

(+)-AFZELECHIN FROM THE RHIZOME OF *CALAMUS ROTANG* LINN.

ABSTRACT

Phytochemical analysis of the rhizome of *Calamus rotang* Linn. afforded β -sitosterol, β -sitosterol 3 β -D-glucopyranoside and (+)-afzelechin. Characterization of (+)-afzelechin is reported for the first time from this plant.

Keywords: *Calamus rotang*, Flavonoid, (+)-Afzelechin

INTRODUCTION

Vetra/ (Tamil: Pirambu) is botanically equated as *Calamus rotang* Linn. Fam. *Arecaceae*. A thorny slender climbing shrub occurring in central and southern parts of India, it is also cultivated in gardens. It bears contorted pieces of horizontally growing, woody rhizome, 4 to 5 cm in length, 2 to 3 cm in width, surface is very rough, longitudinally striated, encircled by prominent, closely arranged leafscars, and well developed tortuous roots, at the lower side; roots are cylindrical, branched, smooth or faintly longitudinally striated, exfoliated, externally dirty earthy brown in colour, internally, pinkish greyish, fracture is hard, fibrous, odour not characteristic, taste, astringent and slightly sour. The rhizomes of the plant are used in various ailments *viz.* piles, burning sensation, cough, leprosy, bleeding disorder and inflammation¹. In the present study characterization of (+)-afzelechin is reported for the first time from the rhizome of *C. rotang* Linn.

EXPERIMENTAL

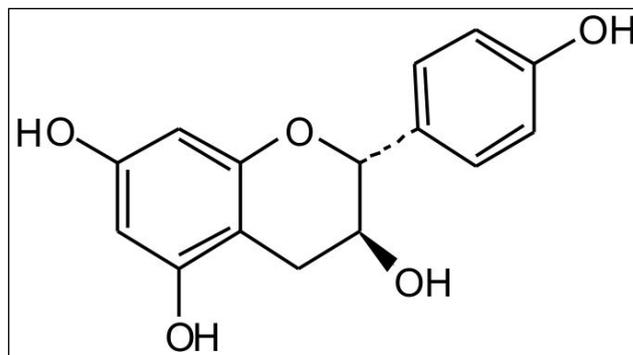
Rhizome of *C. rotang* was collected from Tirunelveli district and authenticated by Dr.V.Chelladurai Ex.Research Officer (Botany) of SMPU unit of Central Council for Research in Ayurvedic sciences at Palayamkottai. The voucher specimen No. Rh3-231 is deposited in the Pharmacognosy department of the institute of Captain Srinivasa Murthi Research Institute for Ayurveda. Coarsely powdered rhizome (1 kg) was extracted with chloroform by cold percolation method for 72 hours. The extraction was repeated and filtered. The combined extract was concentrated by distilling over

a boiling water bath. The last traces of the solvent were removed under vacuum. Extract (7.2 g) was column chromatographed using silica gel (acme 100-200 mesh; 1:22) as the stationary phase and eluted with solvents of increasing polarity in the order *n*-hexane, chloroform, ethyl acetate and methanol and their mixtures. Earlier fractions gave waxy material. *n*-Hexane-chloroform(1:1) eluates on concentration and crystallization yielded β -sitosterol. Fractions eluted with 95% ethyl acetate in chloroform on removal of the solvent gave a solid which on crystallization yielded a compound. It showed a single spot at R_f 0.53 in the mobile phase of toluene: ethyl acetate: formic acid (9:2:0.5). The compound had a sharp melting point at 220°C, (lit. mp. 221-222°C). It gave bluish green colour with alcoholic $FeCl_3$ showing it to be a phenol and answered Shinoda's test for flavonoid. $[\alpha]_D^{20} +0.50^\circ$ (acetone: ethyl acetate 9:1, c: 0.09). IR ν_{max} (KBr) cm^{-1} : 3407(hydroxyl), 2980, 1612, 1454 (aromatic), 1376, 1052, 935, 841, 633. ¹H NMR, δ_{ppm} CD_3OD (400 MHz): 5.86 (1H, d, J= 2.0 Hz, H-6); 5.95 (1H, d, J=2.0 Hz, H-8); 4.61(1H, d, J= 7.6 Hz, H-2); 4.0 (1H, m, H-3); 2.52 (1H, dd, J=16.0, 8.4 Hz, H-2ax); 2.90 (1H, dd, J= 16.0, 5.6 Hz, H-4eq); 7.23 (2H, d, J= 8.8 Hz, H-2' and H-6'); 6.80 (2H, d, J=8.8 Hz, H-3' and H-5'). ¹³C NMR δ_{ppm} CD_3OD (100.62 MHz): 82.89 (C-2), 68.87 (C-3), 28.94 (C-4), 157.59 (C-5), 95.50 (C-6), 157.87(C-7), 96.32 (C-8); 157.01 (C-9), 100.92 (C-10), 131.50 (C-1'), 129.65 (C-2' and C-6'), 116.06 (C-3' and C-5'), 158.41 (C-4').

Further elution of the column with ethyl acetate led to the isolation of β -sitosterol 3 β -D-glucopyranoside, m.p.288°. The known compounds were identified by comparison with authentic samples available (m.m.p, Co-TLC).

RESULTS AND DISCUSSION

The compound m.f. $C_{15}H_{14}O_5$, m.p $220^{\circ}C$ gave positive reaction for phenol and Shinoda's test for flavonoids. IR spectrum showed the presence of hydroxyl at 3407 and absence of carbonyl group. Aromatic system was shown by 1612, 1454 cm^{-1} . The 1H NMR spectrum showed the compound to be a catechin derivative (flavan-3-ol). The ring contained hydroxyls at C-5 and C-7. H-6 and H-8 appeared as one proton doublet ($J=2.0$ Hz) each at δ 5.86 and 5.95. The presence of the C ring of the catechin moiety was shown by the vicinal couplings and chemical shift value of H-2 and H-3. H-2 appeared as a one proton doublet ($J=7.6$ Hz) at δ 4.61. H-3 appeared as a one proton multiplet at δ 4.0. H-4 methylene proton showed geminal coupling of $J=16$ Hz. H-4 axial appeared at δ 2.52 as double doublet with ($J=16.0$ and 8.4 Hz). H-4 equatorial appeared as a double doublet $J=16.0$ and 5.6 Hz at δ 2.90. The presence of A_2B_2 pattern in the ring B was shown by the fact that H-2' and H-6' appeared as a 2 proton ortho coupled doublet $J=8.8$ Hz at δ 7.23. H-3' and H-5' appeared as a 2 proton doublet at δ 6.80 ($J=8.8$ Hz). These data suggested the structure of the compound to be (+)-Afzelechin [1]. The ^{13}C NMR data also confirmed the structure of the compound (see experimental). The identity of the compound was confirmed by comparison of the



(+)-Afzelechin [1]

physical and spectroscopic data with those reported in literature².

ACKNOWLEDGEMENT

Authors are thankful to the Director General, Central Council for Research in Ayurvedic Sciences, Dept. of AYUSH, Ministry of Health and Family Welfare, Govt. of India, New Delhi for providing facilities and ICMR for financial support.

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(Received 09 July 2012) (Accepted 26 November 2012)

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