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樟树叶化学成分的研究

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摘 要:本文研究了樟树叶的化学成分。主要采用硅胶、Sephadex LH-20 凝胶和聚酰胺等柱层析方法对樟树叶的化学成分进行分离纯化,并根据理化性质、波谱数据(^{1}H NMR,^{13}C NMR)鉴定化合物的结构。从樟树叶的甲醇提取物中分离鉴定出 9 个化合物,分别为山柰酚-3-*O*-*β*-D-葡萄糖苷(**1**)、槲皮素-3-*O*-*β*-D-鼠李糖苷(**2**)、槲皮素-3-*O*-*β*-D-葡萄糖苷(**3**)、异鼠李素-3-*O*-*β*-D-葡萄糖苷(**4**)、黑色五味子单体苷(**5**)、山柰酚-3-*O*-*β*-D-芸香糖苷(**6**)、异鼠李素-3-*O*-*β*-D-芸香糖苷(**7**)、新芝麻脂素(**8**)和 maculatin(**9**)。化合物 **1** 为首次从樟属中分离得到,化合物 **4**、**5**、**8** 为首次从樟科植物中分离得到。

关键词:樟树;化学成分;黄酮;木脂素;樟科

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A Study on the Chemical Constituents in the Leaves of *Cinnamomum camphora*

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Abstract: To investigate the chemical constituents of the leaves of *Cinnamomum camphora*. Silica gel, Sephadex LH-20 and Polyamide column chromatography were applied for isolation of the constituents. Nine compounds (**1**-**9**) were isolated and their structures were identified by spectroscopic analysis as following: kaempferol-3-*O*-*β*-D-glucopyranoside (**1**), quercetin-3-*O*-*β*-D-rhamnopyranoside (**2**), quercetin-3-*O*-*β*-D-glucopyranoside (**3**), isorhamnetin-3-*O*-*β*-D-glucopyranoside (**4**), schizandriside (**5**), kaempferol-3-*O*-*β*-D-rutinoside (**6**), isorhamnetin-3-*O*-*β*-D-rutinoside (**7**), neo-sesamin (**8**) and maculatin (**9**). Compound **1** was isolated from *Cinnamomum* spp. for the first time. Compounds **4**, **5** and **8** were isolated from *Lauraceae* for the first time.

Key words: *Cinnamomum camphora*; chemical constituents; flavonoids; lignans; Lauraceae

樟树(*Cinnamomum camphora*)是樟科(Lauraceae)樟属植物,别名:香樟、樟木、芳樟、乌樟、油樟、香蒻、小叶樟,系常绿乔木[1]。樟树分布广泛,主产于我国南方各省区,以福建、台湾为最多,为热带和亚热带常绿阔叶林的代表树种,被誉为江南宝树。樟树的化学成分主要有挥发油、黄酮类、生物碱、有机酸、木脂素及核糖体失活蛋白等,樟树的茎、叶、根、果实、种子等各部分都具有一定的生物活性,主要涉及抑菌[2,3]、食品防腐保鲜[4,5]、驱虫[6,7]、抗氧化[8]、消炎及细胞毒活性[9]等方面,在临床医学应用上也越来越常见[10,11]。

目前,对樟树的研究大多集中在挥发油的化学成分检测分析和粗提物生物活性方面,而对于樟树叶片中非挥发性成分的化学组成相关报道较少。本文以干燥处理的樟树叶为材料,经过醇提、萃取、柱层析对樟树叶中有效的非挥发性成分进行分离及结构鉴定,共分离和鉴定出 9 个化合物,最后与已知文献数据对照,确证化合物结构。分别为山柰酚-3-*O*-*β*-D-葡萄糖苷(**1**),槲皮素-3-*O*-*β*-D-鼠李糖苷(**2**),槲皮素-3-*O*-*β*-D-葡萄糖苷(**3**),异鼠李素-3-*O*-*β*-D-葡萄糖苷(**4**),黑色五味子单体苷(**5**),山柰酚-3-*O*-*β*-D-芸香糖苷(**6**),异鼠李素-3-*O*-*β*-D-芸香糖苷(**7**),新芝麻脂素(**8**)和 maculatin(**9**)。其中,化合物 **1** 为首次从樟属中分离得到,化合物 **4**、**5** 和 **8** 为首次从樟科植物中分离得到。

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1 材料与仪器设备

樟树叶采自安徽合肥市苗圃,经安徽农业大学林学与园林学院黄成林教授鉴定为 *Cinamomum camphora* 的树叶。

核磁共振波谱仪(美国 BROKER 公司,400 MHz ^{1}H NMR,100 MHz ^{13}C NMR),旋转蒸发器(上海申胜生物技术有限公司 R-201 型),核磁测试用氘代试剂 DMSO、氯仿、甲醇(美国 CIL 公司),电子天平(上海天平仪器厂 T6328B 型),电热鼓风干燥箱(上海一恒科技有限公司),实验试剂为分析纯(天津博迪有限公司),硅胶(柱层析用,100 ~ 200、200 ~ 300、300 ~ 400 目,青岛海洋化工厂分厂),葡聚糖凝胶(Sephadex LH-20,美国 Pharmacia 公司),聚酰胺粉(国药集团化学试剂有限公司),聚酰胺薄膜(国药集团化学试剂有限公司)。

2 提取与分离

将 8.4 kg 烘干樟树叶全部粉碎,用甲醇浸提 3 次,得 750 g 樟树叶甲醇粗提浸膏,依次采用石油醚、氯仿、正丁醇 3 种不同极性的有机试剂分级萃取,得到石油醚部分(100 g)、氯仿部分(45 g)、正丁醇部分(160 g)及水相 4 个部分的分级萃取物浸膏。

正丁醇浸膏(160 g)经 HPD-500 型大孔树脂层析柱,得到Ⅰ ~ Ⅴ五个部分。部分 II(51.7 g)湿法过硅胶柱,乙酸乙酯-甲醇-水(10∶0.5∶0.5→6∶0.5∶0.5→2∶0.5∶0.5)梯度洗脱得到组分 1 ~ 13。组分 2(2.7 g),依次通过过硅胶柱(乙酸乙酯-甲醇-水)、聚酰胺柱(二氯甲烷-甲醇)梯度洗脱,得到化合物 **1**(15 mg)和化合物 **2**(39 mg)。组分 3(1.5 g),上聚酰胺柱,用二氯甲烷-甲醇(4∶1)洗脱剂洗脱,得化合物 **3**(10 mg)和化合物 **4**(16 mg)。组分 4(约 200 mg)有白色化合物析出,抽滤得白色沉淀部分,过凝胶柱得到化合物 **5**(10 mg)。组分 7(3.2 g),湿法过硅胶柱,氯仿-甲醇-水(15∶1∶0.1→10∶1∶0.1→7∶1∶0.1→7∶3∶0.5)梯度洗脱,得化合物 **6**(40 mg)。组分 8(1.6g),湿法过硅胶柱,二氯甲烷-甲醇-水(15∶1∶0.1→10∶1∶0.1→7∶1;0.1→8;2∶0.3)梯度洗脱,得化合物 **7**(35 mg)。

氯仿浸膏(45 g),湿法过硅胶柱,丙酮-石油醚(6∶1→4∶1→2∶1→1∶1)梯度洗脱,得化合物 **8**(50 mg)和化合物 **9**(100 mg)。

3 结构鉴定

化合物 1 黄色粉末(甲醇);EIS-MS *m/s*:447 [M-H]$^-$。^{1}H NMR (DMSO-d_6, 400 MHz) δ: 12.61 (1H, s, 5-OH), 8.05 (2H, d, J = 8.8 Hz, H-2′, 6′), 6.89 (2H, d, J = 8.8 Hz, H-3′, 5′), 6.44 (1H, d, J = 2.0 Hz, H-8), 6.21 (1H, d, J = 2.0 Hz, H-6), 5.46 (1H, d, J = 7.6 Hz, H-1′′)。以上数据与文献[12]报道数据一致,故鉴定化合物 **1** 为山柰酚-3-*O*-β-D-葡萄糖糖苷。

化合物 2 黄色粉末(甲醇);EIS-MS *m/s*:447 [M-H]$^-$。^{1}H NMR (DMSO-d_6, 400 MHz) δ: 6.20 (1H, d, J = 2.0 Hz, H-6), 6.39 (1H, d, J = 2.0 Hz, H-8), 7.53 (1H, br s, H-2′), 6.85 (1H, d, J = 8.8 Hz, H-5′), 7.45 (1H, dd, J = 2.4, 8.8 Hz, H-6′), 5.27 (1H, br s, H-1′′), 4.39 (1H, d, J = 1.2 Hz, H-2′′), 3.71 (1H, d, J = 10.4 Hz, H-3′′), 3.06-3.39 (2H, m, H-4′′, 5′′), 0.99 (3H, d, J = 6.4 Hz, H-6′′)。以上光谱数据与文献[13]对照,确定化合物 **2** 为槲皮素-3-*O*-β-D-鼠李糖苷。

化合物 3 黄色固体(甲醇);EIS-MS *m/s*:463 [M-H]$^-$。^{1}H NMR (DMSO-d_6, 400 MHz) δ: 7.59 (1H, dd, J = 2.0, 8.8 Hz, H-6′), 7.59 (1H, d, J = 2.4 Hz, H-2′), 6.87 (1H, d, J = 8.4 Hz, H-5′), 6.39 (1H, d, J = 2.4 Hz, H-8), 6.20 (1H, d, J = 2.0 Hz, H-6), 5.25 (1H, d, J = 7.6 Hz, H-l′′)。以上数据与文献[14]中槲皮素-3-*O*-β-D-葡萄糖苷一致,故鉴定化合物 **3** 为槲皮素-3-*O*-β-D-葡萄糖苷。

化合物 4 黄色粉末(甲醇);EIS-MS *m/s*:477 [M-H]$^-$。^{1}H NMR (DMSO-d_6, 400 MHz) δ: 12.62 (1H, s, 5-OH), 7.95 (1H, d, J = 2.0 Hz, H-2′), 7.51 (1H, dd, J = 2.0, 8.4 Hz, H-6′), 6.92 (1H, d, J = 8.4 Hz, H-5′), 6.45 (1H, d, J = 2.0 Hz, H-8), 6.22 (1H, d, J = 2.0 Hz, H-6), 5.57 (1H, d, J = 7.2 Hz, H-1′′), 3.85 (3H, s, 3′-OCH$_3$); ^{13}C NMR (DMSO-d_6, 100 Hz) δ: 177.4 (C-4), 164.2 (C-7), 161.2 (C-5), 156.4 (C-9), 156.2 (C-2), 149.4 (C-4′), 146.9 (C-3′), 133.0 (C-3), 122.0 (C-1′), 121.1 (C-6′), 115.2 (C-5′), 113.5 (C-2′), 104.0 (C-10), 100.8 (C-1′′), 98.7 (C-6), 93.7 (C-8), 77.4 (C-5′′), 76.4 (C-3′′), 74.3 (C-2′′), 69.8 (C-4′′), 60.6 (C-6′′), 55.7 (3′-OCH$_3$)。以上数据与文献[12,15]报道数据基本一致,故鉴定化合物 **4** 为异鼠李素-3-*O*-

β-D-葡萄糖苷。

化合物5 白色无定型粉末(甲醇);EIS-MS *m/s*:515 [M + Na]$^+$。^{1}H NMR(CD_3OD, 400 MHz)δ: 6.77(1H,d,*J* = 2.0 Hz,H-2),6.74(1H,d,*J* = 8.0 Hz,H-6),6.65(1H,s,H-2′),6.62(1H,dd,*J* = 2.0 Hz,8.0 Hz,H-5),6.16(1H,d,*J* = 0.4 Hz,H-5′),4.05(1H,d,*J* = 10.4 Hz,H-9a) *,4.04(1H,d,*J* = 7.2 Hz,H-1″),3.97(1H,dd,*J* = 2.4,9.6 Hz,H-9′a),3.80(3H,s,3-OCH_3),3.79(3H,s,3′-OCH_3),3.79(1H,m,H-5″a) *,3.75(1H,dd,*J* = 4.0,11.2 Hz,H-9b),3.69(1H,dd,*J* = 6.0,10.8 Hz,H-9′b),3.45(1H,m,H-4′),3.17-3.27(3H,m,H-7),3.11(1H,t,*J* = 11.2 Hz,H-2′),2.82(2H,m,H-7′),2.06(1H,m,H-8),1.85(1H,tt,*J* = 2.8,10.8 Hz,H-8′);^{13}C NMR(CD_3OD,100 MHz):134.4(C-1),114.3(C-2),147.2(C-3),145.2(C-4),116.1(C-5),123.2(C-6),48.0(C-7),45.9(C-8),69.6(C-9),129.1(C-1′),112.5(C-2′),149.0(C-3″),145.9(C-4′),117.4(C-5′),138.6(C-6″),33.8(C-7′),39.7(C-8′),66.9(C-9′),105.8(C-1″),78.0(C-2″),75.0(C-3″),71.3(C-4″),65.2(C-5″),56.5(3-OCH_3),56.4(3′-OCH_3)。以上数据与文献报道[16]的黑色五味子单体苷数据对照,基本一致,确定化合物**5**为黑色五味子单体苷。

化合物6 黄色固体(甲醇);EIS-MS *m/s*:593 [M-H]$^-$。^{1}H NMR(DMSO-d_6, 400 MHz)δ: 12.56(1H,s,5-OH),10.32(1H,s,7-OH),10.10(1H,s,4′-OH),7.98(2H,d,*J* = 8.8 Hz,H-2′,6′),6.88(2H,d,*J* = 8.8 Hz,H-3′,5′),6.41(1H,d,*J* = 2.0 Hz,H-8),6.21(1H,d,*J* = 2.0 Hz,H-6),5.31(1H,d,*J* = 7.2 Hz,H-l″),4.37(1H,d,*J* = 1.2 Hz,H-1‴),3.01-3.70(4H,m,sugar protons),0.98(3H,d,*J* = 6.4 Hz,H-6‴);^{13}C NMR(DMSO-d_6, 100 MHz):157.3(C-2),133.7(C-3),178.0(C-4),161.6(C-5),99.1(C-6),164.6(C-7),94.2(C-8),157.0(C-9),104.3(C-10),121.4(C-1′),131.4(C-2′,6′),115.6(C-3′,5′),160.3(C-4′),101.3(C-1″),74.6(C-2″),76.8(C-3″),70.8(C-4″),76.2(C-5″),67.3(C-6″),101.2(C-1‴),71.1(C-2‴),70.4(C-3‴),72.2(C-4‴),68.7(C-5‴),18.2(C-6‴)。以上数据与文献[12,17]报道数据基本一致。故鉴定化合物**6**为山柰酚-3-*O*-β-D-芸香糖苷。

化合物7 黄色固体(甲醇);EIS-MS *m/s*:623 [M-H]$^-$。^{1}H NMR(DMSO-d_6, 400 MHz)δ: 12.57(1H,s,5-OH),7.85(1H,d,*J* = 2.0 Hz,H-2′),7.50(1H,dd,*J* = 2.0,8.4 Hz,H-6′),6.91(1H,d,*J* = 8.4 Hz,H-5′),6.43(1H,d,*J* = 2.0 Hz,H-8),6.20(1H,d,*J* = 2.0 Hz,H-6),5.43(1H,d,*J* = 7.2 Hz,H-1″),4.49(1H,br s,H-1‴),3.84(3H,s,3′-OCH_3),3.01-3.72(3H,m,sugar protons),0.98(3H,d,*J* = 6.0 Hz,H-6‴);^{13}C NMR(DMSO-d_6,100 MHz)δ: 17.6(C-6‴),55.6(3′-OCH_3),66.8(C-6″),68.2(C-5‴),70.1(C-3‴),70.3(C-2‴),70.6(C-4‴),71.8(C-4‴),74.2(C-2″),75.9(C-3″),76.4(C-5″),93.8(C-8),98.7(C-6),100.8(C-1‴),101.1(C-1″),104.0(C-10),113.3(C-2′),115.2(C-5′),121.0(C-1′),122.3(C-6′),133.0(C-3),146.9(C-4′),149.4(C-3′),156.4(C-2,9),161.2(C-5),164.1(C-7),177.3(C-4)。以上数据与文献[18,19]报道数据基本一致,故鉴定化合物**7**为异鼠李素-3-*O*-β-D-芸香糖苷。

化合物8 无色针状晶体(甲醇);EIS-MS *m/s*: 375 [M + Na]$^+$。^{1}H NMR($CDCl_3$,400 MHz)δ:6.76~6.88(6H,m,H-Ar),5.95(4H,s,2(-OCH_2O-)),5.94(1H,s,H-4β),4.95(1H,d,*J* = 6.8 Hz,H-2),4.83(1H,d,*J* = 7.2 Hz,H-6),4.23(1H,dd,*J* = 6.0,9.2 Hz,H-8β),4.01(1H,dd,*J* = 2.4,9.2 Hz,H-8α),3.23(1H,m,H-1),2.97(1H,m,H-5);^{13}C NMR($CDCl_3$,100 MHz) δ:54.80(C-1,5),72.79(C-8),84.12(C-6),87.99(C-2),101.90(C-2(-OCH_2O)),102.53(C-4),107.03(C-2′),107.77(C-2″),108.43(C-5′),108.78(C-5′),120.26(C-6′),120.42(C-6″),137.66(C-1′),138.56(C-1″),147.77(C-3′),147.94(C-3″),148.74(C-4′),148.91(C-4″)。上述波谱数据与文献[20]一致,故鉴定化合物**8**为新芝麻脂素。

化合物9 白色晶体(甲醇);EIS-MS *m/s*:409 [M + Na]$^+$。^{1}H NMR($CDCl_3$,400 MHz)δ:6.77(1H,d,*J* = 8.0 Hz,H-5),6.75(1H,d,*J* = 7.6 Hz,H-5′),6.69(1H,d,*J* = 2.0 Hz,H-2),6.66(1H,dd,*J* = 2.0,8.0 Hz,H-6),6.55(1H,dd,*J* = 2.0,8.0 Hz,H-6′),6.49(1H,d,*J* = 2.0 Hz,H-2′),4.12(1H,dd,*J* = 7.2,9.2 Hz,H-9′β),3.87(1H,dd,*J* = 1.6,3.6 Hz,H-9′α) *,3.86(3H,s,3-OCH_3),3.85(3H,s,3′-OCH_3),3.84(3H,s,4-OCH_3),3.82(3H,s,4′-OCH_3),2.64(1H,m,H-7′β),2.58(1H,m,H-8),2.53(1H,m,H-7′α),2.48(1H,m,H-8′),2.93(1H,dd,*J* = 6.4,14.0 Hz,H-7α),2.97(1H,

dd, J = 6.0, 14.0 Hz, H-7β); ^{13}C NMR (DMSO-d_6, 100 MHz): 178.68 (C-9), 149.17 (C-3), 149.04 (C-3′), 147.95 (C-4), 147.82 (C-4′), 131.68 (C-1), 131.03 (C-1′), 121.72 (C-6), 120.88 (C-6′), 113.65 (C-2), 112.89 (C-2′), 112.34 (C-5), 112.20 (C-5′), 71.17 (C-9′), 55.94 (4-OCH_3), 55.92 (4′-OCH_3), 55.88 (3-OCH_3), 55.82 (3′-OCH_3), 46.09 (C-8), 41.26 (C-8′), 37.38 (C-7′), 34.14 (C-7)。上述波谱数据与文献[21,22]一致,确定化合物 **9** 为 maculatin。

*:有信号覆盖。

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