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New flavonoids from bioactive extract of Algerian medicinal plant *Launeae arborescens*

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PEER REVIEW

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Comments

The research presented by the authors complements previous work on the phytochemical study of this plant. In fact, after studying the methanol fraction of the extraction of this plant, the authors are interested in the butanol fraction by isolating and identifying new flavonoids using efficient chromatographic and spectroscopic methods. The description of these novel compounds of quite complex structures is a very interesting scientific result to use to search for active substances of this medicinal plant used by the people of these regions.

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ABSTRACT

Objective: To investigate the butanol fraction of the water/acetone extract and isolate of the new flavonoids from *Launeae arboescens*.

Methods: The compounds were isolated by liquid chromatographic methods and their structures were identified by using spectroscopic analysis.

Results: The isolated compounds were identified as: 7-O-[α-rhamnopyranosyl 4′,5,6-Trihydroxy flavone 1,4′,5′-Di-Methoxy 7-(5′′-Me Hexan)1-oyl flavanone 2, 3′′-isopropyl pyrano [1′′:7,4′′:6] 3′,4′,5′,5-Tetrahydroxy flavanone 3,5,4′,5′-Tri-Hydroxy 7-(3′′-Me butan) -yl flavanone 4, 5,7-Dihydroxy-2′,4′,5′ -trimethoxy-isoflavanone 5,5,6,7,4′-tetrahydroxy flavonol 6,7-O-[α-rhamnopyranosyl-(1->6)-β-glucopyranosyl]- 4′,5,7-tri-hydroxy-flavanone 7,7-O-[α-rhamnopyranosyl-(1->6)-β-glucopyranosyl] 3′,5-Dihydroxy 4′-Methoxy flavanone 8.

Conclusions: The presence of different types of bioactive flavonoids in *Launeae arboescens* extract can explain the large ethnopharmacological uses and the potential activity of this medicinal plant.

KEYWORDS

Launeae arborescens, Asteraceae, Flavanone, Isoflavanone, Glycosid flavanone

1. Introduction

Launeae arborescens (*L. arborescens*) is a medicinal plant having capacities of important propagation. Following its biotope, associate to different species, it is frequently notably in the whole region of Algerian southwest of Wadi-Namous until the region of Karzaz. According to our ethnopharmacological survey[1,2], *L. arborescens* is used for treatment of the illnesses gastric. Following our phytochemical works achieved on the polyphenols of the methanolic extract of aerial part of *L. arborescens*, we are also interested to investigate the butanol fraction of the water/acetone extract and isolate of the new flavonoids from this plant[3].

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2. Materials and methods

2.1. General experimental procedure

UV spectra were obtained in MeOH solvent with Unicam UV 300 spectrophotometer. IR spectra were obtained with a Avatar 320 FT-IR spectrophotometer. The NMR spectra were taken on a Bruker GP 250 (^{1}H, 300 MHz; ^{13}C, 125 MHz) Spectrometer. EIMS spectra were obtained on a VG Trio-2 spectrometer. TLC was carried out on silica gel 60 F_{254} plates (Merck, Germany). Column chromatography was performed over silica gel 60 (Merck, particle size 230-400 mesh).

2.2. Plant materials

The aerial part of *L. arborescens* were collected in March 2000 from Bechar (hammada, Oued saoura, Bechar, Algeria). The botanical identification and a voucher specimen was conserved at the phytochemical herbarium of Phytochemistry and Organic Synthesis Laboratory of University Center of Bechar under to accession number CA99/25[2,4].

2.3. Extraction and isolation

The dried aerial part of plants (200 g) of *L. arborescens* were extracted with acetone-water (70:30) using soxhlet apparatus; reflux for 6 h was performed. The residue was evaporated in vacum apparatus until two third, then the third of aqueous residue was partitioned sequentially with *n*-hexan, ethyl ether, EtOAc and *n*-BuOH[5]. To purify and to identify the constituents of butanol fraction (2.36 g), one achieved some separations by liquid chromatography on column, one using a column in glass of type 20 mm/300 mm (29/39) filled with a stationary phase of silica gel (0,20 mm) and the mobile phase chosen for this separation is acetone/toluene/formic acid (60:80:10)[6].

3. Results

The separation has been done previously on a mass of 533 mg of butanol extract in the same conditions. We regrouped the final results after several separations and analysis chromatographic (Table 1).

Table 1

Results of liquid column chromatography (C1,C2,C3: first, second and third column).

Compounds	R_f	Mass (mg)	Yield (%)	Column	Fractions
1	0.16	32	6.00	C2	99-101
2	0.20	40	7.50	C2	68-71
3	0.30	53	9.94	C3	62-67
4	0.40	35	6.57	C3	58-61
5	0.74	77	14.44	C1	40-47
6	0.80	30	5.63	C1	35-37
7	0.90	79	14.82	C1	23-30
8	0.96	167	31.33	C3	6-22

TLC analysis of the samples separated by liquid chromatography on column revealed the existence of eight products to different R_f of which one recovered them after spraying of the solvents for determination of the structures using spectroscopic methods (Table 1).

We have successfully separated three products in the first column (5, 6, 7), two other in the second column (C1, C2) and three in third column (3, 4, 8) by this protocol (Figure 1).

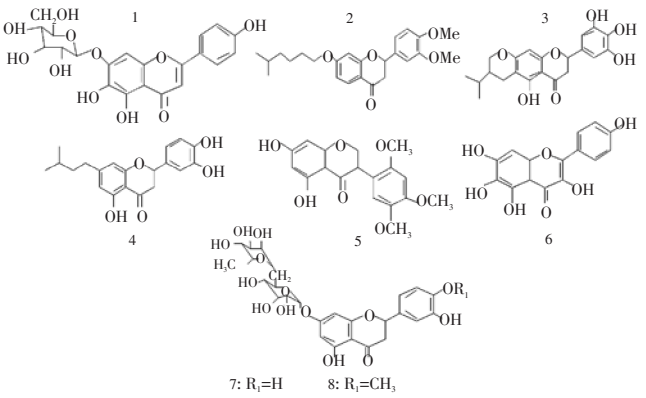

Figure 1. Structures of flavonoids isolated from aerial part of *L. arborescens*.

7-*O*-[α-rhamnopyranosyl 4′,5,6-Trihydroxy flavone. (1): Tf=169 °C. UV_{max} (MeOH): 237, 275, 331 nm. [35] IR(KBr): 3415, 2924, 1613, 1503, 1388, 1257, 1071, 815, 755 cm^{-1}. 1H NMR: 6.59 (H-3 , br s), 6.98 (H-8, s), 7.84 (H-2′, d, *J*= 6.8 Hz), 6.91 (H-3′, d, *J*=7.3 Hz), 6.91 (H-5′, d, *J*=7.3 Hz), 7.84 (H-6′, d, *J*=6.8 Hz), 5.09 (H-1″, d, *J*=5.9 Hz), 3.59 (H-2″, m), 3.56 (H-3″, m), 3.43 (H-4″, t, *J*=8.3 Hz), 3.59 (H-5″, m), 3.74 (H-6″a, m), 3.98 (H-6″b, d, *J*=11.7 Hz). RMN ^{13}C: 166.77 (C-2), 103.49 (C-3), 184.39 (C-4), 147.92 (C-5), 131.86 (C-6), 152.75 (C-7) 95.82 (C-8), 151.75 (C-9), 107.47 (C-10), 123.29 (C-1′), 129.56 (C-2′), 117.00 (C-3′), 162.78 (C-4′), 117.00 (C-5′), 129.56 (C-6′), 102.66 (C-1″), 74.73 (C-2″), 77.52 (C-3″), 71.42 (C-4″), 78.58 (C-5″), 62.55 (C-6″).

4′,5′-Di-Methoxy 7-(5″-Me Hexan)1-oyl flavanone (2) molecular formula $C_{24}H_{30}O_5$, 72.34 (C), 7.59 (H), 20.07(O), MW=398.50, UV spectra: 254, 276, 336, IR (KBr): 3405, 2924, 2853, 1771, 1738, 1509, 1684, 1613, 1252, 1127, 1061, 1383, 755, 815 cm^{-1}, 1H NMR ($CDCl_3$, 300 MHz): 5.36 (t, H-2, 3 Hz, 1.8 Hz), 2.95 (d, H-3a, 1.8), 2.88 (d, H-3b, 3 Hz), 7.392 (d, H-5, 6.8 Hz), 7.325 (d, H-6, 6.8 Hz), 7.28 (s, H-8), 7.455 (d, H-6′, 7.2 Hz), 7.303 (d, H-5′, 7.2 Hz), 7.37 (s, H-2′), 3.738 (t, 7 Hz, H-1″, 2H), 3.668 (s, OCH_3, 6H), 1.67 (m, 1.6 Hz, 2H-2, 2H-3, 2H-4, H-5, 7H), 0.86 (d, 6.6 Hz, H-6, 6H), ^{13}C NMR (CDCl3, 300 MHz):

3″-isopropyl pyrano [1″:7,4″:6] 3′,4′,5′,5-Tetrahydroxy flavanone (3): molecular formula $C_{21}H_{22}O_7$: 65.28 (C), 5.74 (H), 28.98 (O), MW=386.41, UV spectra: 238, 271, 335 IR (KBr): 3404, 2923, 2847, 1738, 1459, 1607, 1170, 1121, 1377, 618, 1H NMR ($CDCl_3$, 300 MHz): 5.32 (H-2), 2.37 (H-3), 7.21 (s, H-8), 7.28 (s, H-2′), 7.28 (s, H-6′), 3.68 (d, H-1″a, 6 Hz), 3.7 (d, H-1″b, 6Hz) , 2.13 (H-3″a, 1.2 Hz) , 2.12 (H-3″b, 1.2 Hz) , 0.75 (H-4″), 0.90 (H-5″) , 1.63 (m, H-2″, 1H), ^{13}C NMR ($CDCl_3$, 300 MHz):