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Isolation of Straight Chain Alcohol and Ester from *Sterculia guttata*

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A straight chain ester, nonacosanoic acid, methyl ester was isolated for the first time from acetone extract of *Sterculia guttata*. Saponification of petroleum ether extract of *Sterculia gutatta* was carried out. Unsaponifiable matter was further purified by repeated column chromatography followed by repeated mixed solvent crystallization to afford docosanol. The structures of both the compounds were confirmed by spectral analysis.

Key Words: Docosanol, Nonacosanoic acid, Methyl ester, *Sterculia guttata*.

INTRODUCTION

Sterculia guttata Roxb. (Sterculiaceae) is a medicinally important plant. It is distributed in India - Maharastra, Assamand and Andamaans. The bark fiber is used for cordage and rough fabrics[1]. Leaves and bark are used as folk medicines. This plant is reported as a famine food[2]. The juice obtained from the bark and phangali (*Pogosteman benghalensis*) leaves by crushing in water, is used in folk medicines, to cure fever and diarrhoea[3]. The seeds are eaten raw or roasted by tribes, especially in times of scarcity. If the seeds are eaten more than a handful quantity at a time, the person feels lethargic[4].

The nonacosanoic acid, methyl ester (**1**) and docosanol (**2**) are isolated for the first time from this plant. Different methyl esters isolated from hexane extract of *Sebastiania argutidens* have been characterized by high resolution gas chromatography (HRGC) and HRGC coupled with mass spectrometry (GC/MS)[5].

Literature survey revealed that the only plant *Sebastiania argutidens* showed the presence of nonacosanoic acid, methyl ester (**1**), which was characterized by high-resolution gas chromatography (HRGC) and HRGC

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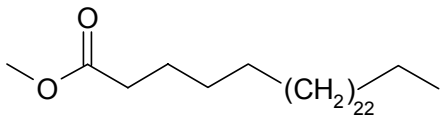
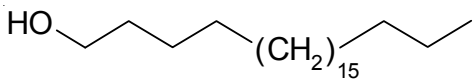
coupled with mass spectrometry (GC/MS)[5]. There are number of acid esters with the same molecular formula were reported, but very little information is mention about nonacosanoic acid, methyl ester (**1**)[6,7].

Docosanol, compound **2** is a saturated 22-carbon, long-chain aliphatic alcohol, isolated for the first time from this plant. It has potential for use in foods as an oil structuring and solidifying agent in fats[8]. It is common in leaf wax, fruit, seed oil. It is used in hair conditioning composition[9] and cosmetics[10]. It has been reported for antiviral activity[11].



Structure of nonacosanoic acdi methyl ester (**1**)



Structure of docosanol (**2**)

EXPERIMENTAL

IR spectrum was recorded on FTIR-8300 Shimadzu spectrometer, NMR spectra on Bruker DRX-500, operating at 500 MHz for ^{1}H and 125 MHz for ^{13}C NMR spectrum and Bruker MSL-300, instrument, operating at 200 MHz for ^{1}H at 24 °C using residual signal of non-deuterated solvents as internal reference. Mass spectrum, LCMS on LCMS-MS Perkin Elmer Applied Biosystems SCIEX- 2000.

Plant material: The leaves of *Sterculia guttata*, Roxb. were collected from deciduous forests near Pune, India in bulk quantity. The plant specimen was authenticated by matching with the voucher specimen BSI/WC/Tech/2000/358 available at the Botanical Survey of India, Pune, India.

Isolation of compound 1: Air shade dried, powered material of leaves of *Sterculia guttata* (300 g) was extracted by refluxing with acetone at 80 °C on water bath for 24 h. The extract was filtered and concentrated under reduced pressure to yield a green mass **A** (15 g). The dried extract (A, 14 g) was subjected to column chromatography over silica gel (60-120 mesh).

Chromatographic separation of constituents of extract A: The extract **A** (14 g) was fractioned over silica gel CC (1:15 g) starting with *n*-hexane-toluene 1:1 (1000 mL), followed by toluene (1000 mL), toluene-ethyl acetate 9:1 (1000 mL), toluene-ethyl acetate 4:1 (1000 mL), toluene-ethyl acetate 7:3 (1000 mL), toluene-ethyl acetate 3:2 (1000 mL), toluene-ethyl acetate 1:1 (1000 mL), toluene-ethyl acetate 1:4 (1000 mL), ethyl

acetate (1000 mL), ethyl acetate-methanol 9:1 (1000 mL), ethyl acetate-methanol 4:1 (1000 mL), ethyl acetate-methanol 1:1 (1000 mL) and finally with methanol. The fractions of 200 mL volume were collected. The progress of the column chromatographic separation was monitored by performing thin layer chromatography of the fractions. Fractions showing similar compositions were combined together to obtain eighteen major fractions. The fourth fraction of column chromatography afforded impure compound **1** (0.038 g) was washed with diethyl ether and further purification was carried out by repeated crystallization using ethyl acetate and methanol to give white amorphous powder of compound **1** (0.014 g).

Isolation of compound 2: Air shade dried powdered leaves material of *Sterculia guttata*, (100 g) was exhaustively extracted with petroleum ether (40-60) (1000 mL). The pet-ether extract was filtered and dried. The residue (B, 5 g) was saponified using 10 % KOH (50 mL) in 50 % EtOH by refluxing for 3 h. After cooling, alkaline solution was extracted with diethyl ether to separate acidic part from extract. Ether layer was washed with distilled water to become neutral and dried over anhydrous sodium sulphate, removal of ether furnished unsaponifiable extract **C** (3.160 g) was used for further separation.

Chromatographic separation of constituents of extract C: Unsaponifiable matter **C** (3 g) was fractionated over silica gel column chromatography (2.5 × 60 cm) (1:30 g) starting with pet-ether-toluene 1:1 (800 mL), followed by toluene (800 mL), toluene-ethyl acetate 9:1 (800 mL), toluene-ethyl acetate 4:1 (800 mL), toluene-ethyl acetate 1:1 (800 mL), ethyl acetate (800 mL), ethyl acetate-methanol 1:1 (800 mL) and finally with MeOH (200mL). The fractions of 200 mL volume were collected. The progress of the column chromatographic separation was monitored by performing thin layer chromatography. Fractions showing similar compositions were combined together to obtain eight fractions. Second fraction of column chromatography afforded impure compound **2** (0.800 g). It was further purified by rechromatography.

Rechromatography of fraction 2: Fraction 2 (0.600g), containing compound **2** was rechromatographed by column chromatography on silica gel (1:70 g) starting with pet-ether (200 mL) followed by pet-ether-toluene 9:1 (100 mL), pet-ether-toluene 4:1 (100 mL), pet-ether-toluene 7:3 (100 mL), pet-ether-toluene 3:2 (100 mL), pet-ether-toluene 1:1 (100 mL), pet-ether-toluene 2:3 (100 mL), pet-ether-toluene 3:7 (100 mL), pet-ether-toluene 1:4 (100 mL), toluene (100 mL), ethyl acetate (100 mL) and finally methanol (100 mL). The fractions of 100 mL volume were collected. The progress of the column chromatographic separation was monitored by thin layer chromatography of the fractions. Fractions showing similar compositions were combined together to obtain twelve major

fractions. Fourth fraction of column chromatography afforded impure compound **2** (0.098 g). It was purified by repeated crystallization using chloroform-methanol as a mixed solvent to give pure compound **2** (0.012 g).

RESULTS AND DISCUSSION

Compound **1**, a straight chain ester, a white solid, was purified by repeated mixed solvent crystallization (methanol and ethyl acetate) having m.p. 87 °C. Elemental analysis is found to be C, 79.86; H, 13.40 %. LCMS showed the molecular ion peak at m/z 452, which coupled with the elemental analysis suggest the molecular formula as $C_{30}H_{60}O_2$. IR spectrum shows a characteristic band at 1703 cm^{-1} (an ester carbonyl) and a peak at 1301 cm^{-1} (C-O stretching). ^1H NMR spectrum shows a broad singlet at δ 2.93 for methyl group attached to ester ethereal oxygen (3H, O=C-O-CH_3), merged triplet at δ 1.55 for methylene protons attached to ester carbonyl (2H, -O=C-CH_2-), a broad singlet at δ 1.22 for methylene proton envelop and a triplet at δ 0.82 (3H, J = 5Hz) for a methyl group. The ^{13}C NMR spectrum of compound **1** shows signals at δ 176.30 (s) for ester carbonyl group, 53.72 (q, O=C-O-CH_3), 33.70 (t, -CH_2-CH_2-C-O-O-CH_3), 31.56 (t, CH_3-CH_2-CH_2-), 29.33 (t, -CH_2- envelop), 29.12 (t, -CH_2-CH_2-C-O-O-CH_3), 29.00 (t, -CH_2-CH_2-CH_2-O-O-CH_3), 28.94 (t, -CH_2-CH_2-CH_2-CH_2-C-O-O-CH_3), 28.80 (t, -CH_2-CH_2-CH_2-CH_2-CH_2-C-O-O-CH_3), 24.56 (t, -CH_2-CH_2-CH_2-CH_2-CH_2-CH_3), 22.31 (t, CH_3-CH_2-) and 13.72 (q, CH_3-CH_2-). This data reveals the compound **1** to be nonacosanoic acid, methyl ester (**1**). This compound has been isolated for the first time from this plant.

Compound **2**, a straight chain alcohol, white fatty mass, $C_{22}H_{26}O$ purified by repeated column chromatography and repeated crystallization, has m.p. 77-79ºC. It shows molecular ion peak at 326 amu by LCMS using +ve and -ve modes, indicates molecular formula $C_{22}H_{46}O$. IR spectrum shows a characteristic band at 3354 cm^{-1} (hydroxyl) and a peak at 1026 cm^{-1} (C-O stretching). ^1H NMR spectrum shows triplet at δ 3.65 for methylene protons attached to hydroxyl group (2H, -CH_2-CH_2-OH, J = 5Hz), multiplet at δ 1.57 for methylene protons (6H, -CH_2-CH_2-OH, -CH_2-CH_2-CH_2-OH, -CH_2-CH_2-CH_2-CH_2-OH), a broad singlet at δ 1.26 for methylene proton envelope and a triplet at δ 0.89 (3H, -CH_2-CH_3, J = 6Hz) for a methyl group. The ^{13}C NMR spectrum of compound **2** shows signals at 62.83 δ (t, -CH_2-OH), 32.53 (t, -CH_2-CH_2-OH), 31.65 (t, CH_3-CH_2-CH_2-), 29.42 (t, -CH_2- envelop), 29.15 (t, CH_3-CH_2-CH_2-CH_2-CH_2-), 25.45 (t, -CH_2-CH_2-CH_2-OH), 22.41 (t, CH_3-CH_2-) and 13.84 (q, CH_3-CH_2-). This data reveals the compound **2** to be 1-docosanol. This compound has been isolated for the first time from this plant.

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