

**CHEMICAL CONSTITUENTS FROM THE ROOTS OF *ELEPHANTOPUS SCABER*, AND
LEAVES OF *FICUS RUMPHII* AND *IPHIONA SCABRA*****Mohammad Ali^{1*}, Shahnaz Sultana^{1,2}, Showkat Rasool Mir¹ and Vinod Damoji Rangari³**¹Phytopharmaceuticals Research Laboratory, School of Pharmaceutical Education and Research, Jamia Hamdard (Hamdard University), New Delhi - 110 062, India.²Present Address: College of Pharmacy, Jazan University, Jazan, Saudi Arabia.³SLT Institute of Pharmaceutical Sciences, Guru Ghasidas Vishwavidyalaya, Koni, Bilaspur, Chhattisgarh – 495 00, India.***Corresponding Author: Mohammad Ali**

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ABSTRACT

Elephantopus scaber L. (family Asteraceae) is an erect, rosetted, perennial, scabrous herb and its roots are used to treat bronchitis, conjunctivitis, cuts, dysmenorrhoea, dysuria, headache, leucorrhoea, menorrhagia, metrorrhagia, piles, pimples, spermatorrhoea, sprue, swelling, stomach disorders, tonsillitis, ulcers and wounds. *Ficus rumphii* Blume (family Moraceae) is a large, deciduous tree, and its leaves are effective to heal bruises, wounds and menstrual problems. *Iphiona scabra* DC. ex Decne. (family Asteraceae) is an annual glandular-hairy, viscid low shrub. The plant is used as an antispasmodic drug. Phytochemical investigation of the roots of *Elephantopus scaber* afforded a new lupene-type triterpenic ester characterized as lup-12, 20(29)-dien- 3 α -olyl 3',7',11',15'-tetramethylhexadecanoate (lup-12, 20(29)-dien- 3 α -olyl phytanoate, **1**). The leaves of *Ficus rumphii* gave a new phenolic compound identified as 1-isopentanyl-3,4-dioxomethylene- 2-phenol (**2**). The leaves of *Iphiona scabra* yielded a new sesquiterpenic carboxylic acid characterized as caryophylla-3-en-2 α -ol 12-oic acid (**3**), a long chain aliphatic alcohol, viz., 1-tetracosanol (lignoceryl alcohol) (**4**), and three phenolic derivatives formulated as 1,3-dihydroxy-4-acetyl benzene (4-acetyl resorcinol, **5**), 1,2-dihydroxy-4,5-diacetyl benzene (4,5-diacetyl catechol, **6**) and 1-(*m*-cresol)-2-(*m*-cresol)-ethane (1,2-bis-*m*-cresol ethane, **7**).

KEYWORDS: *Elephantopus scaber* roots, *Ficus rumphii* leaves, *Iphiona scabra* leaves, phytoconstituents, isolation, characterization.**INTRODUCTION**

Elephantopus scaber L., syn. *E. carolinensis* G. Mey., *E. sordidus* Salisb., *Scabiosa cochinchinensis* Lour., *Asteroccephalus cochinchinensis* Sprengel (Family Asteraceae), known as bhopatri, elephant foot, adhomukha and ban-tambakhu, is a native to tropical Africa, India, south eastern Asia and northern Australia. It is an erect, rosetted, perennial, scabrous herb, up to 60 cm tall; rootstock creeping; plants dichotomously branched; leaves sessile, in radical rosettes, obovate, oblong-lanceolate, cuneate-attenuate at base, subacute at apex, margin serrate, cauline leaves alternate, few, under surface pale-brown, tomentose; flower heads solitary, glomerate, terminal on peduncles, florets pink-purple; achenes truncate, 10-ribbed, cuneate below, crowned by bristly, hairy pappus.^[1] The plant is abortifacient, anthelmintic, antiseptic, astringent, cytotoxic, diaphoretic, diuretic, emmenagogue, emollient, febrifuge, fish poison, hepatoprotective, tonic and vulnerary. It is used to treat conditions such as asthma, colic pain, coughs, diarrhoea, dysentery, dyspepsia, fevers, filariasis, gonorrhoea, loss of appetite, menstrual

derangements due to colds, oedema, pulmonary diseases, skin diseases, urethral discharges and venereal diseases. A plant decoction is utilized to treat fungal skin diseases. The plant is fed to cows to improve lactation.^[2-4] The leaves are diuretic, febrifuge and emollient, effective against bronchitis, diarrhoea, dysentery, dysuria, fevers, malaria, menorrhagia, rheumatism, tetanus, urinary disorders, wounds, as a postpartum remedy, and to induce abortion. Bruised leaves with coconut oil are applied to cure ulcers and eczema. Leaf juice is massaged to prevent hair loss.^[2-4] The roots are antidote, antiseptic, diuretic, emollient, febrifuge and tonic, prescribed to comfort bronchitis, colds, colic, conjunctivitis, coughs, cuts, diarrhoea, dysentery, dysmenorrhoea, dysuria, fevers, flu, headache, intestinal parasites, leucorrhoea, menorrhagia, metrorrhagia, piles, pimples, spermatorrhoea, sprue, swelling, stomach ache, tonsillitis, ulcers and wounds. The root is given to arrest vomiting, to reduce labour pain, to accelerate delivery and to kill maggots; root powder with pepper is applied to prevent tooth-ache. The flowers are taken to overcome

bronchitis, cough, liver problems and ophthalmopathy.^[2,4]

The *Elephantopus scaber* plant contained withanolides,^[5] dipeptides,^[6] elemanolide lactone,^[7] germacranolide scabertopin,^[8] lupeol, stigmasterol and germacranolide 11,13-dihydrodeoxyelephantopin,^[9] sesquiterpene lactone scabertopinol, trans-caffeic acid, methyl 3,4-dicaffeoylquinic acid, luteolin-4'-O- β -D-glucoside, trans-p-coumaric acid, indole-3-carbaldehyde, methyl trans-cafeate, luteolin-7-O-glucuronide 6"-methyl ester and luteolin,^[10] ethyl hexadecanoate, ethyl-9,12-octadecadienoate, ethyl-(Z)-9-octadecenoate, ethyl octadecanoate, lupeol, stigmasterol, stigmasterol glucoside, deoxyelephantopin, 17,19-dihydrodeoxy - elephantopin and iso-17,19-dihydrodeoxyelephantopin,^[11] scabertopin, isoscabertopin, deoxyelephantopin, isodeoxyelephantopin, methyl 3,4,3',4'-tetrahydroxy- δ -truxinate, 5-O-caffeoylquinic acid and chlorogenic acid methyl ester,^[11-15] flavonoids, luteolin, lignan, sesquiterpene lactone, lupeol,^[16, 17] stigmasterol, lupeol, stearic acid, deoxyelephantopin isomers, deoxyelephantopin analogues.^[18] The leaves afforded lupeol, 2-amino-4-hydroxypteridine-6-carboxylic acid, 25-hydroxy-24-methylcholesterol, imidazole and pyrazine derivatives, arginine, methyl jasmonate, haloxazolam, asparagin and carbamic acid methyl ester.^[19,20] The roots yielded sesquiterpenoids, phenols, triterpenoids, caffeoylquinic acids, sterol, 2-butenic acid ester, curcuphenol and patriscabratine.^[21] The plant essential oil was composed of hexadecanoic acid (42.3%), isopropyl dimethyl tetrahydronaphthalenol (14.1%), β -sesquiphellandrene (8.3%), octadecadienoic acid (5.5%) and phytol (5.2%).^[22]

Ficus rumphii Blume, syn. *F. affiniior* Griff., *F. conciliorum* Oken, *F. cordifolia* Roxb. *F. coriacea* Aiton; *F. damit* Gagnep.; *F. populiformis* Schott ex Miq.; *Urostigma cordifolium* (Roxb.) Miq. *U. rumphii* (Blume) Miq. (family Moraceae), known as Kabaipipal, Pilkhan, Asvatthi, Mock peepul tree, Mock bodh tree and Rumpf's fig tree, is found in Indian subcontinent, southern China, Indonesia, Malaysia, Thailand and Vietnam. It is a large, deciduous tree, up to 20 m tall, bark dark grey, flaky; leaves broadly ovate or cordate, alternate, apex acuminate; male flowers few, female flowers ovary white, ovoid, smooth, style persistent; figs axillary on leafy branchlets, in small clusters, dark purple when mature, globose, smooth; achenes thin, tuberculate. The latex and fruits are anthelmintic, emetic and vermifuge, used to treat asthma, diarrhoea, headache, itch and malaria. A fruit powder together with the fruits of *Phyllanthus emblica* is given to control vomiting. The plant is fed to cattle to cure foot and mouth diseases. The plant juice is utilized to kill worms and given internally with turmeric, pepper and ghee to relieve asthma. The bark is taken to comfort biliousness, blood diseases, burning sensations, diarrhoea, headache, haematuria, itching, leprosy,

leucoderma, malaria, snake bites and ulcers. The leaves are boiled with coconut oil and applied to heal bruises, wounds and on the abdomen of a woman suffering from menstrual problems.^[3,23-25] The stem bark afforded β -amyrin, β -sitosterol and flavonol glycoside.^[26] The leaves contained stigmasterol-5-en-3-yl acetate, 1-isopentyl-3,4-dioxomethylene-2-phenol, 3-acetyl-2H-chromen-2-one and 3-(2-hydroxyphenyl)-1-(piperidin-1-yl) propan-1-one.^[27]

Iphiona scabra DC. ex Decne. (family Asteraceae), known as dhafran and Zafra, is distributed from North eastern tropical Africa to central Asia and Arabian Peninsula. It is an annual glandular-hairy, viscid low shrub, up to 25-60 cm high. The plant is used as an antispasmodic drug.^[28] Its extract has anticoagulant, anti-inflammatory and antiplatelet aggregation effects..^[28, 29] The plant infusions are drunk as a rock tea. The plant contained sesquiterpene xylosides,^[30] eudesmane derivatives, a secoeudesmane, sesquiterpene glycosides of iphionane and isoiphionane skeleton,^[31] flavonoids, quercetin and pyrrolizidine alkaloids.^[32] The leaves contained 6-hydroxy-8,11,11-trimethylbicyclo-[7.2.0]undec-4-ene-4-carboxylic acid.^[33] The plant essential oil was composed mainly of camphor, borneol, intermedeol, caffeic acid, α -cadinol, γ -muurolene, α -eudesmol, β -gurjunene, cis-nerolidol and δ -cadinene.^[34] Keeping in view the various therapeutic values of the plants in indigenous medicinal systems and development of ecofriendly, biodegradable and safer herbal preparations it has been aimed to analyze the spectral data to establish structures of the phytoconstituents isolated from the roots of *Elephantopus scaber* and leaves of *Ficus rumphii* and *Iphiona scabra*.

MATERIALS AND METHODS

The protocols of all methodologies (procedures, experimental designs and analysis assays) were adopted from the earlier published work.^[35,36]

Collection and authentication of plant materials

The roots of *E. scaber* were collected from the forest of Achanakmar, Chhattisgarh, India. It was authenticated by the Dr. G. P. Sinha, Scientist, Ministry of Environment and Forests, Botanical Survey of India, Allahabad, Uttar Pradesh. A voucher specimen No. BSI/CRC/TECH /2014-15 has been preserved in the herbarium of SLT Institute of Pharmaceutical Sciences, Guru Ghasidas Vishwavidyalaya, Bilaspur, Chhattisgarh – 495 00, India.

The leaves of *F. rumphii* were collected from the Forest Research Institute, Dehradun, India, and identified by Dr Athar Ali Khan, Taxonomist, Department of Botany, Aligarh Muslim University, Aligarh. A voucher specimen has been deposited in the herbarium of Department of Botany of the University.

The leaves of *Iphiona scabra* were collected from Yemen and identified by Prof. Abdul Nasser Al-Gifri, Professor of Plant Taxonomy, University of Aden,

Yemen. A voucher specimen was deposited in the herbarium of the University of Aden, Yemen.

Extraction and isolation

The air-dried roots of *E. scaber* and the leaves of *F. rumphii* and *I. scabra* were (1.0 kg each) were defatted with *n*-hexane and extracted exhaustively with methanol in a Soxhlet apparatus individually. The methanolic extracts were concentrated independently under reduced pressure to yield dark brown viscous masses (214 g, 347 g, and 281 g, respectively). The dried residues (200 g each) were dissolved in minimum amount of methanol and adsorbed on silica gel column grade (60-120 mesh) separately to obtain slurries. Each slurry was air-dried and chromatographed over silica gel columns loaded in petroleum ether (b. p. 60 – 80 °C) singly. Each column was eluted with petroleum ether, petroleum ether - chloroform (9:1, 3:1, 1:1, 1:3, v/v), chloroform and chloroform - methanol (99:1, 49:1, 19:5, 9:1, v/v). Various fractions were collected separately and matched by TLC to check homogeneity. Similar fractions having the same R_f values were combined and crystallized with solvents. The isolated compounds were recrystallized to get pure compounds.

Isolation of a phytoconstituent from the roots of *Elephantopus scaber*

3-Epi-lup-12-enolyl phytanoate (1)

Elution of the column with chloroform-methanol (19:1) afforded colourless crystals of **1**, recrystallized from chloroform-methanol (1:1), 341 mg, R_f : 0.32 (hexane-ethyl acetate, 4:1); m. p. 210-212 °C, UV λ_{max} (MeOH): 217 nm (log ϵ 5.3); IR ν_{max} (KBr): 2918, 2850, 1730, 1653, 1435, 1363, 1247, 1080, 979, 765 cm^{-1} ; 1H NMR ($CDCl_3$): δ 5.28 (1H, d, J = 5.2 Hz, H-12), 4.71 (1H, s, H₂-29a), 4.68 (1H, s, H₂-29b), 4.50 (1H, dd, J = 5.2, 4.5 Hz, H-3 β), 2.41 (1H, ddd, J = 6.2, 5.8, 8.3 Hz, H-19 β), 2.36 (1H, d, J = 5.8 Hz, H-18 β), 1.67 (1H, brs, Me-30), 1.59 (1H, dd, J = 3.8, 8.9 Hz, H-9 α), 1.04 (3H, brs, Me-23), 1.01 (3H, brs, Me-28), 0.94 (3H, brs, Me-25), 0.82 (3H, s, Me-26), 0.80 (3H, s, Me-27), 0.75 (3H, s, Me-24), 0.67 (1H, m, H-5), 2.06 (2H, t, J = 4.8 Hz, H₂-2'), 1.71 (1H, m, H-3' α), 1.64 (1H, m, H-7' α), 1.61 (1H, m, H-11' α), 1.51 (1H, m, H-15' α), 1.06 (3H, d, J = 6.3 Hz, Me-17'), 0.96 (3H, d, J = 6.1 Hz, Me-18'), 0.90 (3H, d, J = 6.2 Hz, Me-20'), 0.87 (3H, d, J = 6.4 Hz, Me-19'), 0.85 (3H, d, J = 6.2 Hz, Me-16'), 2.33 – 1.15 (36 H, m, 36 \times CH₂); ^{13}C NMR ($CDCl_3$): δ 38.44 (C-1), 27.43 (C-2), 80.93 (C-3), 39.20 (C-4), 55.37 (C-5), 18.21 (C-6), 34.23 (C-7), 40.84 (C-8), 50.35 (C-9), 37.08 (C-10), 21.34 (C-11), 118.90 (C-12), 139.82 (C-13), 42.82 (C-14), 27.03 (C-15), 35.57 (C-16), 43.01 (C-17), 48.67 (C-18), 48.27 (C-19), 150.91 (C-20), 29.83 (C-21), 40.01 (C-22), 27.95 (C-23), 15.98 (C-24), 16.20 (C-25), 16.03 (C-26), 14.71 (C-27), 18.01 (C-28), 109.39 (C-29), 19.30 (C-30), 170.99 (C-1'), 48.01 (C-2'), 42.33 (C-3'), 21.65 (C-4'), 22.55 (C-5'), 23.71 (C-6'), 42.18 (C-7'), 27.61 (C-8'), 34.17 (C-9'), 34.38 (C-10'), 41.08 (C-11'), 36.32 (C-12'), 36.70 (C-13'), 37.01 (C-14'), 38.03 (C-15'), 16.30 (C-16'), 14.51 (C-17'), 17.73 (C-18'), 20.95 (C-19'), 16.53

(C-20'); ESI MS m/z (rel. int.): 718 [M]⁺ (C₅₀H₈₆O₂) (1.3), 423 (15.2), 406 (21.3), 311 (12.8), 295 (21.3), 216 (14.1), 216 (8.7), 207 (16.4), 190 (7.8).

Isolation of a phytoconstituent from the leaves of *Ficus rumphii*

1-Isopentanyl-3,4-dioxomethylene-2-phenol (2)

Elution of the column with petroleum ether gave pale yellow crystals of **2**, recrystallized from chloroform-methanol (1:1), yield 123 mg; m. p. 238 - 239 °C; UV λ_{max} (MeOH): 277 nm; IR ν_{max} (KBr): 3331, 2927, 2843, 1628, 1561, 1443, 1311, 1239, 1134, 1087, 895 cm^{-1} ; 1H NMR ($CDCl_3$): δ 6.77 (1H, d, J = 7.9 Hz, H-5), 6.11 (1H, d, J = 7.9 Hz, H-6), 3.41 (2H, s, O-CH₂-O), 2.55 (2H, t, J = 6.6 Hz, H₂-7), 1.71 (1H, m, H₂-8a), 1.67 (1H, m, H₂-8b), 1.55 (1H, m, H-9), 1.21 (3H, d, J = 6.3 Hz, H₃-10), 1.17 (3H, d, J = 6.7 Hz, H₃-11); ^{13}C NMR ($CDCl_3$): δ 144.69 (C-1), 169.24 (C-2), 156.81 (C-3), 156.77 (C-4), 137.97 (C-5), 106.63 (C-6), 47.58 (C-7), 33.41 (C-8), 41.69 (C-9), 25.31 (C-10), 25.28 (C-11), 99.54 (O-CH₂-O); +ve ESI MS m/z (rel. int.): 208 [M]⁺ (C₁₂H₁₆O₃) (1.4).

Isolation of phytoconstituents from the leaves of *Iphiona scabra*

Caryophylla-3-en-2 α -ol 12-oic acid (3)

Elution of the column with petroleum ether afforded colourless crystals of **3**, recrystallized from chloroform-methanol (1 : 1), yield 63 mg, m. p. 92-93 °C; IR ν_{max} (KBr): 3498, 3241, 2931, 2829, 1696, 1632, 1465, 1263, 1059, 981 cm^{-1} ; 1H NMR ($CDCl_3$): δ 5.81 (1H, d, J = 4.2 Hz, H-3), 4.87 (1H, dd, J = 5.8, 4.2 Hz, H-2 β), 2.79 (1H, dd, J = 11.6, 5.9 Hz, H₂-5 α), 2.31 (1H, dd, J = 5.6, 4.9 Hz, H₂-5 β), 1.81 (1H, dd, J = 5.8, 8.3 Hz, H-1 β), 1.76 (1H, m, H-9 α), 1.57 (1H, m, H-8 α), 1.38 (2H, m, H₂-10), 1.27 (2H, m, H₂-6), 1.23 (2H, m, H₂-7), 1.11 (3H, d, J = 6.4 Hz, Me-13), 0.97 (3H, s, Me-14), 0.94 (3H, s, Me-15); ^{13}C NMR ($CDCl_3$): δ 42.58 (C-1), 78.07 (C-2), 134.38 (C-3), 140.59 (C-4), 29.57 (C-5), 27.31 (C-6), 25.53 (C-7), 38.24 (C-8), 40.25 (C-9), 38.27 (C-10), 48.92 (C-11), 176.11 (C-12), 25.21 (C-13), 18.34 (C-14), 25.82 (C-15); ESI MS m/z (rel. int.): 252 [M]⁺ (C₁₅H₂₄O₃) (11.4), 237 (18.5), 234 (9.2), 222 (23.1), 208 (7.9).

Lignoceryl alcohol (4)

Elution of the column with petroleum ether - chloroform (9:1) gave colourless amorphous powder of **4**, yield 138 mg, m. p. 73 - 74 °C; IR ν_{max} (KBr): 3421, 2926, 2842, 1461, 1253, 1166, 1059, 726 cm^{-1} ; 1H NMR ($CDCl_3$): δ 3.51 (2H, t, J = 6.7 Hz, H₂-1), 1.68 (2H, m, H₂-2), 1.55 (2H, m, H₂-3), 1.34 (2H, m, H₂-4), 1.32 (4H, m, H₂-5, H₂-6), 1.25 (4H, m, H₂-7, H₂-8), 1.22 (30H, br s, 15 \times CH₂), 0.86 (3H, t, J = 6.5 Hz, Me-26); ^{13}C NMR ($CDCl_3$): δ 63.78 (C-1), 32.85 (C-2), 31.94 (C-3), 29.84 (14 \times CH₂), 29.71 (C-18), 29.58 (C-19), 28.53 (C-20), 26.47 (C-21), 25.41 (C-22), 22.72 (C-23), 14.18 (C-24); ESI MS m/z (rel. int.): 354 [M]⁺ (C₂₄H₅₀O) (37.2).

4-Acetyl resorcinol (5)

Further elution of the column with petroleum ether – chloroform (9:1) yielded pale yellow crystals of **5**, recrystallized from chloroform-methanol (1:1), yield 184 mg; m. p. 142 – 143 °C; UV λ_{max} (MeOH): 273 nm; IR ν_{max} (KBr) : 3304, 2926, 2849, 1693, 1633, 1525, 1442, 1378, 1332, 1209, 1145, 1067, 958 cm^{-1} ; ^1H NMR (CDCl_3): δ 7.61 (1H, d, J = 2.8 Hz, H-2), 6.31 (1H, dd, J = 2.8, 9.3 Hz, H-6), 6.25 (1H, d, J = 9.3 Hz, H-5), 2.50 (3H, brs, H_3 -8); ^{13}C NMR (CDCl_3): δ 166.15 (C-1), 114.22 (C-2), 165.89 (C-3), 134.36 (C-4), 109.07 (C-5), 103.48 (C-6), 204.25 (C-7), 25.97 (C-8); +ve ESI MS m/z (rel. int.): 152 $[\text{M}]^+$ ($\text{C}_8\text{H}_8\text{O}_3$) (14.6).

4, 5-Diacetyl catechol (6)

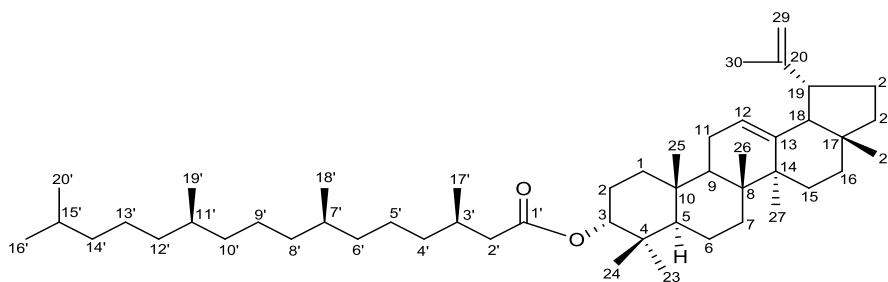
Elution of the column with petroleum ether – chloroform (3:1) furnished pale yellow crystals of **6**, recrystallized from acetone, yield 138 mg; m. p. 180 – 182 °C; UV λ_{max} (MeOH): 277, 286 nm; IR ν_{max} (KBr) : 3321, 2928, 2843, 1698, 1635, 1524, 1447, 1382, 1217, 1146, 1061, 948 cm^{-1} ; ^1H NMR (CDCl_3): δ 7.69 (2H, s, H-3, H-6), 2.43 (3H, s, H_3 -8), 2.41 (3H, brs, H_3 -10); ^{13}C NMR (CDCl_3): δ 165.73 (C-1), 164.92 (C-2), 115.12 (C-3), 134.54 (C-4), 134.50 (C-5), 114.87 (C-6), 204.81 (C-7), 26.11 (C-8), 203 (C-9), 26.04 (C-10); +ve ESI MS m/z (rel. int.): 194 $[\text{M}]^+$ ($\text{C}_{10}\text{H}_{10}\text{O}_4$) (100).

1,2- Bis-(m-cresol) ethane (7)

Elution of the column with chloroform produced pale yellow crystals of **7**, recrystallized from acetone, yield 219 mg; m. p. 206 – 207 °C; UV λ_{max} (MeOH): 281 nm; IR ν_{max} (KBr) : 2921, 1632, 1525, 1446, 1391, 1312, 1285, 1148, 1028 cm^{-1} ; ^1H NMR ($\text{DMSO}-d_6$): δ 3.44 (2H, d, J = 2.8 Hz, H_2 -1), 3.42 (2H, d, J = 2.8 Hz, H_2 -2), 8.14 (1H, d, J = 2.9 Hz, H-2'), 7.58 (1H, m, H-4'), 7.43 (1H, m, H-5'), 7.31 (1H, m, H-6'), 2.55 (3H, s, H_3 -7'), 8.09 (1H, d, J = 2.8 Hz, H-3''), 7.49 (1H, m, H-4''), 7.36 (1H, m, H-5''), 7.21 (1H, m, H-6''), 2.53 (3H, s, H_3 -7''), ^{13}C NMR ($\text{DMSO}-d_6$): δ 62.86 (C-1), 62.86 (C-2), 143.28 (C-1'), 129.25 (C-2'), 164.18 (C-3'), 128.89 (C-4'), 128.11 (C-5'), 122.29 (C-6'), 23.44 (C-7'), 144.06 (C-1''), 129.21 (C-2''), 164.12 (C-3''), 128.69 (C-4''), 127.71 (C-5''), 122.24 (C-6''), 23.39 (C-7''), +ve ESI MS m/z (rel. int.): 242 $[\text{M}]^+$ ($\text{C}_{16}\text{H}_{18}\text{O}_2$) (100), 135 (10.5), 107 (21.6).

RESULTS AND DISCUSSION

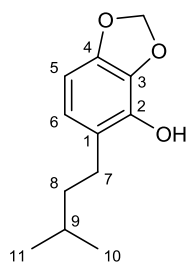
Compound **1**, named 3-epi-lup-12-enolyl phytanoate, showed distinctive IR absorption bands for ester function (1730 cm^{-1}) and unsaturation (1653 cm^{-1}). Its molecular ion peak was determined on the basis of mass and ^{13}C NMR spectra at m/z 718 consistent with a molecular formula of a triterpenic ester, $\text{C}_{50}\text{H}_{86}\text{O}_2$. The ion peaks arising at m/z 423 $[\text{C}_{1'} - \text{O fission}, \text{C}_{30}\text{H}_{47}\text{O}]^+$, 295 $[\text{M} - 423, \text{C}_{20}\text{H}_{39}\text{O}]^+$, 406 $[\text{C}_3 - \text{O fission}, \text{C}_{30}\text{H}_{46}]^+$ and 311 $[\text{M} - 406, \text{C}_{20}\text{H}_{39}\text{O}_2]^+$ indicated that a triterpenol was esterified with a diterpenic C_{20} acid. An ion fragment at m/z 216 $[\text{C}_{30}\text{H}_{46}]^+$ was formed due to retro-Diels-Alder fragmentation of the triterpenoid suggesting the existence of one of the vinylic linkage at C-12 in ring C and another double bond in isopropenyl group of a lupene-type triterpene at C-19 carbon. The ion peaks produced at m/z 207 $[423 - 216]^+$ and 190 $[207 - \text{OH}]^+$ which supported triterpenic nature of the molecule possessing oxymethine group which was placed at C-3 on the basis of biogenetic consideration. The ^1H NMR spectrum of **1** exhibited a one-proton deshielded doublet at δ 5.28 (J = 5.2 Hz) assigned to vinylic H-12 proton, two one-proton singlets at δ 4.71 and 4.68 accounted to exocyclic vinylic methylene H_2 -29 protons of a lupene-type triterpene, a one-proton double doublet at δ 4.50 (J = 5.2, 4.5 Hz) ascribed to β -oriented oxymethine H-3 proton, a three-proton singlet in the downfield region at δ 1.67 and six three-proton singlets between δ 1.04 – 0.75 associated with the tertiary C-23 to C-28 methyl protons. A two-proton doublet at δ 2.06 (J = 4.8 Hz) was ascribed to methylene H_2 -2' adjacent to the ester group. Five three-proton doublets at δ 1.06 (J = 6.3 Hz), 0.96 (J = 6.1 Hz), 0.90 (J = 6.2 Hz), 0.87 (J = 6.4 Hz) and 0.85 (J = 6.2 Hz) were associated with the secondary C-17', C-18', C-20', C-19' and C-16' methyl protons, respectively. The signals from δ 2.41 to 0.67 were designated to the remaining methylene and methine protons. The ^{13}C NMR spectrum of **1** displayed signals for the ester carbon at δ 170.99 (C-1'), vinylic carbons at δ 118.90 (C-12), 139.82 (C-13), 150.91 (C-20) and 109.39 (C-29), oxymethine carbon at δ 80.93 (C-3), and methyl carbons between δ 27.95 – 14.51. The ^1H and ^{13}C NMR spectral values of the triterpenic unit were compared with related lupene-type molecules.^[37-39] On the basis of spectral data analysis and chemical reactions, the structure of **1** was formulated as lup-12, 20(29)-dien- 3 α -olyl 3',7',11',15'-tetramethylhexadecanoate (lup-12, 20(29)-dien- 3 α -olyl phytanoate), a new lupene-type triterpenic ester (Fig. 1).



3-Epi-lupen-12-enol phytanoate (1)

Fig. 1: Structural formula of the phytoconstituent **1** isolated from the roots of *Elephantopus scaber*.

Compound **2** had UV absorption maximum at 277 nm for an aromatic compound, gave positive tests for phenols and showed IR absorption bands for a hydroxy group (3331 cm^{-1}) and aromaticity ($1628, 1561, 1087\text{ cm}^{-1}$). Its molecular ion peak was established on the basis of mass and ^{13}C NMR spectra consistent with a molecular formula of an alkylated dioxymethylene phenol at m/z 208, $\text{C}_{12}\text{H}_{16}\text{O}_3$. The ^1H NMR spectrum of **2** displayed two one-proton doublets at δ 6.77 ($J = 7.9\text{ Hz}$) and 6.11 ($J = 7.9\text{ Hz}$) assigned to ortho-coupled H-5 and H-6 protons, respectively. A two-proton singlet at δ 3.41 was due to dioxymethylene protons. A two-proton triplet at δ 2.55 ($J = 6.6\text{ Hz}$) and two one-proton multiplets at δ 1.71 and 1.67 were associated with the methylene H₂-7 located on the aromatic C-1 carbon and H₂-8 protons. A one-proton multiplet at δ 1.55 was due to methine H-9 proton. The C-10 and C-11 secondary methyl protons resonated as three-proton doublets correspondingly at δ 1.21 ($J = 6.3\text{ Hz}$) and 1.17 ($J = 6.7\text{ Hz}$). The ^{13}C NMR spectrum of **2** exhibited signals for aromatic carbons between δ 169.24 – 106.63, oxymethylene carbon at δ 99.54 (O-CH₂-O), and methyl carbons at δ 25.31 (C-10) and 25.28 (C-11). On the basis of these spectral data analysis, the structure of **2** has been established as 1-isopentenyl-3,4-dioxymethylene-2-phenol, a new phenolic compound (Fig. 2).



1-Isopentenyl-3,4-dioxymethylene-2-phenol (2)

Fig. 2: Structural formula of the phytoconstituent 2 isolated from the leaves of *Ficus ramphii*.

Compound **3** produced effervescences with sodium bicarbonate solution and showed distinctive IR absorption bands for a hydroxyl group (3498 cm^{-1}), carboxylic function ($3241, 1696\text{ cm}^{-1}$) and vinylic bond (1639 cm^{-1}). On the basis of mass and ^{13}C NMR spectra its molecular ions peaks was resolved at m/z 252 consistent with a molecular formula of a sesquiterpenoid. The ions peaks arising at m/z 237 $[\text{M} - \text{Me}]^+$, 222 $[237 - \text{Me}]^+$, 234 $[\text{M} - \text{H}_2\text{O}]^+$ and 208 $[\text{M} - \text{COOH}]^+$ indicated the presence of methyl, hydroxyl and carboxylic functions linked with the sesquiterpene moiety. The ^1H NMR spectrum of **3** exhibited a one-proton doublet δ 5.81 ($J = 4.2\text{ Hz}$) assigned to vinylic H-3 proton. A one-proton double doublet at δ 4.87 (1H, dd, $J = 5.8, 4.2\text{ Hz}$) was ascribed to β -oriented carbinol H-2 proton. Three three-proton signals as a doublet at δ 1.11 ($J = 6.4\text{ Hz}$) and as singlets at δ 0.97 and 0.94 were attributed to secondary C-13 and tertiary C-14 and C-15 methyl protons, respectively. The other methine and methylene protons resonated as one-proton double doublets at δ 2.79 ($J = 11.6, 5.9\text{ Hz}$, H₂-5 α), 2.31 ($J = 5.6, 4.9\text{ Hz}$, H₂-

5 β) and 1.81 ($J = 5.8, 8.3\text{ Hz}$, H-1 β), as one-proton multiplets at δ 1.76 (H-9 α) and 1.57 (H-8 α) and as two-proton multiplets at δ 1.38 (H₂-10), 1.27 (H₂-6) and 1.23 (H₂-7). The ^{13}C NMR spectrum of **3** displayed signals for vinylic carbons at δ 134.38 (C-3) and 140.59 (C-4), carboxylic carbon at δ 176.11 (C-12), carbinol carbon at δ 78.07 (C-2), methyl carbons at δ 25.21 (C-13), 18.34 (C-14) and 25.82 (C-15) and methine and methylene carbons between δ 42.58 – 25.53. On the basis of these evidences the structure of **3** has been established as caryophylla-3-en-2 α -ol 12-oic acid, a new sesquiterpenic carboxylic acid (Fig 3).

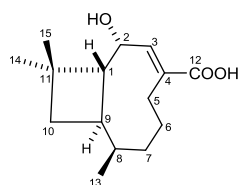
Compound **4** was a long chain aliphatic alcohol characterized as 1-tetracosanol (lignoceryl alcohol) (Fig 3).^[40, 41]

Compound **5** showed UV absorption maximum at 273 nm for aromaticity, responded positive tests for phenols and had IR absorption bands for hydroxy groups (3304 cm^{-1}), carbonyl function (1693 cm^{-1}) and aromaticity ($1633, 1525, 1067\text{ cm}^{-1}$). Its molecular ion peak was found at m/z 152 on the basis of mass and ^{13}C NMR spectra consistent with a molecular formula of an acetyl biphenolic compound, $\text{C}_8\text{H}_8\text{O}_3$. The ^1H NMR spectrum of **5** displayed two one-proton doublets at δ 7.61 ($J = 2.8\text{ Hz}$) and 6.25 ($J = 9.3\text{ Hz}$) and a one-proton doublet at δ 6.31 ($J = 2.8, 9.3\text{ Hz}$) assigned to meta-coupled H-2, ortho-coupled H-5 and meta-, ortho-coupled H-6, respectively. A three-proton singlet at δ 2.50 was ascribed to C-8 methyl protons located on the carbonyl carbon. The ^{13}C NMR spectrum of **5** exhibited signals for aromatic carbons between δ 166.15 – 103.48, carbonyl carbon at δ 204.25 (C-7) and methyl carbon at δ 25.97 (C-8). On the basis of these spectral data evidences, the structure of **5** has been established as 1,3-dihydroxy-4-acetyl benzene (4-acetyl resorcinol), a new phenolic derivative. (Fig. 3).

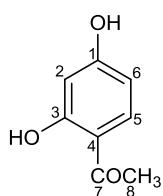
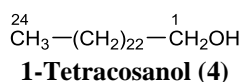
Compound **6**, $[\text{M}]^+$ at m/z 194 ($\text{C}_{10}\text{H}_{10}\text{O}_4$), gave positive tests of phenols and showed UV absorption maxima at 277 and 286 nm for aromaticity and IR absorption bands for hydroxy groups (3321 cm^{-1}), carbonyl function (1698 cm^{-1}) and aromaticity ($1635, 1524, 1061\text{ cm}^{-1}$). The ^1H NMR spectrum of **6** exhibited a two-proton singlet at δ 7.69 assigned to para-coupled H-3 and H-6 protons. Two three-proton singlets at δ 2.43 and 2.41 were ascribed to C-8 and C-10 methyl protons located on the carbonyl C-7 and C-9 carbons, respectively. The ^{13}C NMR spectrum of **6** displayed signals for aromatic carbons between δ 165.73 – 114.87, carbonyl carbons at δ 204.81 (C-7) and 203 (C-9) and methyl carbons at δ 26.11 (C-8) and 26.04 (C-10). On the basis of the foregoing account, the structure of **6** has been elucidated as 1,2-dihydroxy-4,5-diacetyl benzene (4,5-diacetyl catechol), a new catechol derivative. (Fig. 3).

Compound **7**, designated as 1,2-bis-*m*-cresol ethane, showed UV absorption maximum at 281 nm for aromaticity and had IR absorption bands for aromaticity

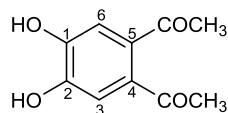
(1632, 1525, 1028 cm^{-1}). Its molecular ion peak was established at m/z 242 on the basis of mass and ^{13}C NMR spectra consistent with a molecular formula of a two cresol units linked with an ethyl group, $\text{C}_{16}\text{H}_{18}\text{O}_2$. The ion fragments arising at m/z 107 [$\text{O} - \text{CH}_2$ fission, $\text{C}_7\text{H}_7\text{O}^+$] and 135 [$\text{M} - 107$, $\text{C}_7\text{H}_7\text{O}-\text{CH}_2-\text{CH}_2^+$] indicated that two cresol units were linked to an ethyl group. The ^1H NMR spectrum of **7** exhibited two two-proton doublets at δ 3.44 ($J = 2.8$ Hz) and 3.42 ($J = 2.8$ Hz) assigned to oxymethylene H_2-1 and H_2-2 protons, respectively. The aromatic protons appeared as one-proton doublets at δ 8.14 ($J = 2.9$ Hz) and 8.09 ($J = 2.8$ Hz) and as multiplets from δ 7.58 to 7.21 assignable to *m*-cresol units. Two deshielded three-protons singlets at δ 2.55 and 2.53 were ascribed correspondingly to C-7' and C-7'' methyl protons linked to the aromatic rings. The ^{13}C NMR spectrum of **7** showed signals for oxymethylene carbons at δ 62.86 (C-1, C-2), aromatic carbons between δ 164.18 – 122.24 and methyl carbons at δ 23.44 (C-7') and 23.39 (C-7''). On the basis of these evidences, the structure of **7** has been characterized as 1-(*m*-cresol)-2-(*m*-cresol)-ethane (1,2-bis-*m*-cresol ethane), a new bis-cresol ethane (Fig. 3).



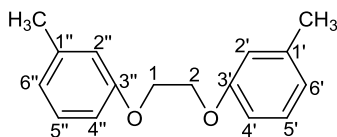
Caryophylla-3-en-2 α -ol 12-oic acid (3)



4-Acetyl resorcinol (5)



4,5-Diacetyl catechol (6)



1,2-Bis-(*m*-cresol) ethane (7)

Fig. 3: Structural formulae of the phytoconstituents 3 - 7 isolated from the leaves of *Iphiona scabra*.

CONCLUSION

Phytochemical investigation of the roots of *Elephantopus scaber* afforded a new lupene-type triterpenic ester characterized as lup-12, 20(29)-dien- 3 α -olyl 3',7',11',15'-tetramethylhexadecanoate (**1**). The leaves of *Ficus rumphii* yielded a new phenolic compound identified as 1-isopentanyl-3,4-dioxomethylene-2-phenol (**2**). The leaves of *Iphiona scabra* furnished a new sesquiterpenic carboxylic acid established as caryophylla-3-en-2 α -ol 12-oic acid (**3**), 1-tetracosanol (lignoceryl alcohol, **4**), and three phenolic derivatives formulated as 1,3-dihydroxy-4-acetyl benzene (4-acetyl resorcinol, **5**), 1,2-dihydroxy-4,5-diacetyl benzene (4,5-diacetyl catechol, **6**) and 1-(*m*-cresol)-2-(*m*-cresol)-ethane (1,2-bis-*m*-cresol ethane, **7**). This work has enhanced understanding about the phytoconstituents isolated for the first time from these plants. These compounds may be used as chromatographic markers for standardization and quality control of these plants.

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