caryophyllene oxide)	$C_{15}H_{24}O$	220	0.040
30	21.829	乙酸顺-9,反-11-十四二烯-1-酯 ((1R)-2,2-dimethyl-3-methylene-bicyclo[2.2.1]heptane)	$C_{10}H_{16}$	136	0.283
31	22.276	未知物(unknow)			0.016
32	22.341	1-羟基-1,7-二甲基-4-异丙基-2,7-环癸二烯 (dodecahydro-acenaphthylene)	$C_{12}H_{20}$	164	0.015
33	22.795	3-蒈烯(7-(1-methylethylidene)-bicyclo[4.1.0]heptane)	$C_{10}H_{16}$	136	0.215
34	23.029	罗汉柏烯(3-(1-methyl-2-propenyl)-1,5-cyclooctadiene)	$C_{12}H_{18}$	162	0.308
35	24.071	未知物(unknow)			0.634
36	24.546	未知物(unknow)			0.112

表 2 长白赤松松针挥发油成分的 GC – MS 分析结果

Tab 2 Analytical results of chemical components of volatile oil from the pine needles of *P. sylvestris* var. *sylvtriforms*

峰号 (peakNo.)	t_R/min	化合物 (compound)	分子式 (molecular formula)	M_r	相对含量 (relativecontent)/%
1	6.383	3 – 蒈烯(3 – carene)	$C_{10}H_{16}$	136	0.56
2	6.669	1 – *R* – α – 蒎烯(1*R* – alpha – Pinene)	$C_{10}H_{16}$	136	17.592
3	7.029	莰烯(camphene)	$C_{10}H_{16}$	136	2.481
4	7.636	1,4 环己二烯,1 – 甲基 – 4 – (1 – 甲基乙基)(4 – methylene – 1 – (1 – methylethyl) – bicyclo [3.1.0] hexane)	$C_{10}H_{16}$	136	0.519
5	7.739	β – 蒎烯(beta – pinene)	$C_{10}H_{16}$	136	5.929
6	8.048	*B* – 月桂烯(beta – myrcene)	$C_{10}H_{16}$	136	2.522
7	9.129	β – 水芹烯(beta – phellandrene)	$C_{10}H_{16}$	136	14.813
8	9.914	1 – 甲基 – 4 – (1 – 甲基亚乙基) 环己烯(1 – methyl – 4 – (1 – methylethyl) – 1,4 – cyclohexadiene)	$C_{10}H_{16}$	136	0.317
9	10.752	异松油烯(1 – methyl – 4 – (1 – methylethylidene) – cyclohexene)	$C_{10}H_{18}O$	154	4.663
10	12.965	冰片(borneol)	$C_{10}H_{18}O$	154	1.09
11	13.285	萜烯醇(4 – methyl – 1 – (1 – methylethyl) – 3 – cyclohexen – 1 – ol)	$C_{10}H_{18}O$	154	0.503
12	13.625	1 – 甲醇 – α,α,4 – 三甲基 – 3 – 环己烯(alpha,alpha,4 – trimethyl – 3 – cyclohexene – 1 – methanol)	$C_{10}H_{18}O$	154	0.411
13	14.795	2 – 异丙基 – 5 – 甲基茴香醚(2 – methoxy – 4 – methyl – 1 – (1 – methylethyl) – benzene)	$C_{11}H_{16}O$	164	0.428
14	16.101	乙酸冰片酯(bornyl acetate)	$C_{12}H_{20}O_2$	196	2.237
15	16.982	未知物(unknow)			0.389
16	17.036	3,7,11 – 三甲基 – 14 – 异丙基 – 1,3,6,10 – 环十四碳烯(3,7,11 – trimethyl – 14 – (1 – methylethyl) – 1,3,6,10 – cyclotetradecatetraene)	$C_{20}H_{32}$	272	0.082
17	17.086	未知物(unknow)			0.11
18	18.383	榄香烯 [1*S* – (1alpha,2beta,4beta)] – 1 – ethenyl – 1 – methyl – 2,4 – bis(1 – methylethenyl) – cyclohexane)	$C_{15}H_{24}$	204	0.942
19	18.987	石竹烯(caryophyllene)	$C_{15}H_{24}$	204	15.398
20	19.6	α – 丁香烯(alpha – caryophyllene)	$C_{15}H_{24}$	204	2.945
21	19.971	1,2,3,4,4a,5,6,8a – 八 H – 7 – 甲基 – 4 – 亚甲基 – 1 – (1 – 甲基乙基) – 萘(1,2,3,4,4a,5,6,8a – octahydro – 7 – methyl – 4 – methylene – 1 – (1 – methylethyl) – naphthalene)	$C_{15}H_{24}$	204	0.588
22	20.09	[*S* – (*E*,*E*)] – 1 – 甲基 – 5 – 亚甲基 – 8 – 异丙基 – 1,6 – 环癸二烯([*S* – (*E*,*E*)] – 1 – methyl – 5 – methylene – 8 – (1 – methylethyl) – 1,6 – cyclodecadiene)	$C_{15}H_{24}$	204	7.573
23	20.185	β – 葎草烯(beta – humulene)	$C_{15}H_{24}$	204	0.344
24	20.291	未知物(unknow)			0.257
25	20.356	[*E*,*E*] – 1,5 – 二甲基 – 8 – 甲基乙二基 – 1,5 – 环癸二烯((*E*,*E*) – 1,5 – dimethyl – 8 – (1 – methylethylidene) – 1,5 – cyclodecadiene)	$C_{15}H_{24}$	204	3.525
26	20.628	1,2,4a,5,6,8a – 六 H – 4,7 – 二甲基 – 1 – (1 – 甲基乙基) – 萘(1,2,4a,5,6,8a – hexahydro – 4,7 – dimethyl – 1 – (1 – methylethyl) – naphthalene)	$C_{15}H_{24}$	204	1.255
27	20.761	1,2,3,5,6,8a – 六氢 – 4,7 – 二甲基 – 1 – (1 – 甲基乙基) – 萘(1,2,3,5,6,8a – hexahydro – 4,7 – dimethyl – 1 – (1 – methylethyl) – naphthalene)	$C_{15}H_{24}$	204	3.806
28	21.656	2 – 异丙基 – 5 – 甲基 – 9 – 亚甲基二环 [4.4.0] 十 – 1 – 烯(2 – isopropyl – 5 – methyl – 9 – methylene – bicyclo [4.4.0] dec – 1 – ene)	$C_{15}H_{24}$	204	0.257
29	21.705	匙柃醇 [1ar – (1a. alpha,4a. alpha,7beta,7a. beta,7b. alpha)] – decahydro – 1,1,7 – trimethyl – 4 – methylene – 1h – cycloprop [e] azulen – 7 – ol)	$C_{15}H_{24}O$	220	0.634
30	21.816	氧化石竹烯(caryophyllene oxide)	$C_{15}H_{24}O$	220	0.759
31	22.468	1,2,3,4,4a,7 – 六 H – 1,6 – 二甲基 – 4 – (1 – 甲基乙基) – 萘(1,2,3,4,4a,7 – hexahydro – 1,6 – dimethyl – 4 – (1 – methylethyl) – naphthalene)	$C_{15}H_{24}$	204	0.283
32	22.667	*T* – 依兰油醇(tau – muurolol)	$C_{15}H_{26}O$	222	3.293
33	22.724	可巴烯(copaene)	$C_{15}H_{24}$	204	0.51
34	22.866	α – 荜澄茄醇(alpha – cadinol)	$C_{15}H_{26}O$	222	3.761

3 讨论

由表1可见,赤松松针挥发油中相对含量较高的为β-水芹烯(20.964%)、檀香三烯(16.471%)、α-蒎烯(16.081%)、3-蒈烯(11.529%)、异松油烯(4.956%)、β-蒎烯(4.600%)、莰烯(3.703%)、7,7-二甲基-2-亚甲基-二环[2,2,1]-庚烷(3.612%)、4-甲基-1-(1-甲基乙基)-1,4-环己二烯(3.431%)、β-月桂烯(2.892%),上述10个成分含量占总含量的88.239%。

由表2可见,长白赤松松针挥发油中相对含量较高的为1-*R*-α-蒎烯(17.592%)、石竹烯(15.398%)、β-水芹烯(14.813%)、[*S*-(*E*,*E*)]-1-甲基-5-亚甲基-8-异丙基-1,6-环癸二烯(7.573%)、β-蒎烯(5.929%)、异松油烯(4.663%)、1,2,3,5,6,8a-六氢-4,7-二甲基-1-(1-甲基乙基)-萘(3.806%)、α-荜澄茄醇(3.761%)、[*E*,*E*]-1,5-二甲基-8-甲基乙二基-1,5-环癸二烯(3.525%)、*T*-依兰油醇(3.293%)、α-丁香烯(2.945%)、β-月桂烯(2.522%)、莰烯(2.481%)、乙酸冰片酯(2.237%),上述14种成分含量占总含量的90.201%。

赤松与长白赤松挥发油的主要化学成分均为通式为$C_{10}H_{16}$和$C_{15}H_{24}$的烯、烷类以及单萜与倍半萜类化合物,化学成分有一定的相近之处,主要化学成分是与其他地域的松种略有不同。云南松松针的主要化学成分为倍半萜[6],兰州油松[5]、广西马尾松和湿地松[7]、山东蒙山黑松[8]、黑龙江樟子松[9]和红松[10]的松针成分都以单萜类化合物为主。

赤松与长白赤松松针挥发油中均检测到多种药理活性成分,如具有抗炎、镇咳、祛痰等作用的α-蒎烯、β-水芹烯、β-蒎烯,具有胆石溶解作用的柠檬烯,这些以萜烯为主的一系列不饱和碳氢化合物及其衍生物可提高体内抗自由基氧化能力,减少脂质过氧化,起到延缓衰老的作用[11]。

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