Vol. 12 (2009) 23-25

Studies on *Viburnum nervosum* Hook: Chemistry and Spectroscopy of Bergenin and its Derivatives

B.K. TIWARI* AND R.L. KHOSA

Department of Pharmacy, Bharat Institute of Technology, Meerut 250103, India.

This paper reports on the isolation of bergenin from *Viburnum nervosum* (Cappriifoliaceae). This is the first time the compound has been isolated from the genus *Viburnum*. Derivatives of bergenin namely bergenin diethyl ether, bergenin pentaacetate and bergenin diethyl ether triacetate were prepared and characterized using spectroscopic data.

Key words: Bergenin, bergenin derivatives, *Viburnum nervosum*

INTRODUCTION

Viburnum nervosum Hook (Cappriifoliaceae), known in the Hindi language as ósHirporna-jayaô, is used as an astringent and emmenagogue in India [1]. It yields an essential oil with potential perfumery applications. In Kashmir, its roots are used in the treatment of acute furunculosis [2]. Several known triterpenes, alcohols and flavones have been isolated from the plant [4]. However, this is the first report of bergenin (**I**) from the roots of *V. nervosum* [2]. Bergenin is known to occur in the genus *Bergenia* (Saxifragaceae). It has been found useful in the treatment of hypercholestrolaemia, kidney stones, fever, diarrhoea, pulmonary infection, as an antioxidant and anticancer agent [3-4]. This paper describes the isolation of bergenin and preparation of some derivatives.

MATERIALS AND METHODS

Plant material

The roots of *Viburnum nervosum* were collected from the hilly area of Srinagar, Kashmir, India and authenticated by S. Saraf (Department of Botany, Kashmir University). A voucher specimen (RLK/VN/77) was deposited in the Department of Pharmaceutics, Banaras Hindu University, Varanasi.

Extraction, isolation and preparation of derivatives

The roots were air-dried, milled to moderately coarse powder and defatted with petroleum ether (60°-80° C) using a Soxhlet extractor. The material was subjected to Soxhlet extraction with rectified spirit. The alcoholic extract was dried and chromatographed over silica gel, eluting the column with a $CHCl_3$-MeOH (5:1) solvent system. Bergenin (**I**) was isolated as a homogeneous white solid which crystallized as colorless rhombohedrons from methanol. Thin layer chromatography confirmed its phenolic nature The compound stained bluish-green after spraying with ferric chloride and blue on exposure to ammonia vapors after spraying with phosphomolybdic acid.

***Bergenin diethyl ether* (II):** Compound **I** (20 mg) was dissolved in 50 ml of dry acetone containing 5 g of anhydrous K_2CO_3 and refluxed with 3 ml of ethyl iodide for 12 h. The mixture was cooled, filtered and the acetone evaporated from the filtrate. The residue crystallized from methanol as grey needles.

***Bergenin pentaacetate (III)*:** A mixture of 10 mg of compound **I**, 3 ml of acetic anhydride and 1 ml of triethylamine was kept at room temperature overnight and then poured into crushed ice till the decomposition of acetic anhydride was complete. The product was extracted into chloroform. The chloroform extract was sequentially washed with aqueous $NAHCO_3$ and distilled water, dried over anhydrous Na_2SO_4 and evaporated to dryness. The resultant residue crystallized from methanol as shiny yellow needles.

Bergenin diethyl ether triacetate (IV): The acetylation and extraction procedure used to

*Author to whom correspondence may be addressed.

obtain compound **III** was carried out on compound **II**. The product crystallized as white flakes from methanol.

Physical properties and spectral data

Bergenin (I): Colorless rhombohedrons from methanol. M.p. 144 °C; soluble in water, alcohol, pyridine; insoluble in apolar organic solvents; $[\breve{U}]_D^{26}$ -47 ° (H_2O); MS, *m/z*: 328 (M^+), 208 (base peak); elemental composition, (%): C (51.53), H (4.47), molecular formula $C_{14}H_{16}O_9$; UV $\breve{o}_{max}$ (methanol, nm): 219, 275; IR (KBr) cm^{-1}: 3450,3270 (br, OH), 2894, 2850 (CH_2), 1720 (Ŭ,ß-unsaturatedŭ-lactone), 1610, 1530 (aromatic C-O), 1345, 1235 (aromatic C-O), 858 (C-H); ^{1}H-NMR (pyridine-d_5), ŭ: 3.98 (3H, s, Ar-OCH_3), 4.50-5.81 (complex m, CH, CH_2 and OH), 7.52 (1H, s, isolated Ar-H).

Bergenin diethyl ether (II): Grey needles from methanol. M.p. 176 °C; IR (KBr, cm^{-1}): 3200-3300 (br, OH), 1725 (Ŭ,ß unsaturated ŭ-lactone); ^{1}H-NMR (pyridine-d_5), ŭ: 3.98 (3H, s, Ar-OCH_3) 1.42, 1.50 (2x3H, t, each) 4.30 (2x2H, q, overlapped, for two ethoxy groups), 4.55-5.75 (complex m, CH, CH_2 and OH), 7.52 (1H, s, isolated Ar-H).

***Bergenin pentaacetate (III)*:** Shiny **y**ellow needles from methanol. M.p. 134-135 °C; IR (KBr) cm^{-1}: 1720 (Ŭ,ß-unsaturated-ŭ-lactone, 1740 (br, ester carbonyl); ^{1}H-NMR ($CDCl_3$) ŭ: 2.08, 2.12, 2.14 (3H, s each) and 2.15 (6H, s) for five acetoxy methyl protons, 3.98 (3H, s, Ar-OCH_3), 7.52 (1H, s, isolated Ar-H).

Bergenin diethyl ether triacetate (IV): White flakes from methanol. M.p. 170 °C; MS, *m/z*: 510 (M^+), 264; elemental composition, (%): C (55.76), H (5.62), molecular formula C_{24} H_{30} O_{12}; IR (KBr) cm^{-1}: 1720 (Ŭ,ß-unsaturated carbonyl), 1740 (br, ester carbonyl); ^{1}H-NMR (pyridine-d_5) ŭ: 2.10, 2.12, 2.13 (3H, s, each for the acetoxy methyl), 4.00 (3H, s, Ar-OCH_3), 4.50-5.82(complex m, CH and CH_2), 7.53 (1H, s, isolated Ar-H).

ACKNOWLEDGEMENT

The authors wish to thank Professor V.B Pandey of the Department of Medicinal Chemistry, Banaras Hindu University, Varanasi, for recording out the mass and nuclear magnetic resonance spectra.

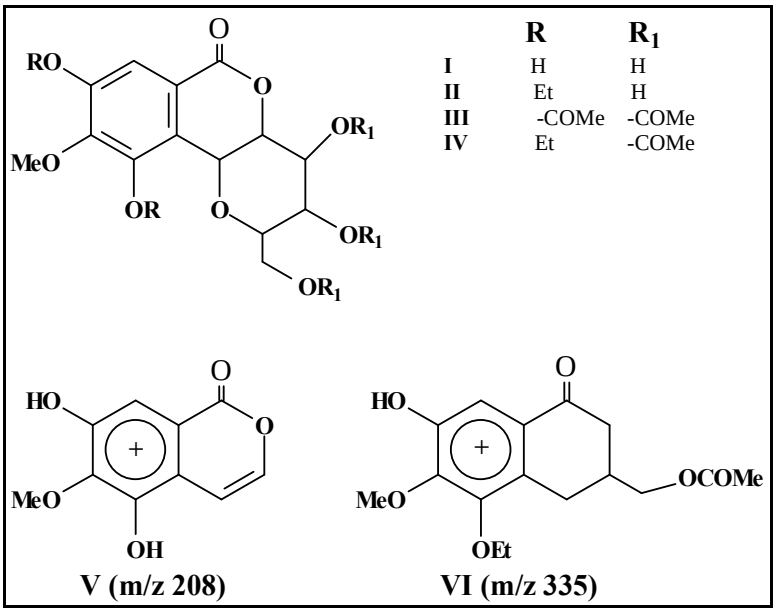

Figure 1: Chemical structures I-VI.

REFERENCES

[1] S.P. Ambasta (Ed.), Wealth of India, Vol II (B) (Revised edition), CSIR, New Delhi. 1988, pp 116-120.

[2] R.L. Khosa, A.K. Wahi, Y. Mohan and A.B. Ray, Indian J. Pharm. Sci. (1978) 67-69.

[3] H. Takahasi, M. Kosaka, Y. Watanabe, K. Nakade and Y. Fukuyama, Bioorg. Med. Chem. 11 (2003) 1781-1788.

[4] Z. Chen, M.Y. Lin, S. Yang, A.B. Song, F.G. Xu and X. Zhou, Bioorg. Med. Chem. 16 (2008) 1337-1344.