

methyl and rhamnose C-1 proton (H-1''). The low J value (J 2 Hz) was due to equatorial-equatorial coupling with H-2'' showing its α -configuration. The anomeric proton of glucose (H-1'') appeared as a doublet at δ 5.35 (J 7 Hz), the large coupling constant being due to *trans*-diaxial coupling with H-2'' suggesting its β -configuration. The above statement was also supported by ^{13}C nmr spectral (DMSO d_6) data. Compound A was thus assigned the structure of quercetin-3-*O*-(6''-*O*- α -L-rhamnopyranosyl)- β -D-glucopyranoside.

Compound B: Compound B, as yellow needles (MeOH), m.p. 153–55°, on hydrolysis with 8% HCl yielded kaempferol (m.p., m.m.p., co-tlc and uv spectra) and two sugars as D-glucose and L-rhamnose (pc). Partial and enzymic hydrolysis as described in compound A, established that the sugars were in disaccharide form with rhamnose as the terminal sugar and D-glucose linked to kaempferol with β -configuration. Permethylation of the compound B followed by Killiani's hydrolysis gave kaempferol 5,7,4'-trimethyl ether, 2,3,4-tri-*O*-methyl-D-glucose and 2,3,4-tri-*O*-methyl-L-rhamnose. The partially methylated aglycone showed a bathochromic shift of 61 nm in band-I with AlCl_3 confirming its C-3 glycosylation⁶. ^1H nmr spectral data of compound B—acetate, m.p. 91°, also supported the above facts. Compound B was thus characterised as kaempferol-3-*O*-(6''-*O*- α -L-rhamnopyranosyl)- β -D-glucopyranoside.

Acknowledgement

The authors wish to express their sincere thanks to Prof. S. M. Osman, Chairman, Department of Chemistry for facilities. One of the authors (F.K.) is thankful to C.S.I.R., New Delhi for financial assistance.

References

1. A. G. R. NAIR, P. RAMESH and S. S. SUBRAMANIAM, *Phytochemistry*, 1976, 15, 839.
2. H. K. DESAI, D. H. GOWAD, B. S. JOSHI, P. G. PARTHASARTHY, K. R. RAVINDERNATH, M. T. SAINDANE, A. R. SIDHAYE and N. VISHWANATHAN, *Indian J. Chem., Sect. B*, 1977, 15, 291.
3. R. M. RANGANATHAN and S. NAGARAJAN, *Ourr. Sci.*, 1980, 49, 309.
4. A. S. R. ANJANEYULU and S. N. RAJU, *Indian J. Chem., Sect. B*, 1984, 23, 1010.
5. R. P. A. INIGO and A. B. POMILIO, *Phytochemistry*, 1985, 24, 347.
6. S. HATTORI in "The Chemistry of Flavonoid Compounds", ed. T. A. GRISMAN, Pergamon, Oxford, 1962, p. 122.

Chemical Investigation of Stem Bark of *Ficus religiosa* and *Prosopis spicigera*

K. D. SWAMI, G. S. MALIK and N. P. S. BISHI*

Department of Chemistry, J V College, Baraut-250 611

Manuscript received 24 November 1988, accepted 19 January 1989

FICUS religiosa Linn.¹ (Moraceae) and *Prosopis spicigera* Linn.² (Leguminosae) are extensively used in the indigenous system of medicine. A hy-

poglycemic response was reported for β -sitosterol-D-glucoside obtained from the bark of *F. religiosa*³. In continuation to our earlier work⁴ on *F. religiosa*, the present communication deals with the chemical investigation of the bark of the title plants.

The shade dried coarsely powdered stem-bark of both the plants were soxhleted with petroleum ether (b.p. 60–80°) for 24 h. The extract was concentrated under reduced pressure and chromatographed over silica gel. The elution was carried out with petroleum ether and its mixture with benzene, furnishing the following compounds.

Ficus religiosa Linn.: Elution with petroleum ether—benzene (9 : 1) afforded a yellow viscous oil; decomposed on heating above 100°; density, 0.96 at 30°; positive colour reaction for quinone⁵. It was identified as vitamin K₁ by ir⁶, pmr and mass⁷ spectral data and degradation reactions.

The petroleum ether—benzene (3 : 1) eluate yielded white crystals ($\text{Me}_2\text{CO}-\text{C}_6\text{H}_6$), m.p. 81–82°, $\text{C}_{28}\text{H}_{58}\text{O}$ (M^+ 410); ν_{max} (KBr) 3 420, 1 040 (OH) and 725 cm^{-1} [$-(\text{CH}_2)_n-$, $n \geq 4$]. It was identified as n-octacosanol by pmr and mass fragmentation pattern; the acetate, m.p. 65°; the iodide, m.p. 59°.

Petroleum ether—benzene (1 : 1) eluate afforded colourless crystals (EtOH), m.p. 202–03°; $\text{C}_{31}\text{H}_{64}\text{O}$ (M^+ 470); positive Liebermann—Burchard (L—B) and TNM tests. It was identified as methyl oleanolate by ir, pmr and mass spectral data and further confirmed by hydrolysis to oleanolic acid, m.p. 312–13°; identical pmr and mass spectra with authentic sample and acetate, m.p. 225–26°.

Elution with petroleum ether—benzene (1 : 3) gave white crystals ($\text{MeOH}-\text{Me}_2\text{CO}$), m.p. 141–42°; positive L—B and TNM tests. The ir, pmr and mass spectral data identified it as lanosterol, further confirmed by m.m.p., co-tlc, preparation of the acetate, m.p. 132–33° and the acetate dibromide, m.p. 170–71°.

The petroleum ether—benzene (1 : 5) fraction yielded colourless plates (MeOH), m.p. 138°; positive L—B and TNM tests, the acetate, m.p. 121–22°. It was identified as β -sitosterol by its superimposable ir, m.m.p., co-tlc with an authentic specimen. Further elution with petroleum ether—benzene (1 : 5) gave colourless needles ($\text{C}_6\text{H}_6-\text{MeOH}$), m.p. 171–72°; positive L—B and TNM tests. Spectral data identified it as stigmasterol and further confirmed by m.m.p., co-tlc with authentic sample, the acetate, m.p. 143–44° and the benzoate, m.p. 158–59°.

The benzene—acetone (1 : 1) eluate afforded colourless crystals (MeOH), m.p. 167–68°; positive L—B and TNM tests. The ir, pmr and mass spectral analyses and the hydrazone, m.p. 340–41°, identified it as lupen-3-one.

Prosopis spicigera Linn.: The petroleum ether—benzene (9 : 1) eluate yielded the yellow viscous oil, identical in all respects with vitamin K₁. Elution with petroleum ether—benzene (3 : 1) furnished

white crystals ($\text{Me}_2\text{CO}-\text{C}_8\text{H}_{18}$), m.p. $61-2^\circ$. The ir, pmr and mass spectra identified it as n-octacosyl acetate (M^+ 452) and further confirmed by co-tlc, m.m.p. of its hydrolysed product, octacosanol, m.p. $84-5^\circ$ with an authentic sample.

The petroleum ether-benzene (1:1) fraction afforded a white solid, m.p. $90-91^\circ$. The infrared spectrum revealed it a long chain aliphatic acid. The mass spectrum of the mixture showed⁹ prominent peaks at m/e 424, 396, 381, 353, 325, 297, 73, 60, 57, 55, 43 and 41. The peaks at m/e 60 (base) and 73 are diagnostic of unbranched long chain fatty acids. Thus, the mass at m/e 424 and 396 are the molecular ion peaks corresponding to n-hexacosanoic acid (relative abundance 86.4%) and n-octacosanoic acid (13.6%) respectively. The identity of the compounds was further confirmed by gc of their methyl esters (CH_3N_2) followed by mass spectrometric analysis.

The alcoholic extract of defatted stem bark, after separating KCl, yielded glucose, rhamnose, sucrose and starch (identified by pc in the usual manner).

The chlorine content of the stem bark was determined by the standard method⁹ and found to be $0.92 \pm 0.01\%$.

Acknowledgement

Authors thanks Prof. K. K. Gandhi, Dr. U. S. Tiwari, I.I.T., New Delhi and Dr. Ramesh Chandra, Chemistry Department, Delhi University for spectral analyses.

References

1. K. P. TRIVEDI, "Dhanvantari Vanaushadhi Visheshank", Dhanvantri Karyalaya, Vijalgarh, 1971, Vol. 4, p. 285.
2. R. N. CHOPRA, I. O. CHOPRA and S. L. NAYAR, "Glossary of Indian Medicinal Plants", O. S. I. R., New Delhi, 1956, p. 204.
3. S. H. AMBIKE and M. R. R. RAO, *Indian J. Pharm.*, 1967, 29, 91.
4. K. D. SWAMI, G. S. MALIK and N. P. S. BISHT, *Z. Naturforsch.*, communicated.
5. F. FRIGL, "Spot Tests in Organic Analysis", Elsevier, Amsterdam, 1960, p. 228.
6. P. G. STRECHER, "The Merck Index", 8th. ed., Merck and Co. Rahway, 1968, p. 1115.
7. K. YAMAGUCHI, "Spectral Data of Natural Products", Elsevier, Amsterdam, 1970, Vol. 1, pp. 428-29.
8. K. BREMANN, "Mass Spectrometry", McGraw-Hill, New York, 1962, pp. 168, 251-253.
9. K. PARCH and M. V. TRACEY, "Modern Methods of Plant Analysis", Springer-Verlag, Berlin, 1955, Vol. 1, p. 487.

Commercial Utilisation of Local Variety *Dioscorea prazeri* for the Production of Diosgenin

LALA P. K. ROY, U. K. SOM and P. K. GHOSH
Government Quinine Factory, Mungpoo, Darjeeling
and

C. P. DUTTA* and MAYA BISWAS (née SINHA)
Department of Chemistry University of Kalyani,
Kalyani-741 235

Manuscript received 12 December 1988, accepted 2 February 1989

DIOSCOREA prazeri Prain and Burk grows abundantly in Darjeeling terai, Jalpaiguri and Malda districts in West Bengal¹, but its use as raw material for the isolation of diosgenin upto the desired limit of purity is not easy because a good percentage of interfering materials lead to the isolation of impure diosgenin². The existing technologies³⁻⁵ fail to produce the desired quality of diosgenin from this species while they work well for the other varieties of *Dioscorea*. The following improved technique can be successfully utilised for the isolation of diosgenin having high degree of purity even in a large scale basis from *D. prazeri*.

Isolation of diosgenin: Dry *D. prazeri* powder was extracted with petroleum ether (b.p. $60-80^\circ$) for 6 h. The resulting defatted mass was hydrolysed with 6 N HCl for 8 h at boiling temperature and then extracted with petroleum ether (b.p. $60-80^\circ$) for 8 h. The crude product was crystallised from methanol to yield pure diosgenin.

Results and Discussion

The results of the process are given in Tables 1-5.

TABLE 1—PRESENCE OF FATTY AND WAXY MATERIALS IN *D. prazeri*

Expt. no.	Weight of <i>D. prazeri</i> powder kg	Weight of defatted <i>D. prazeri</i> powder kg	Weight of fatty and waxy mass g	Average % fatty mass removed
1.	1.200	1.181	19	1.66
2.	1.300	1.279	21	
3.	1.390	1.360	20	
4.	1.430	1.395	25	
5.	1.692	1.661	31	

TABLE 2—RECOVERY OF SOLVENT FROM DEFATTED MASS OF *D. prazeri*

Expt. no.	Amount of plant material kg	Volume of solvent kept absorbed by powdered plant material ml	Volume of solvent recovered ml	Recovery %	Average % recovery of solvent
1.	1.181	1 027.0	955.1	93	90
2.	1.279	1 112.2	1 023.2	92	
3.	1.360	1 182.6	1 064.3	90	
4.	1.395	1 213.0	1 067.4	88	
5.	1.661	1 444.3	1 256.5	87	