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EVALUATION OF CHEMICAL CONSTITUENTS OF *BUTEA MONOSPERMA* (BARK)

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ABSTRACT: From TLC examination of fractions of benzene and benzene: ether (5:5) extract of the stem bark of *B. monosperma* revealed the separable mixture. These fractions were subjected to rechromatography. Isolated compounds were purified and crystallized by chloroform: methanol. After isolation and purification afforded white crystalline substance which was subjected to physical, chemical and spectral analysis and identified as Tetratriacont -15-ene(1), Hepta cos-11-ene(2), 15-hydroxyl ethylheptadec-12-enoate(3), 10-hydroxy dodecyltridec-5 enoate(4), based on spectral evidence.

INTRODUCTION: *Butea monosperma* (lam.) / kuntze (bark) locally known as palas, dhak, and the flame of the forest is belonging to family *Leguminosae*. The bark of this plant has been reported in folk medicine as a cure for snakebite, skin disease, bone fractures, rectal disease, ulcers, tumors, hydrocele, and diabetes. The bark is acrid, bitter, astringent, digestive, constipating, anthelmintic, and tonic [1-3]. Earlier studies on this plant have resulted in the isolation of flavonoids and flavonoid glycosides, as palastrin, isobutein, coreopsin, monospermacide, and isomono-spermacide [4-6]. In this communication, we have discussed the isolation and characterization of novel compounds.

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EXPERIMENTAL: Collection, identification, and preparation of plant materials (extract). The bark of the plant was collected from the nearby area of Ujjain city in March. The plant material bark was identified from the school of studies in Botany Vikram University Ujjain. The bark of Plant was shaded, dried and powdered.

Extraction and Isolation: Powdered (15kg) bark of *B. monosperma* was extracted exhaustively with hexane, hexane: benzene, benzene, and benzene: ether in a Soxhlet extractor. The solvent was recovered by the rotatory evaporator, under vacuum pressure, to afford dark greenish-brown oily mass. In this communication, we have studies only benzene extract and benzene: ether extract of *B. monosperma* (bark).

Spectroscopic Characterization: Different spectroscopic method were used to elucidate the structure of isolated compounds among the spectroscopic techniques IR, ^{1}H-NMR, mass Spectra were carried out IR spectra were recorded

in KBr on Perkin Elmer-377. The ^{1}H-NMR spectra were recorded on 300MH$_Z$ XL spectrometer and 200MHz Brucker WM spectrometer with TMS as an internal standard. The mass spectrum was recorded on Jeol–JMSD-300 mass spectrometer. The column chromatography was carried out on alumina Grade third and TLC on silica gel. The spots were visualized by exposure to iodine vapor or charring with conc. H_2SO_4- Vanillin spray.

Compound (1): The IR spectrum (λ_{max} KBr, cm^{-1}) the IR spectrum showed absorption peak at 3415, 2923, 2853 1639, 1463, 1378 ,1216,1020,761, & 735-20 cm^{-1} .

^{1}HNMR (CDCL$_3$ 200MH$_Z$, ð) ^{1}HNMR has given signals at ð0.90 (t, 6H, 2-CH$_3$), ð1.25 (S, 56H, 28-CH$_2$), ð1.58 (m, 4H, 2-CH$_2$), ð5.18 (m, 2H, CH=CH).

EIMS: m/z (rel. int., %): Mass spectrum showed molecular ion peak at m/z 476 and molecular formula $C_{34}H_{68}$ other ion peaks were also observed at M$^+$ 476 (10), 448 (14.0), 420 (12), 406 (6.0), 364 (2.0), 351 (4.6), 337 (6.7), 323 (8.7), 309 (4.6), 295 (10), 280 (27), 267 (26), 253 (20), 39 (20), 211 (3.8), 183 (5.3), 155 (6.7), 125 (11.6), 111 (28), 97 (13), 85 (42), 71 (56), 57 (100).

Compound (2): The IR spectrum (λ_{max} KBr, cm^{-1}) the IR spectrum showed absorption peak at 2923, 2852, 1463, 1377, 1215, 1019, 761, & 730-20 cm^{-1}.

^{1}HNMR (CDCL$_3$ 200MH$_Z$, ð) ^{1}HNMR has given signals at ð0.90 (t, 6H, 2-CH$_3$), ð1.29 (S, 42H, 21-CH$_2$), ð1.45 (m, 4H, 2-CH$_2$), ð5.02 (t, 2H,CH=CH).

EIMS: m/z (rel. int.,%): Mass spectrum showed molecular ion peak at m/z 378 and molecular formula $C_{27}H_{54}$ other ion peaks were also observed at M$^+$ 378 (12), 364 (31), 351 (28), 337 (4.9), 309 (4.4), 632 406 (6.0), 364 (2.0) 351 (4.6), 337 (6.7), 323 (8.7), 309 (4.4), 295 (7.20), 281 (3.12), 266 (4.6), 253 (9.0), 239 (6.8), 225 (5.8), 211 (7.9), 197 (6.7), 183 (6.71), 169 (78.0), 155 (12.1), 141 (6.8), 125 (84), 111 (34), 97 (64), 85 (75), 71 (62), 57 (100).

Compound (3): The IR spectrum (λ_{max}. KBr, cm^{-1}) the IR spectrum showed absorption peak at 3407, 2921, 2851, 1731, 1635, 1463, 1379, 1247, 1175, 1112, 1015, 980, 757, & 719 cm^{-1}.

^{1}HNMR (CDCL$_3$ 200MH$_Z$, ð) ^{1}HNMR has given signals at ð0.89 (t, 6H, 2-CH$_3$), ð1.25 (S, 14H, 7-CH$_2$), ð1.56 (m, 11H, 5-CH$_2$, H), ð2.30 (t, 2H, CH$_2$-CO-O) ð3.69 (m, 1H, -CHOH), 4.05 (m, 2H, CH$_2$-CO-O), 5.34 (m, 2H, CH=CH).

EIMS: m/z (rel. int., %): Mass spectrum showed molecular ion peak at m/z 312 and molecular formula $C_{19}H_{363}O_3$ other ion peaks were also observed at M$^+$ 312 (10), 284 (14), 254 (5.0), 339 (6.0), 213 (5.6), 185 (5.2), 149 (12.0), 129 (10), 88 (69), 71 (50), 68 (48), 56 (89), 43 (100).

Compound (4): The IR spectrum (λ_{max}. KBr, cm^{-1}) the IR spectrum showed absorption peak at 3465, 2935, 2868, 1753, 1622, 1464, 1377, 1224, 1164, 1042, 907 & 720 cm^{-1} .

^{1}HNMR (CDCL$_3$ 200MH$_Z$,ð) ^{1}HNMR has given signals at ð0.89 (t, 6H, 2-CH$_3$), ð1.25 (S, 22H, 11-CH$_2$), ð1.55 (m, 12H, 6-CH$_2$ 2, H), ð2.30 (t, 2H, CH$_2$-CO-O), ð3.65 (m, 1H, -CH), 4.09 (t, 2H, CH$_2$-CO-O), 5.34 (m, 2H, CH=CH), 1.57 (S, 1H, OH).

EIMS: m/z (rel. int., %): Mass spectrum showed molecular ion peak at m/z 396 and molecular formula $C_{25}H_{48}O_3$ other ion peaks were also observed at M$^+$ 396 (2.5), 395 (74.1), 381 (23.2), 366 (4.5), 330 (14.2), 303 (14.2), 288 (5.2), 254 (9.3), 228 (5.7), 254 (9.3), 186 (7.0), 213 (5.8), 186 (7.0), 168 (68.0), 145 (14.7), 127 (11.1), 109 (24.5), 95 (14.2), 81 (13.5), 71 (15.6), 57 (20.7), 44 (100).

RESULT AND DISCUSSION: The benzene and benzene: ether (5:5) extracts of the bark were subjected to column rechromatography over alumina gr. III and silica gel. We have isolated compounds. Compound (1) and (2) were isolated from benzene extract, and compounds (3 & 4) were isolated from benzene: ether extract.

Compound (1): Tetratriacont – 15 ene, M$^+$ 476, $C_{34}H_{68}$ Compound isolated from rechromatography over silica gel. Column, The column eluate from benzene afforded white wax like compound its M.P. 68- 69 ºC, it exhibited IR absorption bands at 3415, 2953, 2853, 1463, 1020, 761 and 730-720 cm^{-1}, due to long-chain aliphatic nature of the molecule. A band at (1639 cm^{-1}) indicated the presence of unsaturation [12-13]. The PMR spectrum of compound (1) showed triplet at δ 0.9 (t, 6H) for

terminal methyl groups. Multiplet at δ 5.18 (2H) and 1.58 (4H) due to olefinic proton and methylene groups α to olefinic proton. A broad singlet at δ 1.25 for (56-H) protons showed the presence of (28-CH_2) units [14-15]. All the above evidences suggested that the compound contain unsaturated long-chain aliphatic compound. The position of olefinic bond at C-15 was determined by its mass fragmentation pattern, highly abundant fragment at m/z 280 obtain by by α – cleavage and hydrogen transfer, most of the fragments were separated by 14 mass units were appeared at m/z 363, 309, 287, 253, 239, 211, 183 and 155. On the basis of the above evidences, compound (1) was identified as tetratriacont–15- ene.

Compound (2): Heptacos–11-ene, M^+378, $C_{27}H_{54}$ (2:8 ethtlacetate: benzene) fraction was subjected for rechromatography to over silica gel, Column, the hexane eluate afforded white compound, M.P. 60-62 ºC, its IR absorption band at 2923, 2852, 1463 and 730-720 cm^{-1} were considered to be aliphatic nature of the molecule. A band at 1622 cm^{-1} was indicated the position of double bond [12-13]. The PMR spectrum displayed the two triplets at δ 0.9 (6H) and δ 5.2(2H) corresponding to terminal methyl groups and olefinic protons. A multiplet at δ 1.45 was assigned to the methylene proton merged into single peaks at δ 1.29 (42H, 21-CH_2). All these data indicated compound (2) to be long-chain unsaturated aliphatic [14-15]. The position of double bond was determined by its mass spectrum. The presence of relatively high abundant fragments at m/z141, 169, 211 and 239 formed by α cleavage and transfer of hydrogen confirm the position of a double bond at C-11 [16]. Thus, the compound was identified as Heptacos- 11- ene.

Compound (3): 15-hydroxy ethylheptadec-12-enoate, M^+ 312, $C_{19}H_{35}O_3$ isolated from benzene: ether extract, rechromatography of benzene: ethylacetate fraction over silica gel column. (hexane: benzene) eluate afford white crystal of compound (3) M.P.88-89 ºC. The IR bands at 3407 cm^{-1} (OH) 1731 cm^{-1} (ester group) A weak band at 1635 cm^{-1} (double bond), other bands at 2921, 2951, 1463, 1379, 1247, 1175 and 720 c m^{-1} (long-chain aliphatic nature) [12-13]. Its PMR Spectrum showed signal at δ 0.89 were assignable to the presence of two terminal methyl groups, hydroxyl proton was assigned at δ 1.56 as a siglate for (OH group). The carbinolic proton was assigned at δ. 3.69 (m, 1H). The methylene proton of δ to ester group and the hydroxy group were assigned at δ. 1.56. Two triplets at δ 4.05 and δ 2.30 for methlene protons of – CH_2-O-CO and CH_2CO respectively [14-15]. Rest of the methylene merged into a single peak at δ 1.25. All the above evidence suggested that the compound is an unsaturated aliphatic hydrocarbon having hydroxyl group and an ester group. The exact position of the ester group and other group confirm its Mass spectra peaks at m/z 239, 254, were due to the α-cleavage indicated the position of the double bond and a hydroxyl group at C-12 and C-15 respectively [16]. Thus, the compound (3) identified as 15- hydroxy ethyleptadec – 12- enoate.

Compound (4): 10- hydroxy dodecyltridec -5-enoate, M^+396, $C_{25}H_{48}0_3$. Isolated from benzene: ether extract over silica gel Column. The benzene: ethyl acetate fraction (2:8) was subjected to rechromatography (ethylacetate: benzene) eluate afford compound (4), M.P. 98-100 ºC. IR bands at 346cm^{-1} (OH), 1753cm^{-1} (ester group), 1622 cm^{-1} (double bond), other bands at 2935, 2868, 1464, 1377, 1247, 1175 and 720 cm^{-1} (CH stretching and bending vibration suggested that unsaturated aliphatic nature of the molecule [12-13].

It's PMR spectrum the signal at ð.89 (t, 6H) was assignable to the presence of two terminal methyl groups. The carbinolic proton was assigned at δ 3.65 (m, 1H) the methylene proton of ß- to ester group and hydroxyl group were assigned at δ 1.57 the methylene merged into a single peak at δ 1.25. it to be a long chain unsaturated ester [14-15]. All the above evidence suggested that the compound contain unsaturated aliphatic hydrocarbon having hydroxyl and ester group. The exact position of the ester group, hydroxyl group, and double bond were ascertained from its Mass spectrum. It showed an M^+ at m/z 396 abundant fragments at m/z 168, 213 and 109 were obtained due to α- & ß- cleavage of (OH) and (ester group) Indicated the position pf hydroxyl group and ester at C-10 and C-5 respectively [16]. Their fragments obtained at m/z 381, 254, 186, 145 and 95 were all agreement with the proposed structure of the compound(4) Thus the compound(4) identified as 10 hydroxy dodecyl tridec-5- enoate.

CH_3-$(CH_2)_{17}$-CH=CH-$(CH_2)_{13}$-CH_3 tetratriacont - 15-ene (1)

CH_3-$(CH_2)_9$-CH=CH-$(CH_2)_{14}$-CH_3 hepta cos-11-ene (2)

CH_3-CH_2-O-C__CH_2-$(CH_2)_8$-CH_2-CH=CH-CH_2-CH-CH_2CH_3

15-hydroxyl ethylheptadec-12-enoate (3) OH CH_3_$(CH_2)_2$ _CH _$(CH_2)_3$_CH=CH$(CH_2)_3$-CO-$(CH_2)_{11}$-CH_3

10-hydroxy dodecyltridec-5 enoate (4)

CONCLUSION: From the above discussion, these compounds isolated from benzene extract and benzene: ether (5:5) extract fractions after rechromatography and their structures identified by physico-chemical techniques**.**

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CONFLICT OF INTEREST: Nil

REFERENCES:

1. Ayra VR and Kottakkal V: Indian Med. Plants (Lalit Mohan Basu, Allahabad) I. 1994; 314.
2. Kirtikar KR and Basu BD: Indian Medicinal Plants 1936; 1(4): 896.
3. Chopra RN, Nayar SC and IC: Glossary of Indian Medicinal Plants (CSIR New Delhi) 1956.
4. Madhuv TR and Trikha RN: Ind J Chem 1927; 9(11): 1201-03.
5. Murti P, Bhaskar K and Subramanium GDV: Curr Sci 1946; 12(B): 472-76.
6. Lal JB and Dutta SJ: Ind Chem Soc 1935; 12: 262-67.
7. Bellamy LJ: The Infrared Spectra of Complex Organic Molecules, (Chapman and Hall London) 1975; 152.
8. Dyer JR: Applications of absorption Spectroscopy of Organic Compounds, (Prentice Hall of India, New Delhi) 1984; 43.
9. Silverstein RM, Bassler GC and Morril TC: Spectrometric Identification of Organic Compounds, (John Wiley & Sons Inc., New York) 1974; 142.
10. Jackman LM and Sternhell S: Application of Nuclear Magnetic Resonance Spectroscopy in organic Chemistry (Permon press Oxford) 1964.
11. Mclafferty FW" Interpretation of Mass spectrum, (WA Benjamin Inc., New York) 1973; 112.

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