International Journal of Medicine and Medical Sciences Vol.1(2) pp. 028-032, February, 2009
Available online http://www.academicjournals.org/ijmms

Full Length Research paper

Isolation and characterization of a new chalcone from the leaves of *Heteropyxis natalensis*

Adesanwo, J. K.[1]*, Shode, F. O.[2], Aiyelaagbe, O.[3], Oyede, R. T.[2] and Baijnath, H.[4]

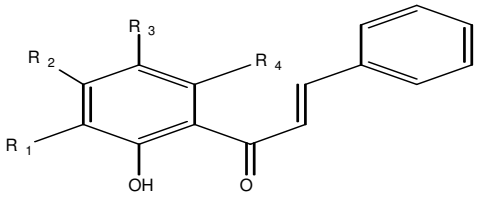
[1]Chemistry Department, Obafemi Awolowo University, Ile-Ife Osun State, Nigeria
[2]School of Chemistry, Westville Campus, University of KwaZulu-Natal, P/Bag X54001, Durban, South Africa.
[3]Chemistry Department University of Ibadan, Nigeria.
[4]School of Conservation and Biological Sciences, Westville Campus, University of KwaZulu-Natal, P/Bag X54001, Durban, South Africa.

Accepted 16 January, 2009

Chromatographic analysis of the defatted dichloromethane extract of the leaves of *Heteropyxis natalensis* afforded the isolation of (*E*)-1-(2',4'-dihydroxy, 5'-methoxy, 3'-methylphenyl)-3-phenylprop-2-en-1-one (R1 = Me; R2 = OH; R3 = OMe; R4 = H) a chalcone. The structure of this compound was established by its spectroscopic data-1D, 2D-NMR, EIMS and HR-EIMS.

Key words: Chalcones, *Heteropyxis natalensis*, heteropyxidaceae, flavone.

INTRODUCTION



[1]: R_1 = Me; R_2 = OH; R_3 = OMe; R_4 = H

[2]: R_1 = H; R_2 = OH; R_3 = Me; R_4 = OMe

[3]: R_1 = Me; R_2 = OMe; R_3 = H; R_4 = OH

Heteropyxis natalensis Harvey (Heteropyxidaceae) commonly known, as lavender tree is a small well-foliated deciduous tree that grows to about 10 meters high (Palgrave, 1977). It occurs naturally on the coastal and inland regions of KwaZulu-Natal province of Southern Africa. Heteropyxidaceae is a small family with only three species known in Southern Africa namely: *Heteropyxis canescens, H. dehniae* and *H. natalensis*.

H. natalensis is used among the Zulus as medicinal tea Its bark is used to treat impotence and as an aphrodisiac. Nose bleeding, is checked by inhaling the steam from a decoction of the roots. The leaves are reputedly used to scent tobacco and dosed to stock in powdered form, to eradicate intestinal worms (Hutchings et al., 1996)

*Corresponding author. E-mail: julius08@oauife.edu.ng.

Previous investigators have examined the essential oils from the leaves of *H. natalensis (*Sibanda et al., 2004; Muzuru Gundidza et al., 2006). The antioxidant activity of the phenolic constituent of *H. natelensis* of Zimbabwe has been determined (Muchuweti et al., 2006). There is no report on the non-volatile phytochemicals from this plant in literature. Our search for non-volatile bioactive natural products as leads for new ethnophamaceuticals, led us to examine the leaves of *H. natalensis* for its phytochemical potentials.

MATERIALS AND METHODS

Experimental

Melting points were determined on a Stuat Scientific SMPI apparatus, IR spectra (KBr) were recorded on a Nicolet Impact 420 spectrophotometer, NMR spectra (both ID and 2D) were obtained on a Varian 300 (300 MHz) spectrometer, using the residual solvent peaks as internal standards, HR-EIMS were determined on a Kratos 9/50 instrument. Column chromatography was carried out using Merck Silica gel 60 (70 – 230 mesh). AnalyticalTLC was carried out on precoated aluminium plates using Merck Silica gel F254; plates were visualized under UV light (λ 254 and 366 nm) and by spraying with anisaldahyde/H_2SO_4 reagent, followed by gentle heating.

Plant material

Fresh leaves of *H. natalensis* were collected in October 2004 from Durban and identified by H. Baijnath. A voucher specimen (MH/06) was deposited in the Wards Herbarium, UKZN- Westville Campus.

Table 1. ^{1}H and ^{13}C NMR of (R_1 = Me; R_2 = OH; R_3 = OMe; R_4 = H).

Position	C-atom	$\delta^{13}C$ (ppm)	$\delta^{1}H$ (ppm), J (Hz)	HMBC
1	C	135.28		
2	CH	128.45	7.64 – 7.62 m	
3	CH	128.95	7.41 – 7.38 m	
4	CH	130.30		
5	CH	128.95		
6	CH	128.45	7.64 – 7.62 m	
α C	CH	126.38	7.82 d, 15.6	193.17, 135.28, 143.27
β C	CH	143.27	7.95 d, 15.6	193.17, 135.28, 126.38, 128.45
1'	C	109.55		
2'	C	161.51		
3'	C	110.25		
4'	C	164.13		
5'	C	161.45		
6'	CH	99.73	6.22 s	164.13, 161.45, 109.55
C=O	C	193.17		
OMe	CH_3	62.24	3.67 s	161.45
Me	CH_3	8.04	2.11 s	161.51, 110.25, 109.55
OH			13.28s	

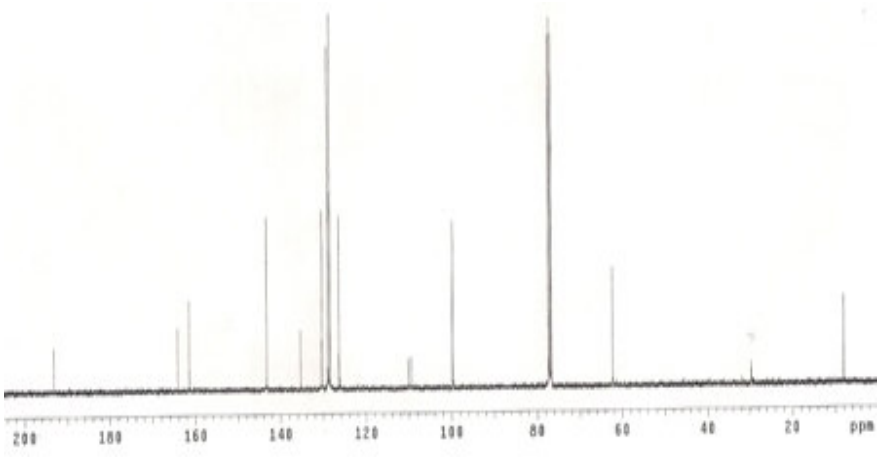


Figure 1. ^{13}C-NMR of (R1 = Me; R2 = OH; R3 = OMe; R4 = H).

Extraction and isolation

The powered air-dried leaves of H. natalensis (1.1 kg) was successively extracted by maceration at room temperature in hexane, dichloromethane, ethyl acetate and methanol to give, after removal of solvent *in-vacuo*, hexane extract (19.1g), DCM extract (39.6 g), ethyl acetate extract (16.5 g) and methanol extract (126.5 g), respectively.

Column Chromatography of the dichloromethane extract (3 g) on silica gel, by gradient elution with hexane and hexane/ethylacetate mixtures followed by purification of fractions gave a yellowish crystalline compound (105 mg), melting point: 139 - 140°C.
IR (KBr): ν_{max}/cm^{-1}: 3252 (br OH), 3106, 3048, 1631, 1505, 1413.
^{1}H and ^{13}C nmr: Table 1 and Figure 1 and 2
EIMS: m/z (rel. int. %, Fig 5) = 284 $[M]^+$ (40), 267$[M\text{-}OH]^+$(05), 207 $[M\text{-}C_6H_5]^+$ (20), 180 $[M\text{-}C_6H_5CH{=}CH_2]^+$ (100), 152 [180- C=O]$^+$ (45).
HREIMS: m/z [M^+, 100] 284.1041 (Cal. 284.1049 for $C_{17}H_{16}O_4$).

RESULTS AND DISCUSSION

Extraction of dried pulverized and defatted leaves of *H. natalensis* (1.1 kg) with dichloromethane afforded 39.6 g extract. Open column chromatographic fractionation of the extract (3.0 g) and purification of fractions led to isolation of R_1 = Me; R_2 = OH; R_3 = OMe; R_4 = H, a yellowish crystalline solid (105 mg) mp 139 - 140°C.

The ^{13}C nmr spectrum (Table 1) of solid R_1 = Me; R_2 = OH; R_3 = OMe; R_4 = H showed 17 signals including two CH signals at δ126.38 and δ143.27 corresponding to the α and β carbons of chalcones (Pelter et al., 1976); six aromatic CH signals at δ130.30, 128.95 (2C), 128.45 (2C) and 99.73; six quaternary carbons at δ164.13, 161.51, 161.45, 135.28, 110.25 and 109.55; two sp^3 carbons at δ62.24 (OCH_3) and δ8.04 (CH_3) and a carbonyl carbon at δ193.17.

Its ^{1}H nmr showed a downfield signal at δ13.28 due to a chelated hydroxyl (this was confirmed by ir spectrum with with a broad OH band at 3252 cm^{-1} and a chelated carbonyl band at 1631 cm^{-1}), 2Hm aromatic (δ7.63), 3Hm

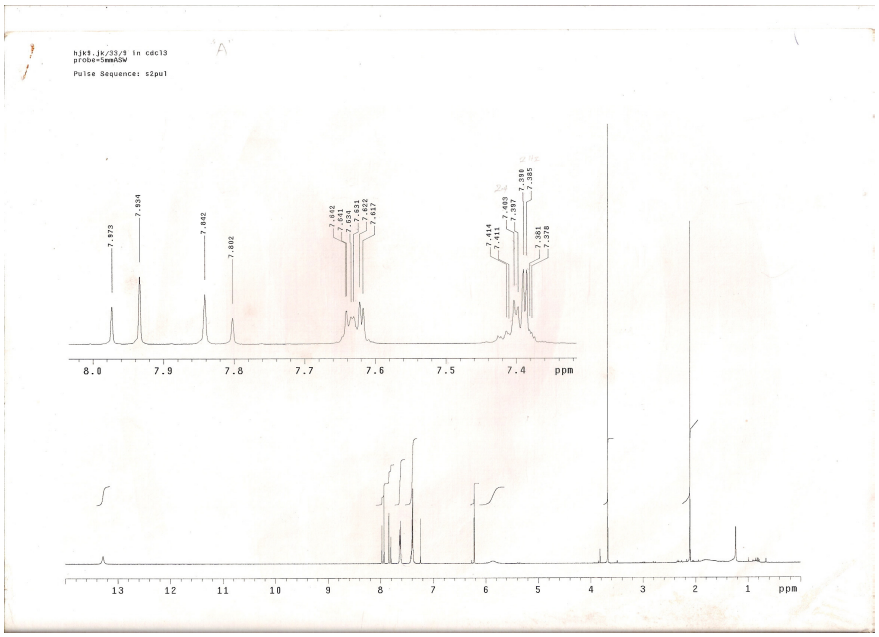


Figure 2. ^{1}H-NMR Spectrum of (R1 = Me; R2 = OH; R3 = OMe; R4 = H).

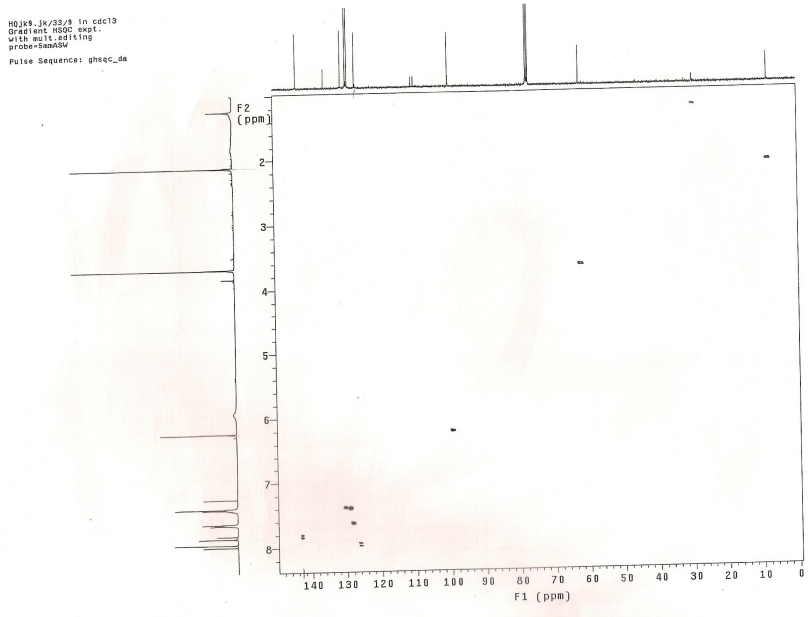


Figure 3. HSQC Spectrum of (R1 = Me; R2 = OH; R3 = OMe; R4 = H).

aromatic (δ 7.40), two doublets at δ7.96 (J 15.6Hz) and δ7.82 (J 15.6Hz), one aromatic singlet at δ6.22, and two other singlets at δ3.67 (methoxy group) and δ2.11 (methyl group). These data are indicative of a 2'-hydroxylated chalcone. Compound R_1 = Me; R_2 = OH; R_3 = OMe; R_4 = H is an isomer of aurentiacin A R_1 = H; R_2 = OH; R_3 = Me; R_4 = OMe and triangularin R_1 = Me; R_2 = OMe; R_3 = H; R_4 = H, chalcones isolated from *Myrica serrata (*Stefan et al., 1996) and *Pityrogramma triagularis* (Star et al., 1978) respectively.

Proton/Carbon Correlations- HSQC and HMBC spectra (Figure 3 and 4): The HSQC spectrum gave clear unambiguous correlations of the carbon atoms with the protons directly attached to them (Table 1) In the HMBC

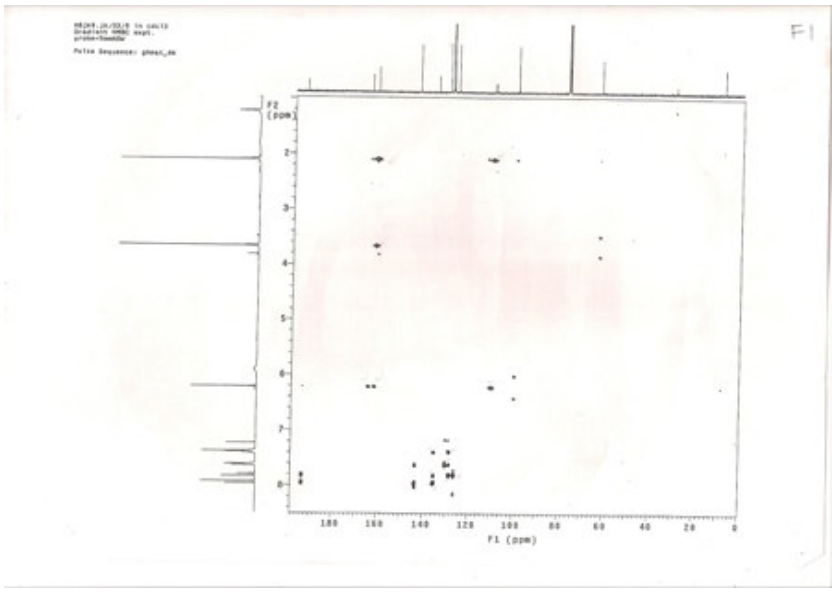


Figure 4. HMBC Spectrum of (R1 = Me; R2 = OH; R3 = OMe; R4 = H).

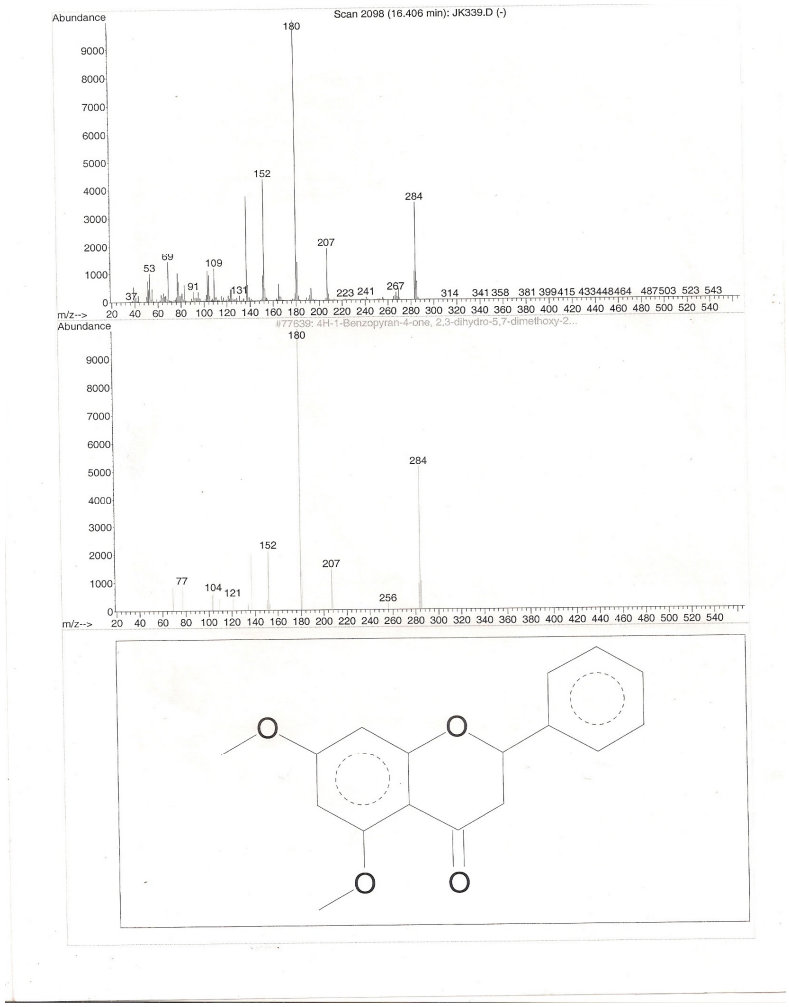


Figure 5. EIMS Spectrum of (R1 = Me; R2 = OH; R3 = OMe; R4 = H).

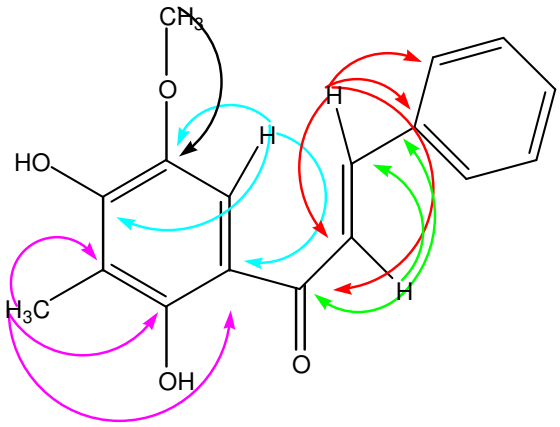


Figure 6. HMBC Correlations in (R1 = Me; R2 = OH; R3 = OMe; R4 = H).

spectrum, the position of the methoxy group at 5' was confirmed by the correlation of the methoxy protons at 3.67 ppm with carbon C-5'. The methyl signal at C-3' was similarly situated with observed correlations of the methyl protons with C-2', C-3', C-4' and C-1' (Table 1, Figure 6).

The base peak fragment ion in the EIMS (m/z 180) corresponds to the carbonyl α-bond cleavage giving the chealation- / resonance-stabilized ion.

We wish to acknowledge financial support from National Research Foundation (NRF) South Africa.

REFERENCES

Hutchings A, Scott AH, Lewis G, Cunningham A (1996). Zulu Medicinal Plants: An Inventory. University of Natal Press, Pietermaritzburg, South Africa. p. 219.

Muzuru Gundidza, Stanley G. Deans, Alan I. Kennedy, Steven Mavi, Peter G, Waterman A, Gray I (2006). The essential oil from *Heteropyxis natalensis* harv: Its antimicrobial activities and phytoconstituents J. Sci. Food Agric. 63 (3): 361-364

Muchuweti Maud, Nyamuk Lynet, Chagoda Larmet S, Ndhlala Ashrrell R, Mupure Chipo and Benhura Mudadi (2006). Total Phenolic content and antioxidant activity in selected medicinal plants of Zimbabwe. Int. J. Food Sci.Technol. 41:33-38

Palgrave KC (1977). Trees of South Africa. Stuik Publishers, Cape Town. pp. 694-695.

Pelter A, Ward RS, Gray IT (1976). The Carbon –13 Nuclear Magnetic Resonance Spectra of Flavonoids and related Compounds. J. Chem. Soc. Perkin I. pp.2475- 2483.

Sibanda S, Chigwada G, Poole M, Gwebu ET, Noletto JA, Schmidt JM, Rea AI, Setzer WN (2004) Composition and Bioactivity of the Leaf Essential Oil of *Heteropyxis dehniae* from Zimbabwe. J. Ethnopharmacol. 92: 107-111.

Star AE, Mabry TJ, Smith DM (1978). Triangularin, a new chalcone from *Pityrograma triagularis*. *Phytochem.* 17:586- 587.

Stefan Gafner, Jean-Luc Wolfender, Stephen Mavi, Kurt Hosteettmann (1996). Antifungal and Antibacterial Chalcones from Myrica serrata. Planta. Med. 62: 67-69.