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RESEARCH ARTICLE

CHARACTERIZATION OF TWO CHALCONE DERIVATIVES ISOLATED FROM FINGER ROOT WITH NUTRACEUTICAL POTENTIALS.

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Abstract

Boesenbergia rotunda is an example of medicinal herbal plants which has been traditionally employed in the treatment of many life-threatening ailments such as diuretic, dysentery, inflammation, aphrodisiac and gastrointestinal disorder.In this study the two chalcone derivatives were isolated from the root of B. rotunda using column-thin chromatography and characterized using different spectroscopy methods such as such as UV-VIS, FTIR, and ^{1}HNMR.The bioactive compound identified were pinostrobinchalcone (1) and cardamone (2). These IR spectra, UV-vis photometry analysis and ^{1}HNMR suggested that the chemical constituent isolated to be a flavonoid derivative, which is similar to the previous studies. The result of the study suggests that B. rotunda rhizome has a potential in drug and nutraceutical applications.

Introduction:-

The increasing demands for chemical diversity in screening characterizations necessitate succinct investigations into the therapeutic activities of herbal plant bio-products. The finger root (*Beosenbergia* rotunda) is an example of herb plants belonging to the family of *Zingiberaceae*. It belongs to the order of Zingiberales herbaceous ground flora plants in the rainforest and is commonly in countries like Thailand, Malaysia, Indonesia and Myanmar (Cheenpracha et al., 2006). There are approximately 150 species of this family with about 23 species found in Peninsular Malaysia (Sukara et al., 2017). The finger-root of *B. rotunda* has been reported for the treatment of peptic-ulcer, colic, oral diseases, urinary disorders, dysentery and inflammation (Chatsumpun et al., 2017). These numerous medicinal benefits are to the presence of some important bioactive components. There is therefore a necessity to isolate these important bioactive constituents at higher purity.In this study, two bioactive constituents from *B. rotunda* were purified and isolated using column chromatography techniques. The extracted metabolites were characterized using FTIR, NMR, and UV-Vis techniques.

Materials and methods:-

Chemical preparation of PinostrobinChalcone

The finger roots (*B. rotunda*) were procurred from Selangor DarulEhsan, Malaysia. The plant sample was identified and authenticated by Assoc. Prof. Dr. Muhammad NadeemAkhtar at the Faculty of Industrial Sciences and Technology, University Malaysia Pahang. The sample was then dried and grounded into smaller size (0.1 mm) using

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a Grindomix grinder (GM-200 model, Germany). The extraction process was conducted with 300 g of the sample and methanol solvent using Soxhlet apparatus. The product of extraction was placed on water-bath at 65 ºC to remove the residual methanol. The residue was then treated with 5% HCl and filtered. The final solution was acidified using HCl(aq) and Meyers reagent/Dragendorff's reagent until precipitation stops. The resulting precipitates was filtered, wash with water and suspended in MeOH-Me_2CO-H_2O (6:2:1) (Isa et al., 2012). A complex mixture was resolved by vacuum column chromatography (VLC) with silica gel 60, packed in slurry with chloroform as the initial solvent and gradually increased the polarity of a solvent. Chromatography separation was performed on pre-coated TLC plates Kieselgel Si 60; 0.25mm (E. Merck, Darmstadt, Germany). The separated spots were detected under UV light (256 & 366 nm). The individual components was purified by preparing TLC on a pre-coated silica-gel plates using MeOH-$CHCl_3$. Column Chromatography was performed on a pre-coated TLC plates Kieselgel Si-60; 0.25mm (E. Merck, Darmstadt, Germany).

Characterization Techniques

The *B. rotunda* extracts and the isolated compounds were characterized using Fourier transform infrared spectroscopy (FTIR), Nuclear Magnetic Resonance (NMR), UV-Vis and GCMS as discussed in the proceeding sections.

Ultraviolet-Visible photometry analysis

The Ultraviolet-Visible photometry analysis was conducted using the theGenesys 10s UV-Vis spectrometer. Prior to the analysis the solid single pure compound isolated was first diluted with acetone which produced a pale yellowish liquid solution. The liquid solution was then pipetted into the 1-cm path length quartz cuvettes. The preparation of the sample and the analysis was carried out in the dark at 21 ± 1 °C. To prevent the evaporation of the solvent (acetone), the reactor was closed and stirred. The solution was irradiated at the wavelength ranged of 200 to 500 nm. The absorption spectrum was displayed by the spectrometer and the data was obtained. The wavelength with maximum absorbance of the single pure compound was then identified.

Fourier Transform Infrared (FTIR) analysis

Fourier transform infrared spectroscopy (FTIR) was carried out on the extracts to determine the functional groups (Olalere et al., 2017; Amani et al., 2018). A Thermo-Nicolet spectrometer (iS5 iD7 ATR, Germany) equipped with OMNIC software was employed in the spectrometry analysis. The analysis was executed using the conventional KBr standard procedure with wavenumber ranging from 4000-600 cm^{-1}. Under this study, the spectrum of the observed bond and associated group frequencies were compared with the table of expected absorption bands (Carol 2000).

Nuclear Magnetic Resonance Spectroscopy (NMR)

Further confirmations of the structure of the chemical constituent of the isolated compounds were performed using ^1H NMR. The sample was diluted be diluted in deuterated chloroform ($CDCl_3$) and then transferred to NMR tube. Based on the NMR spectra, the area under each pattern obtained from the integration of the signal was studied to determine and identify the structure of the compound.

Results and discussion:-

UV-Vis Photometry studies

The pinostrobinchalcone (1**)** was absorbed by UV light wavelength range of 250-380 nm. The observed wavelength range corresponds to the conjugated α, β-unsaturated carbonyl (C=O) chromophore as reported [2]. Figure 1 shows the UV-visible spectrum of pinostrobinchalcone with maximum absorption of 340 nm at wavelength, λ_{max}.

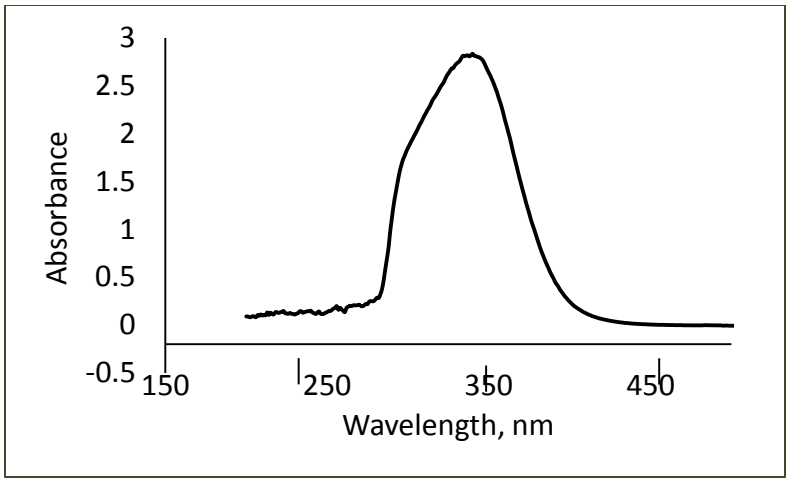

Figure 1:-UV spectrum of pure pinostrobin chalcone (1)

However, cardamone (2) absorbed UV light at a wavelength range of 290-380 nm, which corresponds to the conjugated α, β-unsaturated carbonyl (C=O) chromophore [5]. Figure 2 shows the UV-visible spectrum of cardamone (2) with maximum absorption at wavelength, λ_{max} of 340 nm.

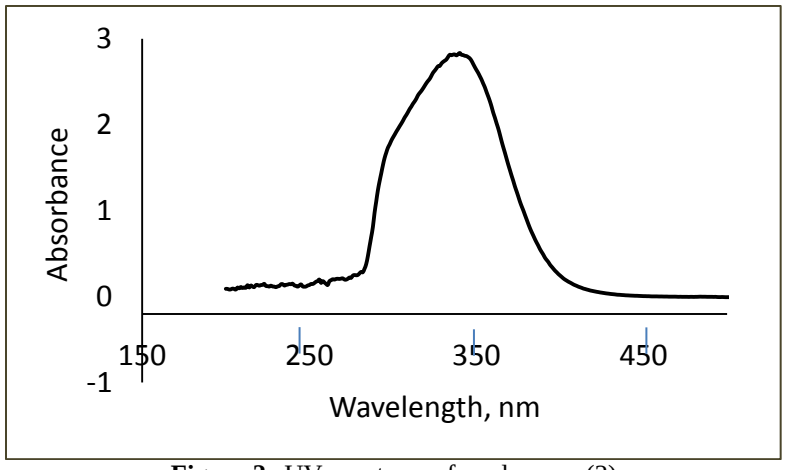

Figure 2:-UV spectrum of cardamone (2)

Functional group analysis

Figure 3 shows the IR spectrum of pinostrobinchalcone (1**)** with an absorption band of 3456 cm^{-1} which corresponds to the hydroxy (O-H). Moreover, the observed absorption band between 2940 and 3092 cm^{-1} corresponds to the aromatic C-H stretching. Absorption band at 1623 cm^{-1} and between 1416-1439 cm^{-1} was indicated the presence of carbonyl (C=O) and aromatic ring C=C, respectively. Absorption band appeared in the range of 1158-1219 cm^{-1} corresponded to (C-O) [1].

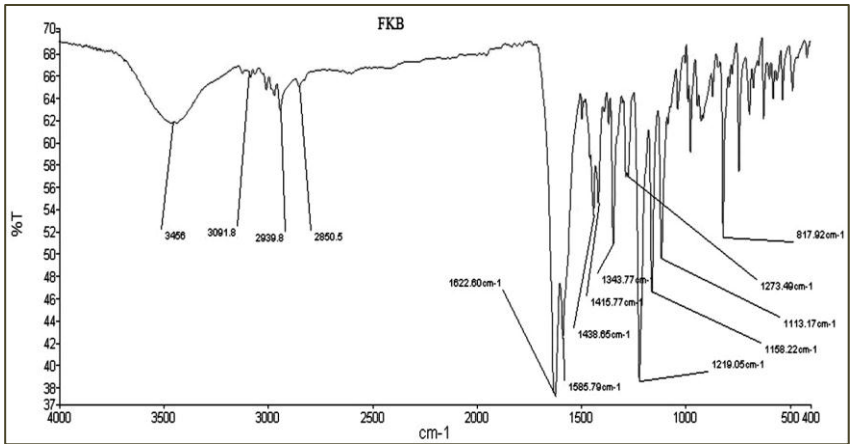

Figure 3 IR spectrum of pure pinostrobin chalcone (1)

IR spectrum of cardamone (2) in Figure 4 shows absorption band at 3456 cm^{-1} which corresponded to hydroxy (O-H). The absorption band between 2940-3092 cm^{-1} corresponds to the aromatic C-H stretch while the absorption band at 1623 cm^{-1} and between 1416-1439 cm^{-1} indicated the presence of carbonyl (C=O) and aromatic ring C=C, respectively. Absorption band appeared in the range of 1158-1219 cm^{-1} corresponded to (C-O) moiety [2].

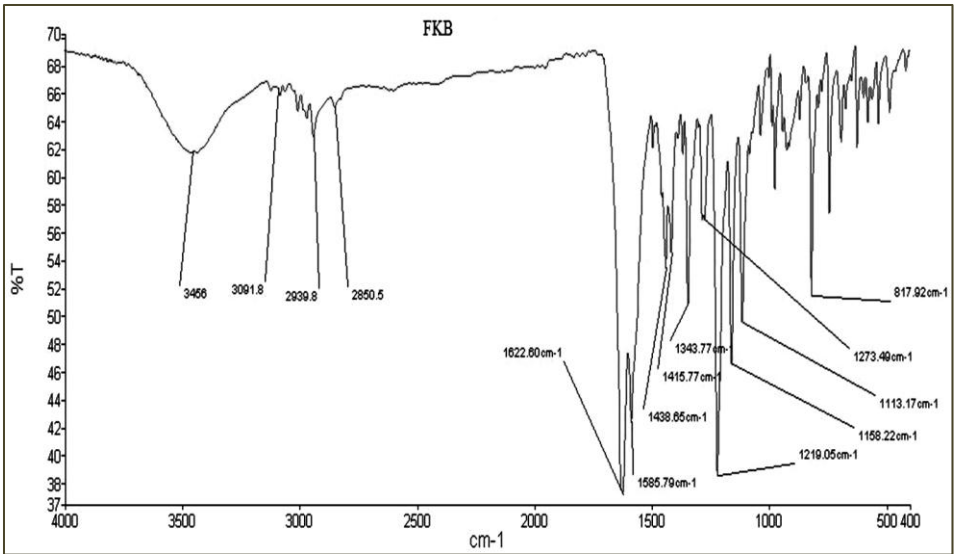

Figure 4:-IR spectrum of cardamone (2)

H Nuclear Magnetic Resonance (NMR)

The ^{1}H-NMR spectrum (600 MHz, CDCl3) of pinostrobinchalcone (1**)** in Figure 5 shows a singlet at δ 3.84, which was assigned to the methoxy protons at C-4′. Two doublet that appeared at 5.96 (d, *J* = 2.46 Hz) and 6.11 (d, *J* = 2.40 Hz) were assigned to C-5′ and C-3′ protons, respectively.

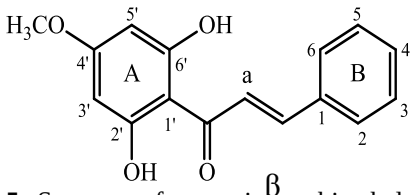

Figure 5:-Structure of pure pinostrobin chalcone (2)

A broad multiplet that appeared in the range of 7.38-7.42 was assigned to C-3, C-4 and C-5 protons. Two doublets observed at 7.62 (d, *J* = 8.04 Hz) and 7.60 (d, *J* = 8.04 Hz) were assigned to C-2 and C-6 protons, respectively. A

downfield doublet at 7.78 (d, *J* = 15.55 Hz) was assigned to proton at α-carbon and another doublet observed at 7.90 (d, *J* = 15.55 Hz) was assigned to proton at β-carbon, which data was supported by previous work [1,2]. A downfield singlet that appeared at 14.30 was assigned to C-2′ hydroxyl proton chelated to carbonyl group. Further details of ^{1}H-NMR and ^{13}C-NMR spectra are shown in Table 1.

Table 1:- NMR data of pure pinostrobin chalcone (2)

Carbon	^{13}C (δ)	^{1}H (δ)	Multiplicity	Designation
1′	108.14	-	-	-
2′	168.90	-	-	-
3′	93.60	6.11	(d, *J* = 2.40 Hz, 1H)	C3′-H
4′	166.70	-	-	-
5′	90.94	5.96	(d, *J* = 2.46 Hz, 1H)	C5′-H
6′	162.50	-	-	-
1	132.90	-	-	-
2	130.20	7.62	(d, *J* = 8.04 Hz, 1H)	C2-H
3	128.10	7.38-7.42	(m, 3H)	C3-H
4	141.00	7.38-7.42	(m, 3H)	C4-H
5	128.10	7.38-7.42	(m, 3H)	C5-H
6	130.20	7.60	(d, *J* = 8.04 Hz, 1H)	C6-H
A	126.60	7.78	(d, *J* = 15.55 Hz, 1H, H-α)	Cα-H
B	142.51	7.90	(d, *J* = 15.55 Hz, 1H, H-β)	Cβ-H
OCH_3	55.70	3.84	(s, 3H)	OCH_3 (C4′)
OH	-	14.30	(s, 1H)	OH (C2′)
OH(C6')				
C=O	192.22	-	-	-

Moreover, the The^{1}H-NMR spectrum (600 MHz, $CDCl_3$) of cardamone (2) in Figure 6 shows a singlet at δ3.84, which was assigned to the methoxy protons at C-6′ atom.

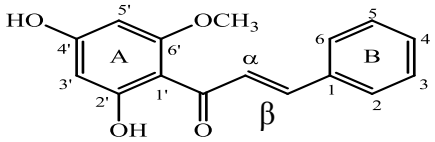

Figure 6:-Structure of pure Cardamone (2)

Two doublet that appeared at 5.96 (d, *J* = 2.46 Hz) and 6.11 (d, *J* = 2.40 Hz) were assigned to C-5′ and C-3′ protons, respectively. A broad multiplet that appeared in the range of 7.38-7.42 was assigned to C-3, C-4 and C-5 protons. Two doublets observed at 7.62 (d, *J* = 8.04 Hz) and 7.60 (d, *J* = 8.04 Hz) were assigned to C-2 and C-6 protons, respectively. A downfield doublet at 7.78 (d, *J* = 15.55 Hz) was assigned to proton at α-carbon and another doublet observed at 7.90 (d, *J* = 15.55 Hz) was assigned to proton at β-carbon, which data is supported by previous work [2,3]. A downfield singlet that appeared at 14.30 was assigned to C-2′ hydroxyl proton chelated to carbonyl group. Further details of ^{1}H-NMR and ^{13}C-NMR spectra are shown in Table 2.

Table 2:-NMR data of Cardamone (**2**)

Carbon	^{13}C (δ)	^{1}H (δ)	Multiplicity	Designation
1′	108.14	-	-	-
2′	168.90	-	-	-
3′	93.60	6.11	(d, *J* = 2.40 Hz, 1H)	C3′-H
4′	166.70	-	-	-

5′	90.94	5.96	(d, *J* = 2.46 Hz, 1H)	C5′-H
6′	162.50	-	-	-
1	132.90	-	-	-
2	130.20	7.62	(d, *J* = 8.04 Hz, 1H)	C2-H
3	128.10	7.38-7.42	(m, 3H)	C3-H
4	141.00	7.38-7.42	(m, 3H)	C4-H
5	128.10	7.38-7.42	(m, 3H)	C5-H
6	130.20	7.60	(d, *J* = 8.04 Hz, 1H)	C6-H
A	126.60	7.78	(d, *J* = 15.55 Hz, 1H, H-α)	Cα-H
B	142.51	7.90	(d, *J* = 15.55 Hz, 1H, H-β)	Cβ-H
OH (C4′)	-	-	-	OH (C4′)
OCH_3 (C6′)	55.53	3.92	(s, 3H)	OCH_3 (C6′)
OH (C2′)	-	14.30	(s, 1H)	OH (C2′)
C=O	193.20	-	-	-

Conclusions:-

The characterizations of two chalcone derivatives were conducted using UV-VIS, FTIR, and ^{1}HNMR. The UV-VIS helped in the identification of pinostrobinchalcone (1) and cardamone (2) by means of its absorption spectra. FTIR revealed the functional groups present to further elucidate and confirm the presence of the two chalcone derivatives in *B. rotunda*. ^{1}H NMR resolved the magnetical dissimilar proton found in the compound isolated. The result obtained from this study indicated the potential of *B. rotunda* rhizome extracts as drug candidate in therapeutic applications and pharmaceutical industries.

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