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“New Long Chain Aliphatic Compounds From *Butea Monosperma* (Lamk).”

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Abstract:

A new long chain aliphatic compounds- triacont-3, 17-diene 1, 25-methyl-dotriacont-6, 20-diene-17-one 2, undecylhexadecanoate 3 and two known compounds- stigmasterol 4, and β -sitosterol 5 have been isolated from the acetone extract of the stem bark *Butea monosperma*. The structures of these compounds have been elucidated by a combination of spectral methods (IR, MS, ^1H , and ^{13}C NMR). The plant showed antioxidant, antidiarrhoeal, dermal, wound healing, antidiabetic, antistress, anticonvulsive, antihepatotoxic, antiestrogenic and antihelmintic activities. Many of them are used in industry for various applications like perfumes, flavors, deodorants, antiseptic and pharmaceuticals. The aim of the present study was to analyze three new long chain aliphatic compounds from the acetone extract of the stem bark of *B. monosperma*.

Keyword: *Butea monosperma* Lamk, triacont-3, 17-diene, 25-methyl-dotriacont-6, 20-diene-17-one, undecyl hexadecanoate.

Introduction

The traditional systems of medicine together with homoeopathy and folklore medicine continue to play a significant role largely in the health care system of the population. *Butea monosperma* (palas) belonging to the family Leguminosae grown wildy in many parts of India. The plant is highly uses by the rural and tribal people in curing various disorders. Flowers are used as drug in many ailments like eye disease, chronic fever, enlargement of spleen, leucorrhoea, epilepsy, leprosy, anti-fungal activity, anti-inflammatory activity, liver disorders antifertility activity and gout etc. *Butea monosperma* Lamk. (Hindi - Palas,

Khakhra and Dhak, Family- Leguminosae) is an indispensable and beautiful tree has been reported to possess anti-inflammatory, antifungal activity, antihelmintic, antifertility and abortifacient activities. Its flowers are known as Kesu, Tesu and Palas ke Phool (kirtikar & Basu 1935, Chopra & Nayar 1956). Almost all the parts of the plant namely root, leaves, fruit, stem bark, flowers, gum, young branches are used as medicine, food, fiber and for other miscellaneous purposes such as fish poison, dye, fodder, utensils, etc. Bark is acrid, bitter, appetizer, aphrodisiac, biliousness, dysmenorrhea, liver disorder, gonorrhea and laxative, useful in

fractures of the bones, dysentery, piles, ulcers and tumours. Stem bark powder is used to apply on injury caused due to axe and in case of body swellings. Various parts of the plant have been explored for antioxidant, antidiarrhoeal, dermal, wound healing, antidiabetic, antistress, anticonvulsive, antihepatotoxic, antiestrogenic and antihelmintic activities. Presence of some new anthocyanin, flavonoids, flavonoid glycosides, terpenes, alkaloids and cyanin chloride have been reported from different parts of this plant (Sahare et al 2012, Sharma & Garg 2009). In some parts of the India these are used for biddies manufacturing by wrapping tobacco leaf. The cattle eagerly eats the palash foliage. The bark of *Butea monosperma* yielding a kind of coarse and brown colored fiber and these are used for rough cordage. The gum of tree (BM) is a dried juice obtained from incisions in the stem of the tree and it posses astringent effect (Wagner & Geyer 1986, Sehrawat et al 2006). The flowers of *Butea monosperma* yielded a red or orange dye which is used as an insecticide and as coloring agent. *Butea monosperma* is a sacred tree, called as a treasurer of the gods. Sacred utensils are made from its wood. The dry stem pieces are offered to make sacred fire. *Butea monosperma* is an anthropogenic tree of several castes. The use of its gum as external astringent application is mentioned by 'Chakradatta'. (Bhargava 1986, Pandey 2001,

Rekha & Jayakar 2011).The leaves are good have astringent, depurate, diuretic and aphrodisiac in pharmacological properties. The seeds are act as a rubefacient when pounded with lemon juice and applied to the skin (Iqbal et al 2006, Sumitra et al 2007, Gunakkunru et al 2005). The present paper describes the isolation and structure elucidation of three new long chain and two known compounds from the acetone extract of the stem bark.

Results and Discussion

Compound 1: Triacont-3, 17-diene, M.P.180 °C, M^+ 418, $C_{30}H_{58}$, IR spectrum showed absorption bands at 760-720 cm^{-1} diagnostic of $(CH_2)_n$ (where n is more then four) and at 1636 cm^{-1} assignable to unsaturation in the molecule. The signal at δ 0.91 (t, 6H) in the ^1H NMR is assignable to the presence of 2 terminal $-CH_3$ groups while the triplet at δ 5.02 was assigned to olefinic protons. The methylene protons α to unsaturation appeared at δ 1.6 as broad peak. Rest of the methylene protons were resonated at δ 1.31 as a broad singlet. The ^{13}C NMR spectrum of compound 1 showed the presence of terminal methyl carbon at 19.9 ppm. The olefinic carbon at C-3 and C-17 were resonated at 117.9 ppm. Mass spectrum showed $[M^+]$ at m/z 418. In the mass spectrum separation of most of the peaks by 14 mass units and appearance of C_nH_{2n-1} ion series confirmed its long chain aliphatic nature. Abundant fragments at m/z

223, 363 formed by allylic cleavage indicated the position of double bond at C-3 and C-17. The compound 1 was brominated and identified as 3, 17-dibromo triacont diene. The formation of dibromo derivative was confirmed by the appearance of a triplet in mass spectrum. On the basis of above evidences, compound 1 was assigned as triacont-3, 17-diene and being reported first time by us.

Compound 2: 25-methyl-dotriacont-6, 20-diene-17-one, M.P. 240 °C, M^+ 474, $C_{33}H_{62}O$, The IR spectrum showed the presence of unsaturation 1627 cm^{-1} and long chain aliphatic nature of the molecule 758 cm^{-1} . Strong band at 1710 cm^{-1} was due to carbonyls group. ^1H NMR spectrum showed a six proton triplet at δ 0.88 for the terminal methyl groups. A multiplet at δ 1.62 accounts for the methine proton. The olefinic proton resonated at δ 5.34 as multiplet. Rest of the methylene merged into a single peak at δ 1.24 as a broad singlet. The triplet at δ 2.29 was assigned to the methylenes attached to carbonyl group. Doublet at δ 0.94 was assigned to branched methyl protons. The ^{13}C NMR spectrum showed the presence of terminal methyl carbon at 19.9 ppm. The carbon of carbonyl group was resonated at 172.0 ppm. The olefinic carbon at C- 6 and C-20 were resonated at 118.0 ppm. Mass spectrum showed the characteristic fragmentation pattern of long chain aliphatic compound. The fragment at m/z 266 was due

to McLafferty rearrangement showed the presence of carbonyl group at C-17. Major fragments were obtained at m/z 265, 319, 375, 446 indicated the position of carbonyl group and unsaturation. The compound 2 was brominated to yield 6, 20-dibromo derivative. The formation of dibromo derivative was revealed by the appearance of a triplet in mass spectrum, thus confirming two double bonds in the molecule. Thus compound 2 was characterized 25-methyl-dotriacont-6, 20-diene-17-one being first time reported.

Compound 3: Undecyl hexadecanoate, M.P. 110 °C, M^+ 410, $C_{27}H_{54}O_2$, The IR spectrum showed bands at 1179 and 720 cm^{-1} for aliphatic nature and at 1737 cm^{-1} for ester group. ^1H NMR spectrum showed a triplet at δ 0.86 ($J=7.5\text{Hz}$) for terminal methyl groups. Methylene protons of $-\text{CH}_2-\text{O}-\text{CO}-$ and $-\text{CH}_2-\text{CO}-\text{O}-$ moieties were resonated as triplets at δ 4.05 and δ 2.29 ($J=7.0\text{Hz}$) respectively. Methylene protons β to ester group were resonated at δ 1.50 as broad multiplet. Rest of the methylenes was resonated at δ 1.25 as singlet. The ^{13}C NMR spectrum of compound 3 showed the presence of terminal methyl carbon at 19.9 ppm. The carbon of ester group was resonated at 174.0 ppm. The methylene carbon, α - and β - to ester group were resonated at 34.5 and 32.0 ppm respectively and rests of the methylene carbons were resonated at 29.8 ppm. Mass spectrum showed the characteristic fragmentation pattern of long chain aliphatic compound. The

abundant peak at m/z 254 was due to the α -cleavage and loss of one hydrogen atom. The fragment at m/z 214 was due to McLafferty rearrangement showed the presence of ester group at C-12. Thus on the basis of above evidences, compound 3 was characterized as undecyl hexadecanoate and being first time reported by us.

Compound 4: Stigmasterol, M.P. 166-167 °C, M^+ 412, $C_{29}H_{48}O$, Comparison of m.p., IR, ^1H NMR, ^{13}C NMR and Mass spectra of compound 4 with those reported in the literature, identified it as Stigmasterol (Sehrawat et al. 2006 & Sharma & Garg 2009)

Compound 5: β -sitosterol, M.P. 138 °C, M^+ 414, $C_{29}H_{50}O$, Comparison of mp., IR, ^1H NMR, ^{13}C NMR and Mass spectra of compound 5 with those reported in the literature, identified it as β -sitosterol.

Experimental Section

All reported mps are uncorrected. The IR spectra were recorded in KBr on Perkin Elmer-377, ^1H NMR spectra were recorded on 300 MHz Bruker WM spectrometer with TMS as internal standard, ^{13}C NMR spectra on varian XL 75 MHz spectrometer in $CDCl_3$ and EIMS on Jeol-JMS D 300 Mass spectrometer at 70 eV. The column chromatography was carried out on alumina Gr. III and TLC on silica gel G. Spots were visualized by exposure to iodine vapour or by

spraying with H_2SO_4 - vanillin solution followed by heating at 105 °C for 5 minutes.

Plant Material: Bark of *B. monosperma* were collected from the nearby area of Ujjain city and identified by IEMPS, Vikram University, Ujjain, and MP. India.

Extraction and isolation: The bark of *B. monosperma* Lamk. (5 kg) were dried, cleaned and powdered coarsely. It was extracted by acetone and excess of solvent was removed by rotatory film evaporator to afford dark brown solid extract (80 gram). The extract showed positive test for the presence of steroids and terpenoids. This extract was fractionated on alumina grade III column. The column was eluted with various solvent in the increasing order of polarity. The fractions were collected in bulk and monitored by TLC. Repeated chromatography afforded 5 compounds in pure form.

Triacont-3, 17-diene 1: M^+ 418, $C_{30}H_{58}$, (chloroform: methanol, 20 mg), mp. 180 °C, isolated from hexane fraction of hexane elute of the column. TLC (hexane: ether 9.8: 0.2 v/v); IR (KBr): 2922, 2852, 1636, 1460, 1020, 760 and 720 cm^{-1} ; ^1H NMR (300 MHz, $CDCl_3$): δ 0.91 (6H, t, 2x- CH_3), 1.31 (40H, s, 20x- CH_2), 1.6 (8H, brs, 4x- CH_2 , α to unsaturation), 5.02 (4H, t, 2x- $CH=CH$); ^{13}C NMR ($CDCl_3$) (100 MHz) (δ ppm): 117.9, 31.9, 29.6 - 29.1 merged, 25.1, 22.2, 19.9, 14.1; EIMS, m/z (rel. int., %): 418 [M^+] (1.2), 404 (1.1), 390 (1.3), 376 (2.0), 362 (2.0), 349

(2.1), 335 (3.6), 307 (3.6), 293 (2.0), 265 (1.9), 251(3.3), 237 (2.2), 233 (2.4), 210 (2.1), 196 (1.8), 182 (3.2), 168 (4.1), 154 (4.8), 136 (3.1), 136 (7.7), 124 (10.8), 122 (4.4), 110 (21.0), 96 (29.0), 71 (70.2), 57 (100); Anal. Found: C, 86.2, H, 13.8. Calcd for $C_{30}H_{58}$: C, 86.1, H, 13.9 %.

Bromination of 1 (1a) compound 1: (1.5 mg) of compound 1 in diethyl ether (10 %) was treated with drop wise addition of 2-3 ml of bromine in cold condition (-10°C) from a finely drawn pipette, with constant stirring the reaction mixture (Sumitra et al. 2007; Wagner & Geyer 1986). Addition of bromine was continued until a persistent reddish – yellow color was obtained. After standing 25 minutes, excess of bromine was destroyed by adding β -amylene and then it was kept in cold condition till over night. After usual work up, the solid was crystallized by adding cold petro naptha in ethereal solution and identified as 3, 17-dibromo triacont diene.

25-methyl-dotriacont-6, 20-diene-17-one 2. M^+ 474, $C_{33}H_{62}O$, (chloroform: methanol, 25 mg), mp. 240°C , isolated from hexane: benzene fraction of hexane: benzene eluate of the column. TLC (hexane: ether: acetic acid 9:1:1 v/v); IR (KBr): 3020, 2928, 2855, 2709, 1627, 1466, 1410, 1215, 1019, 829 and 728 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 0.88 (6H, t, 2x- CH_3), 5.34 (4H, m, 2x- $\text{CH}=\text{CH}$), 2.29 (4H, t, 2x- CH_2 , α to carbonyl group), 0.94 (3H, d, 2x- CH_3), 1.62 (1H, m, - CH_2 -

$\text{CH}(\text{CH}_3)\text{-CH}_2\text{-}$, 1.25 (44H, br s, 22CH_2)); ^{13}C NMR (CDCl_3) (100 MHz) (δ ppm): 172.0, 118.0, 32.8, 29.7 - 29.3 merged, 25.7, 22.7, 19.9, 14.1; EIMS, m/z (rel. int., %): 474 [M^+] (4.2), 460 (4.0), 446 (9.1), 432 (9.0), 418 (9.0), 405 (4.1), 404 (4.6), 390 (4.8), 389 (4.0), 375 (4.1), 359 (3.9), 319 (4.1), 291 (4.6), 265 (3.6), 258 (21.3), 216 (5.9), 183 (6.2), 182 (5.4), 172 (4.5), 165 (6.7), 153 (7.1), 141 (4.8), 139 (11.4), 137 (10.9), 125 (8.81), 123 (19.4), 109 (34.6), 95 (54.2), 70 (69.2), 56 (100); Anal. Found: C, 83.5, H, 13.1, O, 3.37. Calcd for $C_{33}H_{62}O$: C, 83.5, H, 13.0, O, 3.38 %.

Bromination of 2 (2a) compound 2: (1.8 mg) of compound 2 in diethyl ether (10 %) was treated with drop wise addition of 2-3 ml of bromine in cold condition (-10°C) from a finely drawn pipette, with constant stirring the reaction mixture. Addition of bromine was continued until a persistent reddish – yellow color was obtained. After standing 25 minutes, excess of bromine was destroyed by adding β -amylene and then it was kept in cold condition till over night. After usual work up, the solid was crystallized by adding cold petro naptha in ethereal solution and identified as 6, 20-dibromo derivative.

Undecyl hexadecanoate 3. M^+ 410, $C_{27}H_{54}O_2$, (chloroform: methanol, 25 mg), mp. 110°C , isolated from benzene: ether fraction of hexane: benzene eluate of the column. TLC (benzene: methanol: acetic acid 8:2:1 v/v); IR

(KBr): 2935, 2866, 1737, 1464, 1383, 1179, 1215, 1018, 835 and 720 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 0.86 (6H, t, 2x- CH_3), 4.05 (2H, t, $-\text{CH}_2-\text{O}-\text{CO}_2-$, $J=8.0$ Hz), 2.29 (2H, t, $-\text{CH}_2-\text{CO}-\text{O}-$, $J=7.0$ Hz), 1.50 (4H, br m, 2x- CH_2 , β protons), 1.25 (40H, s, 20x- CH_2); ^{13}C NMR (CDCl_3) (100 MHz) (δ ppm): 174.0, 64.4, 34.5, 32.0, 29.7 - 29.1 merged, 22.7, 19.9, 14.1; EIMS, m/z (rel. int., %): 410 [M^+] (6.6), 397 (6.0), 383(6.2), 372 (4.6), 352 (5.6), 337 (4.5), 310 (4.4), 259 (37.9), 254 (73.2), 184 (11.6), 183 (6.8), 167 (6.6), 140 (15.4), 138 (9.8), 82 (40.2), 69 (100), 57 (77.9); Anal. Found: C, 79.1, H, 13.1, O, 7.8. Calcd for $\text{C}_{27}\text{H}_{54}\text{O}_2$: C, 79.0, H, 13.7, O, 7.8 %.

Alkaline hydrolysis of 3 (3a) compound 3:

(2.5 mg) was refluxed with ethanolic KOH

(2.5 ml, 5%) for 1 hr. at the end of reaction; the mixture was diluted with water (3.0 ml) and extracted with chloroform. The chloroform layer was dried over anhydrous magnesium sulphate and concentrated. To separate both compounds it was put in deep freezer. After 4 days, some solid was separated out. After usual work-up it afforded an alcohol, identified as undecanol and acid 3a respectively (IR: 1702 cm^{-1}).

Stigmasterol 4. M^+ 412, $\text{C}_{29}\text{H}_{48}\text{O}$, (chloroform: methanol, 30 mg), M.P. 166-167 $^\circ\text{C}$.

B-sitosterol 5. M^+ 414, $\text{C}_{29}\text{H}_{50}\text{O}$, (chloroform: methanol, 28 mg), M.P.138 $^\circ\text{C}$.

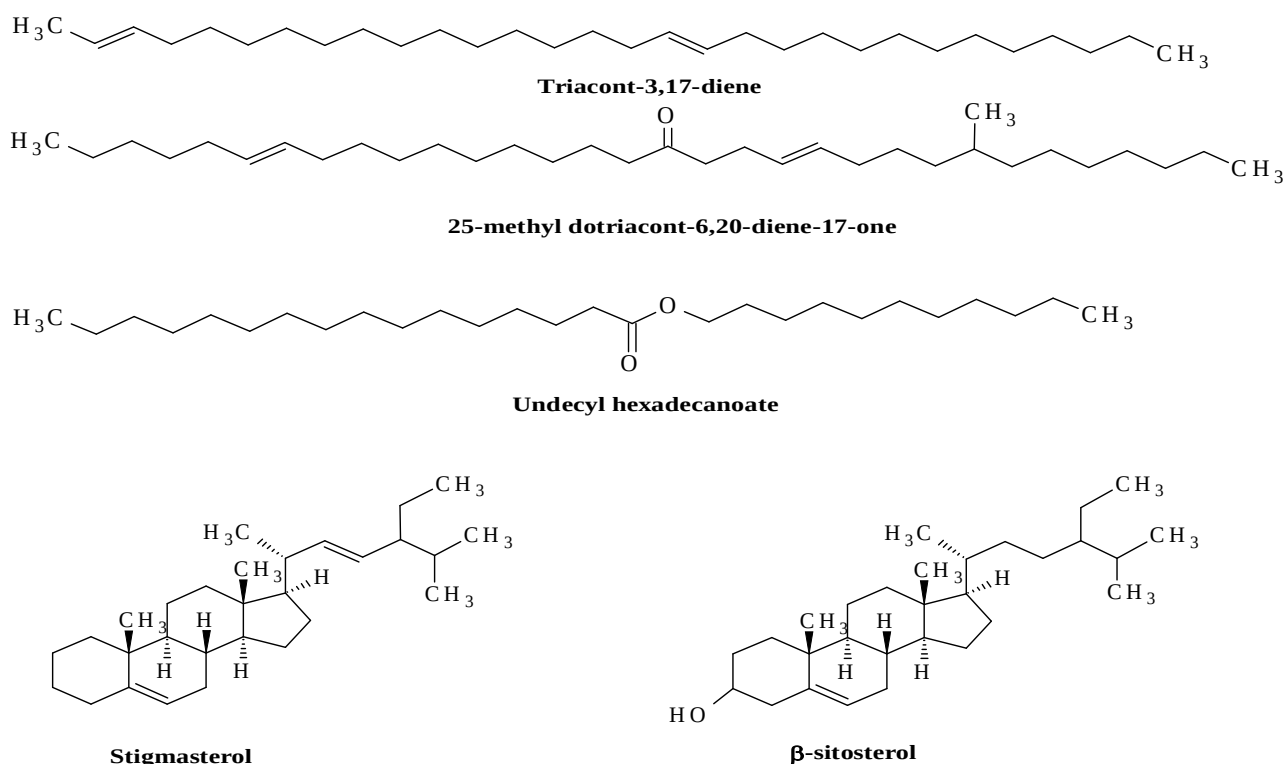


Figure 1: Chemical structure of 1-5

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References

1. Bhargava, S. K. (1986). Estrogenic and postcoital contraceptive activity in rats of butin isolated from *Butea monosperma* seed. *J Ethnopharmacol*, 18(1), 95-101. Retrieved from <http://linkinghub.elsevier.com/retrieve/pii/0378874186900462> pmid:3821138 doi:10.1016/0378-8741(86)90046-2 [Google Scholar]
2. Chopra, R. N., Nayar, S. C., & Chopra, I. C. (1956). *Glossary of Indian Medicinal Plants*. . [Google Scholar]
3. Gunakkunru, A., k Padmanaban,, , & thirumal, P. (2005). Antidiarrhoel activity of butea monosperma in experimental animals. *j of Ethnopharmacology*, 98(3), 241-244. Retrieved from <http://linkinghub.elsevier.com/retrieve/pii/S037887410500019X> pmid:15814254 doi:10.1016/j.jep.2004.12.021[Google Scholar]
4. Iqbal, Z., Lateef, M., & Jabbar, A. (2006). In vivo anthelmintic activity of butea monosperma against trichostrongylid nematodes in sheep. *Fitoterapia*, 77(2), 137-140. Retrieved from <http://www.nlm.nih.gov/medlineplus/herbalmedicine.html> pmid:16388915 doi:10.1016/j.fitote.2005.11.013 [Google Scholar]
5. Kirtikar, K. R., & Basu, B. D. (1935). *Indian medicinal plants*. 2nd ed, 1, 785-88. [Google Scholar]
6. Pandey, H. P. (2001). Seed oil of *Butea monosperma*: A traditional sexual toner and contraceptive. *Ethnobotany*, 13, 118-120.[Google Scholar]
7. Rekha, J. B., & Jayakar, B. (2011). Anti cancer activity of ethanolic extract of Leaves of *Butea monosperma*. *Lam) Taub. Current Pharma Research*, 223, 106-110. [Google Scholar]
8. Sahare, K. N., Anandhraman, V., Meshram, V. G., Meshram, S. U., Singh, V., Reddy, M. V. R., & Goswami, K. (2012). Antifilarial Potential of *Butea monosperma* L. against microfilaria in vitro. *International Journal of PharmTech Research*, 4(3), 1181-1184. [Google Scholar]
9. Sehrawat, A., Khan, T. H., & Sultana, P. S. (2006). *Butea monosperma* and chemo modulation: protective role against thioacetamide-mediated hepatic alterations in wistar rats. *Phytomedicine*, 13(3), 157-163. Retrieved from <http://linkinghub.elsevier.com/retrieve/pii/S0944711305001571> doi:10.1016/j.phymed.2004.11.007 [Google Scholar]
10. Sharma, N., & Garg, V. (2009). Antidiabetic and antioxidant potential of ethanolic extract of *Butea monosperma* leaves in alloxan-induced diabetic mice.. *Indian J Biochem Biophys*, 46(1), 99-105. Retrieved from <http://toxnet.nlm.nih.gov/cgi-bin/sis/search/r?dbs+hsdb:@term+@rn+50-71-5> pmid:19374261 [Google Scholar]
11. Sumitra, M., Manikandan, P., & Suguna, (2007). I.(2007) Efficacy of butea monosperma on dermal wound healing in rats. *Int j biochem cell biol*, 37, 566-73. [Google Scholar]
12. Wagner, H., & Geyer, B. (1986). isoputrin and butrin, the antihepatotoxic principles of butea monosperma flowers. *Planta Med*, 52(2), 77-79. Retrieved from <http://www.thieme-connect.de/DOI/DOI?10.1055/s-2007-969083> pmid:3725938 doi:10.1055/s-2007-969083 [Google Scholar]