# Iridoid, Flavonoid, and Phenylethanoid Glycosides from Wiedemannia orientalis 

Zühal GÜVENALP ${ }^{1 *}$, Hilal ÖZBEK ${ }^{1}$, Türesin ÜNSALAR ${ }^{1}$<br>Cavit KAZAZ ${ }^{2}$, L. Ömür DEMİREZER ${ }^{3}$<br>${ }^{1}$ Atatürk University, Faculty of Pharmacy, Department of Pharmacognosy<br>TR-25240 Erzurum-TURKEY<br>e-mail: guvenalp@atauni.edu.tr<br>${ }^{2}$ Atatürk University, Faculty of Arts and Science, Department of Chemistry TR-25240 Erzurum, TURKEY<br>${ }^{3}$ Hacettepe University, Faculty of Pharmacy, Department of Pharmacognosy TR-06100 Ankara, TURKEY

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#### Abstract

Five iridoid glycosides, lamiide, ipolamiide, ipolamiidoside, $6 \beta$-hydroxyipolamiide, and 5 -hydroxy8 -epi-loganin; 5 flavonoid glycosides, apigenin $7-O-\beta$-glucopyranoside, luteolin $5-O-\beta$ - glucopyranoside, isorhamnetin 3 - $O$-rutinoside, quercetin $3-O$-rutinoside, and apigenin 7 - $O$-( $6^{\prime \prime}$-O-trans- $p$-coumaroyl) $\beta$ glucopyranoside; and a phenylethanoid glycoside, acteoside ( $=$ verbascoside), were isolated from the aerial parts of Wiedemannia orientalis (Lamiaceae). Their structures were identified using spectral methods (UV, 1D- and 2D-NMR, and EI-MS).


Key Words: Lamiaceae, Wiedemannia orientalis, iridoid glycoside, flavonoid glycoside, phenylethanoid glycoside.

## Introduction

The genus Wiedemannia (Lamiaceae) is represented by 2 species in the flora of Turkey. Wiedemannia orientalis Fisch. \& Mey. (Lamiaceae) is an endemic species and is widespread throughout Anatolia ${ }^{1}$. Only one report has been published on the chemical constituents of Wiedemannia orientalis. In that report, waterdistilled essential oil from fresh aerial parts of Wiedemannia orientalis was analyzed by GC and GC-MS, and 31 compounds were identified with germacrene D (38.94\%), geijerene (14.60\%), and pregeijerene (12.90\%) as the major constituents ${ }^{2}$. In the present study, we report on the isolation and structure elucidation of 5 iridoid glycosides, lamiide (1), ipolamiide (2), ipolamiidoside (3), $6 \beta$-hydroxyipolamiide (4), and 5 -hydroxy- 8 -epiloganin (5); 5 flavonoid glycosides, apigenin $7-O-\beta$ - glucopyranoside (6), luteolin 5 - $O$ - $\beta$ - glucopyranoside (7), isorhamnetin 3-O-rutinoside (8), quercetin 3- $O$-rutinoside (9), and apigenin 7-O-( $6^{\prime \prime}$ - $O$-trans- $p$-coumaroyl) $\beta$ glucopyranoside (10); and a phenylethanoid glycoside, acteoside (11), from the aerial parts of Wiedemannia orientalis Fisch. \& Mey.

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## Experimental

General Experimental Procedures: ${ }^{1} \mathrm{H}-$ and ${ }^{13} \mathrm{C}$-NMR spectra were recorded on a Varian Mercury Plus 400 MHz for proton and a 100 MHz for carbon by using TMS as the internal standard. Solvents were $\mathrm{CD}_{3} \mathrm{OD}$ and DMSO-d $\mathrm{d}_{6}$. EI-MS was performed on a Finnigan MAT 95 spectrometer. Silica gel $60(0.063-0.200 \mathrm{~mm}$, Merck) and Sephadex LH-20 (Fluka) were used for open column chromatographic separations. Lichroprep RP-18 ( $25-40 \mu \mathrm{~m}$, Merck) material was used for vacuum liquid chromatography (VLC). TLC was carried out on pre- coated Kieselgel $60 \mathrm{~F}_{254}$ aluminum sheets (Merck) and compounds were detected under UV ( 254 nm ) fluorescence and sprayed with $1 \%$ vanillin- $\mathrm{H}_{2} \mathrm{SO}_{4}$ reagent, followed by heating at $105^{\circ} \mathrm{C}$ for $1-2 \mathrm{~min}$.

Plant Material: Wiedemannia orientalis (Lamiaceae) was collected from Sivrihisar, Eskişehir, Turkey, in May 2004. A voucher specimen was deposited in the herbarium of the Pharmacognosy Department, Faculty of Pharmacy, Hacettepe University, Ankara, Turkey (HUEF 04163).

Extraction and Pre-purification: Open-air-dried and powdered aerial parts of the plant ( 131 g ) were extracted 3 times with MeOH at $40^{\circ} \mathrm{C}(3 \times 2 \mathrm{~L})$. After evaporation of the combined extract in vacuo, 26 g of MeOH extract was obtained. The crude extract was dissolved in water and partitioned with $\mathrm{CHCl}_{3}$ ( $3 \times 0.2 \mathrm{~L}$ ) to give the $\mathrm{CHCl}_{3}$ extract ( 5.0 g ). The aqueous phase was further extracted with n-butanol ( 5 $x 0.25 \mathrm{~L}$ ) and the organic layer was evaporated to dryness ( 12.6 g ). The n-BuOH extract of the plant was chosen for further phytochemical studies as given below.

Isolation of the Compounds: n-Butanol extract was re-dissolved in MeOH and chromatographed on a silica gel column eluting with $\mathrm{CHCl}_{3}-\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}$ mixtures ( $80: 20: 2$ and $61: 32: 7$ ), respectively to yield 5 main fractions (Fr. A: 570 mg ; Fr. B: 1.6 g ; Fr. C: 1.4 g ; Fr. D: 588 mg ; Fr. E: 980 mg ). Fr. A was subjected to a column of Sephadex LH 20 eluting with MeOH to yield Fr. $\mathrm{A}_{1}$ ( 433 mg ) and Fr. $\mathrm{A}_{2}$ ( 83 mg ). Fr. $\mathrm{A}_{1}$ was subjected to VLC on reversed-phase material using $\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}$ mixtures $(0 \%-100 \%)$ to give Fr . $\mathrm{A}_{1.1}(16 \mathrm{mg})$ and $\operatorname{Fr}$. $\mathrm{A}_{1.2}(47.8 \mathrm{mg})$. Further processing of Fr. $\mathrm{A}_{1.1}$ on a silica gel column by eluting with $\mathrm{CHCl}_{3}-\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}(61: 32: 7)$ gave compound $\mathbf{4}(11.5 \mathrm{mg})$. Silica gel chromatography of Fr . $\mathrm{A}_{1.2}$ by eluting with $\mathrm{CHCl}_{3}-\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}(70: 30: 3)$ gave compound $\mathbf{3}(20 \mathrm{mg})$. Fr. $\mathrm{A}_{2}$ was subjected to a column of Sephadex LH 20 by eluting with MeOH to yield Fr. $\mathrm{A}_{2.1}(61 \mathrm{mg})$ and Fr. $\mathrm{A}_{2.2}(15 \mathrm{mg})$. Fr. $\mathrm{A}_{2.2}$ was subjected to VLC using reversed-phase material using a $\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}$ mixture ( $0 \%-100 \%$ ) to give compound $\mathbf{1 0}(7 \mathrm{mg})$. Fr. B was fractioned over RP-VLC using MeOH- $\mathrm{H}_{2} \mathrm{O}$ mixtures $(0 \%-100 \%$ ) as eluent to give 4 fractions (Fr. $\mathrm{B}_{1}: 546 \mathrm{mg}$; Fr. B $\mathrm{B}_{2}: 158 \mathrm{mg} ;$ Fr. B $\mathrm{B}_{3}: 67 \mathrm{mg} ;$ Fr. B $\mathrm{B}_{4}: 64 \mathrm{mg}$ ). Fr. B $\mathrm{B}_{1}$ was subjected to a silica gel column using $\mathrm{CHCl}_{3}-\mathrm{MeOH}$ mixtures ( $90: 10,85: 15 \ldots \ldots .70: 30$ ) to give Fr . $\mathrm{B}_{1.1}\left(455 \mathrm{mg}\right.$ ). Fr. $\mathrm{B}_{1.1}$ was purified by preparative TLC using $\mathrm{CHCl}_{3}-\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}(61: 32: 7)$ mixtures to give compound $\mathbf{1}(70 \mathrm{mg})$. Fr. $\mathrm{B}_{2}$ was subjected to a silica gel column using $\mathrm{CHCl}_{3}-\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}(61: 32: 7)$ mixtures to give Fr. $\mathrm{B}_{2.1}(112 \mathrm{mg})$. Fr. $\mathrm{B}_{2.1}$ was purified by preparative TLC using $\mathrm{CHCl}_{3}-\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}(61: 32: 7)$ mixtures to give Fr . $\mathrm{B}_{2.1 .1}(28 \mathrm{mg})$ and Fr. $\mathrm{B}_{2.1 .2}(38 \mathrm{mg})$. Fr. $\mathrm{B}_{2.1 .1}$ was subjected to VLC using reversed-phase material, by using $\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}$ mixtures $(0 \%-100 \%)$ to give compound $\mathbf{5}(18 \mathrm{mg})$. Fr. $\mathrm{B}_{2.1 .2}$ was subjected to VLC using reversed-phase material by using $\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}$ mixtures $(0 \%-100 \%)$ to give compound $\mathbf{2}(19 \mathrm{mg})$. Fr. $\mathrm{B}_{3}$ was subjected to a column of Sephadex LH 20 by eluting with MeOH to give compound $\mathbf{7}(42 \mathrm{mg})$. Fr. $\mathrm{B}_{4}$ was applied to a silica gel column by employing $\mathrm{CHCl}_{3}-\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}(70: 30: 3)$ mixtures to give Fr. $\mathrm{B}_{4.1}$ ( 15 mg ) and Fr. $\mathrm{B}_{4.2}$ ( 25 mg ). Purification of Fr. B 4.1 by Sephadex LH 20 CC using MeOH gave compound $\mathbf{8}(10 \mathrm{mg})$. Purification of Fr. B4.2 by Sephadex LH 20 CC using MeOH gave compound $\mathbf{6}$ ( 7.5 mg ). Fr. C was subjected to VLC

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on reversed-phase material by using $\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}$ mixtures $(0 \%-100 \%)$ to give Fr . $\mathrm{C}_{1}(153 \mathrm{mg})$. Fr. $\mathrm{C}_{1}$ was purified by preparative TLC using $\mathrm{CHCl}_{3}-\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}$ (61:32:7) mixtures to give compound $\mathbf{1 1}(52 \mathrm{mg})$. Fr. D was subjected to VLC using reversed-phase material, by using $\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}$ mixtures $(0 \%-100 \%)$ to give Fr. $\mathrm{D}_{1}(67 \mathrm{mg})$. Fr. $\mathrm{D}_{1}$ was purified by preparative TLC using $\mathrm{CHCl}_{3}-\mathrm{MeOH}-\mathrm{H}_{2} \mathrm{O}(50: 50: 5)$ mixtures to give compound $9(10 \mathrm{mg})$.

## Results and Discussion

In this study, from the aerial parts of Wiedemannia orientalis, 5 iridoid glycosides, lamiide (1), ipolamiide (2), ipolamiidoside (3), $6 \beta$-hydroxyipolamiide (4), and 5 -hydroxy- 8 -epi-loganin (5); 5 flavonoid glycosides, apigenin $7-O-\beta$-glucopyranoside (6), luteolin $5-O-\beta$ - glucopyranoside (7), isorhamnetin 3-O-rutinoside (8), quercetin 3 - $O$-rutinoside (9), and apigenin $7-O$-( $6^{\prime \prime}$ - $O$-trans- $p$-coumaroyl) $\beta$-glucopyranoside (10); and a phenylethanoid glycoside, acteoside (11), were isolated by fractionation of the butanol extract through an open column chromatograph on silica gel and Sephadex LH-20, followed by VLC (Figure).

Lamiide (1): UV (MeOH) $\lambda_{\max } 232 \mathrm{~nm}$; EIMS m/z 259 [M-Glu] ${ }^{+}$, (calc. for $\mathrm{C}_{11} \mathrm{H}_{15} \mathrm{O}_{7}$ ). ${ }^{1} \mathrm{H}$ NMR $\left(\mathrm{CD}_{3} \mathrm{OD}, 400 \mathrm{MHz}\right): \delta 5.81(1 \mathrm{H}, d, J=<1, \mathrm{H}-1), 7.43(1 \mathrm{H}, s, \mathrm{H}-3), 2.24\left(1 \mathrm{H}, d d, J=15.0 / 2.93 \mathrm{~Hz}^{2}, \mathrm{H}_{a}-6\right)$, $2.35\left(1 \mathrm{H}, d d, J=15.2 / 4.95 \mathrm{~Hz}, \mathrm{H}_{b^{-}}-6\right), 3.52(1 \mathrm{H}, d d, J=4.95 / 2.95 \mathrm{~Hz}, \mathrm{H}-7), 2.78(1 \mathrm{H}$, brs, H-9), 1.08 ( 3 H , $s, \mathrm{H}-10), 3.72(3 \mathrm{H}, s, \mathrm{COOMe}), 4.59\left(1 \mathrm{H}, d, J=7.7 \mathrm{~Hz}, \mathrm{H}-1^{\prime}\right), 3.16-3.40\left(4 \mathrm{H}, m, \mathrm{H}-2^{\prime}, \mathrm{H}-3^{\prime}, \mathrm{H}-4^{\prime}, \mathrm{H}-5^{\prime}\right)$, $3.66\left(1 \mathrm{H}, d d, J=11.7 / 5.9 \mathrm{~Hz}, \mathrm{H}_{a}-6^{\prime}\right), 3.89\left(1 \mathrm{H}, d d, J=11.9 / 1.6 \mathrm{~Hz}, \mathrm{H}_{b}-6^{\prime}\right) ;{ }^{13} \mathrm{C} \operatorname{NMR}\left(\mathrm{CD}_{3} \mathrm{OD}, 100 \mathrm{MHz}\right)$ : Table 1.

Table 1. ${ }^{13} \mathrm{C}$ NMR $\left(\mathrm{CD}_{3} \mathrm{OD}, 100 \mathrm{MHz}\right)$ data of compounds 1-5.

| Atomic Number | $\mathbf{1}$ | $\mathbf{2}$ | $\mathbf{3}$ | $\mathbf{4}$ | $\mathbf{5}$ |
| :--- | :---: | :---: | :---: | :---: | :---: |
| Aglycone |  |  |  |  |  |
| 1 | 93.2 | 93.0 | 93.9 | 95.8 | 94.4 |
| 3 | 151.3 | 151.4 | 153.4 | 152.5 | 152.4 |
| 4 | 114.2 | 114.0 | 111.8 | 112.8 | 114.0 |
| 5 | 68.0 | 70.6 | 71.9 | 65.3 | 70.1 |
| 6 | 45.5 | 37.6 | 35.4 | 63.2 | 46.7 |
| 7 | 76.6 | 39.2 | 37.5 | 41.9 | 76.7 |
| 8 | 77.9 | 77.7 | 87.7 | 75.5 | 42.3 |
| 9 | 56.8 | 60.5 | 58.6 | 53.5 | 50.4 |
| 10 | 20.1 | 22.0 | 19.8 | 16.6 | 12.6 |
| 11 | 166.8 | 166.8 | 166.4 | 166.6 | 166.8 |
| COOMe | 50.5 | 50.4 | 50.4 | 50.5 | 50.3 |
| COMe |  |  | 172.1 |  |  |
| COMe |  |  | 20.9 |  |  |
| Glucose |  |  |  |  |  |
| $1^{\prime}$ | 98.4 | 98.4 | 98.8 | 98.7 | 98.5 |
| $2^{\prime}$ | 73.2 | 73.2 | 73.2 | 73.5 | 73.2 |
| $3^{\prime}$ | 76.2 | 76.2 | 76.3 | 76.6 | 76.3 |
| $4^{\prime}$ | 70.4 | 70.6 | 70.4 | 70.5 | 70.5 |
| $5^{\prime}$ | 77.2 | 77.2 | 77.1 | 77.5 | 77.3 |
| $6^{\prime}$ | 61.5 | 61.7 | 61.5 | 61.8 | 61.7 |

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Lamiide (1)


R Compound
H Ipolamiide (2) Ac Ipolamiidoside (3)


5-Hydroxy-8-epi-loganin (5)

$6 \beta$-Hydroxyipolamiide (4)


Apigenin 7-O- $\beta$ - glucopyranoside (6)


Luteolin 5-O- $\beta$ - glucopyranoside (7)


R Compound
$\mathrm{CH}_{3}$ Isorhamnetin 3-O-rutinoside (8)
H Quercetin 3-O-rutinoside (9)

Figure. Chemical structures of the isolated compounds.


Apigenin 7-O-(6"-O-trans-p-coumaroyl) $\beta$-glucopyranoside (10)


Acteoside (11)
Figure. Contunied
Ipolamiide (2): UV (MeOH) $\lambda_{\max } 229 \mathrm{~nm}$; EIMS m/z 244 [M-Glu] ${ }^{+}$, (calc. for $\mathrm{C}_{11} \mathrm{H}_{15} \mathrm{O}_{6}$ ). ${ }^{1} \mathrm{H}$ NMR ( $\left.\mathrm{CD}_{3} \mathrm{OD}, 400 \mathrm{MHz}\right): \delta 5.80(1 \mathrm{H}, d, J=1.1 \mathrm{~Hz}, \mathrm{H}-1), 7.43(1 \mathrm{H}, s, \mathrm{H}-3), 1.92\left(1 \mathrm{H}, \mathrm{m}, \mathrm{H}_{a}-6\right), 2.26$ $\left(1 \mathrm{H}, \mathrm{m}, \mathrm{H}_{b}-6\right), 1.56\left(1 \mathrm{H}, m, \mathrm{H}_{a}-7\right), 2.08\left(1 \mathrm{H}, m, \mathrm{H}_{b}-7\right), 2.47(1 \mathrm{H}, b s, \mathrm{H}-9), 1.14(3 \mathrm{H}, s, \mathrm{H}-10), 3.72(3 \mathrm{H}, s$, COOMe $), 4.57\left(1 \mathrm{H}, d, J=8.0 \mathrm{~Hz}, \mathrm{H}-1^{\prime}\right), 3.17\left(1 \mathrm{H}, d d, J=9.1 / 8.0 \mathrm{~Hz}, \mathrm{H}-2^{\prime}\right), 3.23-3.38\left(3 \mathrm{H}, m, \mathrm{H}-3^{\prime}, \mathrm{H}-4^{\prime}\right.$, $\left.\mathrm{H}-5^{\prime}\right), 3.65\left(1 \mathrm{H}, d d, J=11.9 / 6.0 \mathrm{~Hz}, \mathrm{H}_{a}-6^{\prime}\right), 3.89\left(1 \mathrm{H}, d d, J=11.8 / 2.2 \mathrm{~Hz}, \mathrm{H}_{b}-6^{\prime}\right) ;{ }^{13} \mathrm{C}$ NMR $\left(\mathrm{CD}_{3} \mathrm{OD}, 100\right.$ MHz ): Table 1.

Ipolamiidoside (3): UV (MeOH) $\lambda_{\max } 229 \mathrm{~nm}$; EIMS m/z 286 [M-Glu] ${ }^{+}$, (calc. for $\mathrm{C}_{13} \mathrm{H}_{15} \mathrm{O}_{7}$ ). ${ }^{1} \mathrm{H}$ NMR $\left(\mathrm{CD}_{3} \mathrm{OD}, 400 \mathrm{MHz}\right): \delta 6.05(1 \mathrm{H}, d, J=1.1 \mathrm{~Hz}, \mathrm{H}-1), 7.56(1 \mathrm{H}, d, J=2.9 \mathrm{~Hz}, \mathrm{H}-3), 2.11(1 \mathrm{H}$, $\left.m, \mathrm{H}_{a}-6\right), 2.39\left(1 \mathrm{H}, m, \mathrm{H}_{b}-6\right), 1.62\left(1 \mathrm{H}, m, \mathrm{H}_{a}-7\right), 2.07\left(1 \mathrm{H}, m, \mathrm{H}_{b}-7\right), 2.71(1 \mathrm{H}, d, J=1.1 \mathrm{~Hz}, \mathrm{H}-9), 1.42$ $(3 \mathrm{H}, \mathrm{s}, \mathrm{H}-10), 3.72(3 \mathrm{H}, s, \mathrm{COOMe}), 2.03(3 \mathrm{H}, s, \mathrm{COMe}), 4.57\left(1 \mathrm{H}, d, J=8.0 \mathrm{~Hz}, \mathrm{H}-1^{\prime}\right), 3.16(1 \mathrm{H}, d d, J=$ $\left.9.1 / 8.0 \mathrm{~Hz}, \mathrm{H}-2^{\prime}\right), 3.26-3.39\left(3 \mathrm{H}, m, \mathrm{H}-3^{\prime}, \mathrm{H}-4^{\prime}, \mathrm{H}-5^{\prime}\right), 3.68\left(1 \mathrm{H}, d d, J=12.0 / 5.5 \mathrm{~Hz}, \mathrm{H}_{a}-6^{\prime}\right), 3.89(1 \mathrm{H}, d d$, $\left.J=12.2 / 2.0 \mathrm{~Hz}, \mathrm{H}_{b^{-}}-6^{\prime}\right) ;{ }^{13} \mathrm{C}$ NMR ( $\left.\mathrm{CD}_{3} \mathrm{OD}, 100 \mathrm{MHz}\right)$ : Table 1.

6 $\beta$-hydroxyipolamiide (4): UV (MeOH) $\lambda_{\max } 231 \mathrm{~nm}$; EIMS $m / z 259[\mathrm{M}-\mathrm{Glu}]^{+}$, (calc. for $\left.\mathrm{C}_{11} \mathrm{H}_{15} \mathrm{O}_{7}\right) .{ }^{1} \mathrm{H}$ NMR $\left(\mathrm{CD}_{3} \mathrm{OD}, 400 \mathrm{MHz}\right): \delta 5.42(1 \mathrm{H}, d, J=8.4 \mathrm{~Hz}, \mathrm{H}-1), 7.47(1 \mathrm{H}, s, \mathrm{H}-3), 3.89(1 \mathrm{H}, d$,

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$\boldsymbol{J}=$ signal pattern unclear due to overlapping, H-6), $2.27\left(1 \mathrm{H}, d d, J=15.4 / 1.8 \mathrm{~Hz}, \mathrm{H}_{a}-7\right), 2.56(1 \mathrm{H}, d, J=$ $\left.15.7 \mathrm{~Hz}, \mathrm{H}_{b}-7\right), 2.35(1 \mathrm{H}, d, J=8.7 \mathrm{~Hz}, \mathrm{H}-9), 1.52(3 \mathrm{H}, \mathrm{s}, \mathrm{H}-10), 3.70(3 \mathrm{H}, s, \mathrm{COOMe}), 4.73(1 \mathrm{H}, d, J=8.0$ $\left.\mathrm{Hz}, \mathrm{H}-1^{\prime}\right), 3.19-3.40\left(4 \mathrm{H}, m, \mathrm{H}-2^{\prime}, \mathrm{H}-3^{\prime}, \mathrm{H}-4^{\prime}, \mathrm{H}-5^{\prime}\right), 3.60\left(1 \mathrm{H}, d d, J=11.9 / 6.6 \mathrm{~Hz}, \mathrm{H}_{a}-6^{\prime}\right), 3.91(1 \mathrm{H}, d d, J=$ $\left.12.1 / 2.2 \mathrm{~Hz}, \mathrm{H}_{b}-6^{\prime}\right) ;{ }^{13} \mathrm{C}$ NMR ( $\left.\mathrm{CD}_{3} \mathrm{OD}, 100 \mathrm{MHz}\right)$ : Table 1.

5-Hydroxy-8-epi-loganin (5): UV (MeOH) $\lambda_{\max } 234 \mathrm{~nm}$; EIMS m/z 244 [M-Glu] ${ }^{+}$(calc. for $\left.\mathrm{C}_{11} \mathrm{H}_{15} \mathrm{O}_{6}\right) .{ }^{1} \mathrm{H}$ NMR $\left(\mathrm{CD}_{3} \mathrm{OD}, 400 \mathrm{MHz}\right): \delta 5.75(1 \mathrm{H}, d, J=1.5 \mathrm{~Hz}, \mathrm{H}-1), 7.47(1 \mathrm{H}, s, \mathrm{H}-3), 2.03(1 \mathrm{H}, d d$, $\left.J=13.6 / 6.5 \mathrm{~Hz}, \mathrm{H}_{a}-6\right), 2.57\left(1 \mathrm{H}, d d, J=13.6 / 5.5 \mathrm{~Hz}, \mathrm{H}_{b}-6\right), 3.54(1 \mathrm{H}, m, \mathrm{H}-7), 2.26(1 \mathrm{H}, m, \mathrm{H}-8), 2.79(1 \mathrm{H}$, $d d, J=10.3 / 1.1 \mathrm{~Hz}, \mathrm{H}-9), 0.95(3 \mathrm{H}, d, J=7.3 \mathrm{~Hz}, \mathrm{H}-10), 3.72(3 \mathrm{H}, s, \mathrm{COOMe}), 4.55\left(1 \mathrm{H}, d, J=8.0 \mathrm{~Hz}, \mathrm{H}-1^{\prime}\right)$, $3.15-3.38\left(4 \mathrm{H}, m, \mathrm{H}-2^{\prime}, \mathrm{H}-3^{\prime}, \mathrm{H}-4^{\prime}, \mathrm{H}-5^{\prime}\right), 3.64\left(1 \mathrm{H}, d d, J=11.7 / 6.2 \mathrm{~Hz}, \mathrm{H}_{a}-6^{\prime}\right), 3.90(1 \mathrm{H}, d d, J=11.9 / 2.0$ $\left.\mathrm{Hz}, \mathrm{H}_{b}-6^{\prime}\right) ;{ }^{13} \mathrm{C}$ NMR ( $\left.\mathrm{CD}_{3} \mathrm{OD}, 100 \mathrm{MHz}\right)$ : Table 1.

Apigenin 7- $\boldsymbol{O}$ - $\beta$-glucopyranoside (6): $\mathrm{C}_{21} \mathrm{H}_{20} \mathrm{O}_{10}$ (mol.wt. 432); EIMS m/z $270[\mathrm{M}-\mathrm{Glu}]^{+} ;{ }^{1} \mathrm{H}$ NMR (DMSO-d $\left.{ }_{6}, 400 \mathrm{MHz}\right): \delta_{H} 6.84(1 \mathrm{H}, s, \mathrm{H}-3), 6.42(1 \mathrm{H}, d, J=2.2 \mathrm{~Hz}, \mathrm{H}-6), 6.81(1 \mathrm{H}, d, J=2.2 \mathrm{~Hz}$, $\mathrm{H}-8), 7.93\left(2 \mathrm{H}, d, J=9.1 \mathrm{~Hz}, \mathrm{H}-2^{\prime}, \mathrm{H}-6^{\prime}\right), 6.90\left(2 \mathrm{H}, d, J=8.8 \mathrm{~Hz}, \mathrm{H}-3^{\prime}, \mathrm{H}-5^{\prime}\right), 5.05(1 \mathrm{H}, d, J=7.3 \mathrm{~Hz}$, $\left.\mathrm{H}-1^{\prime \prime}\right), 3.14-3.39\left(3 \mathrm{H}, m, \mathrm{H}-2^{\prime \prime}, \mathrm{H}-3^{\prime \prime}, \mathrm{H}-4^{\prime \prime}, \mathrm{H}-5^{\prime \prime}\right), 3.55\left(1 \mathrm{H}, d d, J=11.9 / 6.2 \mathrm{~Hz}, \mathrm{H}_{a}-6^{\prime \prime}\right), 3.73(1 \mathrm{H}, d d, J=$ $\left.11.6 / 1.8 \mathrm{~Hz}, \mathrm{H}_{b}-6^{\prime \prime}\right) ;{ }^{13} \mathrm{C}$ NMR (DMSO-d ${ }_{6}, 100 \mathrm{MHz}$ ): Table 2.

Luteolin 5- $\boldsymbol{O}$ - $\beta$-glucopyranoside (7): $\mathrm{C}_{21} \mathrm{H}_{20} \mathrm{O}_{11}$ (mol.wt. 448); EIMS m/z $286[\mathrm{M}-\mathrm{Glu}]^{+} ;{ }^{1} \mathrm{H}$ NMR (DMSO-d $\left.{ }_{6}, 400 \mathrm{MHz}\right): \delta_{H} 6.54(1 \mathrm{H}, s, \mathrm{H}-3), 6.67(1 \mathrm{H}, d, J=2.2 \mathrm{~Hz}, \mathrm{H}-6), 6.77(1 \mathrm{H}, d, J=2.2 \mathrm{~Hz}$, $\mathrm{H}-8), 7.33\left(1 \mathrm{H}, d, J=2.2 \mathrm{~Hz}, \mathrm{H}-2^{\prime}\right), 6.85\left(1 \mathrm{H}, d, J=8.4 \mathrm{~Hz}, \mathrm{H}-5^{\prime}\right), 7.35\left(1 \mathrm{H}, d d, J=8.4 / 2.2 \mathrm{~Hz}, \mathrm{H}-6^{\prime}\right)$, $4.69\left(1 \mathrm{H}, d, J=7.0 \mathrm{~Hz}, \mathrm{H}-1^{\prime \prime}\right), 3.21-3.63\left(4 \mathrm{H}, m, \mathrm{H}-2^{\prime \prime}, \mathrm{H}-3^{\prime \prime}, \mathrm{H}-4^{\prime \prime}, \mathrm{H}-5^{\prime \prime}\right), 3.45\left(1 \mathrm{H}, m, \mathrm{H}_{a}-6^{\prime \prime}\right), 3.80(1 \mathrm{H}$, $\left.d, J=10.0 / 1.8 \mathrm{~Hz}, \mathrm{H}_{b}-6^{\prime \prime}\right) ;{ }^{13} \mathrm{C}$ NMR (DMSO-d ${ }_{6}, 100 \mathrm{MHz}$ ): Table 2.

Isorhamnetin 3- $\boldsymbol{O}$-rutinoside (8): $\mathrm{C}_{28} \mathrm{H}_{32} \mathrm{O}_{16}$ (mol.wt. 624); EIMS m/z $316[\mathrm{M}-(\mathrm{Glu}+\mathrm{Rh})]^{+} ;{ }^{1} \mathrm{H}$ NMR $\left(\mathrm{CD}_{3} \mathrm{OD}, 400 \mathrm{MHz}\right): \delta_{H} 6.18(1 \mathrm{H}, d, J=2.2 \mathrm{~Hz}, \mathrm{H}-6), 6.37(1 \mathrm{H}, d, J=2.2 \mathrm{~Hz}, \mathrm{H}-8), 7.92(1 \mathrm{H}, d, J$ $\left.=1.8 \mathrm{~Hz}, \mathrm{H}-2^{\prime}\right), 6.90\left(1 \mathrm{H}, d, J=8.4 \mathrm{~Hz}, \mathrm{H}-5^{\prime}\right), 7.62\left(1 \mathrm{H}, d d, J=8.6 / 2.0 \mathrm{~Hz}, \mathrm{H}-6^{\prime}\right), 3.94\left(3 \mathrm{H}, s, \mathrm{OCH}_{3}\right)$, $5.21\left(1 \mathrm{H}, d, J=7.3 \mathrm{~Hz}, \mathrm{H}-1^{\prime \prime}\right), 3.21-3.63\left(4 \mathrm{H}, m, \mathrm{H}-2^{\prime \prime}, \mathrm{H}-3^{\prime \prime}, \mathrm{H}-4^{\prime \prime}, \mathrm{H}-5^{\prime \prime}\right), 3.45\left(1 \mathrm{H}, m, \mathrm{H}_{a}-6^{\prime \prime}\right), 3.80(1 \mathrm{H}$, $\left.d, J=10.0 / 1.8 \mathrm{~Hz}, \mathrm{H}_{b}-6^{\prime \prime}\right), 4.52\left(1 \mathrm{H}, d, J=1.5 \mathrm{~Hz}, \mathrm{H}-1^{\prime \prime \prime}\right), 3.21-3.53\left(4 \mathrm{H}, m, \mathrm{H}-2^{\prime \prime \prime}, \mathrm{H}-3^{\prime \prime \prime}, \mathrm{H}-4^{\prime \prime \prime}, \mathrm{H}-5^{\prime \prime \prime}\right)$, $1.09\left(3 \mathrm{H}, d, J=6.2 \mathrm{~Hz}, \mathrm{CH}_{3}-6^{\prime \prime \prime}\right) ;{ }^{13} \mathrm{C}$ NMR $\left(\mathrm{CD}_{3} \mathrm{OD}, 100 \mathrm{MHz}\right)$ : Table 2.

Quercetin 3- $\boldsymbol{O}$-rutinoside (9): $\mathrm{C}_{27} \mathrm{H}_{30} \mathrm{O}_{16}$ (mol.wt. 610); EIMS m/z $301[\mathrm{M}-(\mathrm{Glu}+\mathrm{Rh})]^{+} .{ }^{1} \mathrm{H}$ NMR (DMSO-d $\left.{ }_{6}, 400 \mathrm{MHz}\right): \delta_{H} 6.16(1 \mathrm{H}, d, J=2.2 \mathrm{~Hz}, \mathrm{H}-6), 6.35(1 \mathrm{H}, d, J=2.2 \mathrm{~Hz}, \mathrm{H}-8), 7.53(1 \mathrm{H}$, $\left.d, J=1.8 \mathrm{~Hz}, \mathrm{H}-2^{\prime}\right), 6.81\left(1 \mathrm{H}, d, J=8.0 \mathrm{~Hz}, \mathrm{H}-5^{\prime}\right), 7.50\left(1 \mathrm{H}, d d, J=8.0 / 1.8 \mathrm{~Hz}, \mathrm{H}-6^{\prime}\right), 5.32(1 \mathrm{H}, d, J=$ $\left.7.4 \mathrm{~Hz}, \mathrm{H}-1^{\prime \prime}\right), 3.01-3.37\left(4 \mathrm{H}, m, \mathrm{H}-2^{\prime \prime}, \mathrm{H}-3^{\prime \prime}, \mathrm{H}-4^{\prime \prime}, \mathrm{H}-5^{\prime \prime}\right), 3.30\left(1 \mathrm{H}, m, \mathrm{H}_{a}-6^{\prime \prime}\right), 3.68(1 \mathrm{H}, d, J=10.3 \mathrm{~Hz}$, $\left.\mathrm{H}_{b}-6^{\prime \prime}\right), 4.36\left(1 \mathrm{H}, d, J=1.8 \mathrm{~Hz}, \mathrm{H}-1^{\prime \prime \prime}\right), 3.01-3.37\left(4 \mathrm{H}, m, \mathrm{H}-2^{\prime \prime \prime}, \mathrm{H}-3^{\prime \prime \prime}, \mathrm{H}-4^{\prime \prime \prime}, \mathrm{H}-5^{\prime \prime \prime}\right), 0.97(3 \mathrm{H}, d, J=6.2$ $\left.\mathrm{Hz}, \mathrm{CH}_{3}-6^{\prime \prime \prime}\right) ;{ }^{13} \mathrm{C}$ NMR ( $\mathrm{DMSO}_{\mathrm{d}}^{6}, 100 \mathrm{MHz}$ ): Table 2.

Apigenin 7- $\boldsymbol{O}$-( $\mathbf{6}^{\prime \prime}$ - $\boldsymbol{O}$-trans- $\boldsymbol{p}$-coumaroyl) $\beta$-glucopyranoside (10): $\mathrm{C}_{30} \mathrm{H}_{26} \mathrm{O}_{12}$ (mol.wt. 578); EIMS $m / z 578[\mathrm{M}]^{+}, 149,267,311 .{ }^{1} \mathrm{H}$ NMR (DMSO-d $\left.{ }_{6}, 400 \mathrm{MHz}\right): \delta_{H} 6.81(1 \mathrm{H}, s, \mathrm{H}-3), 6.45(1 \mathrm{H}, d, J=$ $1.8 \mathrm{~Hz}, \mathrm{H}-6), 6.79(1 \mathrm{H}, d, J=1.8 \mathrm{~Hz}, \mathrm{H}-8), 7.92\left(2 \mathrm{H}, d, J=8.7 \mathrm{~Hz}, \mathrm{H}-2^{\prime}, \mathrm{H}-6^{\prime}\right), 6.89(2 \mathrm{H}, d, J=8.7 \mathrm{~Hz}$, $\left.\mathrm{H}-3^{\prime}, \mathrm{H}-5^{\prime}\right), 5.14\left(1 \mathrm{H}, d, J=7.3 \mathrm{~Hz}, \mathrm{H}-1^{\prime \prime}\right), 3.20-3.40\left(3 \mathrm{H}, m, \mathrm{H}-2^{\prime \prime}, \mathrm{H}-3^{\prime \prime}, \mathrm{H}-4^{\prime \prime}\right), 3.81(1 \mathrm{H}, t, J=8.1 \mathrm{~Hz}$, $\left.\mathrm{H}-5^{\prime \prime}\right), 4.13\left(1 \mathrm{H}, d d, J=11.9 / 7.1 \mathrm{~Hz}, \mathrm{H}_{a}-6^{\prime \prime}\right), 4.43\left(1 \mathrm{H}, d, J=10.6 \mathrm{~Hz}, \mathrm{H}_{b}-6^{\prime \prime}\right), 7.34(2 \mathrm{H}, d, J=8.4 \mathrm{~Hz}$, $\left.\mathrm{H}-2^{\prime \prime \prime}, \mathrm{H}-6^{\prime \prime \prime}\right), 6.64\left(2 \mathrm{H}, d, J=8.4 \mathrm{~Hz}, \mathrm{H}-3^{\prime \prime \prime}, \mathrm{H}-5^{\prime \prime \prime}\right), 6.30(1 \mathrm{H}, d, J=15.7 \mathrm{~Hz}, \mathrm{H}-\alpha), 7.46(1 \mathrm{H}, d, J=15.7$ $\mathrm{Hz}, \mathrm{H}-\beta) ;{ }^{13} \mathrm{C}$ NMR $\left(\mathrm{DMSO}_{\mathrm{d}}\right.$, 100 MHz$)$ : Table 2.

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Acteoside (11): $\mathrm{C}_{29} \mathrm{H}_{36} \mathrm{O}_{15}$ (mol.wt.: 624 ); ${ }^{1} \mathrm{H}$ NMR $\left(\mathrm{CD}_{3} \mathrm{OD}, 400 \mathrm{MHz}\right)$ and ${ }^{13} \mathrm{C}$ NMR $\left(\mathrm{CD}_{3} \mathrm{OD}\right.$, 100 MHz ): Table 3.

Table 2. ${ }^{13} \mathrm{C}$ NMR data of compounds $\mathbf{6 - 1 0}$.

| Atomic Number | 6 | 7 | 8 | 9 | 10 |
| :---: | :---: | :---: | :---: | :---: | :---: |
| Aglycone |  |  |  |  |  |
| 2 | 165.0 | 163.4 | 157.4 | 157.1 | 164.9 |
| 3 | 103.6 | 106.4 | 134.2 | 134.0 | 103.6 |
| 4 | 182.6 | 177.6 | 178.0 | 178.0 | 182.6 |
| 5 | 162.5 | 159.0 | 161.8 | 161.9 | 162.0 |
| 6 | 100.1 | 105.2 | 99.2 | 99.4 | 100.1 |
| 7 | 163.6 | 162.0 | 166.0 | 165.0 | 163.3 |
| 8 | 95.5 | 98.8 | 94.0 | 94.3 | 95.4 |
| 9 | 157.6 | 159.3 | 157.5 | 157.3 | 157.5 |
| 10 | 106.0 | 108.9 | 104.2 | 104.5 | 106.0 |
| $1^{\prime}$ | 121.3 | 122.1 | 121.8 | 121.8 | 121.6 |
| $2^{\prime}$ | 129.3 | 113.8 | 113.6 | 116.9 | 129.2 |
| $3^{\prime}$ | 116.7 | 146.3 | 147.2 | 145.4 | 116.6 |
| $4^{\prime}$ | 161.7 | 149.9 | 149.7 | 149.1 | 161.8 |
| $5^{\prime}$ | 116.7 | 116.6 | 114.9 | 115.9 | 116.6 |
| $6^{\prime}$ | 129.3 | 119.2 | 122.8 | 122.3 | 129.2 |
| $\mathrm{OCH}_{3}$ |  |  | 55.6 |  |  |
| Glucose |  |  |  |  |  |
| $1^{\prime \prime}$ | 100.5 | 105.0 | 103.3 | 101.9 | 100.1 |
| $2^{\prime \prime}$ | 73.7 | 74.3 | 74.7 | 74.7 | 73.6 |
| $3^{\prime \prime}$ | 77.8 | 76.3 | 76.2 | 76.6 | 76.8 |
| $4^{\prime \prime}$ | 70.2 | 70.4 | 70.4 | 70.6 | 70.6 |
| $5^{\prime \prime}$ | 77.1 | 78.2 | 77.0 | 77.1 | 74.4 |
| $6^{\prime \prime}$ | 61.2 | 61.5 | 67.4 | 67.6 | 64.0 |
| Rhamnose |  |  |  |  | Acyl |
| $1^{\prime \prime \prime}$ |  |  | 101.3 | 101.4 | 125.5 |
| $2^{\prime \prime \prime}$ |  |  | 70.8 | 71.0 | 130.7 |
| $3^{\prime \prime \prime}$ |  |  | 71.1 | 71.2 | 116.3 |
| $4^{\prime \prime \prime}$ |  |  | 72.6 | 72.5 | 160.4 |
| $5^{\prime \prime \prime}$ |  |  | 68.6 | 68.9 | 116.3 |
| $6^{\prime \prime \prime}$ |  |  | 16.7 | 18.4 | 130.7 |
| $\alpha$ |  |  |  |  | 114.4 |
| $\beta$ |  |  |  |  | 145.6 |
| $\mathrm{C}=\mathrm{O}$ |  |  |  |  | 167.1 |

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Table 3. ${ }^{1} \mathrm{H}$ NMR ( $400 \mathrm{MHz}, \mathrm{CD}_{3} \mathrm{OD}$ ) and ${ }^{13} \mathrm{C}$ NMR ( $100 \mathrm{MHz}, \mathrm{CD}_{3} \mathrm{OD}$ ) data for compound 11.

| Atomic Number | DEPT | $\begin{gathered} \delta_{C} \\ (\mathrm{ppm}) \end{gathered}$ | $\begin{gathered} \delta_{H} \\ (\mathrm{ppm}) \end{gathered}$ | $\begin{gathered} J \\ (\mathrm{~Hz}) \end{gathered}$ |
| :---: | :---: | :---: | :---: | :---: |
| Aglycone |  |  |  |  |
| 1 | C | 130.3 |  |  |
| 2 | CH | 115.9 | 6.69 d | 1.1 |
| 3 | C | 144.9 |  |  |
| 4 | C | 143.4 |  |  |
| 5 | CH | 115.3 | 6.67 d | 7.7 |
| 6 | CH | 120.1 | 6.55 dd | 7.7/1.1 |
| $\alpha$ | $\mathrm{CH}_{2}$ | 70.9 | 3.72 m 4.05 m |  |
| $\beta$ | $\mathrm{CH}_{2}$ | 35.4 | 2.78 t | 6.0 |
| Glucose |  |  |  |  |
| $1{ }^{\prime}$ | CH | 103.0 | 4.37 d | 7.7 |
| $2^{\prime}$ | CH | 75.0 | 3.39 m |  |
| $3^{\prime}$ | CH | 80.5 | 3.81 t | 9.2 |
| $4^{\prime}$ | CH | 69.4 | 4.92 t | 9.5 |
| $5^{\prime}$ | CH | 74.8 | 3.55 m |  |
| $6 \mathrm{a}^{\prime} 6 \mathrm{~b}^{\prime}$ | $\mathrm{CH}_{2}$ | 61.2 | 3.53 m 3.61 m |  |
| Rhamnose |  |  |  |  |
| $1^{\prime \prime}$ | CH | 101.9 | 5.18 d | 1.1 |
| $2^{\prime \prime}$ | CH | 71.2 | 3.91 m |  |
| $3^{\prime \prime}$ | CH | 71.1 | 3.57 m |  |
| $4^{\prime \prime}$ | CH | 72.6 | 3.30 m |  |
| $5^{\prime \prime}$ | CH | 69.3 | 3.54 m |  |
| $6^{\prime \prime}$ | $\mathrm{CH}_{3}$ | 17.3 | 1.08 d | 6.2 |
| Acyl moiety |  |  |  |  |
| $1^{\prime \prime \prime}$ | C | 126.4 |  |  |
| $2^{\prime \prime \prime}$ | CH | 115.1 | 7.05 d | 1.1 |
| $3^{\prime \prime \prime}$ | C | 145.7 |  |  |
| $4^{\prime \prime \prime}$ | C | 148.8 |  |  |
| $5^{\prime \prime \prime}$ | CH | 114.0 | 6.77 d | 7.7 |
| $6^{\prime \prime \prime}$ | CH | 122.1 | 6.95 dd | 7.7/1.1 |
| $\alpha^{\prime}$ | CH | 113.4 | 6.27 d | 15.7 |
| $\beta^{\prime}$ | CH | 146.9 | 7.59 d | 15.7 |
| $\mathrm{C}=\mathrm{O}$ | C | 167.2 |  |  |

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Chemical structures of compounds 1-11 were identified by comparing their spectral (UV, ${ }^{1} \mathrm{H}$ and, ${ }^{13} \mathrm{C}$ NMR) data with those reported in previous studies as: Lamiide $(\mathbf{1})^{3}$, ipolamiide $(\mathbf{2})^{4}$, ipolamiidoside $(\mathbf{3})^{5}$, $6 \beta$-hydroxyipolamiide $(4)^{6-7}, 5$-hydroxy-8-epi-loganin $(5)^{6-8}$, apigenin $7-O$ - $\beta$ - glucopyranoside $(6)^{9}$, luteolin $5-O-\beta$ - glucopyranoside $(\mathbf{7})^{10}$, isorhamnetin 3 - $O$-rutinoside $(\mathbf{8})^{11}$, quercetin 3 - $O$-rutinoside $(\mathbf{9})^{12}$, apigenin $7-O$-( $6^{\prime \prime}$-O-trans-p-coumaroyl) $\beta$ - glucopyranoside $(\mathbf{1 0})^{13}$, and acteoside $(\mathbf{1 1})^{14}$, respectively.

Lamiide, ipolamiide, ipolamiidoside, $6 \beta$-hydroxyipolamiide, 5 -hydroxy-8-epi-loganin, apigenin 7-O-$\beta$-glucopyranoside, luteolin 5-O- $\beta$-glucopyranoside, isorhamnetin 3- $O$-rutinoside, quercetin 3-O-rutinoside, apigenin $7-O$ - ( $6^{\prime \prime}$ - O-trans-p-coumaroyl) $\beta$ - glucopyranoside, and acteoside were isolated for the first time from a Wiedemannia species.

Isolated compounds from Wiedemannia orientalis show different activities. Quercetin 3-O-rutinoside is known to possess antioxidant activity ${ }^{15}$. Lamiide showed anti-inflammatory activity and inhibited lipid peroxidation ${ }^{16}$. Ipolamiide showed anti-inflammatory activity ${ }^{17}$. Ipolamiidoside is reported to have antiviral activity ${ }^{18}$. Acteoside is shown to possess various activities such as anti-inflammatory ${ }^{17}$, antioxidant ${ }^{19}$, antimutagenic ${ }^{19}$, anticarcinogenic ${ }^{19}$, and neuroprotective effects ${ }^{20}$. Consequently, Wiedemannia orientalis can be a good source for various activities.

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[^0]:    *Corresponding author

