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Isolation and structural elucidation of 20 hydroxyecdystone from *Vitex doniana* sweet stem bark (black plum)

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Vitex doniana sweet, a plant commonly known black plum, in English, prunier noir in French, dinya in Hausa, ucha koro in Igbo, oori-nla in Yoruba and ngarmi in Kanuri, is a medium-sized deciduous tree, 8-18 m high, with a heavy rounded crown and a clear bole up to 5 m. *V. doniana* is from Verbenaceae family and abundantly occurring in savannah regions. It can be found throughout tropical Africa. The ethanolic extract of *Vitex doniana* stem bark (11.9 g) was subjected to a silica gel accelerated column chromatography and eluents fractions (150 ml aliquots) obtained were collected and monitored with thin layer chromatography (TLC). Fractions with similar R_f values from same solvents system were pooled together. Phytochemical test of all the fractions were performed. Complete elution yielded 48 fractions (150 ml/fraction) which were pooled to 24 fractions and finally to eight fractions and coded. Fraction Vd_{8-a} (56 mg) has given a single spot a white crystal compound coded V_1 on checking with TLC and observed under Ultraviolet lamp. The R_f value was calculated to be 0.433 and melting point was found to be 241-243°C uncorrected. The infrared spectrum of compound V_1 shows prominent peaks that correspond to OHstr (3365 cm^{-1}) and C=O (1652 cm^{-1}). The ^1H NMR (400 MHz) spectrum of compound V_1 in DMSO- d_6 displayed five singlet signals. It further showed a broad singlet at δ 5.58 integrated for 1 H is due to an olefinic H-atom adjacent to the carbonyl carbon atom. Three signals at δ 3.10 (d, $J=9.0\text{ Hz}$, H-22), 3.59 (m, 1H, 2H-a) and 3.72 (m, 1H, 3H-e) each integrating for one proton is due to oxymethine protons indicating that three oxymethine H-atoms were present in the compound. The ^{13}C -NMR spectrum showed the presence of 27 carbon atoms, suggesting that may be steroid skeleton and DEPT-135 spectra showed the presence of five CH_3 , eight CH_2 , and seven CH groups, and seven quaternary C-atoms. The molecular formula was established as $\text{C}_{27}\text{H}_{44}\text{O}_7$ by HRES-MS positive ion mode m/z 481.3179. Based on the spectral analysis, the compound V_1 is thus concluded to have ecdysteroids skeleton and conclusively confirms with 2β , 3β , 14α , 20R , 22R , 25-hexahydroxy-5 β cholest-7-ene-6-one, commonly known as 20-hydroxyecdysone. This is the first time this compound was isolated from *Vitex doniana* sweet.

Biography

Mustapha A Tijjani has expertise in "Extraction, phytochemical analysis, isolation of compounds & structural identification and elucidation as well as pharmacological evaluation of compounds or extracts from plants". He has worked with many indigenous plants in North Eastern Nigeria that have analgesic, anti-inflammatory, anticonvulsant and antipyretic properties.

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