

Flavonol Glycosides of *Reseda arabica* (Resedaceae)

Djemaa Berrehal¹, Assia Khalfallah¹, Ahmed Kabouche¹, Anastasia Karioti²,
Anna Rita Bilia², Ahmet C. Goren³ and Zahia Kabouche^{1*}

¹Laboratoire d'Obtention de Substances Thérapeutiques (L.O.S.T), Faculté des sciences, Université Mentouri - Constantine, Campus Chaabat Ersas, 25000 Constantine, Algérie.

²Department of Pharmaceutical Sciences, via Ugo Schiff 6, Sesto Fiorentino, 50019 Florence, Italy.

³TUBITAK, Ulusal Metrology Institute, Chemistry Group, Organic Chemistry Laboratory, PO Box 54, 41470 Gebze, Kocaeli, Türkiye

(Received October 14, 2011; Revised May 6, 2012; Accepted June 15, 2012)

Abstract: Five flavonol glycosides, kaempferol 3,7-di-O- α -L-rhamnopyranoside (**1**), isorhamnetin 3,7-di-O- α -L-rhamnopyranoside (**2**), kaempferol 3-O- β -D-glucopyranoside-7-O- α -L-rhamnopyranoside (**3**), isorhamnetin 3-O- β -D-glucopyranoside-7-O- α -L-rhamnopyranoside (**4**), Kaempferol 3-O- β -xylopyranosyl-(1" \rightarrow 2")-O- α -L-rhamnopyranoside-7-O- α -L-rhamnopyranoside (**5**), have been isolated from the aerial parts of *Reseda arabica*. Their structures were established on the basis of physical and spectroscopic analysis, and by comparison with the literature data.

Keywords: *Reseda Arabica*; Resedaceae; flavonoid glycosides.

1. Plant Source

The Resedaceae family, are represented by six genus which are *Reseda*, *Caylusea*, *Oligomeris*, *Astrocarpus* and *Randonia*, and distributed in the Algerian Sahara. The genus *Reseda* is found in the Mediterranean and the South Western Asian areas. There are twenty two species and subspecies in flora of Algeria and the species *R. villosa*, *R. duriaeana* and *R. arabica* are endemic [1,2]. *R. arabica* was collected from Ghardaia (Septentrional Algerian Sahara) in March 2007. The plant was identified by Prof. Gerard De Belair (University Badji-Mokhtar, Annaba, Algeria). A voucher specimen was deposited in the Herbarium of the faculty of sciences, University Mentouri-Constantine (LOST Rar/03L07).

2. Previous Studies

Reseda species have been reported to possess various pharmacological effects such as anti-inflammatory [3-5], antioxidant [6], antibacterial [7] and antimicrobial [8]. The extracts of *Reseda muricata* are being used in external treatments of haemorrhoids and for stomach aches and diarrhea in traditional medicine [9, 10]. There is no chemical report on *R. arabica* in the literature.

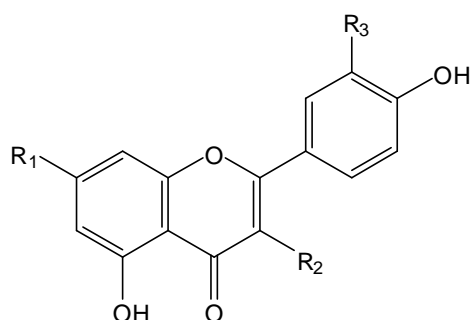
* Corresponding author : E-Mail : zkabouche@yahoo.com

3. Present Study

The air dried and powdered aerial parts of *R. arabica* (1700 g) were macerated in a methanolic solution (70%). The extract was successively concentrated to dryness (under low pressure); the residue was dissolved in boiling water and extracted with ethyl acetate and n-butanol, successively. The butanolic extract (20 g) was fractionated on polyamide SC6 column and eluted with toluene and gradients of methanol with increasing polarity to give ten fractions (F1-F10). Fraction F8 (0.2 g) and F9 (0.18 g) were further TLC chromatographed on silica gel eluted with EtOAc-MeOH-H₂O (10:1:1) to obtain compounds **1** (27 mg) and **3** (20 mg). Compounds **2** (25 mg) and **5** (40 mg) were obtained from fractions F10 (3g) and F11(2.4g) which were combined and subjected to silica gel column chromatography using EtOAc-MeOH-H₂O (10:1:0.5) as eluant. Compound **4** (30 mg) was obtained as a yellow precipitate from fraction F12 (3.4g).

For the acid hydrolysis, each compound (~2 mg) was refluxed with 4N HCl (2 mL) in water for 1 h. The reaction mixture was fractionated by ethyl acetate. Each ethyl acetate soluble fraction was concentrated and examined by TLC with authentic samples. The remaining aqueous layers were examined by TLC with authentic sugars.

Their structures (**1-5**) (Figure 1) were elucidated as kaempferol 3,7-di-O- α -L-rhamnopyranoside (**1**), isorhamnetin 3,7-di-O- α -L-rhamnopyranoside (**2**), kaempferol 3-O- β -D-glucopyranoside-7-O- α -L-rhamnopyranoside (**3**), isorhamnetin 3-O- β -D-glucopyranoside-7-O- α -L-rhamnopyranoside (**4**), Kaempferol 3-O- β -xylopyranosyl-(1" \rightarrow 2")-O- α -L-rhamnopyranoside-7-O- α -L-rhamnopyranoside (**5**) by the physico-chemical techniques (R_f , fluorescence), UV spectroscopy and acid hydrolysis and 1D and 2D NMR techniques (¹H-NMR and ¹³C-NMR, DEPT, COSY, HMQC, ROESY and HMBC) [11,12] (see support information).



Compound	R1	R2	R3
1	O-Rha	O-Rha	H
2	O-Rha	O-Rha	OMe
3	O-Rha	O-Glu	H
4	O-Rha	O-Glu	OMe
5	O-Rha	O-Xyl-O-rha	H

Figure 1. Isolated compounds of *R. arabica*

Compounds **2** and **5** are isolated for the first time from the genus *Reseda*. Compound **1** was found only in the Algerian species *R. villosa* [14] while compound **3** was isolated from three species namely *R. villosa* [14], *R. durieana* [6] and *R. luteola* [15]. Compound **4** was also reported from three *Reseda* species, *R. villosa* [14], *R. lutea* [16] and *R. odorata* [17].

From the present study and previous reports, it appears that, except for the single trioside **5**, bearing a xylose moiety, only two sugars, glucose and rhamnose, are found in flavonol glycosides of *Reseda* which are distributed through 4 monoglycosides and 7 diglycosides.

Acknowledgments

We are grateful to ANDRS (Algeria) and MESRS-DG/RSDT (Algeria) for financial support.

Supporting Information

Supporting Information accompanies this paper on <http://www.acgpubs.org/RNP>

References

- [1] P. Quezel and S. Santa (1963). Nouvelle Flore de l'Algérie et des Régions Désertiques Méridionales. C.N.R.S Paris, **2**, pp. 815-816.
- [2] P. Ozenda (1958) Flore du Sahara septentrional et central. Ed. C.N.R.S. Paris pp :274-276.
- [3] C. Susplugas, C. Taillade, P. Susplugas and F. Michel (1988). Anti-inflammatory activity of an ether extract of *Reseda phyteuma* L., *Pharm. Acta Helv.* **63**, 59-63.
- [4] P. Susplugas, I. J. Mongold, C. Taillade and J. J. Serrano (1993). Anti-inflammatory and analgesic activity of *Reseda phyteuma*, *Plant. Med. et Phytother.* **26**, 375-82.
- [5] J. P. Masse (1986). Anti-inflammatory plant extract, FR. Patent 2581310 AI. 11.
- [6] D. Berrehal, A. Khalfallah, S. Bencharif-Betina, Z. Kabouche, N. Kacem, A. Kabouche, J-C. Calliste and J-L. Duroux (2010). Comparative antioxidant activity of two Algerian *Reseda* species, *Chem. Nat. Compds.* **4**, 456-458.
- [7] Y. Kumarasamy, P. J. Cox, M. Jaspars, M. L. Nahar and S. D. Sarker (2002). Screening seeds of Scottish plants for antibacterial activity, *J. Ethnopharmacol.* **83**, 73-7.
- [8] J. M. Watt and M. G. Breyer (1962). The Medicinal and Poisonous Plants of Southern and Eastern Africa, 2nd Edition. Livingstone, E.S., Edinburgh and London.
- [9] L. Boulos (1983). *Medicinal Plants of North Africa*. Reference Publications, Algonac, MI
- [10] N. H. El-Sayed, N. M. Omara, A. K. Yousef, A. M. Farag and T. J. Mabry (2001). Kaempferol triosides from *Reseda muricata*, *Phytochemistry.* **57**, 575-578.
- [11] K. R. Markham (1982). Techniques of flavonoid identification. Academic Press London.
- [12] K. R. Markham and H. Geiger (1993). ¹H nuclear magnetic resonance spectroscopy of flavonoids and their glycosides in hexadeuterodimethylsulfoxide. In the flavonoids: Advances in research since 1986. Harborne J.B. Ed. Chapman & Hall. London. pp 441.
- [13] D. Berrehal, A. Khalfallah, A. Kabouche, Z. Kabouche, A. Karioti and A.R. Bilia (2010). Flavonoid glycosides from *Randonia africana* Coss. (Resedaceae). *Biochem. Syst. Ecol.*, **38**, 1007-1009.
- [14] D. Berrehal, A. Kabouche, Z. Kabouche and C. Bruneau (2006). Flavonoid glycosides from *Reseda villosa* (Resedaceae), *Biochem. Syst. & Ecol.* **34**, 777-779.
- [15] M. P. Yuldashev, E. Kh. Batirov, V. M. Malikov and N. P. Yuldasheva (1996). Flavonoids of *Psoralea drupaceae* and *Reseda luteola*, *Khim. Prir. Soedin.* **6**, 949-951.
- [16] H. Rzadkowska-Bodalska (1969). Flavonoids in flowers of weld (*Reseda lutea*) III. Identification of compound, *C. Pharm. Pharmacol.* **21**, 169-172.
- [17] V. Plouvier (1966). Flavone heterosides: kaempferol 3-rhamnoglucoside, myricitrin, linarin and saponarin, *Compt. Rend., Ser D.* **262**, 1368-1371.

A C G
publications

© 2012 Reproduction is free for scientific studies