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# CHEMICAL CONSTITUENTS FROM THE LEAVES OF *BUTEA MONOSPERMA* (LAM.) TAUB., PODS OF *CAESALPINIA DIGYNA* ROTTLER AND TUBERS OF *CYPERUS ROTUNDUS* L.

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## ABSTRACT

*Butea monosperma* (Lam.) Taub. (family Fabaceae) is a small-sized, deciduous tree, and its leaves are used to treat colds, colic, boils, eye diseases, inflammation, lumbago, irregular menstruation, piles, pimples, sore throat and worm infestations. *Caesalpinia digyna* Rottler (family Caesalpiniaceae) is a large scandent prickly shrub. Its pods are useful as an astringent, febrifuge, physical rejuvenative, tonic and to heal wounds. *Cyperus rotundus* L. (family Cyperaceae) is a perennial sedge and its tubers are taken as an appetizer, anti-inflammatory and to relieve thirst, pyrexia, cough, vomiting and rheumatoid. Our study was planned to isolate chemical constituents from the methanolic extracts of these plants and to characterize their structures on the basis spectral data analysis. Phytochemical investigation of the leaves of *B. monosperma* led to isolate a fatty acid ester identified as undecanyl oleate **(1).** The pods methanolic of *C. digyna* afforded two fatty acid ester recognized as *n*-nonadecanyl stearate (**2**) and *n*-heptacosanyl stearate (**3**) and a new aromatic ester characterized as 12-(3′,5′-dihydroxyphenyl)-*n*-dodecanyloxy pentanoyloxy methylene (**4**). The methanolic extract of the tubers of *Cyperus rotundus* furnished a fatty acid ester characterized as hexadecyl (Z)-octadec-9-enoate (palmityl oleate, **5**) and a new acyl hexaglucoside structurally elucidated as *(Z)*-tetracos-6-enoyl -O-α-D-glucopyranosyl-(6a→1b)-O-α-D-glucopyranosyl-(6b→1c)-O-α -D-glucopyranosyl-(6c→1d)-O- α -D-glucopyranosyl-(6d→1e)-O- β -D-glucopyranosyl-(6e→1f)-O-β-D-glucopyranoside (an *n*-teracos-6-enoyl O-β-D-hexaglucoside, (**6**).

**KEYWORDS:** *Butea monosperma* leaves, *Caesalpinia digyna* pods, *Cyperus rotundus* tubers, phytoconstituents, isolation, characterization.

## INTRODUCTION

*Butea monosperma* (Lam.) Taub. (family Fabaceae), known as dhak, Bengal kino, palash, and Flame of the forest, is a native to tropical and sub-tropical Indian subcontinent, Cambodia, Laos, Indonesia, Malaysia, Sri Lanka, Thailand and Vietnam. It is a small-sized, deciduous tree, trunk crooked, branches irregular; bark rough, ash coloured; leaves are pinnate, petiole 8 – 16 cm long, with three leaflets; flowers bright orange-red, racemose; fruit is a pod.[1] Its flowers are aphrodisiac, astringent, depurative, diuretic, emmenagogue, expectorant and tonic, used to treat biliousness, diarrhoea, eye diseases, gout, gonorrhoea, inflammation, leprosy, leucorrhoea, difficult micturition, skin diseases, strangury, sun stroke and thirst sensation.[2-4] The seeds are anthelmintic, antiparasitic, diuretic and purgative, utilized to subside eye diseases, inflammation, piles, scorpion sting and skin diseases.[5] The seed oil, known as Moodsga oil, is efficacious to cure abdominal troubles, skin diseases and tumours.[4] The roots are beneficial to relieve night blindness, defected eyes, elephantiasis, impotency and as an antidote for snake bite. Butea gum has anthelmintic, anti-conceptive, anticonvulsive, antidiabetic, antidiarrheal, antiestrogenic, antifertility, antimicrobial, antistress, aphrodisiac, astringent, haemagglutinating, hepatoprotective, liver tonic and wound healing activities. It is prescribed to cure coughs, diarrhoea, dysentery, bladder and stomach haemorrhage, inflammation, excessive perspiration, phthisis, piles, ringworm, stomatitis, thoracic diseases and ulcers.[2,5] The leaves are considered as an anthelmintic, aphrodisiac, appetizer, astringent, carminative, diuretic and tonic, recommended to relieve colds, colic boils, eye diseases, inflammation, lumbago, irregular menstruation, piles, pimples, sore throat and worm infestations. Petiole juice is sucked to overcome colds, cough, diabetes and stomach disorders.[2,4,6,7-10] The stem bark is anthelmintic, aphrodisiac, appetiser, astringent, bitter tonic, blood purifier, digestive, emollient and laxative, useful to prevent biliousness,

body swelling, bone fractures, catarrh, colds, cough, dysmenorrhea, dysentery, gonorrhoea, hydrocele, injury, liver disorders, piles, scorpion sting, ulcers and tumours.[2,4,7-10] The root bark is considered as an aphrodisiac, analgesic and anthelmintic; used against dropsy, piles, tumours and ulcers.[8]

The palash flowers contained butrin, butein, butin, isobutrin, coreopsin, isocoreopsin, sulphurein (glycoside), monospermoside (butein 3-β-D-glucoside) and isomonospermoside, chalcones, aurones, flavonoids (palasitrin, prunetin), sugars, amino acids and steroids.[9-14]

The pods yielded an imide.[15] The seeds afforded a fixed oil, β−sitosterol, fatty acids and palasonin.[16,17] The seed coats possessed allophanic acid derivatives.[18] The stems furnished stigmasterol, its beta-D-glucoside, nonacosanoic acid, 3-α-hydroxyeuph-25-ene and 2,14-dihydroxy-11,12-dimethyl-8-oxo-octadec-11-enylcyclohexane.[19] The gum and stem bark produced leucocynidin, tannins, mucilaginous material, pyrocatechin, kino-tannic acid, gallic acid, (-)-3 hydroxy-9-methoxypterocarpan [(-)-medicarpin], lupenone, lupeol, β-sitosterol, 5-methoxygenistein and prunetin.[20]

*Caesalpinia digyna* Rottler, syn. *C. gracilis* Miq.; *C. oleosperma* Roxb.; *Moullava digyna* (Rottler) E. Gagnon et G. P. Lewis (family Caesalpiniaceae), known as Vakeri-mul, Teri pod, is found in Tanzania, eastern Asia in China, Indian subcontinent and Malaysia to Indonesia. It is a large scandent prickly shrub, branches glabrous or slightly downy, bark brown; leaves 12-20 cm, with recurved prickles, puberulent or glabrous, leaflets obtuse, pale beneath, subsessile; flowers in simple axillary racemes, petals orbicular, yellow; pods oblong, turgid, seeds 2–4. Its root is astringent. It is taken to treat phthisis, scrofula and diabetes. Teri pods are regarded as an astringent, febrifuge, physical rejuvenative and tonic, useful to heal wounds quickly.[2,21,22] Teri pods yielded a glycoside bergenin, caesalpinines A and C. The leaves contained 2,3-dihydro-7-hydroxy-3-[(4- methoxyphenyl) methylene]-4 H-1- benzopyran-4-one. The roots afforded homoisoflavonoids - isointricatinol [(*Z*)-7,8-dihydroxy-3-(4′- methoxybenzyl) chroman-4-one], intricatinol, (Z) and (E) -8-methoxybonducellins, isobonducellin, bonducellin and (Z) and (E )-eucomines, flavonoids, bergenin, 11-*O*-galloylbergenin and gallic acid derivatives,[23-25] friedelin, hexacosanoic acid, β-sitosterol and stigmasterol.[26] The plant produced caesalpinine A, cellallocinnine, ellagic acid, gallic acid, pipecolic acid, bergenin and tannins.[27]

*Cyperus rotundus* L., syn. *C. maritimus* Bojer (family Cyperaceae), known as nagarmotha and nut grass, occurs as a weed in India and other temperate regions of the world. It is a smooth, erect and perennial herb with wiry, slender, scaly, creeping, dark and persistent rhizomes.[27] Its tubers are used as an analeptic, analgesic, anthelmintic, antimalarial, anti-obesity, antiseptic, antispasmodic, antitussive, aphrodisiac, aromatic, astringent, carminative, diaphoretic, diuretic, emmenagogue, febrifuge, litholytic, sedative, stimulant, stomachic, vermifuge, tonic and to treat amenorrhea, loss of appetite, arthritis, blisters, boils, bronchitis, cervical cancer, colic, constipation, cough, diarrhea, dysentery, dysmenorrhea, dyspepsia, dysuria, epilepsy, erysipelas, itching, eye diseases, fever, flatulence, food toxicity, indigestion, infertility, inflammation, insect bites, intestinal parasites, deficient lactation, malaria, loss of memory, menstrual disorders, metritis, nausea, nervous gastralgia, renal and vesical calculi, rheumatism, skin diseases, stomach disorders, loose teeth, thirst, urinary tenesmus, excessive thirst, vomiting, worm infestation and wounds.[28-31]

The Cyperus rhizomes contained essential oil consisted of cyperene, cyperone, nor-rotundone, isorotundone, cypera-2,4(15)-diene, cyperadione, β- selinene, anethole, cuminaldehyde and arachidic and stearic acids[28-36], sugetriol triacetate, caryophyllene, caryophylla-6-one, patchoulenone, 4,7-dimethyl-1-tetralone, 10,12-peroxycalamenene, 4,5-secoeudesmanolide, 10-epi-4,5-secoeudesmanolide, cyclic acetal cyperolone, musktakone, nootkatone, rotunols[37-39], β-sitosterol, oleanolic acid-3-O-neohesperidoside, rhamnetin 3-O-rhamnosyl rhamnopyranoside, rotundines, flavonoids, alkaloids[40,41], phenylpropanoids, phenolic acids, triterpenic glycosides[42-45], sesquiterpenic keto acid, aliphatic ketones, fatty esters, steroids and a lupenyl glycosidic ester[46, 47], (*Z*)-*n*-pentacos-19-ene-10-one, arachidyl xyloside, β-sitosterol 3-β-D-glucopyranoside and three phenyl xylosides *n*-nonadecanyl salicylate xyloside, *n*-heneicosanyl salicylate xyloside and *n*-pentacosanyl salicylate xyloside.[48]

The presence of herbal chemical constituents vary due to many factors such as geographic regions, soils, seasonal changes, plant species and application of fertilizers. Keeping in views the various therapeutic values and variation aspects of chemical constituents of the plants and development of ecofriendly, biodegradable and safer herbal preparations, it has been aimed to establish chemical structures of phytoconstituents isolated from the leaves of *Butea monosperma*, pods of *Caesalpinia digyna* and tubers of *Cyperus rotundus*.

## MATERIALS AND METHODS

The protocols of all methodologies (procedures, experimental designs and spectral data analysis) were adopted from the earlier published work.[46-48]

### Collection and authentication of plant materials

The leaves of *Butea monosperma* were collected from a wild plant in Ghaziabad (U.P.) region. The pods of *Caesalpinia digyna* and tubers of *Cyperus rotundus* were obtained from Moradabad (U.P.) on the banks of the Ramganga river. The plant materials were identified and authenticated by Prof. M. P. Sharma, Taxonomist, Department of Botany, Jamia Hamdard, New Delhi. Voucher specimens of the plant material were preserved

in the herbarium of the Department of Pharmacognosy and Phytochemistry, Jamia Hamdard, New Delhi.

**Extraction and isolation**

The leaves of *Butea monosperma*, pods of *Caesalpinia digyna* and tubers of *Cyperus rotundus* (1 kg each) were dried in air, coarsely powdered and extracted separately and exhaustively with methanol in a Soxhlet apparatus. The extracts were concentrated under reduced pressure to get dark brown masses, 211.3 g, 116.1 g and 123.8 g, respectively. Each dried residue (100 g each) was 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) individually. 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, 19:1, 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 leaves of *Butea monosperma***

**Undecanyl oleate (1)**

Elution of the column with petroleum ether gave a pale yellow semisolid mass of **1**, yield 211 mg, IR $\upsilon_{max}$ (KBr) : 2920, 2853, 1739, 1617, 1457, 1394, 1244, 1187, 1115, 722 $cm^{-1}$; $^1H$ NMR ($CDCl_3$): δ 5.37 (1H, m, H-9), 5.32 (1H, m, H-10), 4.04 (2H, t, J = 6.9 Hz, $H_2$ -1′), 2.28 (2H, t, J = 6.5 Hz, $H_2$ -2), 2.01 (2H, m, $H_2$ -8), 1.96 (2H, m, $H_2$ -11), 1.61 (6H, m, $H_2$-3, $H_2$-7, $H_2$-12), 1.34 (2H, m, $H_2$-4), 1.25 (32H, brs, 16 x $CH_2$), 0.96 (3H, t, J = 6.8 Hz, Me-11′), 0.87 (3H, t, J = 6.5 Hz, Me-18); $^{13}C$ NMR ($CDCl_3$): δ 179.20 (C-1), 33.99 (C-2), 29.23 (C-3), 29.28 (C-4), 29.67 (C-5), 29.67 (C-6), 29.67 (C-7), 31.90 (C-8), 129.99 (C-9), 128.38 (C-10), 31.18 (C-11), 29.69 (C-12), 29.63 (C-13), 29.23 (C-14), 29.11 (C-15), 25.19 (C-16), 22.66 (C-17), 14.07 (C-18), 63.81 (C-1′), 29.57 (C-2′), 29.61 (C-3′), 29.35 (C-4′), 29.45 (C-5′), 29.57 (C-6′), 29.41 (C-7′), 29.32 (C-8′), 27.19 (C-9′), 24.68 (C-10′), 15.26 (C-11′); ESI MS *m/z* (rel.int.): 436 $[M]^+$ ($C_{29}H_{56}O_2$) (1.5), 281 (8.3), 265 (14.7).

**Isolation of phytoconstituents from the pods of *Caesalpinia dygna***

***n*-Nonadecanyl stearate (2)**

Elution of the column with chloroform furnished colourless amorphous mass of **2**, 178 mg, $R_f$ 0.55 (chloroform – benzene – methanol, 2.5:2: 1.5); m. p. 126-128 °C; IR $\upsilon_{max}$ (KBr): 2926, 2854, 1726, 1630, 1493, 1445, 1381, 1276, 1187, 1081, 967, 727 $cm^{-1}$; $^1H$ NMR ($CDCl_3$): δ 4.24 (2H, t, J = 7.8 Hz, $H_2$-1′), 2.23 (2H, t, J = 7.3 Hz, $H_2$-2), 1.61 (2H, m, $H_2$-3), 1.56 (4H, m, $H_2$-2′, $H_2$-3′), 1.33 (16H, brs, 8 x $CH_2$), 1.28 (18H, brs, 9 x $CH_2$), 1.22 (24H, brs, 12 x $CH_2$), 0.89 (3H, t, J = 6.5 Hz, Me-18), 0.84 (3H, t, J = 6.3 Hz, Me-19′); $^{13}C$ NMR ($CDCl_3$): δ 172.83 (C-1), 62.14 (C-1′), 34.29 (C-2), 31.42 (C-3), 29.84 (17 x $CH_2$), 28.76 (11 x $CH_2$), 25.88 (C-16), 24.81 (C-18′), 22.67 (C-17), 16.23 (C-18), 14.17 (C-19′); ESI MS *m/z* (rel. int.): 550 $[M]^+$ ($C_{37}H_{74}O_2$) (1.8).

***n*-Heptacosanyl stearate (3)**

Elution of the column with chloroform – methanol (99:1) yielded a colourless mass of **3**, yield 211 mg, recrystallized from chloroform-methanol (1:1), m. p. 78 - 79 °C; $R_f$ 0.65 (benzene-chloroform- methanol, 2:2.5:0.5); IR $\upsilon_{max}$ (KBr) : 2925, 2853, 1726, 1445, 1363, 1276, 1086, 967, 763 $cm^{-1}$; $^1H$ NMR ($CDCl_3$): δ 4.28 (2H, t, J = 8.3 Hz, $H_2$ -1′), 2.24 (2H, t, J = 7.2 Hz, $H_2$ -2), 1.64 (2H, m, $H_2$-3), 1.59 (4H, m, $H_2$-2′, $H_2$-4), 1.33 (8H, brs, 4 x $CH_2$), 1.28 (12H, brs, 6 x $CH_2$), 1.25 (54H, brs, 27 x $CH_2$), 0.88 (3H, t, J = 6.8 Hz, Me-18), 0.84 (3H, t, J = 6.5 Hz, Me-27′); $^{13}C$ NMR ($CDCl_3$): δ 171.28 (C-1), 62.59 (C-1′), 34.31 (C-2), 33.76 (C-3), 32.85 (C-2′), 31.77 (C-4), 31.44 (C-5), 30.21 (C-3′), 29.71 (C-4′), 29.37 (29 x $CH_2$), 29.33 (C-25′), 28.68 (C-16), 25.94 (C-17), 22.70 (C-26′), 14.28 (C-18), 14.13 ( C-27′); ESI MS *m/z* (rel.int.): 662 $[M]^+$ ($C_{45}H_{90}O_2$) (2.1), 379 (4.9), 283 (2.6), 267 (8.9).

**12-(3′,5′-Dihydroxyphenyl)-n-dodecanyloxy pentanoyloxy methylene (4)**

Elution of the column with chloroform - methanol (48:1) furnished colourless yellowish green semisolid mass of **4**, recrystallized from ethyl acetate, yield 148 mg; $R_f$ 0.45 (benzene-chloroform-methanol, 2:2.5:1.5); UV λ max (MeOH): 269 nm; IR υmax (KBr): 3445, 2921, 2850, 1726, 1643, 1525, 1462, 1382, 1260, 1219, 1080, 968, 772 $cm^{-1}$; $^1H$ NMR ($CDCl_3$) : δ 7.52 (1H, dd, J = 2.1, 3.0 Hz, H-4′), 7.35 (1H, dd, J = 2.1, 2.2 Hz, H-2′), 7.13 (1H, dd, J = 2.2, 3.0 Hz, H-6′), 4.32 (2H, brs, O-$CH_2$-O), 3.74 (2H, t, J = 7.2 Hz, $H_2$-1), 2.23 (2H, t, J = 7.5 Hz, $H_2$-2″), 2.17 (2H, m, $H_2$-12), 1.57 (4H, m, $H_2$-11, $H_2$-3″), 1.33 (4H, m, $H_2$-10, $H_2$-4″), 1.29 (6H, m, $H_2$-2, $H_2$-9, $H_2$-5″), 1.25 (10 H, brs, 5 × $CH_2$), 0.86 (3H, t, J = 6.7 Hz, $H_3$-5″); $^{13}C$ NMR ( $CDCl_3$) : δ 68.92 (C-1), 37.88 (C-2), 31.44 (C-3), 31.34 (C-4), 31.43 (C-4), 30.21 (C-5), 29.71 (C-6), 29.71 (C-7), 29.71 (C-8), 29.65 (C-9), 29.57 (C-10), 27.63 (C-11), 46.24 (C-12), 138.69 (C-1′), 132.77 (C-2′), 163.18 (C-3′), 136.82 (C-4′), 151.41 (C-5′), 112.26 (C-6′), 171.98 (C-1″), 42.15 (C-2″), 29.37 (C-3″), 22.70 (C-4″), 14.13 (C-5″), 99.97 (O-$CH_2$-O); ESI MS *m/z* (rel. int.) : 408 $[M]^+$ ($C_{24}H_{40}O_5$) (91.8), 395 (1.8), 277 (1.9), 299 (8.8).

**Isolation of phytoconstituents from the tubers of *Cyperus rotundus***

**Palmityl oleate (5)**

Elution of the column with petroleum ether – chloroform (1:1) afforded a pale yellow semisolid mass of **5**, yield 139 mg, $R_f$ 0.71 (benzene-chloroform-methanol, 5:4:1); IR $\upsilon_{max}$ (KBr): 2957, 2853, 1723, 1634, 1445, 1381, 1277, 1216, 1188, 1079, 968, 761 $cm^{-1}$; $^1H$ NMR ($CDCl_3$): δ 5.92 (1H, m, H-9), 5.81 (1H, m, H-10), 4.35 (2H, t, J = 8.5 Hz, $H_2$ -1′), 2.30 (2H, t, J = 7.2 Hz, $H_2$ -2),

2.18 (2H, m, $H_2$ -8), 2.13 (2H, m, $H_2$ -11), 1.63 (2H, m, $H_2$-3), 1.57 (2H, m, $H_2$-7), 1.37 (4H, m, $H_2$-4, $H_2$ -12), 1.31 (4H, brs, $H_2$-10, $H_2$-13), 1.29 (38H, brs, 19 x $CH_2$), 0.87 (3H, t, J = 6.5 Hz, Me-18), 0.84 (3H, t, J = 6.4 Hz, Me-16′); $^{13}$C NMR ($CDCl_3$): δ 172.24 (C-1), 38.73 (C-2), 31.93 (C-3), 30.36 (C-4), 29.70 (C-5), 29.66 (C-6), 29.70 (C-7), 34.87 (C-8), 124.46 (C-9), 119.08 (C-10), 34.52 (C-11), 30.19 (C-12), 29.52 (C-13), 29.41 (C-14), 29.32 (C-15), 29.21 (C-16), 22.69 (C-17), 14.13 (C-18), 67.13 (C-1′), 31.44 (C-2′), 29.70 C-3′), 29.71 C-4′), 29.69 C-5′), 29.66 C-6′), 29.57 C-7′), 29.46 C-8′), 29.44 C-9′), 29.36 C-10′), 29.25 C-11′), 29.07 C-12′), 28.92 C-13′), 25.53 (C-14′), 22.99 (C-15′), 14.02 (C-16′); ESI MS *m/z* (rel.int.): 506 $[M]^+$ ($C_{34}H_{66}O_2$) (51.2), 265 (3.1).

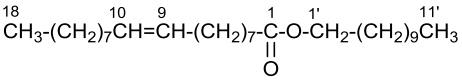
***n*-Tetracos-6-enoyl O-β-D-hexaglucoside (6)**

Elution of the column with chloroform - methanol (1:3) furnished light brown semisolid of **6**, yield 89 mg, IR $\nu_{max}$ (KBr): 3510, 3431, 3261, 2927, 2851, 1723, 1638, 1445, 1382, 1262, 1056, 923, 818, 776 cm$^{-1}$; $^1$H NMR (DMSO d-$_6$): δ 5.53 (1H, m, $w_{1/2}$ = 9.1 Hz, H-6), 5.35 (1H, m, $w_{1/2}$ = 8.7 Hz, H-7), 2.31 (2H, t, J = 7.2 Hz, $H_2$ -2), 2.27 (2H, m, $H_2$ -5), 2.18 (2H, m, $H_2$ -8), 1.65 (2H, m, $H_2$-3), 1.57 (2H, m, $H_2$-4), 1.55 (2H, m, $H_2$-9), 1.32 (8H, brs, $H_2$-10 to $H_2$-13), 1.25 (10H, brs, $H_2$-14 to $H_2$-18), 1.22 (10H, brs, $H_2$-19 to $H_2$-23), 0.87 (3H, t, J = 6.4 Hz, Me-24), 5.32 (1H, d, J = 3.6 Hz, H-1a), 4.27 (1H, m, H-5a), 3.99 (1H, m, H-2a), 3.72 (2H, m, H-3a, H-3b), 3.63 (1H, m, H-4a), 3.44 (2H, d, J = 9.6 Hz, $H_2$-6a), 5.08 (1H, d, J = 3.9 Hz, H-1b), 4.25 (1H, m, H-5b), 3.97 (1H, m, H-2b), 3.72 (2H, m, H-3a, H-3b), 3.60 (1H, m, H-4b), 3.41 (2H, d, J = 6.8 Hz, $H_2$-6b), 5.01 (1H, d, J = 4.1 Hz, H-1c), 4.17 (1H, m, H-5c), 3.94 (2H, m, H-2c, H-2d), 3.70 (2H, m, H-3c, H-3d), 3.58 (1H, m, H-4c), 3.33 (2H, d, J = 5.6 Hz, $H_2$-6c), 4.92 (1H, d, J = 3.8 Hz, H-1d), 4.15 (1H, m, H-5d), 3.94 (2H, m, H-2c, H-2d), 3.70 (2H, m, H-3c, H-3d), 3.55 (1H, m, H-4d), 3.31 (2H, d, J = 8.0 Hz, $H_2$-6d), 4.45 (1H, d, J = 7.7 Hz, H-1e), 4.10 (2H, m, H-5e, H-5f), 3.91 (2H, m, H-2e, H-2f), 3.68 (2H, m, H-3e, H-3f), 3.52 (1H, m, H-4e), 3.28 (2H, d, J = 6.8 Hz, $H_2$-6e), 4.43 (1H, d, J = 7.6 Hz, H-1f), 4.10 (2H, m, H-5e, H-5f), 3.91 (2H, m, H-2e, H-2f), 3.68 (2H, m, H-3e, H-3f), 3.50 (1H, m, H-4f), 3.05 (2H, dd, J = 6.3, 6.5 Hz, $H_2$-6f),

$^{13}$C NMR (DMSO d-$_6$): δ 171.43 (C-1), 34.93 (C-2), 31.52 (C-3), 31.24 (C-4), 33.96 (C-5), 127.18 (C-6), 118.25 (C-7), 34.07 (C-8), 29.94 (C-9), 29.94 (C-10), 29.94 (C-11), 29.78 (C-12), 29.45 (C-13), 29.37 (C-14), 29.23 (C-15), 29.12 (C-16), 29.06 (C-17), 28.92 (C-18), 28.47 (C-19), 27.18 (C-20), 25.02 (C-21), 24.89 (C-22), 22.32 (C-23), 13.89 (C-24), 105.48 (C-1a), 74.71 (C-2a), 70.21 (C-3a), 65.08 (C-4a), 81.74 (C-5a), 63.39 (C-6a), 103.80 (C-1b), 72.83 (C-2b), 70.13 (C-3b), 64.81 (C-4b), 81.22 (C-5b), 63.14 (C-6b), 101.88 (C-1c), 74.75 (C-2c), 69.33 (C-3c), 64.48 (C-4c), 78.52 (C-5c), 62.47 (C-6c), 99.74 (C-1d), 72.06 (C-2d), 68.01 (C-3d), 64.48 (C-4d), 76.52 (C-5d), 62.39 (C-6d), 97.96 (C-1e), 71.97 (C-2e), 69.33 (C-3e), 64.81 (C-4e), 76.35 (C-5e), 61.37 (C-6e), 96.68 (C-1f), 74.71 (C-2f), 68.01 (C-3f), 64.09 (C-4f), 76.35 (C-5f), 61.30 (C-6f); ESI MS *m/z* (rel.int.): 1338 $[M]^+$ ($C_{60}H_{106}O_{32}$) (1.3), 365 (100), 101 (13.6).

## RESULTS AND DISCUSSION

Compound **1** showed IR absorption bands for an ester group (1739 cm$^{-1}$) and long aliphatic chain (722 cm$^{-1}$). Its mass spectrum exhibited a molecular ion peak at *m/z* 436 consistent with the molecular formula of a fatty acid ester, $C_{29}H_{56}O_2$. The generation of the ion peaks at *m/z* 281 [$C_{1'}$ - O fission, $CH_3(CH_2)_7$-CH=CH$(CH_2)_7$-$COO]^+$ and 265 [$C_1$ - O fission, $CH_3(CH_2)_7$-CH=CH$(CH_2)_7$-$CO]^+$ indicated that oleic acid was esterified with undecanol. The $^1$H NMR spectrum of **1** displayed two one-proton deshielded multiplets at δ 5.37 and 5.32 assigned to vinylic H-9 and H-10 protons and two two-proton triplets at δ 4.04 (J = 6.9 Hz, $H_2$ -1′) and 2.28 (J = 6.5 Hz, $H_2$ -2) ascribed to oxymethylene $H_2$-1′ and methylene $H_2$ -2 protons adjacent to the ester function, respectively. The remaining methylene protons appeared as multiplets at δ 2.01 (2H), 1.96 (2H), 1.61 (6H) and 1.34 (2H) and as a broad singlet at δ 1.29 (32H). Two three-proton triplets at δ 0.96 (J = 6.8 Hz) and 0.87 (J = 6.50 Hz) were due to correspondingly C-11′ and C-18 primary methyl protons. The $^{13}$C NMR spectrum of **1** showed signals for ester carbon at δ 179.20 (C-1), vinylic carbons at δ 129.99 (C-9) and 128.38 (C-10), oxymethylene carbon at δ 63.81 (C-1′), other methylene carbons between δ 33.99 - 22.66 and methyl carbons at δ 14.07 (C-24) and 15.26 (C-11′). On the basis of these spectral data analysis, the structure of **1** has been elucidated as undecanyl oleate, a rare fatty acid ester (Fig. 1).




Undecanyl oleate **(1)**

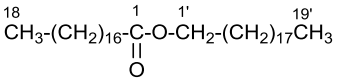
**Fig. 1: Chemical constituent 1 isolated from the leaves of *Butea monosperma* (Lam.) Taub.**

Compound **2** is a known fatty acid ester identified as *n*-nonadecanyl stearate (Fig. 2), earlier isolated from the roots of *Saussurea costus*.[49]

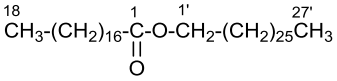
Compound **3**, designated as *n*-heptacosanyl stearate, displayed distinctive IR absorption bands for an ester function (1726 cm$^{-1}$) and long aliphatic chain (763 cm$^{-1}$). Its mass spectrum showed a molecular ion peak at *m/z* 662 consistent with a molecular formula of a fatty acid ester, $C_{45}H_{90}O_2$. The formation of the ion peaks at *m/z* 267 [$C_1$ - O fission, $CH_3(CH_2)_{16}$-$CO]^+$, 283 [$C_{1'}$ - O fission, $CH_3(CH_2)_{16}$-CO) $]^+$ and 379 [M – 283, $CH_2$-$(CH_2)_{25}$-$CH_3]^+$ indicated that stearic acid was esterified with *n*-heptacosanol. The $^1$H NMR spectrum of **3** exhibited two two-proton triplets at δ 4.28 (J = 8.3 Hz) and 2.24 (J = 7.2 Hz) assigned to oxymethylene $H_2$-1′ and methylene $H_2$ -2 protons nearby to the ester function, respectively. The remaining methylene protons appeared as multiplets at δ 1.64 (2H) and 1.59 (4H) and as broad singlets at δ 1.33 (8H), 1.28 (12H) and 1.25 (54H). Two three-proton triplets at δ 0.88 (J = 6.8 Hz) and 0.84 (J =

6.5 Hz) were due to C-18 and C-27′ primary methyl protons, respectively. The $^{13}$C NMR spectrum of **3** showed signals for the ester carbon at δ 171.28 (C-1), oxymethylene carbon at δ 62.59 (C-1′), other methylene carbons between δ 34.31 – 22.70 and methyl carbons at δ 14.28 (C-18) and 14.13 (C-27′). The absence of any $^{1}$H NMR signal beyond δ 4.28 and carbon signals between δ 171.28 – 62.59 supported the saturated nature of the molecule. On the basis of these spectral data analysis evidences, the structure of **3** was elucidated as *n*-heptacosanyl stearate (Fig. 2).

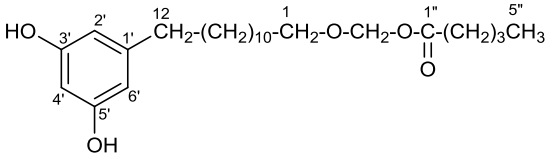
Compound **4** gave positive tests of phenols, showed UV absorption maximum at 269 nm for aromatic ring and IR absorption bands for hydroxyl groups (3445 cm$^{-1}$), ester function (1726 cm$^{-1}$), aromatic ring (1643, 1525 cm$^{-1}$) and aliphatic chain (772 cm$^{-1}$). On the basis of mass and $^{13}$C NMR spectra its molecular ion peak was determined at *m/z* 408 consistent with a molecular formula of a dihydroxyphenyl dodecanoyl ester, $C_{24}H_{40}O_5$. The ion peaks arising at *m/z* 299 [$C_{12}$- $C_{1'}$ fission, $(CH_2)_{12}$-$OCH_2O$-CO-$(CH_2)_3CH_3$]$^+$ and 277 [$C_1$ - O fission, $C_6H_3(OH)_2$-$CH_2$-$(CH_2)_{10}$-$CH_2$]$^+$ indicated that dihydroxyphenyl dodecanoyl was esterified with oxymethylene pentanoate. The $^{1}$H NMR spectrum of **4** displayed three one-proton double doublets at δ 7.52 (J = 2.1, 3.0 Hz), 7.35 (J = 2.1, 2.2 Hz) and 7.13 (J = 2.2, 3.0 Hz) assigned to meta-coupled aromatic H-4′, H-2′ and H-6′ protons, respectively, a two-proton singlet at δ 4.32 ascribed to dioxymethylene protons, two two-proton triplets at δ 3.74 (J = 7.2 Hz) and 2.23 (J = 7.5 Hz) attributed to oxymethylene $H_2$-1 and methylene $H_2$-2″ protons adjacent to the ester function, other methylene protons as multiplets at δ 2.17 (2H), 1.57 (4H), 1.33 (4H), 1.29 (6H) and as a singlet 1.25 (10H) and a three-proton triplet δ 0.86 (J = 6.7 Hz) accounted to terminal C-5″ primary methyl protons. The $^{13}$C NMR spectrum of **4** exhibited signals for the ester carbon at δ 171.98 (C-1″), aromatic carbons between δ 163.18 – 112.26, di-oxymethylene carbon at δ 99.97 (O-$CH_2$-O), oxymethylene carbon at δ 68.92 (C-1), other methylene carbons from δ 42.15 to 22.70 and methyl carbon at δ 14.13 (C-5″). On the basis of the aforementioned spectral data analysis the structure of **4** has been elucidated as 12-(3′,5′-dihydroxyphenyl)-*n*-dodecanyloxy pentanoyloxy methylene, a new aromatic ester (Fig. 2).




*n*-Nonadecanyl oleate **(2)**




*n*-Heptacosanyl stearate **(3)**



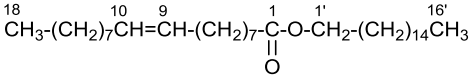

12-(3′,5′-Dihydroxy phenyl) *n*-dodecanyloxy pentanoyloxy methylene **(4)**

**Fig. 2: Chemical constituents 2, 3 and 4 isolated from the pods of *Caesalpinia digyna* Rottler.**

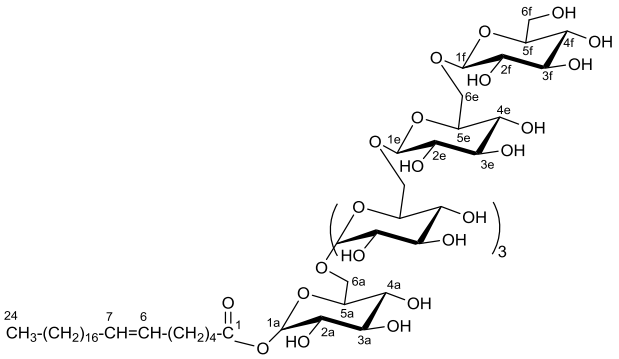
The compounds **5** was a common fatty ester characterized as hexadecyl (Z)-octadec-9-enoate (palmityl oleate) (Fig. 3).[50]

Compound **6**, named *n*-teracos-6-enoyl O-β-D-hexaglucoside, gave positive tests for glycosides and showed IR absorption bands for hydroxyl groups (3510, 3431, 3261 cm$^{-1}$), ester function (1723 cm$^{-1}$), unsaturation (1638 cm$^{-1}$) and aliphatic chain (776 cm$^{-1}$). On the basis of mass and $^{13}$C NMR spectral data, the molecular ion peak of **6** was established at *m/z* 1338 consistent with a molecular formula of an acyl hexaglycoside, $C_{60}H_{106}O_{32}$. An ion peak generating at *m/z* 365 [O-$C_1$ fission, $C_{24}H_{45}O_2$]$^+$ suggested that *n*-tetracosenoyl unit was linked with a hexaglycosidic moiety. An ion fragment arising at *m/z* 101 [$(CH_2)_4$-COO]$^+$ indicated the presence of the vinylic linkage at C-6 carbon position. The $^{1}$H NMR spectrum of **6** exhibited two one-proton multiplets at δ 5.53 ($w_{1/2}$ = 9.1 Hz) and 5.35 ($w_{1/2}$ = 8.7 Hz) were ascribed to *cis*-oriented vinylic H-6 and H-7 protons, respectively. A two-proton triplet at δ 2.31 (J = 7.2 Hz) was attributed to methylene $H_2$-2 protons adjacent to the ester group. A three-proton triplet at δ 0.87 (J = 6.4 Hz) was accounted to terminal C-24 primary methyl protons. The remaining methylene protons appeared as two - protons multiplets at δ 2.27 ($H_2$ -5), 2.18 ($H_2$ -8), 1.65 ($H_2$-3), 1.57 ($H_2$-4) and 1.55 ($H_2$-9) and as broad singlets at δ 1.32 (8H), 1.25 (10H) and 1.22 (10H). Six one - proton doublets at δ 5.32 (J = 3.6 Hz), 5.08 (J = 3.9 Hz), 5.01 (J = 4.1 Hz), 4.92 (J = 3.8 Hz), 4.45 (J = 7.7 Hz) and 4.43 (J = 7.6 Hz) were assigned correspondingly to α-oriented sugar anomeric H-1a, H-1b, H-1c and H-1d and β-oriented H-1e and H-1f protons. The other sugar protons resonated from δ 4.27 to 3.05. The $^{13}$C NMR spectrum of **6** displayed signals for ester carbon at δ 171.43 (C-1), vinylic carbons at δ 127.18 (C-6) and 118,25 (C-7), methylene carbons from δ 34.93 to 22.32, terminal methyl carbon at δ 13.89 (C-24), six anomeric carbons at δ δ 105.48 (C-1a), 103.80 (C-1b), 101.88 (C-1c), 99.74 (C-1d), 97.96 (C-1e) and 96.68 (C-1f), sugar oxymethine carbons between δ 81.74 – 64.09 and sugar oxymethylene carbons in the range of δ 63.39 to 61.30. The presence of downfield signals of oxymethylene protons as two-proton doublets at δ 3.44 (2H, d, J = 9.6 Hz, $H_2$-6a), 3.41 (2H, d, J = 6.8 Hz, $H_2$-6b), 3.33 (2H, d, J = 5.6 Hz, $H_2$-6c), 3.31 (2H, d, J = 8.0 Hz, $H_2$-6d) and 3.28 (2H, d, J = 6.8 Hz, $H_2$-6e) in the $^{1}$H NMR spectrum and carbon signals at δ 63.39 (C-6a), 63.14 (C-6b), 62.47 (C-6c), 62.39 (C-6d) and 61.37 (C-6e) in the $^{13}$C NMR spectrum

suggested the (6→1) linkages among sugar units. Acid hydrolysis of **6** yielded D-glucose, $R_f$ 0.26 (*n*-butanol-acetic acid – water, 4: 1: 5). On the basis of above discussion, the compound **6** was structurally elucidated as *(Z)*-tetracos-6-enoyl -O-α-D-glucopyranosyl-(6a→1b)-O-α-D-glucopyranosyl-(6b→1c)-O-α -D-glucopyranosyl-(6c→1d)-O- α -D-glucopyranosyl-(6d→1e)-O- β -D-glucopyranosyl-(6e→1f)-O-β-D-glucopyranoside, a new acyl hexaglucoside (Fig 3).



Palmityl oleate **(5)**



*n*-Tetracos-6-enoyl *O*-β-D-hexaglucoside **(6)**

**Fig. 3: Chemical constituents 5 and 6 isolated from the tubers of *Cyperus rotundus* L.**

## CONCLUSION

Phytochemical investigation of the leaves of *Butea monosperma* gave a fatty acid ester identified as undecanyl oleate **(1).** The pods methanolic of *Caesalpinia digyna* afforded two fatty acid ester recognized as *n*-nonadecanyl stearate (**2**) and *n*-heptacosanyl stearate (**3**) and a new aromatic ester characterized as 12-(3′,5′-dihydroxyphenyl)-*n*-dodecanyloxy pentanoyloxy methylene (**4**). The methanolic extract of the tubers of *Cyperus rotundus* furnished a fatty acid ester and its structure was established as hexadecyl (Z)-octadec-9-enoate (palmityl oleate, **5**) and a new acyl hexaglucoside structurally elucidated as *(Z)*-tetracos-6-enoyl -O-α-D-glucopyranosyl-(6a→1b)-O-α-D-glucopyranosyl-(6b→1c)-O-α -D-glucopyranosyl-(6c→1d)-O- α -D-glucopyranosyl-(6d→1e)-O- β -D-glucopyranosyl-(6e→1f)-O-β-D-glucopyranoside (an *n*-teracos-6-enoyl O-β-D-hexaglucoside, **6**). This work has enhanced understanding about the chemical constituents of the undertaken plants. Further research is recommended to screen bioactivities of the isolated phytoconstituents with a view for supplementing conventional drug development especially in developing countries.

## ACKNOWLEDGEMENT

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