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E: ISSN NO.: 2455-0817 *Remarking An Analisation*

Kanerocin-3-0-β-D-Glucopyranosyla (1...>4)-O-(L-Arabino Pyranosla (28...>1)-β-D-Glucopyranosyla Ester of Delonix Indica Flower

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Abstract

The plant Delonix Indica Linn[1-3], belongs to natural order papleonacial. Different parts of this plant are used in the treatment of ulcers, leprosy swellings, skin diseases, etc. It has also been reported to possess insecticidal and parasiticidal properties.

Keywords: Schiff Base, Spectrophotmeteric Determination, Molybdenum (Mo) Polymethane.

Introduction

Extràction and Isolation

The air-dried powdered flowers (2.0 Kg) of Delonix Indica were extracted with 95 % hot ethanol. The concentrated extract was evaporated under reduced pressure. The brown syrupy mass obtained was partitioned with benzene, chloroform, acetone, ethyl acetate and methanol. The benzene solable fraction was concentrated to remove solvent and crude compound obtained was subjected to column chromatography with mixture of different solvents in varying proportions. Fractions eluted with $CHCl_3$: C_6H_6 (3:2) were mixed and concentrated when saponin AS-I precipitated out. Crystallisation of AS-I with acetone gave light brown coloured crystals which showed a single homogeneous sport on TLC over silica gel using C_6H_6: $(CH_3)_2CO$: CH_3OH (4:3:3) as solvent system and I_3 vapours as visualising agent.

Aim of the Study

The present paper deals with the isolation and stractural characterizaiton of a pentacyclic triterpenoidal saponin, Kanerocin- 3-O-B-D-glucopyranosyl (...>4)-0-a-1 arabinopyranosyl (28→ 1)-β-D-glucopyranosl ester from the flower of this plant.

The compound AS-I analysed for molecular formula C47H74017 75.18% , H- 9.87 %, Calcualied C-61.97 % , H = 8.13 %) , mp 270- fourd C 271dand [M*1910 (FABMS), It responded positive to all characteristic colour reactions of triterpenoids and also gave positive Molisch's test, confirming it saponin. It's IR spectrum showed absorption bands at to be a triter 3377.8 (free-OH), 2933.9 (C-H str.), 1725.1 (>C-0), 1629.6 (C=C), 1405.6 (- CH6), 1370.2 (C-H bending), 867.2 (cyclohexane ring). IH-NMR (DMSO, 300 MHz) 6 0.73 (3H, s. H-23 Me), 0.83 (3H, s. H-24 Me), 0.87 (3H, s, H-25 Me), 0.90 (3H, s, H-26 Me), 0.92 (3H, s, H-27 Me), 1-59 (3H, s, H-25 Me), 167 (3H, s, H-30 Me), 1.24(2H, m, H-1), 1.90 (2H, m, H-2) 3.69 (1H, t, H-3), 1.63 (1H, m, H-5), 1.45 (2H, m, H-6), 1.32 (2H, m, H-7), 1.54 (1H, m H-9), 2.00 (211, m, H-11), 1.20 (2H, m, H-12), 2.40 (1H, t, H-13), 1.14 (2H, m, 11- 15), 1.40 (211, m, H-16), 5.23 (1H, t, H-21). 1.71 (2H, m. H-22), 6.33 (1H, anomeric proton arabinose 11-1), 5.23 (111, 11-1" glucose anomeric proten). 3.80 4.60 (4H, m, arabinose protons), 4.08-4.48 (5H, m, glucose rotons), 6.23 (1H, H-1" anomeric proton glucose), 3.83-4.44 (5H m, glucose protons). 1PC-NMR (DMSO, 300 MHz) 39.8 (C-1), 26.6 (C-2) 91.6 (C-3), 40.3 (C-4), 56.2 (C-5), 19.3 (C-6), 32.2 (C-7), 42.0 (C-8), 51.8 (C-9), 38.0(C-10), 22.2 (C-11), 31.0 (C-12), 401 (C-13), 42.8 (C-14), 28.8 (C-15), 33.5 (C-16), 50.5 (C-17), 127.7 (C-18), 138.5 (C-19), 141.8 (C-20) (118.2 (C-21), 38.7 (C-22),16.7 (C-23), 28.7 (C-24), 16.4 (C-25), 16.9 (C-26, 15.2 (C-27), 176.3 (C-28), 23.4 (C-29), 20.7 (C-30), 101.5 (C-1'), 76.8 (C-2'), 72.5 (C-3'), 67.3 (C-4"), 63.1 (C-5'), 92.8 (C-1"), 79.5 (C-2"), 77.3 (C-3"), 70.5 (C-4), 78.0 (C-5), 61.1 (C-6"), 105.6 (C-), 79.4 (C-2"), 76.8 (C-3"), 73.5 (C-4"), 76.5 (C-5""), and 70.7 (C-6"). PABMS : m/z 910, 586, 454, 437, 436, 410, 409, 246, 239, 201, 190 and 189.

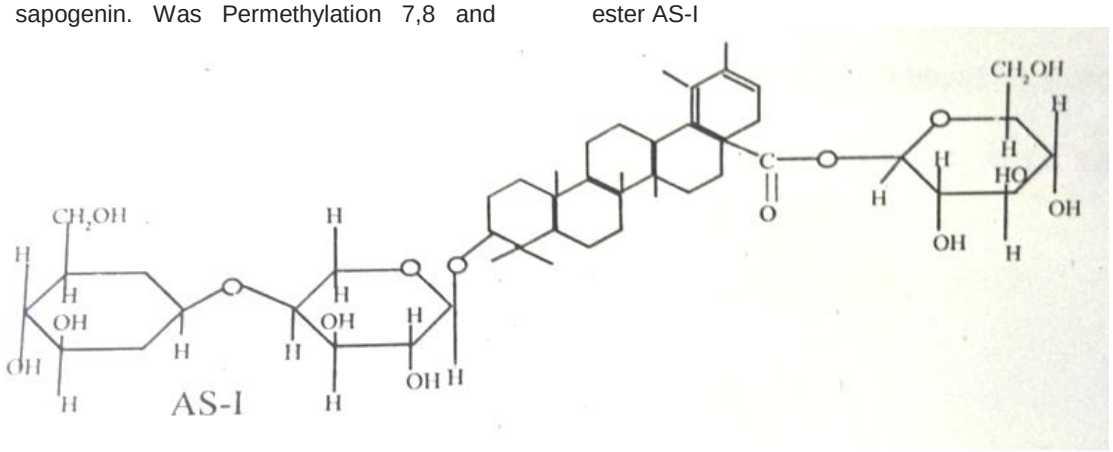
Acid hydrolysis of compound AS-I : the saponin As-I was dissolved in ethanol and treated with 7% H,SO, and refluxed on water bath for 10-12 hrs.

The reaction mixture was concentrated and allowed to cool and the residue was examined separately for was extracted with ether. The aqueous layer was washed with water, identification of sugar portion and etheronl layer evaporated to dryness and the residue was subjected to column chromatography using CHCI, : C,H (3:2) yielded sapogenin AS-I (a), molecular formula CHO Found C- 79.28 % , H-10.14; calcualted C- 79.29%, H= 10.13]. mp 258° - 259°. [M*] 454 (FABMS). It was identified as Kanerocin by chemical degradation and spectral analysis.4 The aqueous hydrolysate obtained, after acid hydrolysis of saponin AS1, was neutralized with BaCO3 and BaS04 to paper was filtered. The filtrate was concentrated and subjected chromatography. The sugars present were identified as Dglucose and L 6 (KU.18 and 0.22)- arabinose Alkaline hydrolysis of AS-I : The saponin AS-I (300 mg) was treated with methanolic KOH (20 ml) and refluxed for 5 hrs. The amorphous mass obtained was dissolved in MeOH and concentrated. Residue obtained was washed with water, evaporated to dryness and crystallized from acctone, yielding prosnpogenin AS-I (b), which analysed for mi. f. CH O,m. p. 2360- 2370 IM'] 586 (FABMS) and D-glucose which was i nthe hydrolysate. Acid hydrolysis of Prosapogenin AS-I (b): On acid hydrolysis with 7% H,SO, the prosapogenin AS-I (b) furnished sapogenin AS-I (a) (Kanerocin) and L-arabinose in equimolar ratio. The IABMS of AS-I showed a fragment ion peak at m/z 586 indicating that in prosapogenin, L-arabinose directly attached to C-3-OH of sapogenin. Was Permethylation 7,8 and hydrolysis of AS-I : 50 mg of saponin AS-I, 5 mg of Mel and 20 mg of Ag20 in 5 ml of DMF wer: refluxed for 24 hrs. at room temperature and when worked up it yielded sapogenin and methylated sugars identified as 2, 3,4,6 tetra O-methyl, D-glucose and 2,3 di-O-methyl- L-arabinose (COPC and COTLC) in the ratio of 2:1. It also indicated that C4-OH of L-arabinose and C1-OH of D-glucose were involved in formation of the glycoside. Saponin AS-I on graded hydrolysis with Killiani mixture liberated first two molecules of D-glucose followed by one molecule of L-arabinose, suggesting that D-glucose was terminal sugar and L-arabinose was linked to the sapogenin AS-I (a) Compilation of all above facts concluded that saponin AS-I has bisdesmosidic nature and a diasaccharide 4-D- glucopyranosyl L-arabinose was attached via C-l' anomeric hydroxyl of arabinose to C-3-OH of sapogenin and also that a molecule of D-glucose was involved in an ester linkage with -COOH group of sapogenin (Kanerocin).

Enzymatic hydrolysis of saponin AS-I: The saponin AS-I (30 mg) was dissolved in ethanol and treated with almond emulsin (40ml) and the contents were allowed to stand for 4 days at room temperature, when prosapogenin and D-glucose were liberated indicating B-linkage between D-glucose units and prosapogenin. Prosapogenin on further hydrolysis with Takadiastase yielded sapogenin kanerocin and L-arabinose confirming a-linkage between sapogenin and L-arabinose.

Conclusion

Thus the structure of the saponin AS-I was established as; Kanerocin -3-O-B-D-glucopyranosyl (1 4)-0-a-L-arabinopyranosyl (28.>1)-B-D glucopyranosyl ester AS-I



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References

1. *R. N. Chopra, S. L. Nayar and I. C. Chopra, Glossary of Indian Medicinal 1.Plants, CSIR Publication New Delhi, P. 175. (1956).*
2. *R. D. Chaudhary, Herbal Drug Industry, Bastern Publisher, New Delhi, P- 2. 204 (1998)*
3. *K. R. Kirtikar and B. D. Basu, Indian Medicinal Plants, Third Edition, Internaitonal Book Distributors, Dehradun, Vol. II, p 1584 1586 (1988).*
4. *R. Silverstech, M. Bassler and G. Clayton, Spectrometric Identification of Organic Compounds, John Willey Sons Inc., New York (1964)*
5. *H. Lederer and M. Lederer, Chomatography, 2nd Ed., Eleselver Publication, New York (1957).*
6. *H. C. Srivastava and F. Smith, J. Am. Chem. Soc. 79, p. 982, (1957)*
7. *B. Abegaz and B, Tecle, Phytochemistry, 19, p. 155401555, (1980)*
8. *C.C.Nweely, R. Hentle, M. Nak!ta and W. W. Wells, J. Am, Chem. Soc, 85, p. 2497 (1963). 41*