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Research Article

GLYCOSIDES FROM THE LEAVES OF *ARTOCARPUS HETEROPHYLLUS* LAM.

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ABSTRACT

Phytochemical investigation of the leaves of *Artocarpus heterophyllus* furnished six compounds from different combinations of petroleum ether, chloroform and methanol. Structures of these compounds were elucidated and established by standard spectroscopic methods. Isolated compounds are n-Octadec-9-enoyl α-L-rhamnopyranoside(1), n-octadec-9,12-dienoyl-α-L-rhamnopyranoside (2), n-octadec-9,12-dienoyl-β-D-glucopyranoside (3), n-octadec-9-enoyl-β-D-glucopyranoside (4), n-octadec-9-enoyl-β-D-arabinopyranoside (5) and n-octadec-9-enoyl-α-D-xylopyranoside (6) respectively. The structures of all the phytoconstituents are elucidated on the basis of spectral data analyses and chemical reactions.

Keywords: *Artocarpus heterophyllus,* Leaves, Column chromatography, Phytoconstituents

INTRODUCTION

Artocarpus heterophyllus Lam (Moraceae) commonly known as jackfruit is native to western ghats of India, Malaysia and also found in central and eastern Africa, southeastern Asia, the Caribbean, Florida, Brazil, Australia, Puerto Rico and many Pacific islands[1]. It is a large, evergreen tree, 10-15m in height, indigenous to the evergreen forests at altitude of 450 - 1200m throughout the hotter parts of India. Stem of this plant is straight, rough whereas bark is green or black, 1.25 cm thick exuding milky latex, leaves broad obovate[2]. Jackfruit is essentially a carbohydrate food and therefore useful as a source of energy. They contain vitamin C, protein, fat, calcium, phosphorus and iron in quantities normally present in other fruits[3]. Jackfruit *(Artocarpus heterophyllus* Lam) produces heavier yield than any other tree species, and bear the largest known edible fruit (up to 35 kg)[4].

The plants of *Artocarpus* species have been used by traditional folk medicine in Indonesia against inflammation, malarial fever, stomachache, ulcers, abscesses, dysentery, diarrhoea, defective urinary secretion, skin disease[5,6] and asthma[7].The plant parts contain Flavonoids[8], Triterpenic compounds[9],Sapogenins[10], Steroids[11] and Carotenoids[12].The present paper describes the isolation and characterization of six compounds from the leaves of *A. heterophyllus.*

MATERIAL AND METHODS

Plant material

The leaves of *Artocarpus heterophyllus* were collected from Patna, Bihar and identified by Dr. H.B. Singh, Scientist and Head, Niscair, NewDelhi. A voucher specimen of the plant leaves (NISCAIR/RHMD/consult/-20-09-10/1322/124) was deposited in the herbarium of NISCAIR, India.

Extraction and isolation

Dried powder of Artocarpus heterophyllus leaves (1.7 kg) was extracted with methanol (4 L) at 50 °C for 1 day. Extract was concentrated to dryness under reduced pressure to obtain slurry (148 g). The slurry was dissolved in minimum amount of methanol and was adsorbed on silica gel (60–120 mesh). The slurry was chromatographed over silica gel column packed in petroleum ether. The column was eluted successively with petroleum ether/$CHCl_3$/MeOH gradient system (1:0:0, 3:1:0, 1:1:0, 1:3:0, 0:1:0, 0:99:1, 0:49:1, 0:97:3, 0:19:1, 0:97:7, 0:91:9, 0:89:11, 0:87:13 and 0:17:3; 2.0 L for each gradient system). Six different compounds were isolated at different fractions. Compond **1** (110 mg) from $CHCl_3$, compound **2** (90 mg) from $CHCl_3$/MeOH (23:2), compound **3** (286 mg) from $CHCl_3$/MeOH (19:1) were obtained as pale yellow crystals; compound **4** (100 mg) from $CHCl_3$/MeOH (93:7) was obtained as colorless crystals, compound **5** (500 mg) from $CHCl_3$/MeOH (9:1), compound **6** (90 mg) from $CHCl_3$/MeOH (43:7) were obtained as pale yellow crystals.

RESULT AND DISCUSSION

Characterization of compound 1

It was obtained as a pale yellow crystalline mass from chloroform eluant.

M.P: 78 – 80°C

Rf: 0.57 (Petroleum ether/$CHCl_3$, 1:1)

UV λmax (MeOH): 275 nm (log ε 4.8)

IR γmax (KBr): 3415, 3320, 2927, 2857, 1727, 1606, 1454, 1376, 1252, 1217, 1081, 971, 762cm^{-1};

1H NMR (CDCl3): δ 5.38(1H, m, H-9), 5.36 (1H, m, H-10), 5.10 (1H, d, J= 7.0 Hz, H-1^1), 4.57 (1H, m, H-5^1), 4.23(1H, m, H-2^1), 4.07(1H, m, H-3^1), 3.75(1H, m, H-4^1), 2.72(1H,d, J= 6.6 Hz, H_2-2a), 2.67(1H, d, J= 7.5 Hz, H_2-2b), 2.30(2H, m, H_2-8), 2.04(2H,m, H_2-11), 1.68(2H, brs, CH_2), 1.60(2H, m, CH_2), 1.41(2H, brs, CH_2), 1.33(2H, brs, CH_2), 1.29(4H,brs, 2x CH_2), 1.04(3H,d, J= 6.6Hz, Me- 6^1), 0.80(3H, t, J= 6.3Hz, Me- 18).

It gave positive tests for glycosides. Its IR spectrum displayed characteristic absorption bands for hydroxyl groups (3415, 3320 cm^{-1}), ester group (1727 cm^{-1}), unsaturation (1606 cm^{-1}) and long aliphatic chain (762 cm^{-1}). On the basis of Mass spectrum of the structure of compound **1**, its molecular formula was determined as $C_{24}H_{44}O_6$. It indicated three double bond equivalents which were adjusted one each in the vinylic linkage, ester group and sugar unit. The ^{1}H-NMR spectrum of compound **1** showed two one proton- multiplets at δ 5.38 and 5.36 assigned to vinylic H-9 and H-10 respectively. A one proton doublet at δ 5.10 with coupling interaction of 7.0 Hz was ascribed to anomeric H-1' proton. Four one-proton multiplets at δ 4.57, 4.23, 4.07 and 3.75 were due to carbinol proton of the sugar unit. Two one proton doublets at δ 2.72 (J=6.6 Hz) and 2.67 (J=7.5Hz) were attributed to methylene H_2-2 protons adjacent to the ester groups. The remaining methylene protons resonated as two proton multiplets at δ 2.30, 2.04 and 1.60 and as broad signals from δ 1.68 to 1.25. A three proton doublet at δ 1.04 (J= 6.6Hz) and a three proton triplet at δ0.90 (J=6.3 Hz) were accounted to secondary methyl Me-6' and primary methyl Me-18 protons. Acid hydrolysis yielded Oleic acid and D-rhamnose (C_0- TLC comparable). On the basis of spectral data analysis and chemical reactions, the structure of compound **1** is elucidated as n-octadec-9-enoyl α-L-rhamnopyranoside.

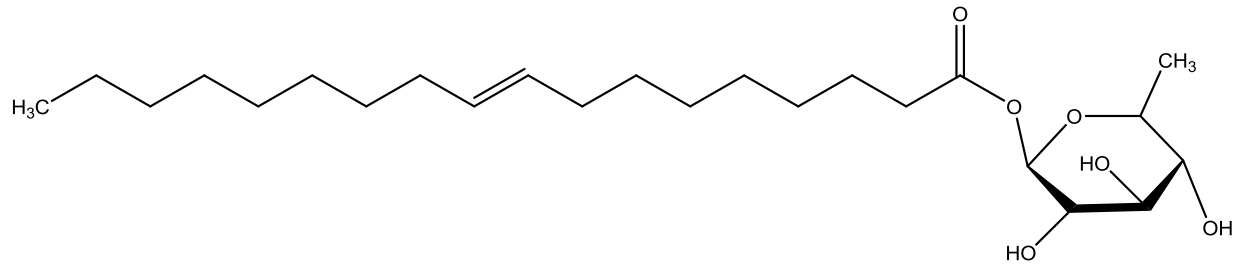

Fig. 1: n-octadec-9-enoyl α-L-rhamnopyranoside

Characterization of compound 2

It was obtained as Pale yellow crystalline mass in chloroform: Methanol (23:2) eluants.

M.P: 75 – 76°C

Rf: 0.85 ($CHCl_{3/MeOH}$, 19:1);

UV λmax (MeOH): 276 nm (log ε 2.1);

IR γmax (KBr): 3450, 3393, 2930, 2862, 1721, 1657, 1455, 1377, 1217, 1078, 1024,763 cm^{-1};

1H NMR (CDCl3): δ 5.40 (1H, m, H-13), 5.35 (1H, m, H-12), 5.33 (1H, m, H-10), 5.25 (1H, m, H-9), 5.10 (1H,d, J= 7.1Hz, H-1^1), 4.66 (1H, m, H-5^1), 4.25 (1H, m, H-2^1), 4.13 (1H,m, H-3^1), 3.77 (1H, m, H-4^1), 2.76 (2H, m, H_2-11), 2.32 (1H, d, J= 7.2Hz, H_2-2a), 2.27 (1H, d, J= 7.2Hz, H_2- 2b), 2.04 (2H, m, H_2- 8), 2.01 (2H, m, H_2- 14), 1.68 (2H, brs, CH_2), 1.60 (2H, brs, CH_2), 1.44 (2H, brs, CH_2), 1.29 (4H, brs, 2x CH_2), 1.25 (6H, brs, 3x CH_2), 1.04 (3h, d, J= 7.8 Hz, Me- 6^1), 0.87 (3H, t, J= 6.3 Hz, Me- 18).

It gave positive tests for glycosides. Its IR spectrum showed characteristic absorption bands for hydroxyl group at 3450, and 3393, ester group at 1721 cm $^{-1}$, unsaturation at 1657 cm $^{-1}$ and long aliphatic chain. On the basis of mass spectrum, the molecular formula of compound 2 was determined as $C_{24}H_{42}O_6$. The ^{1}H-NMR spectrum showed four one-proton multiplets at δ 5.40, 5.35, 5.33 and 5.25 assigned to vinylic H-13 , H-12, H-10 and H-9 protons, respectively. A one proton doublet at δ5.10 (J=7.1 Hz) was due to anomeric H-1' proton. The other sugar protons appeared as multiplets between δ 4.66- 3.77. A two proton multiplet at δ 2.76 was ascribed to methylene H_2-11 protons located between two vinylic carbons. Two one –proton doublets at δ 2.32 (J=7.2 Hz) and 2.27 (J= 7.2Hz) were accounted to methylene H_2-2 protons adjacent to the ester group. The other methylene proton appeared from δ 2.04 to 1.25. A three proton doublet at δ 1.04 (J=7.8 Hz) and a three proton triplet at δ 0.57 (J= 6.3Hz) were accounted to C-6' secondary and C-18' primary methyl protons, respectively. Acid hydrolysis yielded linoleic acid and L-rhamnose. On the basis of the above discussion, the structure of compound **2** has been characterized as n-octadec-9, 12-dienoyl-α-L-rhamnopyranoside.

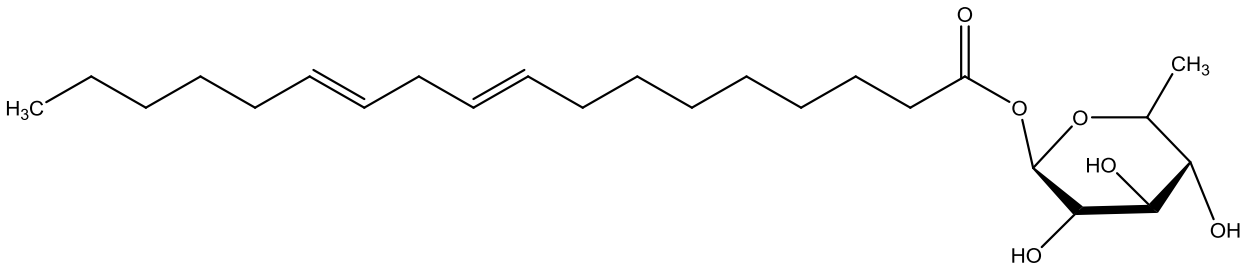

Fig. 2: n-octadec-9, 12-dienoyl-α-L-rhamnopyranoside

Characterization of compound 3

It was obtained as Pale yellow crystals from chloroform: Methanol (19:1) eluants.

M.P.: 76 – 77 ° C

Rf: 0.87 ($CHCl_{3/MeOH}$, 19:1)

UV λmax (MeOH): 271 nm (log ε 2.3)

IRγmax (KBr): 3410, 3335, 2925, 2855, 1721, 1635, 1457, 1378, 1219, 1078, 768 cm^{-1}

1H NMR (CDCl3): δ 5.35 (3H, brs, H-9,H-8,H-12), 5.11 (2H, m, H-13), 5.02 (1H, d, J=7.0Hz,H-1^1), 4.40 (1H, m, H-5^1), 4.31 (1H, m, H-2^1), 4.31 (1H, m, H-2^1), 3.85 (1H, m, H-3^1), 3.74 (1H,m, H-4^1), 3.40 (1H, d, J=6.6 Hz, H_2-6^1a), 3.35 (1H, d, J=7.2 Hz, H_2-6^1b), 2.80 (2H, m, H_2-11), 2.34 (1H, d, J= 7.8Hz, H_2- 2a), 2.29 (1H, d, J=7.2 Hz, H_2- 2b), 2.06 (2H, m, H_2- 8), 2.03 (2H, m, H_2-14), 1.60 (2H, m,CH_2), 1.30 (4H, brs, 2x CH_2) ,1.25 (10H, brs, 5xCH_2), 0.87 (3H, t, J=6.6 Hz, Me – 18).

It gave positive tests for glycosides. Its IR spectrum showed distinctive absorption bands for hydroxyl group at 3410 and 3335cm-^{1}, ester group at 1721 cm $^{-1}$, unsaturation at 1635 cm $^{-1}$ and long aliphatic chain at 768cm-^{1}. On the basis of mass spectrum, the molecular formula of compound **3** was determined as $C_{24}H_{42}O_7$. It indicated four double bond equivalents ; two of them were adjusted in the vinylic linkages and the remaining one each in the ester group and sugar unit. The ^{1}H-NMR spectrum of compound 3 exhibited a three-proton broad signal at δ 5.35 and a one proton multiplet assigned to four vinylic protons. A one proton doublet at δ 5.02 (J=7.0 Hz) was ascribed to anomeric H-1' proton. Four one- proton multiplets at δ 4.40, 4.31, 3.85 and 3.74 were attributed to carbinol protons of the sugar unit. Two one – proton doublets at δ 3.40 (J=6.6 Hz) and 3.35 (J= 7.2Hz) were accounted to hydroxymethylene H_2-6' protons. A one proton multiplet at δ 2.80 was accommodated to methylene H_2-11 protons located between two vinylic carbons. Two one poroton doublets at δ 2.34 (J= 7.8 Hz) and 2.29 (J=7.2 Hz) were ascribed to methylene H_2-2 protons adjacent to the ester group. The other methylene protons resonated from δ 2.06 to 1.25. A three proton triplet at δ 0.87 (J=6.6 Hz) was associated with C-18 primary methyl protons. Acid hydrolysis yielded linoleic acid and glucose. On the basis of these evidences, the structure of compound **3** has been characterized as n-octadec-9, 12-dienoyl-β-D-glucopyranoside.

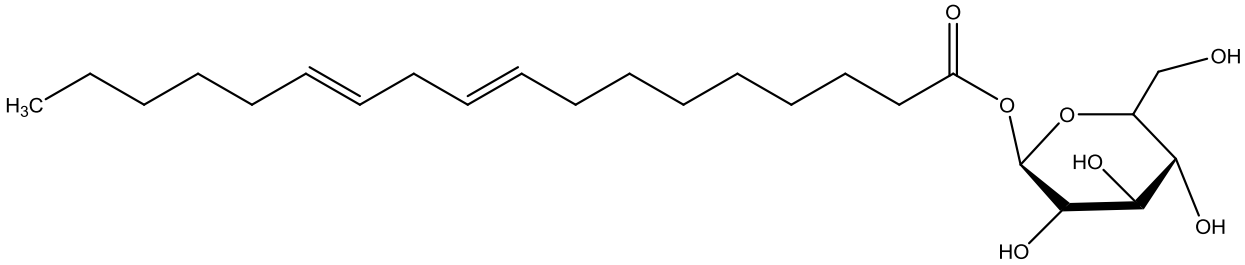

Fig. 3: n-octadec-9,12-dienoyl-β-D-glucopyranoside

Characterization of compound 4

It was obtained as colourless crystalline mass in chloroform: Methanol (93:7) eluants.

M.P: 88–90° C

Rf: 0.17 ($CHCl_3$/MeOH, 19:1)

UV λmax (MeOH): 205 nm (log ε 2.9)

IRγmax (KBr): 3480, 3376, 3255, 2928, 2850, 1725, 1667, 1445, 1371, 1219,1107,769 cm^{-1};

1H NMR (CDCl3): δ 5.36 (2H, m, H-9, H-10), 5.03 (1H, d, J=7.1 Hz,H-1^1), 4.87 (1H, m,H-5^1), 4.34 (1H, m, H-2^1), 4.17 (1H, m, H-3^1), 3.70 (1H, m, H-4^1), 3.41 (1H,d, J=6.9Hz, H_2-6^1a), 3.36 (1H,d, J=7.2Hz,H_2-6^1b), 2.41 (1H, d, J=7.8 Hz, H_2-2^1a), 2.36 (1H, d, J=8.4 Hz, H_2-2^1b), 2.20 (2H, m, H_2-8), 2.14 (2H, m, H_2- 11), 1.62 (2H, m,CH_2), 1.58 (2H, m, CH_2), 1.31 (4H, brs,2x CH_2), 1.25 (14H, brs,7x CH_2), 0.82 (3H, t, J=6.9 Hz, Me -18).

It gave positive tests for glycosides. Its IR spectrum showed characteristic absorption bands for hydroxyl groups at 3480, 3376 and 3255, ester group at 1725 cm $^{-1}$, unsaturation at 1667 cm $^{-1}$ and long aliphatic chain at 769 cm^{-1}. On the basis of mass spectrum, the molecular formula of compound **4** was determined as $C_{24}H_{44}O_7$.It has three double bonds equivalents, each of them were adjusted in the vinylic linkage, ester group and glycosidic unit. The ^{1}H-NMR spectrum showed a two-proton multiplets at δ 5.36 assigned to vinylic H-9 and H-10. A one proton doublet at δ5.03 (J=7.1 Hz) was due to anomeric H-1' proton. The other sugar protons appeared as multiplets between δ 4.87- 3.70 and one proton doublets at δ 3.41 (J=6.9Hz) and 3.36 (J=7.2Hz) due to hydroxymethylene H_2-6' protons.

Two one proton doublets at δ 2.41(J=7.8Hz) and 2.36 (J=8.4Hz) were attributed to methylene H_2-2'protons adjacent to the ester function. Four two – proton multiplets between δ 2.20 - 1.58 and two broad signals at δ 1.31(4Hz) and 1.25 (14Hz) were associated with the other methylene protons. A three proton triplet at δ 0.82 (J=6.9 Hz) was due to terminal C-18 primary methyl protons. Acid hydrolysis of compound 4 yielded oleic acid and D-glucose. On the basis of above discussion, the structure of compound **4** has been characterized as n-octadec-9-enoyl-β-D-glucopyranoside.

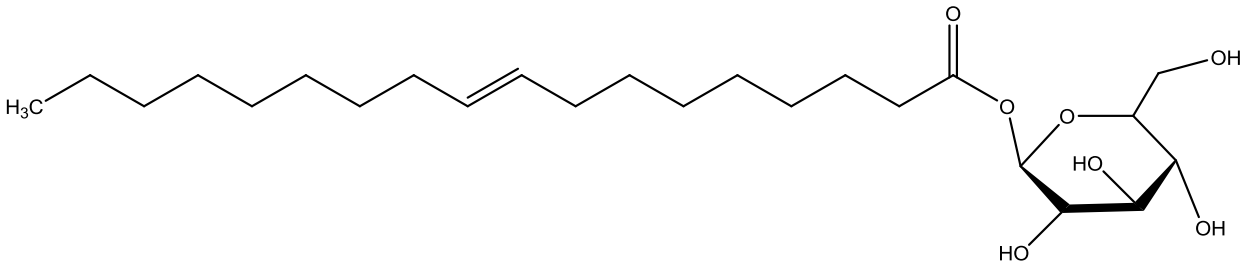

Fig. 4: n-octadec-9-enoyl-β-D-glucopyranoside

Characterization of compound 5

It was obtained as Pale yellow crystalline mass in chloroform: Methanol (9:1) eluants.

M.P: 93–95°C

Rf: 0.25 ($CHCl_3$/MeOH, 19:1)

UV λmax (MeOH): 204 nm (log ε 2.6)

IRγmax (KBr): 3410, 3360, 2920, 2855, 1721, 1625, 1440, 1220, 1105, 770 cm^{-1}

1H NMR (CDCl3): δ 5.35 (2H, brs, H-9, H-10), 4.94 (1H, d, J=10.8 Hz,H-1^1), 4.30 (1H, d, J=9.3,10.8Hz,H-2^1), 3.92 (1H, m, H-3^1), 3.83 (1H, m, H-4^1), 3.66 (1H, d, J=8.1Hz, H_2-5^1a), 3.6 (1H,d, J=10.2 Hz, H_2-5^1b), 2.30 (1H,d, J=9.6Hz,H_2-2a), 2.27 (1H, d, J=9.5 Hz, H_2-2b), 2.18 (2H, m, H_2-8), 2.05 (2H, m, H_2-11), 1.64 (2H, m, CH_2), 1.59 (2H, m,CH_2), 1.41 (2H, m, CH_2), 1.28 (6H, brs,3x CH_2), 1.25 (12H, brs,6x CH_2), 0.87 (3H, t, J=6.5 Hz, Me -18).

It responded positively to glycosidic units. Its IR spectrum showed characteristic absorption bands for hydroxyl groups (3408, 3365 cm^{-1}) , ester function (1722) cm $^{-1}$, unsaturation at 1603 cm $^{-1}$ and long aliphatic chain at 768 cm^{-1}. On the basis of mass spectrum, the molecular formula of compound **5** was determined as $C_{23}H_{42}O_6$. It has three double bonds equivalents, each of them were adjusted in the vinylic linkage, ester group and glycosidic unit. The ^{1}H-NMR spectrum of compound 5 showed a two-proton broad signal at δ 5.35 assigned to vinylic H-9 and H-10 protons. A one proton doublet at δ4.94 (J=10.8 Hz) was due to anomeric H-1' proton. A one proton double doublets at δ 4.30 (J=9.3, 10.8Hz), two one proton muliplets at δ 3.92 and 3.83and two one proton doublets at δ 3.66 (J=8.1Hz) and 3.60 (J=10.2Hz) were attributed to carbinol protons H-2', H-3'and H-4' and to oxygenated methylene H_2-5' protons, respectively.

Two one proton doublets at δ 2.30 (J=9.6Hz) and 2.27 (J=9.5Hz) were accounted to methylene H_2-2 protons adjacent to the ester group. The other methylene protons appeared between δ 2.18-1.25. A three proton triplet at δ 0.57 (J=6.5 Hz) was due to terminal C-18 primary protons. Acid hydrolysis yielded oleic acid and arabinoside. On the basis of the above discussion, the structure of compound **5** has been identified as n-octadec-9-enoyl-β-D-arabinopyranoside.

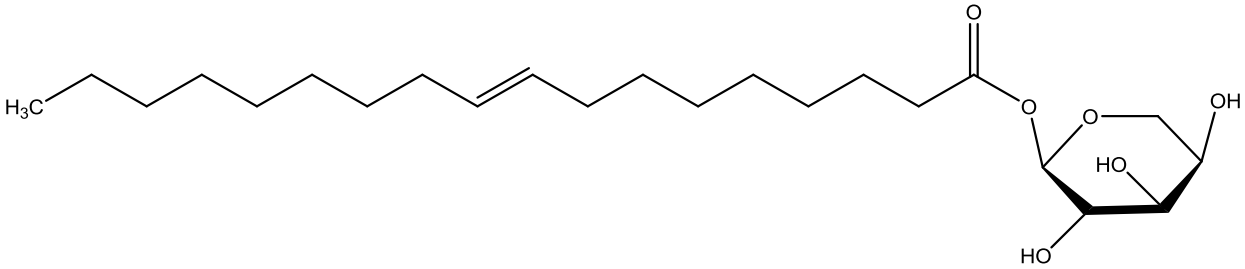

Fig. 5: n-octadec-9-enoyl-β-D-arabinopyranoside

Characterization of compound 6

It was obtained as Pale yellow crystalline mass in Chloroform: Methanol (43:7) eluants.

M.P: 83–85° C

R_f: 0.76 ($CHCl_3$/MeOH, 19:1)

UV λmax (MeOH): 205 nm (log ε 2.6)

IR γmax (KBr): 3410, 3360, 2920, 2855, 1721, 1625, 1440, 1220, 1105, 770 cm^{-1}

^{1}H-NMR($CDCl_3$): δ 5.35 (2H, brs, H-9, H-10), 5.01 (1H, d, J=7.5 Hz,H-1^1), 4.32 (1H, m, H-2^1), 4.1 (1H, m, H-3^1), 3.81 (1H, m, H-4^1), 3.66 (1H, d, J=9.9Hz, H_2-5^1a), 3.64 (1H,d, J=9.9 Hz, H_2-5^1b), 2.30 (1H,d, J=8.7Hz,H_2-2a), 2.27 (1H, d, J=8.7 Hz, H_2-2b), 2.07 (2H, brs, H_2-8), 2.05 (2H, m, H_2-11), 1.61 (2H, m, CH_2), 1.58 (2H, m,CH_2), 1.41 (2H, brs, CH_2), 1.29 (6H, brs,3x CH_2), 1.25 (10H, brs,10x CH_2), 0.55 (3H, t, J=6.7 Hz, Me -18); ^{13}C-NMR ($CDCl_3$) : δ 173.09 (C-1), 130.56 (C-9), 122.05 (C-10), 103.58(C-1'), 80.41(C-2') , 78.36 (C-3'), 69.50 (C-4'), 66.12 (C-5'), 33.25 (C-2), 31.89 (C-3), 29.65 (6xCH_2), 29.47 (CH_2), 28.97 (CH_2), 28.61(CH_2), 28.33(CH_2), 25.44(CH_2), 22.65(CH_2), 14.07 (Me-18).

It responded positively to glycoside tests. Its IR spectrum showed characteristic absorption bands for hydroxyl groups (3410, 3360 cm^{-1}) , ester function (1721) cm^{-1}, unsaturation at 1625 cm^{-1} and long aliphatic chain at 770 cm^{-1}. On the basis of mass spectrum, the molecular formula of compound **6** was determined as $C_{23}H_{42}O_6$. It has three degrees of unsaturation, each of them were adjusted in the vinylic linkage, ester group and glycosidic unit. The ^{1}H-NMR spectrum of compound 6 showed a two-proton broad signal at δ 5.35 assigned to vinylic H-9 and H-10 protons. A one proton doublet at δ 5.01 (J=7.5 Hz) was due to anomeric H-1' proton. Three one proton muliplets at δ 4.32, 4.10 and 3.81 and two one proton doublets at δ 3.66 (J=9.9Hz) and 3.64 (J=9.9Hz) were attributed to carbinol protons H-2', H-3'and H-4' and to oxygenated methylene H_2-5' protons of the sugar unit, respectively. Two one proton doublets at δ 2.30 (J=8.7Hz) and 2.27 (J=8.7Hz) were accounted to methylene H_2-2 protons adjacent to the ester group..The other methylene protons appeared between δ 2.07-1.25. A three proton triplet at δ 0.57 (J=6.7 Hz) was due to terminal C-18 primary methyl protons. The ^{13} C NMR spectrum exhibited signals for ester carbon at δ 173.09 (C-1), vinylic carbons at δ 130.56 (C-9) and 122.05 (C-10), anomeric carbon at δ 103.58 (C-1'),other sugar carbons from δ 80.41 to δ 66.12, methylene carbon between δ 33.25-22.65 and methyl carbon at δ 14.07 (C-18). Acid hydrolysis yielded oleic acid and α-D-xylose. On the basis of the above discussion, the structure of compound **6** has been identified as n- octadec-9-enoyl-α-D-xylopyranoside.

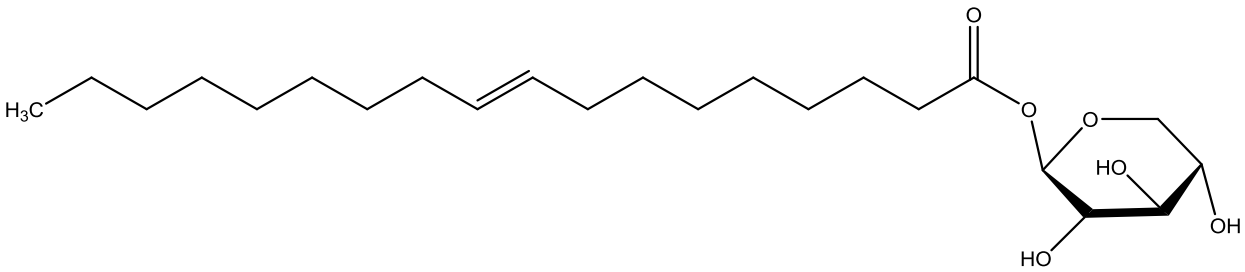

Fig. 6: n- octadec-9-enoyl-α-D-xylopyranoside

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