CHEMICAL STUDIES OF NEW ISOLATED FLAVONE

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ABSTRACT
A new compound 5, 7, 4′-trihydroxy-3-(3′-methyl butyl) flavone has been isolated from the bark of Pterocarpus marsupium. Pterocarpus marsupium is big tree. Different parts of this tree are used to cure many diseases as diarrhoea, elephantiasis, leucoderma, erysipelas urethrorrhoea, diabetes, rectalgia, rectitis, opthalmopathy, dysentery, cough, asthma, greyness of hair, fractures bruises, inflammations, leprosy and skin disease, etc. The structure of new isolated 5, 7, 4′-trihydroxy-3-(3′-methyl butyl) flavone compound has been studied with the help of synthesis, Chemical degradation and spectra.

Keywords: ¹H NMR, IR, KBr and Methanol.

I INTRODUCTION
Pterocarpus marsupium is multipurpose tree which gives very good timber to society. It’s called by different names depending upon the name of region as, Malbar Kino tree (English), Venga (Malyalam and Bijasal (Hindi) etc. Tribal people residing in the Jodhalal forest of Karnataka use stem bark to treat the wounds, fever, stomachache, diabetes and elephantiasis (Mankani et al.) [1]. the juice of the bark is applied in the mouth (Prusti and Behera) [1]. The powdered bark is mixed with Schleichera oleosa and taken with cold water to treat dysentery (Mohanta) [3]. Gum Kino is used in the treatment of polyurea. Flowers are bitter, sweet, cooling, appetizing and febrifuge (Warrier) [4]. Bark is useful in urinary discharge and piles. The gum Kino is externally applied to leucorrhoea (Pullaiah) [5]. Leaf juice is given in purulent discharges from ear, plant is useful in snakebite and scorpion sting. Fruit cures biliousness and kapha (Kirtikar and Basu) [6]. Bruised leaves are useful in boils sores, skin diseases, stomachic and cholera (Jain) [7]. Zaid et al. reported that lowered activities of erythrocytic membrane Ca++-ATpase leads to cardiomyopathy indicated by reduction in contractibility, relaxation, cardiac work and diastolic complications in Type-2 diabetes mellitus. When the normal and diabetic type-2 patients treated with 1nm (-) epicatechin, the Ca++-ATpase activity increased both in normal and diabetic type-2 patients [8]. According to Grover et al., increase in glycogen content in renal and decrease in glycogen content in hepatic and skeletal muscle was partly prevented by aqueous extract of Pterocarpus treatment [9]. Srikrishna and Mathew synthesized a dimethyl ether of marsupin [10]. Rajalakshmi et al. studied the antioxidant activity of P. marsupium on isolated frog heart and found that the plant extract protected the cardiac muscles from oxidative stress induced by H₂O₂. While, the cardiac arrest time was prolonged by 14 minutes in the presence of plant extract than control, indicating the antioxidant activity of the methanolic extract.
of marsupium bark [11]. Apart from this many other researchers proved the antidiabetic nature of the Pterocarpus marsupium (Sharma and Kumar) [12].

II EXPERIMENTAL PROCEDURE

It has following steps:

2.1 Extraction and Purity: The plant material was collected from forest of M.P., India and was identified by the botanical survey of India, Central zone Allahabad. The Bark shaving P. marsupium were extracted in ethanol at reflux temperature. The extract was concentrated under pressure and the concentrated extract was subject to continuous liquid-liquid extraction employing petroleum ether, hexane, benzene, ethyl acetate and acetone as solvents. The dark brown coloured ethyl acetate extract was concentrated with chromatographed on silica gel column with different solvents and their mixtures to yield the compound A. Purity of the compound was checked by TLC.

2.2 Melting point: Melting point of pure isolated compound is 350 °C.

2.3 Molecular Formula: C_{21}H_{20}O_{5}. Homogeneous on TLC R_{f} 0.85 (solvent a) and 0.62 (solvent b). Spot appearance (UV) deep purple. (UV/ NH_{3}) yellow green (found-C: 71.58%, H: 5.60 %requires C-71.59 %; H-5.68 %

2.4 Instrumentation: The collected fractions were analyzed by UV, IR Spectral, H1NMR, Mass, Synthesis and Chemical degradation.

2.5 Λmax (MeOH) nm: 267, 295 (sh), 336; +NaOMe: 275, 320,392; +AlCl3: 276, 348, 384; +AlCl3/HCl: 276, 299,338,331; +NaOAc: 274, 300, 376; +NaOAc/H_{3}BO_{3}:268,301(Sh), 338.

2.6 1H NMR 90 MHz, CDCl3 Values in δ: 6.4 (d, J= 2Hz, 1H, C-8), 6.1 (d, J=2Hz, 1H, C-6), 6.7 (d, J=9Hz, 2H, C-3’ 5’), 7.8 (d, J= 9Hz, 2H, C-2’), 0.95 (6H, d, J=6Hz, (CH3)2CH), 1.41 (3H (CH3)2 CHCH2) and 2.83 (2H, d, J=8 Hz, Benzylic CH2) ppm.

Fig. (1): 1H NMR spectrum of Compound C
2.7 $^{13}$C NMR: 27.96712, 120.3, 28.32273, 154.18776, 114.3899, 97.77164, 22.5586, 22.5586, 127.69405, 128.54778, 105.05714, 164.60408, 30.0158, 163.58, 182.81625, 162.9, 94.66786, 128.14576, 158.95, 104.26364 ppm.

**Fig. (2):** $^{13}$C NMR spectrum of Compound C

2.8 Mass (70 eV) direct inlet: m/z 340, 153, 162, 131, 124, 123, 121.

**Fig. (3):** Mass Spectrum of compound C

2.9 IR Spectrum: ν KBr cm$^{-1}$: 3445, 2910, 2820, 1750, 1600, 1500, 1300.

\[\text{max}\]
RESULT AND DISCUSSION

A green colour with aqueous Ferric chloride indicated the presence of a hydroxy group at position-5 which was evidenced by UV spectrum exhibiting a bathochromic shift of 45 nm in Band I, in the presence of aluminium chloride-hydrochloric Acid. Compound (C) showed M^+ in the mass spectrum at m/z 352 in agreement with the formula C_{12}H_{20}O_{5}. Its colour reactions indicated that it to be a flavone. The presence of 7-hydroxy group was shown by a bathochromic shift of 7 nm in Band II with sodium acetate relative to its methanol spectrum, while the presence of 4"-hydroxy group was confirmed by bathochromic shift of 56 nm and 40 nm in band I with sodium methoxide and sodium acetate respectively.

The ^1H NMR of compound A displayed signals at 0.95 (6H, d, J= 6 Hz, (CH$_3$)$_2$ CH); 1.42 (3H, (CH$_3$) CHCH$_2$) and 2.85 (2H, d, J= 8 Hz benzylic CH$_2$) for the presence of a 3-methyl butyl side chain. Aromatic protons H-6 and H-8 appeared as doublet J= 2 Hz at 6.7 (d, 2H, J= 9Hz) and 7.8 ppm (d, 2H, J= 9 Hz) were observed for 3’, 5’ and 2’, 6’ protons respectively.

Thus UV spectrum and diagnostic shift are as Apigenin with free 5, 7 and 4’-hydroxy groups.

Compound (Z) on alkylation degradation with 50 % aqueous potassium hydroxide afforded p-hydroxy benzoic acid strongly supporting the presence of hydroxyl group at 4’ position.

It was concluded therefore that 3-methyl butyl side chain must be present at C-3, since no signal due to H-3, could be observed near 6.2-6.3 ppm in its ^1H NMR.

Thus compound (C) is 5, 7, 4’-trihydroxy-3-(3’-methyl butyl) flavone (fig 5).
IV CONCLUSION
Different parts of *Pterocarpus marsupium* has different medical properties. Compound 5, 7, 4’-trihydroxy-3-(3’-methyl butyl) flavones has been isolated from *Pterocarpus marsupium* first time. The chemical and spectral identification also has been done first time. Medicinally properties of this compound will be checked for further studies.

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REFERENCES

Fig. 5 (5, 7, 4’-trihydroxy-3-(3’-methyl butyl) flavones)


