Available online at www.scholarsresearchlibrary.com

Scholars Research Library

Der Pharmacia Lettre, 2015, 7 (12):183-186
(http://scholarsresearchlibrary.com/archive.html)

Der Pharmacia Lettre
Scholars Research Library
ISSN 0975-5071
USA CODEN: DPLEB4

Lipids and sterol from *Berberis vulgaris* L. var. asperma

Consolacion Y. Ragasa[1,2*], Virgilio D. Ebajo Jr.[1], Maria Carmen S. Tan[1], Glenn Oyong[3,4], Robert Brkljača[5] and Sylvia Urban[5]

[1]Chemistry Department, De La Salle University, Taft Avenue, Manila, Philippines [2]Chemistry Department, De La Salle University Science & Technology Complex Leandro V. Locsin Campus, Biñan City, Laguna, Philippines
[3]Center for Natural Science and Ecological Research, De La Salle University, Taft Avenue, Manila, Philippines
[4]Biology Department, De La Salle University, Taft Avenue, Manila, Philippines
[5]School of Applied Sciences (Discipline of Chemistry), Health Innovations Research Institute (HIRi) RMIT University, Melbourne, Victoria, Australia

---

ABSTRACT

*Chemical investigation of the dichloromethane extract of the fruit of Berberis vulgaris L. var. asperma affordedmixtures of saturated carboxylic acid, (2Z)-3-(4-hydroxyphenyl)-2-propen-1-yl esters (**1**), fatty acid esters (**2**), fatty alcohols (**3**), triacyl glycerols (**4**), monoacyl glycerols (**5**), fatty acids (**6**); and β-sitosterol (**7**). The structures of **1**and**2** were elucidated by extensive 1D and 2D NMR spectroscopy, while those of **3-7** were identified by comparison of their NMR data with literature data.*

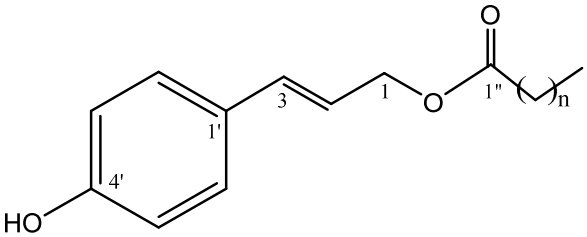
Keywords: *Berberis vulgaris*, Berberidaceae, carboxylic acid, (2*Z*)-3-(4-hydroxyphenyl)-2-propen-1-yl esters, fatty acid esters, fatty alcohols, triacyl glycerols, monoacyl glycerols, fatty acids, β-sitosterol

---

INTRODUCTION

Berberis vulgaris also known as barberry is a shrub which produces edible berries. *B. vulgaris* fruits have been used as tea, jelly or syrup for treatment of the respiratory tract infection, fever, cold, and flu [1].The root, leaf, bark, and fruit have been used as folk medicine for the treatment and prevention of cardiovascular, gastrointestinal, respiratory, skin, renal, and infectious diseases [2]. Earlier studies reported the isolation ofquaternary protoberberine and bisbenzylisoquinolinealkaloids from the root bark of *Berberis vulgaris*subsp. australis [3].Another study reported the presence phenolics and anthocyanins from *B. vulgaris* which exhibited antioxidant properties [4].Recently, a newisoquinoline-isoquinolone alkaloid was isolated from the roots of *B. vulgaris* [5]. Berberine, an isoquinoline alkaloid isolated from *B. vulgaris* exhibited anti-diabetes [6-8], anticancer [9], anti-inflammatoty [10], and antiatherosclerosis [11].

In this study, mixtures of saturated carboxylic acid, (2*Z*)-3-(4-hydroxyphenyl)-2-propen-1-yl esters (**1**), fatty acid esters (**2**), fatty alcohols (**3**), triacyl glycerols (**4**), monoacyl glycerols (**5**), fatty acids (**6**), and β-sitosterol (**7**)(Fig. 1) were obtained from *Berberis vulgaris* L. var. asperma. To the best of our knowledge, this is the first report on the isolation of **1** from *B. vulgaris.*



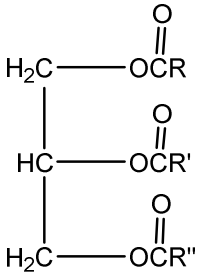
1

2 RCOOR'

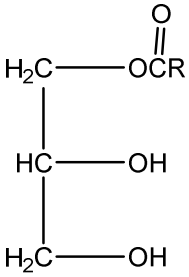
3 RCH_2OH

6 RCOOH

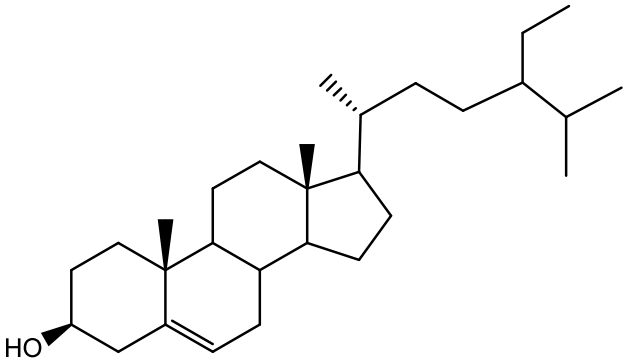
2 R, R' = long chain fatty acids
3, **6** R = long chain fatty acids



4 R, R', R" = long chain fatty acids



5 R = long chain fatty acids



7

Fig. 1. Chemical constituents of *B. vulgaris*:saturated carboxylic acid, (2*Z*)-3-(4-hydroxyphenyl)-2-propen-1-yl esters (1), fatty acid esters (2), fatty alcohols (3), triacyl glycerols (4), monoacyl glycerols (5), fatty acids (6), and β-sitosterol (7)

MATERIALS AND METHODS

General Experimental Procedures

^1H (300 MHz) and ^{13}C (75 MHz) NMR spectra were acquired in $CDCl_3$ on a Bruker Ultrashield 300 MHz magnet equipped with a Bruker Avance III 300 consolewith referencing to solvent signals (δ 7.26 and 77.0 ppm). Column chromatography was performedwith silica gel 60 (70-230 mesh). Thin layer chromatography was performed with plastic backed plates coated with silica gel F_{254} and the plates were visualizedby spraying with vanillin/H_2SO_4 solution followed by warming.

General Isolation Procedure
A glass column 18 inches in height and 1.0 inch internal diameter was used for the fractionation of the crude extracts. Ten milliliter fractions were collected. Fractions with spots of the same Rf values were combined and rechromatographed in appropriate solvent systems until TLC pure isolates were obtained. A glass column 12 inches in height and 0.5 inch internal diameter was used for the rechromatography. Five milliliter fractions were collected. Final purifications were conducted using Pasteur pipettes as columns. One milliliter fractions were collected.

Sample Collection
The fruits of *Berberis vulgaris* L. var. asperma were harvested from Mashhad, Khorasan Province, Iran and identified at the Mashhad Medical University of Sciences, Iran.

Isolation
Freeze-dried *B. vulgaris* fruits wereground in a blender, soaked in $CHCl_3$ for three days and then filtered. The filtrate was concentrated under vacuum to afford a crude extract (2.3 g) which was chromatographed by gradient elution using increasing proportions of acetone in CH_2Cl_2(10% increments) as eluents.The CH_2Cl_2 fraction wasrechromatographed (3 ×) using 2.5% EtOAc in petroleum ether to afford **2** (9 mg) after washing with petroleum ether. The 10% acetone in CH_2Cl_2 fraction was rechromatographed (2 ×) using 10% EtOAc in petroleum ether to yield **4** (12 mg). The 20% acetone in CH_2Cl_2 fraction was rechromatographed (3 ×) using10% EtOAc in petroleum ether yield **1** (8 mg) after washing with petroleum ether. The 30% acetone in CH_2Cl_2 fraction was rechromatographed using 20% EtOAc in petroleum ether. The less polar fractions were combined and rechromatographed (2 ×) using 20% EtOAc in petroleum ether yield **3**(10 mg) after washing with petroleum ether. The more polar fractions were combined and rechromatographed (3 ×) usingCH_3CN:Et_2O:CH_2Cl_2 (1:1:8 by volume ratio) to afford **7** (5 mg). The 40% acetone in CH_2Cl_2 fraction was rechromatographed using CH_3CN:Et_2O:CH_2Cl_2 (1:1:8 by volume ratio). The less polar fractions were combined and rechromatographed (2 ×) using CH_3CN:Et_2O:CH_2Cl_2 (1:1:8 by volume ratio) to yield **6** (9 mg) after washing with petroleum ether. The more polar fractions were combined and rechromatographed (3 ×) using CH_3CN:Et_2O:CH_2Cl_2 (2:2:6 by volume ratio) to yield **5 (**4 mg).

Carboxylic acid, (2*Z*)-3-(4-hydroxyphenyl)-2-propen-1-yl esters (**1**):^{1}HNMR ($CDCl_3$, 500 MHz): δ 4.70 (dd, *J* = 0.6, 3.9 Hz, H_2-1), 6.13 (m, H), 6.60 (d, *J* = 9.3 Hz, H-3),6.85(d, *J* = 5.1 Hz, H-2', H-6'), 7.28(d, *J* = 5.1 Hz, H-3', H-5'), 2.33 (t, *J* = 4.5 Hz, H_2-2"), 1.65 (H_2-3"), 1.25-1.36 $(CH_2)_n$, 0.89 (t, *J* = 4.2 Hz , CH_3"); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 65.2 (C-1), 120.6 (C-2), 133.9 (C-3), 128.5 (C-1'), 115.5 (C-2', C-6'), 127.8(C-3', C-5'), 155.2 (C-4'), 173.5 (C-1"), 34.2 (C-2"), 24.5 (C-3"), 29.2-29.7 $(CH_2)_n$, 14.0 (CH_3").

RESULTS AND DISCUSSION

Silica gel chromatography of the dichloromethane extract of the fruit of *B. vulgaris* yielded mixtures of saturated carboxylic acid, (2*Z*)-3-(4-hydroxyphenyl)-2-propen-1-yl esters (**1**), fatty acid esters (**2**), fatty alcohols (**3**) [12], triacyl glycerols (**4**) [13], monoacyl glycerols (**5**) [14], fatty acids (**6**) [15]; and β-sitosterol (**7**) [16]. The structures of **1**and **2**were elucidated by extensive 1D and 2D NMR spectroscopy, while those of **3**-**7** were identified by comparison of their NMR data with literature data.

Acknowledgement
A research grant from the De La Salle University Science Foundation through the University Research Coordination Office is gratefully acknowledged.

REFERENCES

[1] S. Vogl, P. Picker, J. Mihaly-Bison, N. Fakhrudin, A. G. Atanasov, E. H. Heiss, C. Wawrosch, G. Reznicek, V. M. Dirsch, J. Saukel, B. Kopp, *J. Ethnopharmacol.*, **2013**,149(3), 750–71.
[2] M. Imanshahidi, H. Hosseinzadeh, *Phytother. Res*., **2008**, 22(8), 999–1012.
[3]R. Suau, R. Rico, J. M. L. Pez-Romero, F. N. Jera, A. Cuevas. *Phytochem*., **1998**,49(8), 2545–2549.
[4]M. Özgen, O. Saraçoğlu, E. N. Geçer**.** *Res. Rep. Cult. Physiol., Hort. Environ. Biotechnol*.,**2012**,53(6), 447-451.
[5] A. Host'álková, Z. Novák, M. Pour, A. Jirosová, L. Opletal, J. Kunes, L. Cahliková,*Nat. Prod. Commun*., **2013**,8(4), 441–442.
[6] H. Zhang, J. Wei, R. Xue R, *Metabolism*. **2010**, 59(2), 285-292.

[7] Y. Zhang, X. Li, D. Zou D, *J Clin Endocrinol Metab.* **2008**, 93(7), 2559-2565.
[8] J. Yin, H. Xing, J. Ye, *Metabolism*, **2008**, 57(5), 712-717.
[9] H. S. Kim, M. J. Kim, E. J. Kim, Y. Yang, M. S. Lee, J. S. Lim, *Biochem Pharmacol.* **2012**, 83(3), 385-394.
[10] H. W. Jeong, K. C. Hsu, J. W. Lee, *Am J Physiol Endocrinol Metab*. **2009**, 296(4), E955-964.
[11] S. Guan, B. Wang, W. Li, J. Guan, X. *Am J Chin Med*. **2010**, 38(6), 1161-1169.
[12] C. Y. Ragasa, V. A. S. Ng, M. M. De Los Reyes, E. H. Mandia, G. G. Oyong, C.-C. Shen, *Der Pharma Chemica.***2014**, 6(5), 182-187.
[13] V. A. S. Ng, E. M. G. Agoo, C.-C. Shen, C. Y. Ragasa, *J. Appl. Pharm. Sci*., 2015, 5(Suppl 1), 12–17.
[14] C. Y. Ragasa, G. S. Lorena, E. H. Mandia, D. D. Raga, C.-C. Shen, *Amer. J. Essent. Oils Nat. Prod.*, **2013**, 1(2), 7-10.
[15] C. Y. Ragasa, M. P. Medecilo, C.-C. Shen,*Der Pharma Chemica.* **2015**, 7(7), 395-399.
[16] C. Y. Ragasa, V. A. S. Ng, M. M. De Los Reyes, E. H. Mandia, G. G. Oyong, C.-C. Shen, *Der Pharma Chemica.* **2014**, 6(5), 182-187.