Chemical Investigation of the Flowers of Striga senegalensis Benth⁺

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The ethylacetate extract of flowers of the plant *Striga senegalensis* Benth after defatting with n-hexane yielded a yellow coloured crystalline compound (from methanol), mp. 350°, characterized as apigenin (4', 5, 7-trihydroxyflavone, $C_{15}H_{10}O_5$) on the basis of spectral analysis (UV, IR, NMR and Mass.).

The plant Striga senegalensis Benth (family: Scrophulariaceae) is an erect annual, widespread in the tropics of Egypt, Gambia, Ghana, Mali, Nigeria, Niger Republic, Senegal and Sudan. It is a parasitic weed of food crop cereals such as rice, millet, maize and sorghum roots1-3. The local names of the plant in Hausa are 'Kudiji,' 'Makasar dawa' (killer of guinea corn), 'Dodon dawa' and 'Wuta wuta.' In some parts of Africa, the plant is used for the treatment of leprosy and leprous ulcers. In East Africa, a decoction and infusion of the roots is administered orally as an abortifacient and in the treatment of prieumonia. In Northern Nigeria a decoction of the plant is drunk and fresh leaves are rubbed on to the skin for the treatment of fungal infections4. The pharmacological studies of this plant as an abortifacient tested on albino rat uterus5 and antifertility activity in female albino rats⁶ have been reported recently. The present article describes the isolation and characterization of apigenin (4, 5, 7-trihydroxyflavone, C₁₅H₁₀O₅, Fig. 1) from the flowers of Striga senegalensis on the basis of UV, IR, NMR (proton, C-13) spectral data and Mass spectrometry.

All solvents obtained from BDH, England were distilled before use. Silica gel for column chromatography (M and B Laboratory Chemicals, May and Baker Ltd, Dagenham, England), silica gel for TLC (E. Merck, Germany) and iodine for visualization of the spots were used. Melting points were determined using a Gallenkamp apparatus. The UV spectrum was recorded on a Pye Unicam 5000 Spectrometer SP 8-100.

The flowers of the plant were collected from maize fields in Ahmadu Bello University campus, Zaria, Nigeria in the months of October to November and authenticated by the herbarium at the Department of Biological Sciences, Ahmadu Bello University. A herbarium sample was prepared and a voucher deposited. The air dried and powdered plant material (39.2 g) was extracted with n-hexane (Soxhlet) for 48 h. The defatted marc was dried in air and then extracted with ethylacetate (Soxhlet) for 48 h. The

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solvent was concentrated to give a green solid (1.28 g) which was purified by column chromatography. Elution with hexane-ethylacetate (1:1) afforded an amorphous yellow coloured compound which furnished a yellow crystal after crystallization from methanol (200 mg), mp, 350°.

The compound gave a positive Shinoda test for flavonoid and on tlc it gave a R₁ of 0.5 in benzeneethylacetate 1:1 solvent system. UV absorbance maxima were found at 220, 276 and 348 nm in MeOH.

MeOH+anhydrous AlCl $_3$ +HCl : 235, 250, 332, 395 nm MeOH + fused NaOAc : 230, 283, 392 nm MeOH + 2 drops dil NaOH : 228, 282, 400 nm.

IR (Nujol): 3303 (4'-OH), 3083 (7-OH), 2954 (weak, 5-OH), 1658 (4-keto chelated to 5-(OH), 1556 (aromatic).

PMR (DMSO-d_e, 400 MHz, δ): 6.2 (1H, s, H-6), 6.5 (1H, s, H-8), 6.8 (1H, s, H-3), 6.89 and 6.94 centered at 6.92 (2H, d, J = 8.8 Hz, H-3, H-5, ortho coupling with H-2' and H-6' respectively), 7.91 and 7.93 centered at 7.92 (2H, d, J = 8.8 Hz, H-2, H-6, ortho coupling with H-3' and H-5 respectively), 10.35 (1H, s, 4'-OH), 10.85 (1H, s, 7-OH), 12.97 (1H, s, 5-OH).

Decoupling PMR: Irradiation of the signal centered at 7.92 gave a singlet at 6.92. Similarly, irradiation at 6.92 resulted a singlet at 7.92 indicating the mutual coupling between H-24 and H-3'; H-5' and H-6.'

C-13 NMR (DMSO-d_g, 100 MHz, DEPT, δ); 93.91 (C-8, CH), 98.80 (C-6, CH), 102.81 (C-3, CH), 103.69 (C-10, QC), 115.92 (C-3, C-5, CH), 121.16 (C-1, QC), 128.40 (C-2, C-6, CH), 157.28 (C-5, QC), 161.13 (C-4, QC), 161.44 (C-9, QC), 163.71 (C-7, QC), 164.09 (C-2, QC), 181.71 (C-4, CO).

Mass (EI): m/z 270 (M+, 100%, base peak).

The compound gave a positive Shinoda test for flavonoids and a violet black colouration with dilute FeCl₃ indicating the presence phenolic hydroxyl group(s). The UV spectrum in methanol exhibited two major absorption bands at 276 nm (Band II) and 348 nm (Band I), typical of a 5-hydroxyflavone^{7,8}. The bathochromic shift of 47 nm in Band I (ring B) on addition of anhydrous AlCl₃+HCl

showed the presence of 5-OH in ring A^{7,9}. Addition of fused NaOAc shifted the absorption of Band II (in ring A) by 7 nm indicating the presence of 7-OH group. On addition of 2 drops of dil NaOH, Band I shifted 52 nm towards longer wave length (bathochromic shift). This was typical of flavones containing a free 4'-OH group in ring B¹⁰. The UV, IR and NMR (proton, C-13) spectra revealed the presence of three OH groups situated at C-4,' C-5 and C-7 in the flavone nucleus. The compound showed a molecular ion peak (M⁺) at m/z 270 (100%). No phytochemical study was reported before on this plant. This is the first report of a flavonoid compound, apigenin in *Striga senegalensis*. Further work is in progress in isolation and determining the active constituent present in the plant.

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