Available on line www.jocpr.com

Journal of Chemical and Pharmaceutical Research

ISSN No: 0975-7384
CODEN(USA): JCPRC5

J. Chem. Pharm. Res., 2011, 3(2):762-764

Isolation and characterization of flavanone compounds from the leaf extract of *Polygonum barbatum*

Queen Rosary Sheela X[1*], Arockiasamy P[2], Kanmani R[1], Charles A[1]and Alex Ramani V[1]

[1]*PG & Research Department of Chemistry, St. Joseph's College, Trichy, Tamil Nadu, India*
[2]*PG & Research Department of Chemistry, Auxilium College, Vellore, Tamil Nadu, India*

ABSTRACT

The objective of the present study was to isolate and characterize phytoconstituents from the ethanol extract of Polygonum barbatum leaves. Phytochemical investigation of the leaf extract of Polygonum barbatum yields a two flavanone. The structures of these compounds were determined on the basis of spectral and chemical studies. This is the first report of the presence of flavonone in the leaves of Polygonum barbatum.

Key Words: *Polygonum barbatum*, Flavanone, Extraction and Isolation, Spectroscopy.

INTRODUCTION

Polygonum barbatum (Family: Polygonaceae) which was distributed throughtout the hotter parts of India, particularly in wet places. The plant was used as medicine to relieve the gribring pains of colic and in the treatment of ulcers [1]. The dichloromethane extract of *Polygonum barbatum* shown to have brine shrimp toxicity and spasmolypic activity .The methanolic extract of plant possess cholinergic activity [2]. The antinociceptive, anti-inflammatory and diuretic properties were also studied [3]. No history of compound isolation has been reported in the literature.

Flavonoids and derivatives are among the most ubiquitous of polyphenolic natural products, occurring widely within the plant kingdom [4]. The present study deals with isolation and characterization of flavanones from the leaves of *Polygonum barbatum*.

EXPERIMENTAL SECTION

Plant material

The leaves of *Polygonum barbatum* were collected along the beds of Cauvery river, near Trichy, Tamil Nadu in February 2008. The plant was identified and authenticated by Dr.G.V.S.Moorthy, Joint Director, Botanical Survey of India (BIS), Agriculture University campus, Coimbatore, India. The voucher specimen number was BIS/SC/5/23/08-09/Tech-1614, and the specimen was deposited at herbarium.

Extraction and isolation

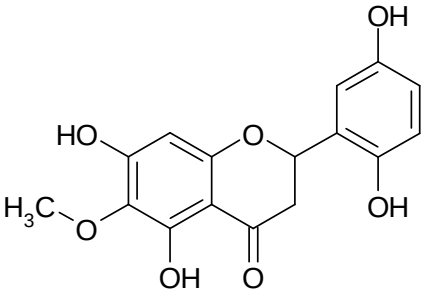
The leaf of *Polygonum barbatum* (1kg) were ground into powder and then extracted with ethanol (5 l x 3) at room temperature. The yield of alcoholic extract was found to be 100 g, and then partitioned in turn with chloroform (500 ml x 3) and ethyl acetate (600 ml x 3), to afford chloroform soluble (50 g), ethyl acetate soluble (35 g). The chloroform soluble part of the crude ethanolic extract was chromatographed on silica gel (Merck 230-400 mesh) using the solvents with increasing polarity. The compound **1** was eluted at 10% ethyl acetate in chloroform. The compound obtained was purified and re-crystallized in methanol. The ethyl acetate soluble extract was subjected to silica gel coloumn chromatography (Merck 230-400 mesh). The fraction eluted at $CHCl_3$: EtOH (25:1) was further subjected to coloum chromatography using petroleum ether: acetone (3:1) and purified over Sephadex LH-20 coloumn using methanol to afford compound **2**. The structure of the isolated compound was established on the basis of spectroscopic evidences (IR, UV, ^{1}HNMR, ^{13}CNMR, MS).

RESULTS AND DISCUSSION

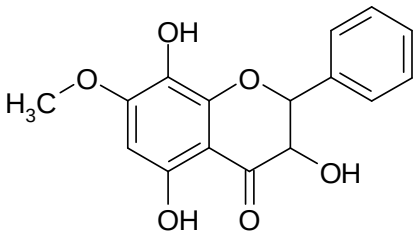
Compound **1** was identified as 5, 7, 2', 5'-tetrahydroxy-6-methoxyflavanone. A molecular formula of $C_{16}H_{14}O_7$ [(M+ at m/z 318, mp 120-122 °C)] was isolated as colorless crystals. It gave the absorption bands of hydroxyl and carbonyl group in the infrared spectrum (IR). The IR spectrum indicated the presence of carbonyl (C=O) group and hydroxyl group at 1652 cm^{-1} and 3300 cm^{-1} respectively. The OH substitution pattern in the A-ring was established by UV analysis. The UV spectrum of compound **1** was characteristic of the 5, 7 dihydroxy flavanone series. Compound **1** in MeOH gave a major absorption band at 294 nm with a shoulder at 342 nm [5]. 5, 7 dihydroxy flavanone was indicated by ^{13}C NMR signals for C-2 and C-3 at 74.5 and 41.7 ppm respectively and a low field signal at 197.3 ppm for the C=O (C-4)[6]. The dihydro flavanol skeleton of compound **1** was easily distinguish from the ^{1}H spectrum by the characteristic set of doublet of doublet at 5.48 (12.0, 4.5 Hz) for H-2, at 2.88 for H-3. In the ^{1}H nmr spectrum showed the signals of one methoxyl (3.61), hydroxyl (9.02, 11.75). The EI-MS of compound **1** showed in addition to molecular ion at 318, significant fragments at m/z: 300, 285, 264, 257, 236, 222, 213, 183, 167, 152, 136, 129, 111, 97, 83, 69 and 55. The mass spectrum exhibited the fragment ion peak originating from the B ring at m/z 136 ($C_8H_8O_2$) and A ring at m/z 183 (C_8H_7O5)

Compound **2** was obtained as pale yellow powder was identified as 7-methoxy-3, 5, 8-trihydroxyflavanone. A molecular formula of $C_{16}H_{14}O_6$ [($M+H^+$) peak at m/z 303.0869, mp 2226-228 °C] was assigned for the compound. The ^{1}H NMR spectrum of compound **2** displayed signals of two oxygenated methine doublets at 5.17 (1H, d, J=10.4 Hz) for H2 and 4.62 (1H, d,

J=7.2 Hz) for H3, a methoxy group at 3.82 (3H, S), a hydroxyl group at 11.59 (1H, S). The 13C NMR spectrum of compound **2**, also displayed two oxygenated methines at 82.9 (C-2) and 71.8 (C-3), a methoxy group at 56.1 (OMe), a conjugated ketone at 198.3, the signals of two aromatic rings 137.3 and 127.0 respectively. Accordingly, this suggested that compound **2** is a flavanone with an unsubstitued B ring [7]. EI-Mass spectra of this compound suggested that its characteristic fragments observed at m/z: 302, 285, 183, 156, 139, 136, 120, 111, 91 and 69. The UV-VIS absorption bands at λ_{max} 290, and 321 nm. The UV data was supporting the presence of a flavanone. In the IR spectrum absorption due to a chelated carbonyl appered at 1640 cm^{-1} and a hydroxyl appeared at 3327 cm^{-1}.



Compound 1



Compound 2

Flavanones were isolated and characterized from chloroform and ethyl acetate extracts of *Polygonum barabtum* leaves. Therefore, it may be concluded that *Polygonum barabtum* leaves have more biological activities due to its flavonone.

Acknowledgements
The authors thank Indian Institute of Science, Bangalore for providing spectral facilities to carry out this work.

REFERENCES

[1]. AK Nadkarni. Indian Material Medica, Bombay Popular Prakashan, Bombay, India, **1991**; 999.
[2]. Bashir Ahmad Chaudhry; Muhammad Younas Syad; Khalid Hussain Janbaz; Altaf Ahmed Dasti; Bashir Ahmad Loothar. *Journal of Research (Science).*, **2003**, 14,169-175.
[3]. M Abdul Mazid; Bidyut K. Datta, S Lutfun Nahar; AM Khairul Bashar; Sitesh C. Bachar; Satyajit D. Sarker. *Brazilian Journal of Pharmacognosy.*, **2009**, 19, 749-754.
[4]. JB Harborne; H Baxter. The Handbook of Natural Flavonoids, Wiley and sons, Chichester, United Kingdom, **1991**.
[5] JB Harborne; TJ Mabry; H Mabry. The flavonoids, Chapman and Hall, London, **1975;** 78-126.
[6]. PK Agrawal. In C-13 NMR of flavanoids, Elsevier, Amsterdam, **1998;** 41-182.
[7]. M Kuroyanagi; Y Yamamoto; S Fukushima; A Ueno; T Noro; T Miyase. *Chem. Pharm. Bull.*, **1982**, 30, 1602–1608.