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Photochemical Investigation of the Roots of Jatropha Integerrima Jacq

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Abstract

Jatropha integerrima Jacq. (Euphorbiaceae) is grown in many tropical and subtropical countries and used to treat eczema, herpes, ringworm, rheumatism, pruritus, scabies, toothaches, tumors and warts. Its latex is toxic. A methanolic extract of the roots was chromatographed over silica gel column packed in petroleum ether. The column was eluted with petroleum ether, chloroform and methanol successively in order of increasing polarity to isolate n-pentadecanyl oleate (**1**), α-D-glucopyranosyl-(2→1')-O-α-D-glucopyranoside (α-D-diglucoside, **2**), n-hexanoyl-O- α-D-glucopyranosyl-(2a→1b)-O- α-D-glucopyranosyl-(2b→1c)-O- α-D-glucopyranosyl-(2c→1d)-O- α-D-glucopyranosyl-(2d→1e)-O- α-D-glucopyranoside (n-caproyl O-α-D-pentaglucoside, **3**), n-hexanoyl-O-β-D-glucopyranosyl-(2a→1b)-O-β-D-glucopyranosyl-(2b→1c)-O-β-D-glucopyranosyl-(2c→1d)-O-β-D-glucopyranosyl-(2d→1e)-O-β-D-glucopyranosyl-(2e→1f)-O-β-D-glucopyranoside (n-caproyl O-β-D-hexaglucoside, **4**), α-D-glucopyranosyl-(2→1a)-O- α-D-glucopyranosyl-(2a→1b)-O- α-D-glucopyranosyl-(2b→1c)-O- α-D-glucopyranosyl-(2c→1d)-O- α-D-glucopyranosyl-(2d→1e)-O- α-D-glucopyranoside (α-D-hexaglucoside **5**) and α-D-glucopyranosyl-(2→1a)-O- α-D-glucopyranosyl-(2a→1b)-O- α-D-glucopyranosyl-(2b→1c)-O- α-D-glucopyranosyl-(2c→1d)-O- α-D-glucopyranosyl-(2d→1e)-O- α-D-glucopyranosyl-(2e→1f)-O-α-D-glucopyranoside (α-D-heptaglucoside, **6**) .The structures of these compounds have been established on the basis of spectral data analysis and chemical means.

Keywords: Jatropha Integerrima; Roots; Chemical Constituents; Isolation; Characterization.

1. Introduction

Jatrophaintegerrima Jacq., syn. J. acuminate Desr., J. coccinea Link, J. diversifolia A.Rich., J.pandurifolia Andrews(Euphorbiaceae), commonly known as peregrina, spicy jatropha, firecracker jatroph and coral plant, is a native to the West Indies, Cuba, Dominican Republic and Haiti, but widely grown for ornament in many tropical and subtropical countries including southern India, Philippines. Thailand and South America. It is a slender-stemmed, multi-trunked tropical evergreen tree or large shrub, up to 5 m in height with an equal spread, and has unusual long glossy green, oblong, fiddle-shaped, or even-lobed leaves with beautiful clusters of scarlet flowers and seeded capsules [1-3]. It should be kept out of the reach of children. It is related to Jatropha cruces that are grown commercially to produce oil used in biodiesel fuel. The plant is used as an emetic, purgative, styptic and to treat eczema, herpes, ringworm, rheumatism, pruritus, scabies, toothaches, tumors and warts [4, 5]. Its latex is toxic, the leaves are purgative and if accidentally chewed, can cause squeamish and stomachalgia [6]. The latex contained cyclic peptides, integerrimides A, B and C [5, 7 - 9]. The leaf essential oil was composed of pentad canal, 1, 8-cineole, and β-ionone as the major components. The seed oil was comprised mainly of aliphatic hydrocarbons pentacosane, hexacosane, octacosane and heptacosane and fatty acids viz., palmitic, ricinoleic, steraric, oleic, linoleic, linolenicand arachidic acids [5]. However, the defatted seed oil was consisted of the monoterpenes 1, 8-cineole, p-cymene and α-pinene [10]. The trunks possessed sesquineolignan enantiomers, jatrointelignans A and B, neolignans jatrointelignans C and D and schisphenlignan [11]. The roots yielded

hydroxyjatropholones, 1,5-dioxo-2,3-dihydroxyrhamnofola-4(10),6,11(18),15-tetraene, 2-keto-5-hydroxyguai-3,11-diene, an jatrophadioxan, integerrimene, endoperoxide 2-epicaniojane, caniojane, 1,11-bisepicaniojane; 8,9-seco-rhamnofolane and endoperoxide 2-epicaniojane [6, 12]. The stem bark contained 3-O- acetylaleuritolic acid, jatropholones A and B, hydroxyljatropholone, scopoletin and aleuritolic acid 3-p- hydroxycinnamate [13]. The physicochemical parameter has been generated to ascertain the authenticity and quality of the roots [14]. The manuscript describes isolation and characterization of a fatty ester, caproyl glucosides and di-, hexa- and heptaglucosides from the roots of J. integerrima.

2 . Experimental

2.1 General Procedures

Melting points were determined on a Perfit melting apparatus (Ambala, Haryana, India) and are uncorrected. UV spectra were measured with a Lambda Bio 20 spectrophotometer (Perkin Elmer-Rotkreuz, Switzerland) in methanol. Infrared spectra were recorded on Bio-Rad FTIR 5000 spectrophotometer (FTS 135, Kawloon, and Hong Hong) using KBr pellets; γmax values are given in cm^{-1}. The ^{1}H and ^{13}C NMR spectra were screened on Advance DRX Bruker spectrospin 400 and 100 MHz, respectively, instruments (Karlesruthe, Germany) using $CDCl_3$ or DMSO-d_6 as a solvent and TMS as an internal standard. Mass spectra were scanned by effecting ionization at 70 eV on a JEOL-JMS-DX 303 spectrometer (Japan) equipped with direct inlet probe system. Column chromatography was performed on silica gel (60-120 mesh; Qualigen, Mumbai, India). TLC was run on silica gel G (Qualigen). Spots were visualized by exposing to iodine vapours, UV radiation and spraying with ceric sulphate solution.

2.2 Plant Material

The roots of J.integerrima were collected from Hissar, Haryana and identified by Dr. H. B. Singh, Head, Raw Materials Herbarium and Museum, NISCAIR, New Delhi under voucher specimen no. NISCAIR/RHMD/11/12/1887/187. A voucher specimen has been retained in the Department of Pharmaceutical Sciences, G J University of Science and Technology, Hissar, Haryana.

2.3 Preparation of Extract

The dried roots (1.0 kg) were coarsely powdered and exhaustively extracted in a Soxhlet apparatus with methanol. The methanolic extract was concentrated under reduced pressure to yield a dark brown viscous mass (118 g). A small portion of the extract was analyzed chemically to determine the presence of different chemical constituents.

2.4 Isolation of phytoconstituents

The viscous dark brown extract (100 g) was dissolved in small quantity of methanol and adsorbed onto silica gel (60 - 120 mesh) for preparation of a slurry. The slurry was air dried and subjected to chromatography over silica gel column packed in petroleum ether. The column was eluted successively with petroleum ether, mixture of petroleum ether - chloroform (9:1, 3:1, 1:1, 1:3), chloroform and the mixture of chloroform - methanol (99:1, 97:3, 95:5, 92:8, 9:1, 3:1, 1:1, 1:3). Various fractions were collected separately and matched by TLC to check the homogeneity. Similar fractions having the same R_f values were combined and crystallized. The isolated compounds were recrystallized to get the following compounds:

2.5 n-Pentadecanyl oleate (1)

Elution of the column with chloroform afforded colourless crystals of **1**, 223 mg (0.29 % yield), R_f: 0.21 (chloroform – methanol, 19 : 1), m.p. 96-97°C; IR ν_{max}(KBr): 2924, 2839, 1725, 1641, 1435, 1365, 1258, 1031, 721 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 5.33 (1H, m, H-9), 5.29 (1H, m, H-10), 3.69 (2H, t, J = 6.4 Hz, H_2-1'), 2.46 (2H, t, , J = 7.3 Hz, H_2-2), 2.20 (2H, m, H_2-8), 2.01 (2H, H_2-11), 1.49 (2H, m, CH_2), 1.24 (46H, brs, 23×CH_2), 0.86 (3H, t, J = 6.1 Hz, Me-18), 0.83 (3H, t, J = 6.3 Hz, Me-15');^{13}C NMR ($CDCl_3$): δ 173.83 (C-1), 51.84 (C-2), 123.62 (C-9), 117.65 (C-10), 68.21 (C-1'), 32.01 – 22.57 (26 x CH_2), 14.68 (C-18), 14.41 (C-15'); TOF MS m/z (rel.int.): 492 $[M]^+$ ($C_{33}H_{64}O_2$) (60.3), 265 (8.9).

2.6 Diglucoside (2) □-D-

Elution of the column with chloroform - methanol (9:1) afforded light brown crystals of **2**, 269 mg (0.36 % yield), R_f: 0.24 (chloroform – methanol, 17 : 3), m.p. 120 – 121°C; IR ν_{max}(KBr): 3515, 3312, 3260, 2920, 2837, 1460, 1355, 1260, 835 cm^{-1}; ^{1}H NMR (DMSO d-$_6$): δ 5.23 (1H, d, J = 3.2 Hz, H-1), 4.56 (1H, m, H-2), 4.45 (1H, m, H-5), 3.89 (1H, dd, J = 7.2, 7.6 Hz, H-3), 3.66 (1H, m, H-4), 3.18 (2H, dd, J = 5.6, 7.6 Hz, H_2-6), 5.19 (1H, d, J = 4.0 Hz, H-1'), 4.41 (1H, m, H-5'), 4.05 (1H, m, H-2'), 3.78 (1H, m, H-3'), 3.52 (1H, m, H-4'), 3.13 (2H, d, J = 9.2 Hz, H_2-6');^{13}C NMR (DMSO d-$_6$): δ 104.50 (C-1), 83.01 (C-2), 73.27 (C-3), 70.31 (C-4), 77.58 (C-5), 62.57 (C-6), 92.22 (C-1'), 73.33 (C-2'), 72.11 (C-3'), 70.31 (C-4'), 64.79 (C-5'), 60.93 (C-6'); TOF MS m/z (rel.int.): 342 $[M]^+$ ($C_{12}H_{22}O_{11}$) (1.2).

2.7 n-Caproyl O-α-D-pentaglucoside(3)

Elution of the column with chloroform - methanol (19:1) furnished light brown crystals of **3**, 245 mg (0.327 % yield), R_f: 0.46 (chloroform – methanol, 19 : 1), m.p. 125-126°C; IR ν_{max}(KBr): 3510, 3455, 3317, 2970, 2842, 1724, 1636, 1435, 1279, 1035 cm^{-1}; ^{1}H NMR (DMSO d-$_6$): δ 5.33 (1H, d, J = 4.8 Hz, H-1a), 5.31 (1H, d, J = 3.6 Hz, H-1b), 5.21 (1H, d, , J = 4.4 Hz, H-1c), 5.18 (1H, d, , J = 3.6 Hz, H-1d), 4.97 (1H, d, , J = 2.0 Hz, H-1e), 4.79 (1H, m, H-2a), 4.68 (1H, m, H-2b), 4.55 (1H, m, H-2c), 4.48 (1H, m, H-2d), 4.36 (3H, m, H-5a, H-5b, H-5c), 4.32 (1H, m, H-5d), 4.28 (1H, m, H-5e), 3.44 (1H, m, H-3a), 3.41 (2H, m, H-3b, H-3c), 3.39 (1H, m, H-3d), 3.37 (1H, m, H-3e), 3.36 (1H, m, H-4a), 3.35 (1H, m, H-4b), 3.31 (2H, m, H-4c, H-4d), 3.30 (1H, m, H-4e), 3.19 (2H, d, J = 6.5 Hz, H_2-6a), 3.17 (2H, d, J = 7.3 Hz, H_2-6b), 3.12 (2H, d, J = 6.8 Hz, H_2-6c), 3.10 (2H, d, J = 6.5 Hz, H_2-6d), 3.50 (2H, d, J = 8.8 Hz, H_2-6e), 2.51 (2H, t, J=7.2 Hz, H_2-2), 1.33 (2H, m, CH_2), 1.23 (4H, brs, 2×CH_2), 0.81 (3H, t, J = 6.3 Hz, Me-6); ^{13}C NMR (DMSO d-$_6$): δ 173.16 (C-1), 33.41 (C-2), 29.44 (C-3), 29.44 (C-4), 22.67 (C-5), 18.21 (C-6), 102.45 (C-1a), 82.51 (C-2a), 72.95 (C-3a), 68.15 (C-4a), 77.19 (C-5a), 60.15 (C-6a), 98.51 (C-1b), 82.35 (C-2b), 72.83 (C-3b), 68.27 (C-4b), 75.79 (C-5b), 61.66 (C-6b), 97.35 (C-1c), 82.35 (C-2c), 72.40 (C-3c), 69.63 (C-4c), 76.12 (C-5c), 61.63 (C-6c), 94.49 (C-1d), 81.23 (C-2d), 72.01 (C-3d), 63.52 (C-4d), 75.30 (C-5d), 61.68 (C-6d), 93.03 (C-1e), 73.53 (C-2e), 71.04 (C-3e), 63.45 (C-4e), 75.30 (C-5e), 61.66 (C-6e); TOF MS m/z (rel.int.): 926 $[M]^+$ ($C_{36}H_{62}O_{27}$) (2.1).

2.8 n-Caproyl O-β-D-hexaglucoside (4)

Elution of the column with chloroform - methanol (3:1) furnished brown crystals of (**4**), 381 mg (0.51 % yield), R_f: 0.11 (chloroform – methanol, 3 : 1), m.p. 121-123°C; IR ν_{max}(KBr): 3490, 3365, 3280, 2927, 2842, 1721, 1636, 1455, 1365, 1259, 1072 cm^{-1}; ^{1}H NMR (DMSO d-$_6$): δ 5.21 (1H, d, J = 7.2 Hz, H-1a), 5.09 (1H, d, J = 7.3 Hz, H-1b), 5.02 (1H, d, J = 7.1 Hz, H-1c), 4.98 (1H, d, J = 7.5 Hz, H-1d), 4.90 (2H, brs, H-1e, 1f), 4.53 (1H, m, H-2a), 4.45 (2H, m, H-2b, H-2c), 4.28 (1H, m, H-2d), 4.19 (1H, m, H-2e), 4.84 (1H, m, H-5a), 3.81 (1H, m, H-5b), 3.78 (2H, m, H-5c, H-5d), 3.75 (1H, m, H-5e), 3.70 (1H, m, H-5f), 3.68 (1H, m, H-2f), 3.63 (1H, m, H-3a), 3.61 (1H, m, H-3b), 3.58 (1H, m, H-3c), 3.55 (1H, m, H-3d), 3.51 (1H, m, H-3e), 3.47 (1H, m, H-3f), 3.44 (2H, m, H-4a, H-4b), 3.41 (1H, m, H-4c), 3.37 (2H, m, H-4d, H-4e), 3.32 (1H, m, H-4f), 3.20 (2H, d, J = 6.5 Hz, H_2-6a), 3.18 (2H, d, J = 6.8 Hz, H_2-6b), 3.16 (2H, d, J = 6.8 Hz, H_2-6c), 3.14 (2H, d, J = 6.6 Hz, H_2-6d), 3.12 (2H, d, J = 6.5 Hz, H_2-6e), 3.09 (2H, d, J = 6.3 Hz, H_2-6f), 2.35 (2H, t, J = 7.3 Hz, H_2-2), 1.23 (6H, brs, 3×CH_2), 0.85 (3H, t, J = 6.5 Hz, Me-6); ^{13}C NMR (DMSO d-$_6$): δ 170.27 (C-1), 31.83 (C-2), 29.45 (C-3), 29.45 (C-4), 22.69 (C-5), 18.49 (C-6), 104.63 (C-1a), 83.38 (C-2a), 71.95 (C-3a), 64.47 (C-4a), 77.22 (C-5a), 62.43 (C-6a), 102.45 (C-1b), 82.36 (C-2b), 70.91 (C-3b), 64.86 (C-4b), 76.39 (C-5b), 62.41 (C-6b), 98.49 (C-1c), 81.45 (C-2c), 70.67 (C-3c), 63.78 (C-4c), 76.25 (C-5c), 62.38 (C-6c), 97.36 (C-1d), 81.45 (C-2d), 70.38 (C-3d), 63.78 (C-4d), 75.82 (C-5d), 62.36 (C-6d), 93.49 (C-1e), 80.31 (C-2e), 69.65 (C-3e), 63.54 (C-4e), 75.82 (C-5e), 61.58 (C-6e), 92.02 (C-1f), 72.93 (C-2f), 68.16 (C-3f), 63.48 (C-4f), 73.75 (C-5f), 61.58 (C-6f); TOF MS m/z (rel.int.): 1088 $[M]^+$ ($C_{42}H_{72}O_{32}$) (1.3).

2.9 α-D-Hexaglucoside(5)

Elution of the column with chloroform - methanol (1:1) furnished colourless crystals of **5**, 452 mg (0.6 % yield), R_f: 0.47 (chloroform – methanol, 1 : 1), m.p. 108-109°C; IR ν_{max}(KBr): 3470, 3347, 3290, 2917, 2823, 1412, 1311 1320, 1271, 1132, 1051, 941 cm^{-1}; ^{1}H NMR (DMSO d-$_6$): δ 5.17 (1H, d, J = 3.6 Hz, H-1), 4.91 (1H, d, J = 3.2 Hz, H-1a), 4.88 (1H, d, , J = 3.2 Hz, H-1b), 4.86 (1H, d, , J = 3.9 Hz, H-1c), 4.71 (2H, brs, H-1d, H-1e), 4.28 (1H, m, H-2), 4.26 (2H, m, H-2a, H-2b), 4.24 (2H, m, H-2c, H-2d), 3.86 (1H, m, H-5), 3.83 (1H, m, H-5a), 3.81 (1H, m, H-5b), 3.79 (1H, m, H-5c), 3.75 (1H, m, H-5d), 3.73 (1H, m, H-5e), 3.71 (1H, m, H-2e), 3.65 (1H, m, H-3), 3.62 (1H, m, H-3a), 3.58 (1H, m, H-3b), 3.55 (1H, m, H-3c), 3.52 (1H, m, H-3d), 3.50 (1H, m, H-3e), 3.46 (1H, m, H-4), 3.43 (2H, m, H-4a, H-4b), 3.34 (1H, m, H-4c), 3.31 (1H, m, H-4d), 3.25 (1H, m, H-4e), 3.18 (2H, d, J = 10.8 Hz, H_2-6), 3.15 (2H, d, J = 8.0 Hz, H_2-6a),3.13 (J = 9.2 Hz, H_2-6b), 3.11 (2H, d, J = 10.8 Hz, H_2-6c), 3.08 (2H, d, J = 10.8 Hz, H_2-6d), 3.05 (2H, d, J = 8.0 Hz, H_2-6e); ^{13}C NMR (DMSO d-$_6$): δ 104.19 (C-1), 83.21 (C-2), 73.26 (C-3), 69.54 (C-4), 77.07 (C-5), 61.64 (C-6), 102.36 (C-1a), 82.87 (C-2a), 73.19 (C-3a), 68.18 (C-4a), 77.05 (C-5a), 62.60 (C-6a), 98.48 (C-1b), 82.44 (C-2b), 72.67 (C-3b), 64.69 (C-4b), 76.43 (C-5b), 62.56 (C-6b), 97.23 (C-1c), 82.22 (C-2c), 72.28 (C-3c), 64.04 (C-4c), 75.69 (C-5c), 61.57 (C-6c), 92.58 (C-1d), 81.47 (C-2d), 71.04 (C-3d), 63.50 (C-4d), 75.52 (C-5d), 61.54 (C-6d), 92.20 (C-1e), 74.76 (C-2e), 71.16 (C-3e), 63.29 (C-4e), 75.07 (C-5e), 60.86 (C-6e); TOF MS m/z (rel.int.): 990 $[M]^+$ ($C_{36}H_{62}O_{31}$) (2.8).

2.10 α-D-Heptaglucoside(6)

Elution of the column with chloroform - methanol (3:1) yielded brown crystals of **6**, 429 mg (0.573 % yield), R_f: 0.34 (chloroform – methanol, 1 : 1), m.p. 99-101°C; IR ν_{max}(KBr): 3550, 3465, 3327, 3245, 2917, 2842, 1455, 1050, 952 cm^{-1}; ^{1}H NMR (DMSO d-$_6$): δ 5.17 (1H, d, J = 3.2 Hz, H-1), 4.91 (1H, d, J = 3.2 Hz, H-1a), 4.87 (1H, d, , J = 3.2 Hz, H-1b), 4.85 (1H, d, J = 4.3 Hz, H-1c), 4.81 (1H, d, J = 3.8 Hz, H-1d), 4.68 (1H, d, J = 4.4 Hz, H-1e), 4.65 (1H, d, J = 4.1 Hz, H-1f), 4.28 (2H, m, H-2, H-2a), 4.26 (2H, m, H-2b, H-2c), 3.92 (2H, m, H-2d, H-2e), 3.88 (1H, m, H-5), 3.86 (1H, m, H-5a), 3.82 (1H, m, H-5b), 3.80 (1H, m, H-5c), 3.78 (1H, m, H-5d), 3.74 (1H, m, H-5e), 3.72 (1H, m, H-5f), 3.67 (1H, m, H-2f), 3.64 (1H, m, H-3), 3.62 (1H, m, H-3a), 3.58 (1H, m, H-3b), 3.54 (1H, m, H-3c), 3.51 (2H, m, H-3d, H-3e), 3.48 (1H, m,

H-3f), 3.46 (1H, m, H-4), 3.43 (2H, m, H-4a, H-4b), 3.41 (2H, m, H-4c, H-4d), 3.35 (2H, m, H-4e, H-4f), 3.21 (2H, d, J = 6.8 Hz, H_2-6), 3.19 (2H, d, J = 5.6 Hz, H_2-6a), 3.15 (2H, d, J = 5.8 Hz, H_2-6b), 3.13 (2H, d, J = 5.6 Hz, H_2-6c), 3.11 (2H, d, J = 5.4 Hz, H_2-6d), 3.08 (2H, d, J = 8.2 Hz, H_2-6e), 3.04 (2H, d, J = 7.6 Hz, H_2-6f); ^{13}C NMR (DMSO d-$_6$): δ 104.66 (C-1), 83.15 (C-2), 73.23 (C-3), 71.79 (C-4), 76.44 (C-5), 60.86 (C-6), 104.38 (C-1a), 82.81 (C-2a), 73.15 (C-3a), 71.13 (C-4a), 76.26 (C-5a), 61.56 (C-6a), 103.51 (C-1b), 82.16 (C-2b), 72.77 (C-3b), 70.86 (C-4b), 75.64 (C-5b), 61.53 (C-6b), 98.46 (C-1c), 81.48 (C-2c), 72.61 (C-3c), 70.23 (C-4c), 75.18 (C-5c), 62.54 (C-6c), 97.17 (C-1d), 77.64 (C-2d), 72.44 (C-3d), 69.96 (C-4d), 74.74 (C-5d), 62.59 (C-6d), 92.54 (C-1e), 77.01 (C-2e), 72.23 (C-3e), 69.51 (C-4e), 76.44 (C-5e), 60.86 (C-6e), 92.19 (C-1f), 73.43 (C-2f), 72.13 (68.63), 74.74 (C-5f), 62.54 (C-6f); TOF MS m/z (rel.int.): 1152 $[M]^+$ ($C_{42}H_{72}O_{36}$) (1.9).

2. Results and Discussion

Compound **1**showedIR absorption bands for ester function (1725 cm^{-1}), unsaturation (1641cm^{-1}) and long aliphatic chain (721 cm^{-1}). On the basis of mass and ^{13}C NMR spectra, the molecular ion peak of **1** was determined at m/z 492 consistent to the molecular formula of an aliphatic ester ($C_{33}H_{64}O_2$). The ion fragments generating at m/z 265 [C_1- O fission, CH_3 $(CH_2)_7$ CH = CH $(CH_2)_7$ CO]$^+$suggested that oleic acid was esterified with C_{15}-aliphatic alcohol. The ^{1}H-NMR spectrum of **1** exhibited two one-proton multiplets at δ 5.33 and 5.29 assigned to vinylic H-9 and H-10 protons, respectively. A two-proton triplet at δ 3.69 (J = 6.4 Hz) was ascribed to oxygenated methylene H_2-1′ protons. A two-proton triplet at δ 2.46 (J = 7.3 Hz) was accounted to C-2 methylene protons adjacent to the ester group. The signals between δ 2.20 – 1.24 were due to other methylene protons. Two three-proton triplets at δ 0.86 (J = 6.1 Hz) and 0.83 (J=6.1 Hz) were associated with terminal C-18 and C-15' primary methyl protons, respectively. The ^{13}C-NMR spectrum of **1** displayed signals for ester carbon at δ 173.83 (C-1), vinylic carbons at δ 123.62 (C-9) and 117.65 (C -10), oxygenated methylene carbon at δ 68.21 (C-1'), methyl carbons at δ 14.68 (Me-18) and 14.41 (Me-15') and other methylene carbons between δ 29.90-22.57. On the basis of spectral analysis the structure of compound **1**has been characterized as n-pentadecanyl oleate(Fig. 1).

Compound **2**, named □-D-diglucoside, gave positive tests for glycosides and showed IR absorption bands for hydroxyl groups (3515, 3312, 3260 cm^{-1}). On the basis of mass and ^{13}C-NMR spectra, the molecular ions peak of **2** was determined at m/z 342 consistent to the molecular formula of a disaccharide, $C_{12}H_{22}O_{11}$. The ^{1}HNMR spectrum of compound **2** exhibited anomeric signals as one-proton doublets at δ 5.23 (J= 3.2 Hz) and 5.19 (J= 4.0 Hz) assigned to H-1 and H-1′, respectively, supported the existence of α-glycosidic units of the disaccharide. The other sugar protons resonated between δ 4.56-3.13. The ^{13}CNMR spectrum of compound **2** displayed signals for anomeric carbons at δ 104.50 (C-1) and 92.22 (C-1’) and the remaining sugar carbons from δ 83.01 to 60.93. The presence of the sugar H-2 signal in the deshielded region at δ 4.56 in the ^{1}H NMR spectrum and C-2 carbon signal at δ 83.01 in the ^{13}C NMR spectrum suggested (2→1′) linkage of the sugar units. Acid hydrolysis of **2** yielded D-glucose, R_f 0.26 (n-butanol- acetic acid – water, 4 : 1 : 5). On the basis of these evidences the structure of 2 has been formulated as α-D-glucopyranosyl-(2→1’)-O-α-D-glucopyranoside(Fig. 1).

Compound **3**, named n-caproyl O-α-D-pentaglucoside, gave confirmatory tests for glycosides and had characteristic IR absorption bands for hydroxyl groups (3510, 3455, 3317 cm^{-1}) and ester function (1724 cm^{-1}). The molecular ion peak of **3**was determined at m/z 926 on the basis of mass and ^{13}C NMR spectra corresponding to the molecular formula of an acyl pentaglycoside, $C_{36}H_{62}O_{27}$. The 1HNMR spectrum of **3** displayed five one-proton anomeric H-1a to H-1e proton signals doublets at δ 5.33 (J= 4.8 Hz), 5.31 (J= 3.6 Hz), 5.21 (J= 4.4 Hz) 5.18 (J= 3.6 Hz) and 4.97 (J= 2.0 Hz) indicating α-glycosidic units of the pentaglycoside chain. Other sugar protons appeared as multiplets between δ 4.79-3.10 and as five two-proton doublets at δ 3.19 (J = 6.5 Hz), 3.17 (J = 7.3 Hz), 3.12 (J = 6.8 Hz), 3.10 (J = 6.5 Hz), 3.05 (J = 8.8 Hz) due to hydroxymethylene H_2-6a to H_2-6e protons. A three-proton triplet at δ 0.81 (J=6.3 Hz) was accounted to C-6 primary methyl protons. The remaining methylene protons appeared as a two-proton triplet at δ 2.51 (J=7.2 Hz), as a two-proton multiplet at δ 1.33 and as a four – proton broad singlet at δ 1.23. The ^{13}CNMR spectrum of compound **3** showed signals for anomeric carbons from δ 102.45 to 93.03, other sugar carbons between δ 82.51-60.15, ester carbon at δ 173.16 (C-1), methyl carbon at δ 18.21 (C-6) and methylene carbons between δ 33.41-22.67. The presence of the sugar protons from H-2a to H-2d in the deshielded region from δ 4.79 to 4.48 in the ^{1}H NMR spectrum and carbon signals C-2a to C-2d between δ 82.51 – 81.23 in the ^{13}C NMR spectrum suggested (2→1) linkages of the sugar units. Acid hydrolysis of **3** yielded D-glucose, R_f 0.26 (n-butanol- acetic acid – water, 4 : 1 : 5).On the basis of above mentioned discussion, the structure of compound**3**has been characterized as n-hexanoyl-O-□-D-glucopyranosyl-(2a→1b)-O-□-D-glucopyranosyl-(2b→1c)-O-□-D-glucopyranosyl-(2c→1d)-O-□-D-glucopyranosyl-(2d→1e)-O-□-D-glucopyranoside, a new fatty acid tetraglucoside (Fig. 1).

Compound **4**, designated as n-caproyl O-β-D-hexaglucoside, $[M]^+$ at m/z 1088 ($C_{42}H_{72}O_{32}$), gave affirmative chemical tests for glycosides and exhibited distinctive IR absorption bands for hydroxyl groups (3490, 3365, 3280 cm^{-1}) and ester function (1721 cm^{-1}). The ^{1}HNMR spectrum of **4** exhibited four one-proton doublets at δ 5.21 (J= 7.2 Hz), 5.09 (J= 7.3 Hz), 5.02 (J= 7.1 Hz) and 4.98 (J= 7.5 Hz) and a two-proton broad singlet at δ 4.90 assigned to anomeric H-1a, H-1b, H-1c, H-1d, H-1e and H-1f protons, respectively. The other sugar protons displayed signals between δ 4.84-3.09. A three-proton triplet at δ 0.85 (J = 6.5 Hz), a two-proton triplet at δ 2.35 (J = 7.3 Hz) and a six – proton singlet at δ 1.23 were attributed to terminal C-6 primary methyl and methylene protons. The ^{13}CNMR spectrum of compound **4** showed signals

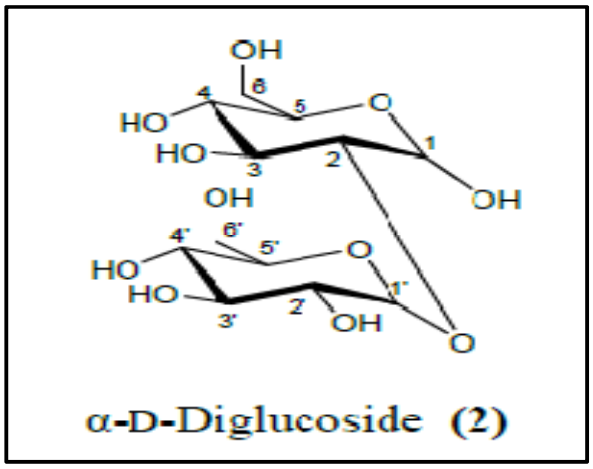
for anomeric carbons from δ 104.63 to 92.02, other sugar carbons between δ 83.38-61.58, ester carbon at δ170.27 (C-1), methyl carbon at δ 18.49 (C-6) and methylene carbons between δ 31.83-22.69. The presence of H-2a to H-2e proton in the deshielded region between δ 4.53 – 4.19 in the ^{1}H NMR spectrum and C-2a to C-2e from δ 83.38 to 80.31 in the ^{13}C NMR spectrum indicated (2→1) linkages of the sugar units.Acid hydrolysis of **4** yielded D-glucose, R_f 0.26 (n-butanol- acetic acid – water, 4 : 1 : 5).On the basis of above mentioned discussion, the structure of compound **4** has been characterized as n-hexanoyl-O-β-D-glucopyranosyl-(2a→1b)-O-β-D-glucopyranosyl-(2b→1c)-O-β-D-glucopyranosyl-(2c→1d)-O-β-D-glucopyranosyl-(2d→1e)-O-β-D-glucopyranosyl-(2e→1f)-O-β-D-glucopyranoside, a new acyl hexaglucoside (Fig. 1).

Compound **5,** named α-D-hexaglucoside,$[M]^+$ at m/z 990 ($C_{36}H_{62}O_{31}$), gave positive tests for glycosides and showed IR absorption bands for hydroxyl groups (3470, 3347, 3290 cm^{-1}). The ^{1}HNMR spectrum of compound **5** exhibited six one – proton doublets for anomeric proton signals as one-proton doublets at δ 5.17 (J= 3.6 Hz), 4.91 (J= 3.2 Hz), 4.88 (J= 3.2 Hz) and 4.86 (J= 3.9 Hz) and as a two-proton broad singlet at δ 4.71 (H-1d, H-1e) assigned to α-oriented anomeric protons H-1 and H-1a to H-1e. . The other sugar protons resonated as multiplets between δ 4.28-3.25 due to oxymethine and as two proton doublets at δ 3.18 (J = 10.8 Hz), 3.15 (J = 8.0 Hz), 3.13 (J = 9.2 Hz), 3.11 (J = 10.8 Hz), 3.08 (J = 10.8 Hz) and 3.05 (J = 8.0 Hz) associated with the hydroxymethylene H_2-1 and H_2-1a to H-1e protons, respectively. The ^{13}C NMR spectrum of compound **5** displayed signals for anomeric carbons from δ 104.19 to 92.20 and other sugar carbons between 83.21-60.86. The presence of the sugar protons from H-2 to H-2d in the deshielded region from δ 4.28 to 4.24 in the ^{1}H NMR spectrum and carbon signals C-2 to C-2d between δ 83.21 – 81.47 in the ^{13}C NMR spectrum suggested (2→1) linkages of the sugar units. Acid hydrolysis of 5 yielded D-glucose, R_f 0.26 (n-butanol- acetic acid – water, 4: 1: 5). On the basis of the foregoing discussion the structure of **5** has been established as □-D-glucopyranosyl-(2→1a)-O-□-D-glucopyranosyl-(2a→1b)-O-□-D-glucopyranosyl-(2b→1c)-O-□-D-glucopyranosyl-(2c→1d)-O-□-D-glucopyranosyl-(2d→1e)-O-□-D-glucopyranoside, a new α-D-hexaglucoside derivative(Fig. 1).

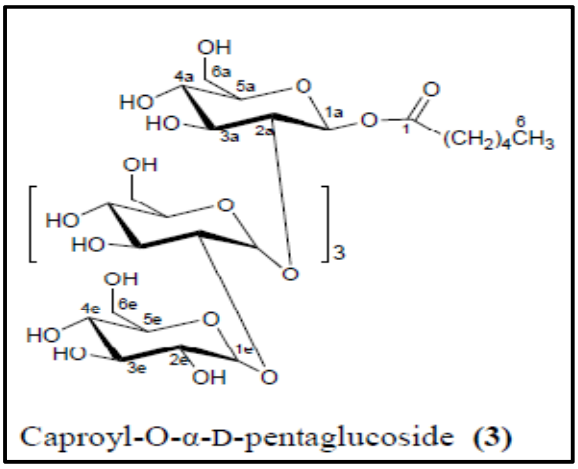
Compound **6,** designated asα-D-heptaglucoside,$[M]^+$ at m/z 1152 ($C_{42}H_{72}O_{36}$), was a homologue of compound **5**. It gave positive tests for glycosides and showed IR absorption bands for hydroxyl groups (3555, 3327, 3245 cm^{-1}). The ^{1}H-NMR spectrum of **6**exhibited seven anomeric proton signals as one-proton doublets from δ 5.17 to 4.65 with coupling interactions between 4.4 – 3.2 Hz indicating α-oriented sugar units and other sugar protons in the range of δ 4.28-3.04. The ^{13}CNMR spectrum of 6 displayed seven signals for anomeric carbons from δ 104.66 to 92.19 and other sugar carbons between 83.15-60.86. The presence of the sugar H-2 to H-2e protons in the deshielded region from δ 4.28 to 3.92 in the ^{1}H NMR spectrum and carbon signals C-2 to C-2e between δ 83.15 – 77.01 in the ^{13}C NMR spectrum suggested (2→1) linkages of the 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 this discussion the structure of **6** has been formulated as □-D-glucopyranosyl-(2→1a)-O-□-D-glucopyranosyl-(2a→1b)-O-□-D-glucopyranosyl-(2b→1c)-O-□-D-glucopyranosyl-(2c→1d)-O-□-D-glucopyranosyl-(2d→1e)-O-□-D-glucopyranosyl-(2e→1f)-O-□-D-glucopyranoside, a new α-D-heptaglucoside derivative(Fig. 1).

$$\overset{18}{C}H_3(CH_2)_7\overset{10}{C}H{=}{=}\overset{9}{C}H(CH_2)_7\overset{1}{C}OO\overset{1'}{C}H_2(CH_2)_{13}\overset{15'}{C}H_3$$

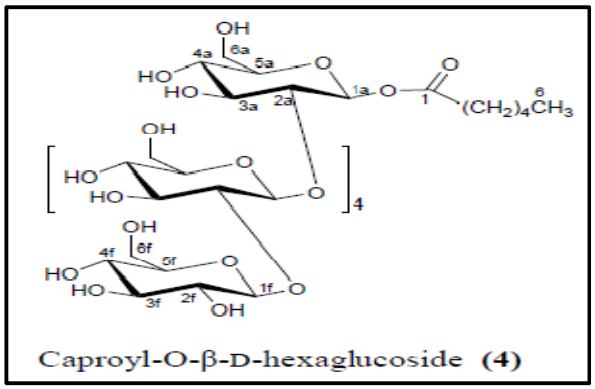
N-Pentadecanyloleate **(1)**

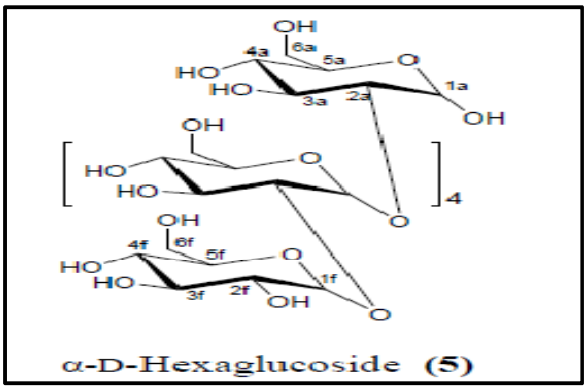


α-D-Diglucoside **(2)**



Caproyl-O-α-D-pentaglucoside **(3)**





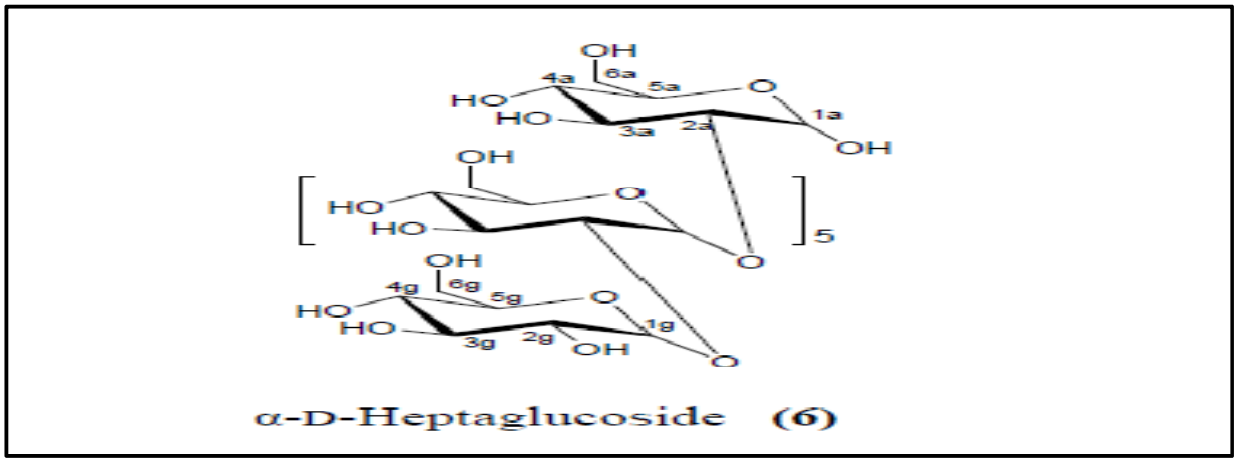


Fig. (1). Structural Formulae of the Chemical Constituents **1** – **6**.

4 Conclusion

Phytochemical investigation of a methanolic extract of the roots of Jatropha integerrima led to isolate a fatty ester, two caproyl glucosides and one each of di-, hexa- and heptaglucosides. This work has enhanced understanding about the phytoconstituents of the plant. These compounds may be used as chromatographic markers for standardization of the roots of the plant.

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