综合以上数据,确定化合物Ⅰ为 6-甲氧基-7-*O*-*β*-*D*-(4′-甲氧基)吡喃葡萄糖基香豆素[6-methoxy-7-*O*-*β*-*D*-(4′-methoxy)-glucopyranosyl coumarin],为一新化合物,结构式见图 1。

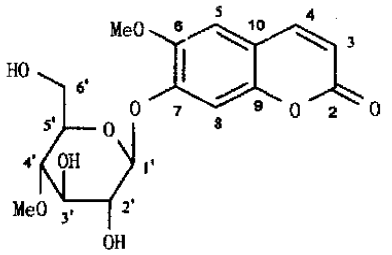

图 1 化合物Ⅰ的化学结构

Fig. 1 Structure of compound Ⅰ

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Insecticidal active constituents from twig of *Aglaia odorata*

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Abstract: **Object** To study the insecticidal activity constituents from the twigs of traditional Chinese folk medicine "Mizilan" or "Shu-Lan" (*Aglaia odorata* Lour.). **Methods** Seperation and purification under bioassay-directed were performed on silica gel CC, Sephadex LH-20 CC and preparative TLC. Their structures were established on the basis of physicochemical and spectral analysis. **Results** Seven compounds were isolated and identified as rocaglamide (Ⅰ), desmethyl rocaglamide (Ⅱ), 8-methoxy-marikarin (Ⅲ), 7-hydroxy-6-methoxy-coumarin (Ⅳ), 3′-hydroxy-methylrocaglate (Ⅴ), 3′-hydroxy-rocaglamide (Ⅵ), marikarin (Ⅶ), respectively. **Conclusion** Compound Ⅳ is isolated from the plant of *Aglaia* Lour. for the first time and compounds Ⅲ and Ⅶ are isolated from *A. odorata* for the first time.

Key words: *Aglaia odorata* Lour.; insecticide; chemical constituent

米仔兰枝条中杀虫活性成分研究

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摘 要: 目的 研究中国传统民族植物药米仔兰(又称树兰)*Aglaia odorata* Lour. 枝条中杀虫活性成分。方法 以生物测定为指导,采用硅胶柱、Sephadex LH-20 柱以及制备薄层分离板对其成分进行分离和纯化,并通过物化性质和光谱分析对其结构鉴定。结果 从中分离得到 7 个化合物,分别鉴定为 rocaglamide (Ⅰ)、desmethyl rocaglamide (Ⅱ)、8-methoxymarikarin (Ⅲ)、7-羟基-6-甲氧基-香豆素 (Ⅳ)、3′-hydroxy-methylrocaglate (Ⅴ)、3′-hydroxy-rocaglamide (Ⅵ)、marikarin (Ⅶ)。结论 化合物Ⅳ为首次从 *Aglaia* Lour. 属植物中分离得到,化合物Ⅲ和Ⅶ为首次从米仔兰植物中分离得到。

关键词: 树兰; 杀虫; 化学成分

中图分类号: R284.1 文献标识码: A 文章编号: 0253-2670(2004)11-01207-05

收稿日期: 2004-03-15

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Aglaia odorata Lour., a slow-growing member of Meliaceae, native to China but widely cultivated throughout Malaysia and Thailand, is a favourite of the Chinese on account of its fragrance. The flowers are used to perfume clothes and for scenting tea. It also figures prominently in traditional folk pharmacopeias throughout Southeast Asia. Known as Mizilan or Shu-Lan in Fujian and Taiwan Provinces of China, it has been used as a herbal remedy for treatment of human cough, inflammation and traumatic injury in Chinese Folklore. In Thailand the same plant is traditionally prescribed as a heart stimulant and febrifuge, and as an expectorant.

For the past two decades, in Thailand and Vietnam, a series of rocaglamide derivatives which featured the cyclopentatetrahydrobenzofuran skeleton had been isolated from several species of the *Aglaia* Lour., and were shown to exhibit the insecticidal activity[1-7]. This activity reported for the rocaglamide congeners is comparable to that of azadirachtin, a powerful natural insecticide that is of considerable commercial interest and has prompted us to screen for further active derivatives. In order to search for new potential insecticidal compounds of plant origin and compare the chemical constituents of *A. odorata*. cultivated in China with which cultivated at abroad, the insecticidal constituents of the twigs of traditional Chinese folk medicine "Shu-Lan" (*A. odorata*) had been studied. Seven compounds from the crude methanolic extracts of swigs of *A. odorata* were isolated by bioassay-guided fractionation and identical as rocaglamide (Ⅰ), desmethyl rocaglamide (Ⅱ), 8-methoxymarikarin (Ⅲ), 7-hydroxy-6-methoxy-coumarin (Ⅳ), 3′-hydroxy-methylrocaglate (Ⅴ), 3′-hydroxyrocaglamide (Ⅵ), marikarin (Ⅶ), respectively. Among them, compound Ⅳ was isolated from the plants of *Aglaia* Lour. for the first time and compounds Ⅲ and Ⅶ were isolated from *A. odorata* for the first time.

1 Instruments and materials

NMR spectra were recorded on Bruker DRX-400 MHz spectrometers. EI-MS spectra (70 eV) were obtained by direct inlet on a HP 5970 mass spectrometer. Optical rotation was WXG-4 polarimeter. Mps were carried out using a B-545 melting point apparatus. CC: silica gel (200-300 mesh, from Qingdao Marine Chemical Factory). Sephadex LH-20 (Pharmacopia 17-0090-01).

The stwigs of *A. odorata* was collected in Wuyishan, Fujian Province, China and identified by Dr. WANG Wei-feng in May of 2001.

2 Extraction and isolation

Air-dried and powdered twigs of *A. odorata* (2.5 kg) were ground and extracted with MeOH (12 L×2) at room temperature for two days. The methanolic extract was concentrated to 1 L in vacuo, diluted with an equal volumn of H_2O, and extracted successively with hexane (500 mL×3), EtOAc (500 mL×3) and water saturated *n*-butanol, respectively. Each obtained fraction was subjected to a bioassay with neonate larvae of *S. littoralis*. From this bioassay the insecticidal activity was found to reside in the EtOAc fraction. The EtOAc fraction was concentrated to 100 mL in vacuo, diluted with 100 mL 5% NaOH solution, and extracted successively with Et_2O (200 mL×3). The Et_2O solution washed with H_2O (200 mL), and dried with anhydrous. Na_2SO_4, and evaporated in vacuo to give a pale brownish gum (17.3 g). Isolation of the compounds from the pale brownish gum was accomplished by repeated chromatographic separation employing silica gel (mobile phase: CH_2Cl_2-iso-PrOH 95∶5 and 90/10), Sephadex LH-20 (Me_2CO). Fractions were monitored by TLC on precoated TLC plates with silica gel 60 F_{254} (mobile phase: CH_2Cl_2-iso-PrOH 95∶5 or hexane-iso-PrOH-EtOAc 9∶1∶1) or after spraying with anisaldehyde reagent. Final purification was obtained using preparative TLC [silica gel 60 F_{254}, CH_2Cl_2-EtOAc-MeOH (75∶20∶5), or EtOAc-MeOH (45∶1)]. Yields of compounds were Ⅰ: 13.2 mg; Ⅱ: 6.3 mg; Ⅲ: 7.4 mg.

The NaOH solution was acidified with diluted HCl and extracted with Et_2O (200 mL×3), the Et_2O layer was evaporated in vacuo. Isolation of the compounds from the residue was accomplished

by repeated chromatographic separation employing silica gel (mobile phase: CH_2Cl_2–iso-PrOH 90∶10), Sephadex LH-20 (Me_2CO). Fractions were monitored by TLC on precoated TLC plates with silica gel 60 F_{254} (mobile phase: CH_2Cl_2–iso-PrOH 90∶10 or hexane–iso-PrOH–EtOAc 8∶1∶1) or after spraying with anisaldehyde reagent. Final purification was obtained using preparative TLC [silica gel 60 F_{254}, CH_2Cl_2–EtOAc–MeOH (70∶25∶5), or EtOAc–MeOH (40∶1)]. Yields of compounds were Ⅳ: 15.6 mg; Ⅴ: 7.4 mg; Ⅵ: 10.5 mg; and Ⅶ: 5.3 mg.

3 Experiments with insects

Larvae of *S. litoralis* were from a laboratory colony reared on an artificial diet under controlled conditions at 26 ℃. Feeding studies were conducted with neonate larvae (n= 20 for each treatment). Neonate larvae were kept on diets containing extracts under study. After six days, survival and weight of survived larvae were recorded and compared with the controls.

4 Structure elucidation

Compound Ⅰ: Pale yellow powder, mp 115–117 ℃ ($CHCl_3$). $[\alpha]_D^{20}$ – 89.0 °($CHCl_3$). EI-MS m/z (%): 505 $[M]^+$ (5), 478 (12), 390 (51), 313 (55), 300 (69), 285 (24), 205 (17), 181 (22), 176 (100), 135 (11), 131 (14), 103 (61), 77 (62), 72 (95), 55 (71), 44 (54). ^{1}H-NMR (acetone-d_6): δ 2.71 (1H, br, s, OH), 2.76 (1H, br, s, OH), 2.89 (3H, s, N-Me), 3.41 (3H, s, N-Me), 3.82 (3H, s, O-Me), 3.88 (3H, s, O-Me), 3.91 (3H, s, O-Me), 4.13 (1H, dd, J= 6.6, 13.2 Hz, H-2), 4.51 (1H, 13.6 Hz, H-3), 4.96 (1H, 6.6 Hz, H-1), 6.25 (1H, 1.4 Hz, H-5), 6.37 (1H, d, 1.4 Hz, H-7), 6.72 (2H, d, 8.9 Hz, H-3′和 H-5′), 6.86–7.05 (5H, m, H-2″–6″), 7.16 (2H, d, 8.9 Hz, H-2′和 H-6′). ^{13}C-NMR (acetone-d_6): δ 34.7, 36.2 (2×N-Me), 42.9 (C-2), 55.4, 55.7, 56.3 (3×OMe), 57.2 (C-3), 79.1 (C-1), 89.7 (C-5), 93.4 (C-7), 94.9 (C-8b), 102.7 (C-3a), 109.8 (C-8a), 114.7 (C-3′, 5′), 126.1 (C-4″), 127.6 (C-2″, 6″), 128.9 (C-3″, 5″), 130.6 (C-1′), 131.1 (C-2′, 6′), 140.4 (C-1″), 158.1, 158.9, 159.2, 162.4 (C-4′, 4a, 6, 8), 172.5 (C= O). Comparing with the reported data, the structure of compound Ⅰ was identified as rocaglamide[1,2].

Compound Ⅱ: Pale yellow powder; mp 117–119 ℃($CHCl_3$). $[\alpha]_D^{20}$ – 54.7 °($CHCl_3$). EI-MS m/z (%): 491 $[M]^+$ (8), 473 (15), 390 (51), 313 (59), 301 (84), 300 (100), 285 (41), 242 (56), 191 (36), 181 (53), 162 (89), 131 (44), 162 (96), 77 (45), 58 (74). ^{1}H-NMR ($CDCl_3$): δ 2.01 (1H, br, s, OH), 2.69 (3H, s, N-Me), 3.57 (1H, br, s, OH), 3.78 (3H, s, O-Me), 3.81 (3H, s, O-Me), 3.85 (3H, s, O-Me), 3.92 (1H, dd, J= 5.8, 14.6 Hz, H-2), 4.29 (1H, 14.6 Hz, H-3), 4.88 (1H, 5.8 Hz, H-1), 6.23 (1H, d, 2.1 Hz, H-5), 6.34 (1H, d, 2.1 Hz, H-7), 6.68 (2H, d, 9.5 Hz, H-3′和 H-5′), 6.98–7.06 (5H, m, H-2″–6″), 7.12 (2H, d, 9.5 Hz, H-2′和 H-6′). ^{13}C-NMR ($CDCl_3$): δ 26.9 (N-Me), 52.9 (C-2), 55.8, 56.2, 56.3 (3×OMe), 56.8 (C-3), 80.3 (C-1), 89.3 (C-5), 93.1 (C-7), 95.4 (C-8b), 103.1 (C-3a), 109.9 (C-8a), 113.6 (C-3′, 5′), 126.4 (C-4″), 129.5 (C-2″, 6″), 129.9 (C-3″, 5″), 130.3 (C-1′), 131.4 (C-2′, 6′), 139.5 (C-1″), 158.4, 158.7, 160.9, 163.7 (C-4′, 4a, 6, 8), 173.8 (C= O). All of these data were in good agreement with those reported data of desmethyl rocaglamide, so compound Ⅱ was identified as desmethyl rocaglamide[2].

Compound Ⅲ: Colourless oil. $[\alpha]_D^{20}$ – 49.9 °($CHCl_3$). EI-MS m/z (%): 524 $[M]^+$ (47), 506 (27), 389 (74), 370 (100), 343 (19), 313 (42), 285 (41), 135 (46), 77 (41), 58 (63). ^{1}H-NMR ($CDCl_3$): δ 2.37 (1H, m, H-3‴B), 2.41 (1H, m, H-3‴A), 2.72 (1H, br, s, OH), 3.17 (1H, m, H-4‴B), 3.29 (1H, m, H-4‴A), 3.74 (3H, s, OMe-4′), 3.79 (3H, s, OMe-8), 3.82 (3H, s, OMe-6), 4.12 (1H, m, H-2‴B), 4.19 (1H, m, H-2‴A), 4.51 (1H, s, H3-H), 6.19 (1H, d, 2.0 Hz, H-7), 6.36 (1H, d, 2.0 Hz, H-5), 6.54 (2H, d, 9.2 Hz, H-3′和 H-5′), 6.84–7.02 (5H, m, H-2″–6″), 7.09 (2H, d, 9.2 Hz, H-2′和 H-6′). ^{13}C-NMR ($CDCl_3$): δ 19.7 (C-3‴), 34.7 (C-4‴), 50.2 (C-2‴), 55.1, 55.9, 56.1 (3×OMe), 59.4 (C-3), 92.4 (C-7), 94.1 (C-5), 96.6 (C-8b),

105.4 (C-3a), 108.9 (C-8a), 113.8 (C-3′, 5′), 120.4 (C-2), 127.6 (C-4″), 128.3 (C-3″, 5″), 129.2 (C-1′), 130.4 (C-2′, 6′), 131.5 (C-2″, 6″), 139.1 (C-1″), 159.4, 159.7, 162.3, 164.8 (C-4′, 4a, 6, 8), 161.8 (C-1), 167.2 (C-5‴), 171.5 (C=O). Comparing with the reported data, the structure of compound Ⅲ was identified as 8-methoxymarikarin[3].

Compound Ⅳ: Colourless needle crystals. mp 204-206 ℃ ($CHCl_3$). It showed blue fluorescence under UV 254. ^{1}H-NMR ($CDCl_3$): δ 3.91 (3H, s, OCH_3-6), 6.29 (1H, d, 9.2 Hz, H-3), 7.65 (1H, d, 9.2 Hz, H-4), 6.81 (1H, s, H-8), 6.97 (1H, s, H-5), 6.28 (1H, brs, OH). EI-MS *m/z* (%): 192 $[M]^+$ (100), 177 (44), 164 (21), 149 (19), 121 (39), 79 (62), 69 (51). By comparing its mp, ^{1}H-NMR, EI-MS, and Rf values with those of the authentic sample, the compound Ⅳ was identified as 7-hydroxy-6-methoxy-coumarin.

Compound Ⅴ: Pale yellow powder; mp 124-126 ℃ ($CHCl_3$). $[\alpha]_D^{20}$ - 55.1 ° ($CHCl_3$). EI-MS *m/z* (%): 508 $[M]^+$ (6), 490 (20), 406 (17), 329 (27), 316 (100), 301 (41), 283 (34), 218 (14), 181 (19), 77 (51), 58 (67). ^{1}H-NMR ($CDCl_3$): δ 2.45 (1H, br, s, OH), 2.89 (1H, br, s, OH), 3.69 (3H, s, CO-OCH_3), 3.74 (3H, s, OCH_3-4′), 3.82 (3H, s, OCH_3-8), 4.07 (1H, dd, 14.3, 6.6 Hz, H-2), 4.26 (1H, d, 14.3 Hz, H-3), 4.91 (1H, d, 6.6 Hz, H-1), 6.19 (1H, d, 1.9 Hz, H-5), 6.32 (1H, d, 1.9 Hz, H-7), 6.64 (1H, d, 8.2 Hz, H-5′), 6.71 (1H, dd, 2.1, 8.2 Hz, H-6′), 6.82 (1H, d, 2.1 Hz, H-2′), 6.94-7.04 (5H, m, H-2″-6″), 12.69 (1H, br, s, Ar-OH). ^{13}C-NMR ($CDCl_3$): δ 52.4 (C-2), 53.6 (CO-CH_3), 55.9, 56.1, 56.4 (3×OMe), 57.2 (C-3), 81.4 (C-1), 90.7 (C-5), 93.6 (C-7), 95.2 (C-8b), 102.5 (C-3a), 109.4 (C-8a), 112.2 (C-5′), 116.3 (C-2′), 121.7 (C-5′), 127.9 (C-4″), 129.1 (C-3″, 5″), 130.3 (C-2″, 6″), 130.9 (C-1′), 139.3 (C-1″), 145.3 (C-3′), 148.4, 158.9, 161.3, 164.7 (C-4′, 4a, 6, 8), 172.1 (C=O). Comparing with the reported data, the structure of compound Ⅴ was identified as 3′-hydroxy-methylrocaglate[6].

Compound Ⅵ: Pale yellow powder; mp 126-129 ℃ ($CHCl_3$). $[\alpha]_D^{20}$ - 89.8 ° ($CHCl_3$). EI-MS *m/z* (%): 521 $[M]^+$ (11), 503 (31), 406 (62), 329 (59), 316 (78), 301 (21), 283 (13), 181 (57), 176 (100), 131 (23), 77 (44), 58 (61). ^{1}H-NMR ($CDCl_3$): δ 2.37 (1H, br, s, OH), 2.74 (1H, br, s, OH), 2.96 (3H, s, N-Me), 3.44 (3H, s, N-Me), 3.81 (3H, s, OCH_3-4′), 3.84 (3H, s, OCH_3-6), 3.93 (3H, s, OCH_3-8), 4.13 (1H, dd, 13.9, 7.2 Hz, H-2), 4.37 (1H, d, 13.9 Hz, H-3), 4.92 (1H, d, 7.2 Hz, H-1), 6.24 (1H, d, 1.9 Hz, H-5), 6.36 (1H, d, 1.9 Hz, H-7), 6.66 (1H, d, 8.6 Hz, H-5′), 6.77 (1H, dd, 2.3, 8.6 Hz, H-6′), 6.84 (1H, 2.3 Hz, H-2′), 6.90-7.06 (5H, m, H-2″-6″), 12.49 (1H, br, s, Ar-OH). ^{13}C-NMR ($CDCl_3$): δ 28.2 (*N*-Me), 34.8 (*N*-Me), 39.5 (C-2), 55.4, 55.7, 56.2 (3×OMe), 57.4 (C-3), 80.7 (C-1), 89.6 (C-5), 92.9 (C-7), 96.1 (C-8b), 102.7 (C-3a), 110.3 (C-8a), 113.4 (C-5′), 116.9 (C-2′), 120.7 (C-6′), 127.3 (C-4″), 128.9 (C-3″, 5″), 129.6 (C-2″, 6″), 130.4 (C-1′), 140.1 (C-1″), 146.4 (C-3′), 147.4, 159.9, 163.3, 165.2 (C-4′, 4a, 6, 8), 172.1 (C=O). All of these data were in good agreement with those reported data of 3′-hydroxyrocaglamide, so compound Ⅵ was identified as 3′-hydroxyrocaglamide[3].

Compound Ⅶ: Colourless oil. $[\alpha]_D^{20}$ - 44.9 ° ($CHCl_3$). EI-MS *m/z* (%): 510 $[M]^+$ (25), 494 (23), 492 (67), 415 (82), 370 (100), 343 (11), 341 (47), 135 (26), 77 (54), 58 (59). ^{1}H-NMR ($CDCl_3$): δ 2.31 (1H, m, H-3‴B), 2.37 (1H, m, H-3‴A), 2.92 (1H, br, s, OH), 3.21 (1H, m, H-4‴B), 3.28 (1H, m, H-4‴A), 3.71 (3H, s, OMe-4′), 3.74 (3H, s, OMe-6), 4.09 (1H, m, H-2‴B), 4.14 (1H, m, H-2‴A), 4.62 (1H, s, H-3), 6.11 (1H, d, 1.9 Hz, H-7), 6.28 (1H, d, 2.0 Hz, H-5), 6.59 (2H, d, 8.9 Hz, H-3′和 H-5′), 6.94-7.09 (5H, m, H-2″-6″), 7.14 (2H, d, 8.9 Hz, H-2′和 H-6′), 12.18 (1H, br, s, Ar-OH). ^{13}C-NMR ($CDCl_3$): δ 18.4 (C-3‴), 33.8 (C-4‴), 49.4 (C-2‴), 55.1 (OMe-4′), 56.2 (OMe-6), 59.9 (C-3), 91.3 (C-7), 92.7 (C-5), 95.8 (C-8b), 104.1 (C-3a), 107.2 (C-8a), 113.1 (C-3′,

5′), 121.4 (C-2′), 126.3 (C-4′), 127.0 (C-3″, 5″), 128.8 (C-1′), 129.5 (C-2′, 6′), 130.5 (C-2″, 6″), 137.4 (C-1″), 157.2, 160.8, 165.6 (C-4′, 4a, 6, 8), 162.4 (C-1), 167.7 (C-5‴), 170.1 (C=O). Comparing with the reported data, the structure of compound Ⅶ was identified as marikarin[7].

Acknowledgements: We thank Prof. DENG Guo-cai (from Department of Chemistry, Nankai University, China) for helpful discussions and some useful advices.

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中国南海海绵正丁醇部分中的核苷类成分研究

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摘 要: 目的 对中国南海产海绵 *Cinachyrella australiensis* 的正丁醇部分化学成分进行了系统分离, 为合理开发海洋药物资源, 更好地利用海洋生物体内具有强生理活性的化学成分, 从而开发出适用于人体的安全有效的药物。方法 采用多种色谱方法进行分离纯化, 应用波谱分析技术, 并结合文献对照, 对所分离到的化合物进行了结构鉴定。结果 共分离得到 7 个化合物, 其结构分别为: 2-甲基-6-氨基嘌呤脱氧核苷[2-methyl-6-amino-9-(2-deoxy-β-D-ribofuranosyl)-purine, Ⅰ]、6-氨基嘌呤脱氧核苷(2′-deoxyadenosne, Ⅱ)、6-氨基嘌呤核苷(6-amino-9-β-D-ribofuranosyl-9H-punne, Ⅲ)、尿嘧啶(uracill, Ⅳ)、胸腺嘧啶(thymine, Ⅴ)、胸腺嘧啶脱氧核苷(thymidine, Ⅵ)、尿嘧啶脱氧核苷[1-(2-deoxy-β-D-ribofuranosyl) uracil, Ⅶ]。结论 对中国南海海绵正丁醇部分的化学成分进行了报道, 系统地分离出了 2 大类核苷类化合物。

关键词: 海绵; 分离提取; 结构鉴定

中图分类号: R284.1 文献标识码: A 文章编号: 0253-2670(2004)11-01211-03

Study on nucleotidoids from n-butyl alcohol part of sponge *Cinachyrella australiensis*

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Abstract: Object To search for bioactive secondary metabolites from marine organisms, the marine sponge *Cinachyrella australiensis* was collected from Hainan Island, Southern China Sea to develop the efficient drug for human beings. **Methods** Silic gel flash chromatography in association with reverse phase semipreparative HPLC was performed for the isolation and purification of the ethanol extracts, and extensive spectroscopy including 2D NMR spectra as well as the comparison of the spectral data with those reported in literatures was applied for the structure elucidation. **Results** Seven nucleotidoids were isolated

* 收稿日期: 2004-04-20
基金项目: 国家科技部海洋 863 研究课题(2001AA620403, 2002AA217081)
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