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Chemical Investigation on the Leaves of *Hibiscus Furcatus*

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The petroleum ether (60°–80°C) extract of the leaves of *Hibiscus furcatus* yielded five components. They were identified to be friedelin, β-sitosterol, taraxerol, a mixture of *n*-alkanes in the C_{30}–C_{34} range and a mixture of n-alkanols in the C_{28}–C_{34} range by chemical and physical methods.

INTRODUCTION

Hibiscus furcatus belongs to the section furcaria of the family Malvaceae. It is a prickly trailing herb found in almost all parts of the world having large yellow flowers with purple centre.[1] The leaves of this plant are acidic and eaten after cooking. They are said to improve digestion and are considered to be anthelmintic. The juice of leaves mixed with honey is applied to eye diseases. A decoction of the root bark is given as a remedy for poisons and swellings and for cleansing kidneys.[2] L-Allohydroxycitric acid has been reported to be present in the leaves.[3] The flowers are reported to contain gossypin, gossypitrin and a flavonolglycoside hibiscatin.[4] In the present work, the leaves of *Hibiscus furcatus* have been chemically investigated.

EXPERIMENTAL

The plants were collected from Thrissur district of Kerala State and identified botanically. Air dried and finely powdered leaves of *H. furcatus* (2 kg) were extracted repeatedly with petroleum ether (60°–80°C, 6 L) and the combined extract was concentrated under reduced pressure to about 500 mL. This was then adsorbed on 750 g of neutral alumina and packed tightly in a column (3 cm × 100 cm; d × 1) and eluted with benzene (2 L), 3 : 1 benzene-ethyl acetate (2 L) and with ethyl acetate alone (1 L). Combined benzene fractions were concentrated under reduced pressure and subjected to column chromatography over silicagel column (3 cm × 100 cm; d × 1), eluted with petroleum ether, benzene, benzene-ethylacetate and ethyl acetate alone. Altogether five components were isolated from the petroleum ether extract.

Component 1 (n-alkanes): This component was isolated from the petroleum ether extract on elution with petroleum ether (60°–80°C) and recrystallised from acetone as a white powdery substance (150 mg). It melted at 64°C. Absorptions

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(1918.7, 2849.2, 1478.8, 1464.1 cm^{-1}) in the IR spectrum showed the presence of only C—H and C—C bonds. The absorptions at 734 and 719 cm^{-1} were indicative of the presence of a long hydrocarbon chain. ^1H NMR (absorptions in the region δ 0.9 – 1.65) and ^{13}C NMR spectrum (absorptions at δ 14.1, 22.69, 25.05, 25.45 and a number of absorptions around 29 and another at 31.93) also supported the straight chain hydrocarbon nature. The EI Mass spectrum of the compound has the base peak at m/z 57 and the maximum m/z value at 478. The high intensity of the peaks at m/z 464 and 436 were suggestive of a mixture of compounds. GC-MS analysis of this compound [Varian 3400 GC with 0.3 μm fused silica column (permaphase PVMS/54, I.D. 0.25 mm of Ca 22 m). The mass spectrometer used was funnigan MAT TSQ 70] confirmed that this component was a mixture of hydrocarbons in the C_{30}–C_{34} range, major component being $C_{31}H_{64}$, hentriacontane (M^+ at 436, 60%). Another major component was $C_{33}H_{68}$, tritriacontane (M^+ at 464, 30%). Minor components were corresponding to C_{30}-, C_{32}-, and C_{34}- hydrocarbons.

Component 2 (Friedelin)—This component was isolated from the petroleum ether extract on elution with benzene and recrystallised from acetone as colourless crystalline needles (500 mg) which melted at 252°C. It gave positive Liebermann-Burchard reaction showing persistent pink colour, typical for triterpenes. EI Mass spectrum of this compound showed M^+ at m/z 426. The base peak at m/z 205 and fragmentation pattern with m/z 411, 341, 302, 287 and 274 indicated it to be a friedelin type triterpenoid. IR spectrum showed a sharp peak at 1716.9 cm^{-1}, suggesting the presence of a >C=O group. Other IR absorptions were at 1972.7, 2870.4, 1464.1 1388.9, 1109.2 and 1072.6 cm^{-1}. ^1H NMR (absorptions in the region δ 0.7–1.03) showed the presence of seven tertiary methyl singlets and one secondary methyl doublet. Melting point, IR and all other spectral data were quite comparable with that of friedelin, reported in literature.[5, 6]

Component 3 (Taraxerol): This component obtained on elution with 3 : 1 benzene-ethyl acetate, as colourless crystals, m.p. 272°C, also gave a positive Liebermann-Burchard colour reaction for triterpenes. The molecular mass of this compound was found to be 426 from its EI mass spectrum. The IR spectrum showed the absorption band of a hydroxyl group at 3490 cm^{-1}. The ^1H NMR spectrum showed eight tertiary methyl singlets (δ 0.8 – 1.09) indicating that it belonged to the pentacyclic group of triterpenes. The ^1H and ^{13}C NMR spectroscopy showed the presence of a trisubstituted double bond [^1H NMR δ 5.53 (1H, vinylic proton); ^{13}C NMR: δ 158.2 (unsaturated quaternary carbon), δ 116.9 (unsaturated CH carbon)] and a secondary hydroxyl group [^1H NMR: δ 3.2 (1 H carbinol methine proton), ^{13}C NMR: δ 79.2 carbinol methine carbon]. EI Mass spectrum showed M^+ at m/z 426. Other peaks were at m/z 411 ($M^+ - CH_3$), 408 ($M^+ - H_2O$), 393 ($M^+ - CH_3 - H_2O$), 302, 287, 269. The physical constants, ^1H, ^{13}C NMR and EI mass spectrum were found to be comparable with that of taraxerol in the literature[7, 8].

Component 4 (β-Sitosterol): This component was isolated from the petroleum ether extract on elution with 3:1 benzene-ethylacetate as a white powdery substance. Recrystallised from methanol to colourless needles (600 mg) melting at 139°C. It showed a play of colours with Liebermann-Burchard reagent

indicating that it was a sterol. EI mass spectrum of this compound showed M^+ at m/z 414 and base peak at m/z 55. The other prominent peaks were at m/z 399 ($M^+ - CH_3$), 381 [($M^+ - CH_3 + H_2O$)], 273m ($M^+ - C_{10}H_{21}$), 231 ($273 - C_3H_6$) and 213 ($231 - H_2O$). IR absorption spectrum showed the absorption of a hydroxyl group at 3441.4 cm^{-1}. Bands due to *gem*-dimethyl group (doublet at 1383.1 and 1385.1 cm^{-1}), —C—H stretching and bending bands were also observed in the spectrum. ^1H NMR spectrum of this compound was quite comparable with that for β-sitosterol. The acetate and benzoate[9,10] of this compound were prepared which found to be comparable with β-sitosterol acetate and β-sitosterol benzoate respectively.

Component 5 (n-alkanols): This component was isolated from the petroleum ether extract on elution with 3:1 benzene-ethylacetate as white powdery substance (650 mg); recrystallised from benzene and had m.p. 82°C. This component did not respond colour reaction with Liebermann-Burchard reagent. IR spectrum showed the presence of hydroxyl group (broad absorption at 3450 cm^{-1}). Other prominent absorption bands were at 2918.7, 2849.2 (doublet, —CH stretching), 1478, 1462.2 (doublet, —C—H bending), 1061.9 (C—O stretching), 734.1 and 719 cm^{-1} (long chain aliphatic compound), ^1H NMR (absorptions in the region δ 0.8–1.6) and ^{13}C NMR spectrum (absorptions at δ 14.1, 22.65, 25.72, a number of absorptions around 29 and absorptions at 31.92, 32.31) indicated a long hydrocarbon chain. The prominent ^{13}C NMR absorption at δ 63.11 indicated the presence of C—O group in the compoound. EI Mass spectrum of the component had the base peak at m/z 57 and maximum m/z value at 476. The other high intensity peaks at m/z 448, 420 were suggestive of a mixture of compounds. This was confirmed by GC-MS analysis of this compound (Varian 3400 GC) with 0.3 μm fused silica volume (Permaphase, PVMS/54, ID 0.25 mm of Ca 22 m). The mass spectrum did not show any molecular ion peak but characteristic ions at ($M^+ - H_2O$). This compound was identified as a mixture of long chain 1-alkanols, the major component being dotriacontanol, $CH_3-(CH_2)_{30}-CH_2OH$ (45%). Other major compounds were triacontanol, $CH_3-(CH_2)_{28}-CH_2OH$ (25%), tetratriacontanol, $CH_3-(CH_2)_{32}-CH_2OH$ (15%) and octacosanol, $CH_3-(CH_2)_{26}-CH_2OH$ (8%) together with minor components of this series with 29, 31 and 33 carbon atoms each.

RESULTS AND DISCUSSION

Benzene fraction of the petroleum ether extract, of *Hibiscus furcatus* leaves on column chromatography gave five components. Component 1, m.p. 64°C, was identified to be a mixture of long chain hydrocarbons in the C_{30}—C_{34} range, the major components being $C_{31}H_{64}$ and $C_{33}H_{68}$. Component 2, m.p. 252°C, giving persistent pink colour with Liebermann-Burchard reagent, was identified to be friedelin. Component 3, m.p. 272°C, also giving persistent pink colour with Liebermann-Burchard reagent was identified to be taraxerol. Component 4, m.p. 139°C, giving a play of colours with Liebermann-Burchard reagent, was identified to be β-sitosterol. Component 5, m.p. 82°C, was identified to be a mixture of long chain 1-alkanols, the major components being $C_{32}H_{66}O$ and $C_{30}H_{62}O$.

Although friedelin is a commonly occurring tritèrpenoid, this is the first report of isolation of this compound from *Hibiscus* species of Malvaceae family.

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