

辽东槲木三萜皂苷类化学成分的研究^Δ

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摘要 目的:研究辽东槲木中三萜皂苷类化学成分。方法:采用各种柱色谱对辽东槲木中的化学成分进行分离纯化,通过理化性质和波谱分析鉴定化合物结构。结果:从辽东槲木中分离并鉴定出10个三萜皂苷,分别为齐墩果酸-28-*O*-β-D-吡喃葡萄糖苷(1)、Elatoside E(2)、Elatoside F(3)、3-*O*-β-D-吡喃葡萄糖(1-3)-α-L-吡喃阿拉伯糖-齐墩果酸-28-*O*-β-D-吡喃葡萄糖苷(4)、Araliasaponin IV(5)、屏边三七苷(6)、Tarasaponin IV(7)、Elatoside C(8)、Spinasaponin A 28-*O*-glucoside(9)、槲木皂苷C(10)。其中,化合物4、5为首次从该植物中分离得到。结论:该试验结果可为辽东槲木的进一步研究提供依据。

关键词 辽东槲木;三萜皂苷;化学成分

Study on Saponins Chemical Components from *Aralia elata*

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ABSTRACT OBJECTIVE: To study the saponins chemical components from *Aralia elata*. METHODS: Chemical compounds were isolated and purified by column chromatography, and their structures were identified by means of physical property and spectral data analysis. RESULTS: 10 saponins were isolated and identified as Oleanolic acid 28-*O*-glucoside ester (1), Elatoside E (2), Elatoside F (3), 3-*O*-β-D-Glucopyranosyl (1-3)-α-L-arabinopyranosyl oleanolic acid 28-*O*-β-D-glucopyranosyl ester (4), Araliasaponin IV (5), Stipuleanoside R₂ (6), Tarasaponin IV (7), Elatoside C (8), Spinasaponin A 28-*O*-glucoside (9), Aralioside C (10). Compound 4 and 5 both were obtained from the plant for the first time. CONCLUSION: Results of the trial can provide reference for the further study of *A. elata*.

KEY WORDS *Aralia elata*; Saponins; Chemical components

辽东槲木(*Aralia elata* Seem.)为五加科槲木属植物,又名龙牙槲木、刺龙牙、刺嫩芽,主要分布在我国东北地区,朝鲜、日本等地也有分布。其药用部位为根皮、叶,具有补气安神、活血化痰、除湿止痛的功效,用于治疗神经衰弱、风湿性关节炎、肝炎、糖尿病、外伤出血症等^[1]。本课题组从20世纪80年代就开始了辽东槲木的系统研究^[2-3],通过现代色谱技术分离制备了辽东槲木皂苷,并通过研究发现了其治疗冠心病、心绞痛的新用途。目前,以辽东槲木总皂苷为成分研制的槲木心脉通胶囊已获得药物临床研究批件(批号:2003L01111),并已完成临床观察。为明确槲木总皂苷中具有抗心肌缺血的皂苷类化合物及其构效关系,本试验对辽东槲木的皂苷类成分进行了研究,从其总皂苷中分离得到10个三萜皂苷类化合物,其结构根据理化性质与波谱分析分别鉴定为齐墩果酸-28-*O*-β-D-吡喃葡萄糖苷(1)、Elatoside E(2)、Elatoside F(3)、

3-*O*-β-D-吡喃葡萄糖(1-3)-α-L-吡喃阿拉伯糖-齐墩果酸-28-*O*-β-D-吡喃葡萄糖苷(4)、Araliasaponin IV(5)、屏边三七苷(6)、Tarasaponin IV(7)、Elatoside C(8)、Spinasaponin A 28-*O*-glucoside(9)、槲木皂苷C(10)。其中,化合物4和5为首次从该植物中分离得到。

1 材料

1.1 仪器

AVANCE III 600型超导超屏蔽傅里叶变换核磁共振(NMR)波谱仪(TMS为内标,德国Bruker公司);LTQ-Obi-trap XL型高效液相色谱(HPLC)-质谱(MS)仪(美国Thermo Scientific公司);

1.2 试剂

AB-8大孔吸附树脂(南开大学化工厂);柱层析硅胶、薄层层析硅胶(青岛海洋化工厂);所用试剂均为分析纯。

1.3 药材

试验药材采自辽宁省鞍山市,经中国医学科学院药用植物研究所张本刚研究员鉴定其来源为五加科槲木属植物辽东槲木 *A. elata* Seem.。

2 提取与分离

取辽东槲木药材10 kg,用70%乙醇回流提取3次,每次70 L,减压浓缩得提取液。提取液经大孔吸附树脂柱(AB-8),

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先用水洗,再用20%乙醇洗,最后用80%乙醇洗脱,回收80%乙醇,减压干燥,得到干浸膏,干浸膏加70%乙醇溶解,回收溶剂后减压干燥,制得总皂苷。总皂苷通过硅胶柱,合并得9个洗脱部分(Fr.1~Fr.9)。经过反复硅胶柱层析,Fr.4得到化合物1(200 mg);Fr.6得到化合物2(5.2 mg)、3(8.4 mg)、4(24.3 mg)、5(8.4 mg);Fr.7得到化合物6(46 mg)、7(71.6 mg)、8(30.6 mg)、9(32.5 mg);Fr.8得到化合物10(350 mg)。

3 结构鉴定

化合物1:白色粉末,5%浓硫酸-乙醇溶液喷雾显色后显紫红色斑点。¹H-NMR(*d*₅-pyridine, 600 MHz)δ: 6.33(1H, d, *J*=7.8 Hz), 5.46(1H, s, H-12), 1.24(3H, s, CH₃), 1.22(3H, s, CH₃), 1.13(3H, s, CH₃), 1.02(3H, s, CH₃), 0.92(3H, s, CH₃), 0.90(3H, s, CH₃), 0.89(3H, s, CH₃)。碳谱数据见表1。以上数据与文献^[4]报道基本一致,故确定该化合物为齐墩果酸-28-*O*-β-D-吡喃葡萄糖苷(Oleanolic acid 28-*O*-β-D-glucoside ester)。

表1 化合物1~10的碳谱数据(吡啶)

Tab 1 ¹³C-NMR spectrum data of compounds 1-10(in *d*₅-pyridine)

| C | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|------------|--------|--------|--------|-------|-------|-------|-------|-------|-------|-------|
| 1 | 39.5 | 39.2 | 39.4 | 39.3 | 39.2 | 39.1 | 39.9 | 39.2 | 39.1 | 39.1 |
| 2 | 28.6 | 27.1 | 27.2 | 27.1 | 27.0 | 26.9 | 26.9 | 26.9 | 26.9 | 26.9 |
| 3 | 78.6 | 89.6 | 89.7 | 89.1 | 90.0 | 89.6 | 89.8 | 89.9 | 89.6 | 90.2 |
| 4 | 39.8 | 40.3 | 40.3 | 40.0 | 40.1 | 40.0 | 39.1 | 40.4 | 39.9 | 39.9 |
| 5 | 56.3 | 56.4 | 56.5 | 56.3 | 56.4 | 56.1 | 56.1 | 56.3 | 56.2 | 56.3 |
| 6 | 19.3 | 18.9 | 19.0 | 19.0 | 19.0 | 19.0 | 19.0 | 19.0 | 19.0 | 19.0 |
| 7 | 33.7 | 33.6 | 33.7 | 33.6 | 33.6 | 33.6 | 33.6 | 33.0 | 33.6 | 33.5 |
| 8 | 40.4 | 40.2 | 40.4 | 40.4 | 40.4 | 40.4 | 40.4 | 40.4 | 40.1 | 40.1 |
| 9 | 48.6 | 48.5 | 48.6 | 48.5 | 48.5 | 48.5 | 48.4 | 48.5 | 48.5 | 48.4 |
| 10 | 37.9 | 37.5 | 37.5 | 37.5 | 37.4 | 37.4 | 37.4 | 37.4 | 37.4 | 37.4 |
| 11 | 24.3 | 24.2 | 24.2 | 23.9 | 23.9 | 24.0 | 23.9 | 23.9 | 23.9 | 23.9 |
| 12 | 123.4 | 123.3 | 123.4 | 123.6 | 123.3 | 123.4 | 123.4 | 123.4 | 123.4 | 123.4 |
| 13 | 144.6 | 144.5 | 144.6 | 144.6 | 144.6 | 144.7 | 144.6 | 144.6 | 144.6 | 144.6 |
| 14 | 42.6 | 42.6 | 42.6 | 42.6 | 42.6 | 42.7 | 42.6 | 42.6 | 42.6 | 42.6 |
| 15 | 28.7 | 28.7 | 28.7 | 28.7 | 28.7 | 28.8 | 28.8 | 28.5 | 28.8 | 28.7 |
| 16 | 23.9 | 24.2 | 24.3 | 24.3 | 24.2 | 24.3 | 24.3 | 24.2 | 24.3 | 24.2 |
| 17 | 47.5 | 47.1 | 47.5 | 47.5 | 47.5 | 47.5 | 47.5 | 47.5 | 47.5 | 47.5 |
| 18 | 42.3 | 42.4 | 42.2 | 42.2 | 42.2 | 42.2 | 42.2 | 42.2 | 42.2 | 42.2 |
| 19 | 46.7 | 46.6 | 46.7 | 46.7 | 46.7 | 46.7 | 46.7 | 46.7 | 46.7 | 46.7 |
| 20 | 31.2 | 31.4 | 31.2 | 31.2 | 31.2 | 31.3 | 31.3 | 31.2 | 31.3 | 31.2 |
| 21 | 34.5 | 34.7 | 34.5 | 34.5 | 34.5 | 34.5 | 34.5 | 34.5 | 34.5 | 34.5 |
| 22 | 33.0 | 33.2 | 33.0 | 33.0 | 33.0 | 33.0 | 33.0 | 33.0 | 33.0 | 33.0 |
| 23 | 29.2 | 28.3 | 28.3 | 28.6 | 28.3 | 28.6 | 28.6 | 28.1 | 28.7 | 28.2 |
| 24 | 17.0 | 16.9 | 17.0 | 17.4 | 16.9 | 17.5 | 17.5 | 16.8 | 16.9 | 17.2 |
| 25 | 16.1 | 15.9 | 16.1 | 16.0 | 16.0 | 16.0 | 16.0 | 16.0 | 16.0 | 16.0 |
| 26 | 18.0 | 17.8 | 18.0 | 17.9 | 17.9 | 18.0 | 18.0 | 17.9 | 18.0 | 17.9 |
| 27 | 26.5 | 26.6 | 26.6 | 26.5 | 26.6 | 26.7 | 26.6 | 26.6 | 26.6 | 26.6 |
| 28 | 176.9 | 180.0 | 176.9 | 176.8 | 176.9 | 177.0 | 176.9 | 176.9 | 176.9 | 176.9 |
| 29 | 33.6 | 33.7 | 33.6 | 33.6 | 33.6 | 33.7 | 33.6 | 33.6 | 33.6 | 33.6 |
| 30 | 24.1 | 24.2 | 23.9 | 24.1 | 24.1 | 24.0 | 24.2 | 24.1 | 24.2 | 24.1 |
| 3-O(Sugar) | Ara(p) | Ara(p) | Ara(p) | Glc | GlcA | GlcA | GlcA | GlcA | GlcA | GlcA |
| 1 | 106.1 | 106.0 | 106.1 | 105.4 | 106.7 | 106.7 | 105.0 | 106.7 | 105.4 | |
| 2 | 77.8 | 77.9 | 72.3 | 79.4 | 76.4 | 82.1 | 79.7 | 74.8 | 74.2 | |
| 3 | 84.1 | 84.1 | 84.6 | 89.3 | 82.4 | 76.2 | 88.1 | 88.1 | 88.0 | |
| 4 | 69.3 | 69.4 | 69.7 | 70.6 | 79.3 | 79.3 | 72.7 | 71.8 | 79.7 | |
| 5 | 66.5 | 66.6 | 67.4 | 78.2 | 76.4 | 78.9 | 78.2 | 76.1 | 75.5 | |
| 6 | | | | 62.8 | 177.0 | 176.0 | 176.9 | 176.9 | 176.9 | |

续表1

Continued tab 1

| C | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|------|-------|-------|-------|--------|--------|--------|--------|-------|--------|-----|
| | | Glc | Glc | Glc | Glc | Glc | Glc | Gal | Glc | Gal |
| 1' | 105.5 | 105.5 | 106.8 | 105.2 | 105.9 | 105.6 | 104.8 | 105.8 | 104.9 | |
| 2' | 75.7 | 75.7 | 76.2 | 75.8 | 75.5 | 76.0 | 71.8 | 76.2 | 71.6 | |
| 3' | 78.8 | 78.9 | 78.8 | 79.1 | 78.5 | 78.7 | 73.2 | 79.1 | 73.2 | |
| 4' | 71.8 | 71.8 | 72.1 | 72.1 | 71.3 | 71.4 | 70.9 | 72.4 | 70.9 | |
| 5' | 78.9 | 78.8 | 79.0 | 79.1 | 78.8 | 78.9 | 78.2 | 78.6 | 76.3 | |
| 6' | 63.0 | 63.0 | 63.2 | 63.1 | 63.0 | 62.6 | 62.8 | 63.0 | 62.4 | |
| | Xyl | Xyl | | Xyl(p) | Ara(f) | Ara(f) | Xyl(p) | | Xyl(p) | |
| 1'' | 105.5 | 105.5 | | 105.1 | 108.0 | 108.5 | 104.9 | | 104.6 | |
| 2'' | 76.4 | 76.4 | | 75.8 | 82.1 | 82.1 | 76.7 | | 76.0 | |
| 3'' | 79.4 | 79.4 | | 79.8 | 76.1 | 76.0 | 79.4 | | 78.7 | |
| 4'' | 72.0 | 72.0 | | 71.9 | 88.1 | 88.1 | 71.6 | | 71.4 | |
| 5'' | 67.5 | 67.5 | | 67.7 | 62.6 | 62.8 | 67.6 | | 67.6 | |
| 6'' | | | | | | | | | | |
| 28-O | -Glc | -Glc | -Glc | -Glc | Glc | Glc | -Glc | -Glc | -Glc | |
| 1''' | 96.7 | 96.2 | 96.2 | 96.2 | 96.7 | 96.2 | 96.2 | 96.2 | 96.2 | |
| 2''' | 74.6 | 74.6 | 74.6 | 74.6 | 74.1 | 74.6 | 74.6 | 74.6 | 74.6 | |
| 3''' | 79.4 | 79.3 | 79.4 | 79.4 | 78.8 | 79.3 | 79.0 | 79.1 | 79.1 | |
| 4''' | 71.7 | 71.6 | 71.6 | 71.6 | 72.0 | 71.6 | 70.8 | 71.7 | 71.6 | |
| 5''' | 79.7 | 79.7 | 79.7 | 79.7 | 79.1 | 79.7 | 79.7 | 79.8 | 79.7 | |
| 6''' | 62.7 | 62.7 | 62.7 | 62.7 | 62.7 | 62.7 | 62.7 | 62.7 | 62.7 | |

化合物2:白色粉末,5%浓硫酸-乙醇溶液喷雾显色后显紫红色斑点。¹H-NMR(*d*₅-pyridine, 600 MHz)δ: 5.46(1H, s, H-12), 5.40(1H, d, *J*=7.8 Hz), 5.30(1H, d, *J*=7.8 Hz), 4.75(1H, d, *J*=6.6 Hz), 1.29(3H, s, CH₃), 1.28(3H, s, CH₃), 1.08(3H, s, CH₃), 1.00(3H, s, CH₃), 0.98(3H, s, CH₃), 0.95(3H, s, CH₃), 0.83(3H, s, CH₃)。碳谱数据见表1。以上数据与文献^[5]报道基本一致,故确定该化合物为 Elatoside E。

化合物3:白色粉末,5%浓硫酸-乙醇溶液喷雾显色后显紫红色斑点。¹H-NMR(*d*₅-pyridine, 600 MHz)δ: 6.30(1H, d, *J*=7.8 Hz), 5.41(1H, s, H-12), 5.37(1H, d, *J*=7.8 Hz), 5.28(1H, d, *J*=7.8 Hz), 4.75(1H, d, *J*=7.2 Hz), 1.26(3H, s, CH₃), 1.25(3H, s, CH₃), 1.08(3H, s, CH₃), 1.08(3H, s, CH₃), 0.91(3H, s, CH₃), 0.88(3H, s, CH₃), 0.86(3H, s, CH₃)。碳谱数据见表1。以上数据与文献^[6]报道基本一致,故确定该化合物为 Elatoside F。

化合物4:白色粉末,5%浓硫酸-乙醇溶液喷雾显色后显紫红色斑点。¹H-NMR(*d*₅-pyridine, 600 MHz)δ: 6.33(1H, d, *J*=7.8 Hz), 5.54(1H, s, H-12), 5.38(1H, d, *J*=7.8 Hz), 4.74(1H, d, *J*=7.8 Hz), 1.30(3H, s, CH₃), 1.26(3H, s, CH₃), 1.10(3H, s, CH₃), 1.10(3H, s, CH₃), 0.91(3H, s, CH₃), 0.88(3H, s, CH₃), 0.86(3H, s, CH₃)。碳谱数据见表1。以上数据与文献^[6]报道基本一致,故确定该化合物为 3-*O*-β-D-吡喃葡萄糖(1-3)-α-L-吡喃阿拉伯糖-齐墩果酸-28-*O*-β-D-吡喃葡萄糖苷(3-*O*-β-D-Glucopyranosyl(1-3)-α-L-arabinopyranosyl oleanolic acid 28-*O*-β-D-glucopyranosyl ester)。

化合物5:白色粉末,5%浓硫酸-乙醇溶液喷雾显色后显紫红色斑点。¹H-NMR(*d*₅-pyridine, 600 MHz)δ: 6.32(1H, d, *J*=8.4 Hz), 5.59(1H, d, *J*=7.8 Hz), 5.42(1H, s, H-12), 5.37(1H, d, *J*=7.8 Hz), 4.82(1H, d, *J*=7.8 Hz), 1.26(3H, s, CH₃), 1.25(3H, s, CH₃), 1.08(3H, s, CH₃), 1.06(3H, s, CH₃), 0.90

(3H, s, CH₃), 0.88(3H, s, CH₃), 0.83(3H, s, CH₃)。碳谱数据见表1。以上数据与文献^[7]报道基本一致,故确定该化合物为Araliasaponin IV。

化合物6:白色粉末,5%浓硫酸-乙醇溶液喷雾显色后显紫红色斑点。¹H-NMR(*d*₅-pyridine, 600 MHz)δ: 6.31(1H, d, *J*=7.8 Hz), 5.64(1H, d, *J*=7.8 Hz), 5.44(1H, s, H-12), 5.10(1H, d, *J*=7.8 Hz), 4.80(1H, d, *J*=7.8 Hz), 1.29(3H, s, CH₃), 1.22(3H, s, CH₃), 1.07(3H, s, CH₃), 0.92(3H, s, CH₃), 0.91(3H, s, CH₃), 0.89(3H, s, CH₃), 0.78(3H, s, CH₃)。碳谱数据见表1。以上数据与文献^[8]报道基本一致,故确定该化合物为屏边三七苷(Stipuleanoside R₂)。

化合物7:白色粉末,5%浓硫酸-乙醇溶液喷雾显色后显紫红色斑点。¹H-NMR(*d*₅-pyridine, 600 MHz)δ: 6.29(1H, d, *J*=7.8 Hz), 5.53(1H, d, *J*=7.8 Hz), 5.44(1H, s, H-12), 5.11(1H, d, *J*=7.8 Hz), 4.88(1H, d, *J*=7.8 Hz), 1.31(3H, s, CH₃), 1.25(3H, s, CH₃), 1.08(3H, s, CH₃), 0.95(3H, s, CH₃), 0.92(3H, s, CH₃), 0.89(3H, s, CH₃), 0.78(3H, s, CH₃)。碳谱数据见表1。以上数据与文献^[9]报道基本一致,故确定该化合物为Tarasaponin IV。

化合物8:白色粉末,5%浓硫酸-乙醇溶液喷雾显色后显紫红色斑点。¹H-NMR(*d*₅-pyridine, 600 MHz)δ: 6.31(1H, d, *J*=7.8 Hz), 5.42(1H, d, *J*=7.8 Hz), 5.42(1H, s, H-12), 5.27(1H, d, *J*=7.8 Hz), 4.46(1H, d, *J*=7.8 Hz), 1.28(3H, s, CH₃), 1.19(3H, s, CH₃), 1.06(3H, s, CH₃), 1.01(3H, s, CH₃), 0.90(3H, s, CH₃), 0.87(3H, s, CH₃), 0.81(3H, s, CH₃)。碳谱数据见表1。以上数据与文献^[10]报道基本一致,故确定该化合物为Elatoside C。

化合物9:白色粉末,5%浓硫酸-乙醇溶液喷雾显色后显紫红色斑点。¹H-NMR(*d*₅-pyridine, 600 MHz)δ: 6.31(1H, d, *J*=7.8 Hz), 5.68(1H, d, *J*=7.8 Hz), 5.42(1H, s, H-12), 4.80(1H, d, *J*=7.8 Hz), 1.30(3H, s, CH₃), 1.28(3H, s, CH₃), 1.25(3H, s, CH₃), 1.08(3H, s, CH₃), 0.95(3H, s, CH₃), 0.92(3H, s, CH₃), 0.89(3H, s, CH₃)。碳谱数据见表1。以上数据与文献^[11]报道基本一致,故确定该化合物为Spinasaponin A 28-O-glucoside。

化合物10:白色粉末,5%浓硫酸-乙醇溶液喷雾显色后显紫红色斑点。¹H-NMR(*d*₅-pyridine, 600 MHz)δ: 6.31(1H, d, *J*=7.8 Hz), 5.43(1H, d, *J*=7.8 Hz), 5.39(1H, s, H-12), 5.32(1H, d, *J*=7.8 Hz), 4.80(1H, d, *J*=7.8 Hz), 1.26(3H, s, CH₃), 1.22(3H, s, CH₃), 1.06(3H, s, CH₃), 0.91(3H, s, CH₃), 0.92(3H, s, CH₃), 0.88(3H, s, CH₃), 0.80(3H, s, CH₃)。碳谱数据见表1。以上数据与文献^[11]报道基本一致,故确定该化合物为椴木皂苷C(Araloside C)。

4 讨论

本试验从椴木总皂苷中分离得到10个五环三萜类皂苷,其中2个为首次从该植物中分离得到,进一步丰富了该植物的化学成分组成。目前,辽东椴木总皂苷的抗心肌缺血活性是研究热点,不仅总皂苷被证实作用确切,而且还发现部分活性显著的单体化合物。本试验结果可为椴木皂苷类成分抗心肌缺血的活性及其构效关系的分析奠定物质基础,还将为辽东椴木的深入研究与新药开发提供科学依据。

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