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PHYTOCHEMICAL STUDY OF *CASSIA DIDYMOBOTRYA* FRES. CULTIVATED IN EGYPT. PART II: FLAVONOIDS AND STEROLS.

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ABSTRACT

From the air-dried powdered leaves, stems, flowers and fruits of Cassia didymobotrya Fres. the following compounds were isolated for the first time: isorhamnetin-3-O-glucoside, isorhamnetin-7-O-glucoside, isorhamnetin-3-O-rhamnosyl-O-glucoside, kaempferol-7-O-rhamnosyl-O-glucoside, kaempferol-3-O-rhamnosyl-O-glucoside, stigmasterol and its 3-O-glucoside. In addition kaempferol and quercetin were isolated. All compounds were identified by comprehensive spectral analysis.

INTRODUCTION

In a previous communication[1] the authors reported the isolation and identification of quinones and uracil from the leaves, stems, flowers and fruits of *Cassia didymobotrya* Fres.

The present investigation deals with the isolation and identification of the flavonoids and sterols present in the title plant.

EXPERIMENTAL

Plant material:

The plant material used in this work consists of the dried leaves, stems, flowers and fruits of *Cassia didymobotrya* Fres. collected from public gardens in Assiut during April-May and identified by Prof.Dr.N.E.El-Keltawy, Prof. of Horticulture, Faculty of Agriculture, Assiut University, Assiut-Egypt.

General Experimental Procedures:

Melting points were uncorrected, all UV-spectra were in MeOH (UV,VIS) spectrometer 550 S,Perkin-Elmer). ^{1}H-NMR spectra were run in $CDCL_3$ or DMSO-d_6 on spectrometer WH-90 (Bruker physics) and XL 300 Varian. Mass spectral measurements were on 70 eV spectrometer-MS-50 (Kratoes). ^{13}C-NMR: spectrometer XL-300 (Varian).

Thin layer Chromatographic study :

Adsorbent : Silica gel 60 F 254.

Spray reagents: a) Ammonia vapour b) 1 % alcoholic solution of $ALCl_3$

c) Vanillin-sulphuric acid

d) Thymol-sulphuric acid

Solvent systems:

I) ethyl acetate-ethanol (8:2)

II) cyclohexane-dichloromethane ethyl formate-formic acid (35:30:5).

III) methylene chloride-methanol-water (40:10:1)[2]

IV) petroleum ether-ethyl acetate (9:1).

V) n-butanol-acetone-formic acid-water (60:17:8:15)[8].

VI) chloroform-methanol (95:5)

VII) acetonitrile-water (85:15)[4]

Extraction and isolation :

The powdered organs of C.didymobotrya Fres. viz. (leaves 8.5 kg , stems 2.5 kg, flowers 2.0 kg and fruits 1.0 kg) were first defatted with petroleum-ether and the marc in each case was extracted with ethanol(70%). The alcohol-free residues were successively fractionated with ether , ethyl acetate and n-butanol. Individual fractions of each organ were subjected to TLC using silica gel and the previously mentioned solvent systems and spray reagents. It was found that the extracts of both leaves and stems have almost the same components so they were mixed together.

The concentrated fractions were separately chromatographed over silica gel columns. Elution was accomplished by pet-ether followed by petroleum-ether-ethyl acetate gradient in the case of ether fractions and with

ethyl acetate followed by ethyl acetate-ethanol gradient in the case of ethyl acetate and n-butanol fractions. Compounds 1-6 were obtained successively from ether fractions.

Compounds 7-9 were obtained successively from ethyl acetate fractions and compounds 10-11 were obtained from n-butanol.

The presence and amount of these compounds in the different organs as well as their physical characters are cited in Tables 1 & 2. UV spectral data of the isolated flavonoids 3-11 are given in Table 3. ^{1}H-NMR spectral analysis for compounds 1 & 2 are listed in Table 4, and for compounds from 3-11 are listed in Table 5. ^{13}C-NMR for compound 1 is listed in Table 6 and for compounds 7 & 9 are summarized in Table 7.

Acid hydrolysis[5] :

Each isolated glycoside (5 mg) was dissolved in 5.0 ml MeOH to which 20% HCl solution was added and the mixture was refluxed on a boiling water bath for 8 hrs. A sample of the hydrolysate was withdrawn every 30 minutes and subjected to TLC study. After complete hydrolysis, the mixture was cooled and the aglycone was separated by successive extraction with $CHCl_3$. Chromatographic studies of aglycone and sugars were carried out using systems V and VII and thymol-H_2SO_4 as spray reagent.

RESULTS AND DISCUSSIONS

The defatted, air-dried powdered organs: leaves, stems, flowers and fruits of C.didymobotrya Fres. were separately extracted with alcohol (70%). The residues of the alcoholic extracts were successively fractionated with ether, ethyl acetate and n-butanol and examined by TLC. Each fraction was subjected to column chromatography to isolate the corresponding compounds. Two sterols (1&2) and four flavonoids (3-6) were isolated from ether fractions. Three flavonoidal glycosides (7-9) were isolated from ethyl acetate fractions while another two (10-11) were isolated from n-butanol fractions.

Identification of the isolated compounds :

Compound 1:

The IR spectrum of compound 1 showed the absorption bands at 3350 cm^{-1} (OH streching vibration), at 2940 (C-H streching vibration), at 1460-1360 assigned to CH_3 groups in the side chain[6] and from 1380-1360 cm^{-1} (reported for geminal methyl groups)[7].

The results of ^{1}H-and ^{13}C-NMR of compound 1 are summarized in Tables 4 & 6 respectively. Mass spectrum of compound 1 showed [M^+] at m/z=412.37 coresponding to the mol. formula $C_{29}H_{48}O$. This was confirmed by the number of carbon atoms in ^{13}C-NMR. TLC revealed that compound 1 appeared as a single spot, R_f=0.14 in system II Table 1.

From the above mentioned spectral studies, chromatographic study as well as comparison with published data[8,9], it can be concluded that compound 1 is stigmasterol.

Compound 2 :

Mass spectrum of compound 2 showed [M^+] at m/z =412.38 ($C_{29}H_{48}O$), Results of ^{1}H-NMR are summarized in Table 4. From IR spectrum of compound 2 band at (γ)=1610 cm^{-1} was assigned to ring vibration of pyranose sugar[10]. The other bands are nearly similar to that of compound 1. From ^{1}H-NMR of compound 2, the anomeric proton of glucose appeared at 5.07 ppm J=7,5 Hz (B-linked glucose).

Acid hydrolysis of compound 2 followed by chromatographic studies of the aglycone and the sugar revealed that the aglycone was identical with stigmasterol (compound 1), where the sugar part was identical with authentic glucose.

From the aforementioned spectral and chromatographic studies as well as comparison with the published data, it can be concluded that compound 2 is stigmasterol-3-O-B-D-glucoside.

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Compound 3 :

The UV spectral study (Table 3) indicated that it is a flavonol with free OH group at C-3,5,7 and 4'[11]. ^{1}H-NMR (Table 5) showed identical data with that reported for kaempferol[11]. Ms : m/=286.05 (100%). Other peaks at 285,153,152 and 121. Based on these data, it can be concluded that compound 3 is kaempferol.

Compound 4 :

The UV spectrum (Table 3) suggested that it is a flavonol with free OH groups at C-3,5,7,4' and 3'[11]. ^{1}H-NMR spectrum (Table 5) showed similar data to that reported for quercetin[11]. In addition, the mass spectrum showed [M^+] at m/z=302.04 corresponding to mol. formula $C_{15}H_{10}O_7$ and a fragmentation pattern comparable with quercetin[12]. Accordingly it can be concluded that compound 4 is quercetin.

Compound 5 :

UV spectrum of compound 5 (Table 3) showed a maximum absorption band at (353 nm) indicating a flavone or 3-substituted flavone[11]. A bathochromic shift at band 1 (+ 54 nm) was obtained on addition of NaOMe due to the presence of free OH group at C-4'. Addition of $AlCl_3$ or $AlCl_3$/HCl produced a shift at band I (+ 47 nm), indicating free OH group at C-5 and absence of dihydroxy group at ring B.

A free OH group at C-7 was indicated by the shift (+ 13 nm) on addition of sodium acetate.

^{1}H-NMR spectrum (Table 5) shows that it is a flavonoidal compound with a methoxy group probably at C-3' and a glucose molecule linked at C-3. The anomeric proton appeared as a doublet at 5.18 ppm with coupling constant of 7.5 H_2 indicating that it is B-linked glucose[11].

Mass spectrum of compound 5, showed a base peak at m/z=316 [M^+] corresponding to molecular formula $C_{16}H_{12}O_7$. Other ions,

including $(M-H)^+$, $(M-CH_3)^+$ at 301 and $(M-CH_3CO)^+$ at 273 provide considerable structural information and other characteristic peaks at 153,137,121,85 and 60. Acid hydrolysis of compound 5 and chromatographic study of the sugar revealed a single spot comparable with the same characters of glucose.

The available data are comparable with that published for isorhamnetin glycosides. So, compound 5 could be identified as isorhamnetin-3-O-glucoside.

Compound 6 :

The UV spectrum (Table 3) shows absorption band at 365 nm indicating its flavonol nature[11]. $ALCl_3$/HCl produced a bathochromic shift in band 1 (+60 nm) indicating a free OH at C-3, addition of sodium acetate gave no shift showing absence of OH group at C-7. The other bands were similar to compound 5.

^{1}H-NMR of compound 6 (Table 5) revealed that it is a flavonoidal compound with OCH_3 at C-$\bar{3}$ and a sugar molecule linked at C-7. Mass spectrum showed the same fragmentation pattern as mentioned under compound 5. Also, the chromatographic studies of the products of hydrolysis gave similar results as compound 5 So, it can be concluded that compound 6 is isorhamnetin-7-O-glucoside.

Compound 7 :

From the UV spectrum (Table 3) it can be suggested that compound 7 is a 3-substituted flavonol with free OH groups at C-5, C-7 and C-$\bar{4}$.

HRMS showed a molecular formula of $C_{15}H_{10}O_6$ and a fragmentation pattern similar to that of kaempferol, $[M]^+$ at m/z=286 , other peaks at 258,153,152,134 and 121. Both ^{1}H-and ^{13}C-NMR (Table 7) showed signals assignable to a flavonoid similar to kaempferol with substitution at C-3, in addition to those of a sugar[10].

The anomeric proton of glucose appeared at 5.25 ppm with J=7.5 Hz indicating a B-linkage. Chromatographic study of the hydrolysate showed that the aglycone is kaempferol and the sugar is glucose.

These data, indicate that compound 7 is kaempferol-3-O-glucoside

Compound 8 :

The UV spectral study (Table 3) indicated that compound 8 is a flavonol with free hydroxyl groups at C-3,C-5 and C-4'.

The ^{1}H-NMR spectrum (Table 5 showed a doublet at 1.13 ppm assignable to a CH_3 of rhamnose. The coupling constant for rhamnose anomeric proton was 1.5 Hz, indicative of α-linked rhamnose. The anomeric proton (H-1)of glucose at 5.11 ppm confirmed its direct attachement to the aglycone. Mass spectrum showed [M^+] at m/z=285 and other peaks at 153, 152 and 121. The fragmentation pattern for the aglycone of compound 8 is identical to that of kaempferol.

From these data in hand as well as the chromatographic studies of the aglycone and the sugar, it can be concluded that compound 8 is kaempferol-7-O- gluco-rhamnoside.

Compound 9 :

UV absorption spectrum (Table 3) indicates a 3-substituted flavonol with free OH groups at C-5,C-7 and C-4'. From ^{1}H-and ^{13}C-NMR spectral data (Tables 5,7) it can be suggested that compound 9 has a gluco-rhamnosyl disaccharide moiety at C-3,C-6 of glucose is linked to C-1 of rhamnose as evidenced by the downfield shift of C-6 of glucose (68.57 ppm)(glucose, C-6 at 62.9 ppm)[13]. The presence of kaempferol is evidenced by mass spectroscopy, [M^+] at m/z=286.04 and other peaks at 258,153,152, 134 and 121. This fragmentation pattern is identical with that of kaempferol. Chromatographic study of the hydrolysates proved that the aglycone is kaempferol and glucose and rhamnose are the sugars. Compound 9 could be identified as kaempferol-3-O-rhamnosido-O-glucosyl or rutinoside[14].

Compound 10 :

The UV spectrum (Table 3) indicating its flavone nature[11], with free OH group at C-4̃,C-5 and C-7 with absence of orthodihydroxy groups at ring B. ^{1}H-NMR (Table 5) revealed that compound 10 is a flavonoid with a methoxyl group at C-3̄ and substitution at C-3. A doublet at 1.13 ppm is assignable to a CH_3 of rhamnose. The signals at 5.03 and 4.49 ppm are assignable to the anomeric protons of glucose and rhamnose, respectively. The upfield resonance from H-1'' proton (4.49, ppm with J=2 Hz) of the H-1̿ proton of the terminal sugar which appears relatively remoted by the enfluence of the flavonoid neucleus indicates that rhamnose should be a second moiety of the disaccharide. The diaxial coupling (J=7.5 Hz) between H-1 and H-2 indicated B-configuration of glucose[11].

After acid hydrolysis and chromatographic study, the presence of two sugars corresponding to glucose and rhamnose confirms the forementioned suggestions.

From the above data, it can be concluded that compound 10 is isorhamnetin-3-O-rhamnosido-O-glucosyl.

Compound 11 :

The UV spectrum Table 3 suggested a 3-substituted flavonol with free hydroxyl groups at C-5,C-7 and C-4̃.

From the ^{1}H-NMR Table 5 it was found that compound 11 is a flavonoidal glycoside with a methyl group at C-3. The sugar appears as two molecules of glucose and one molecule of rhamnose.

Compound 11 is still under investigation.

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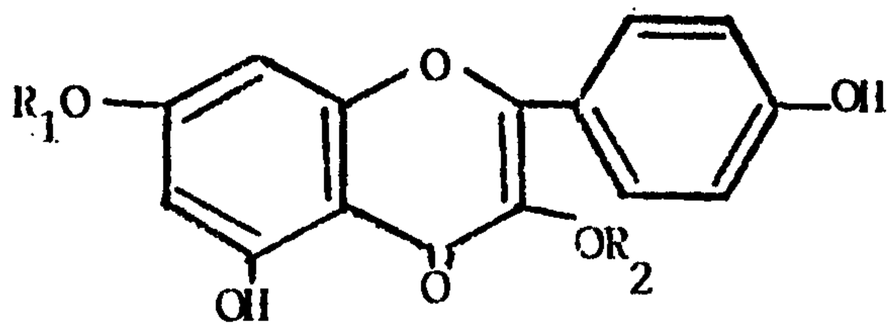
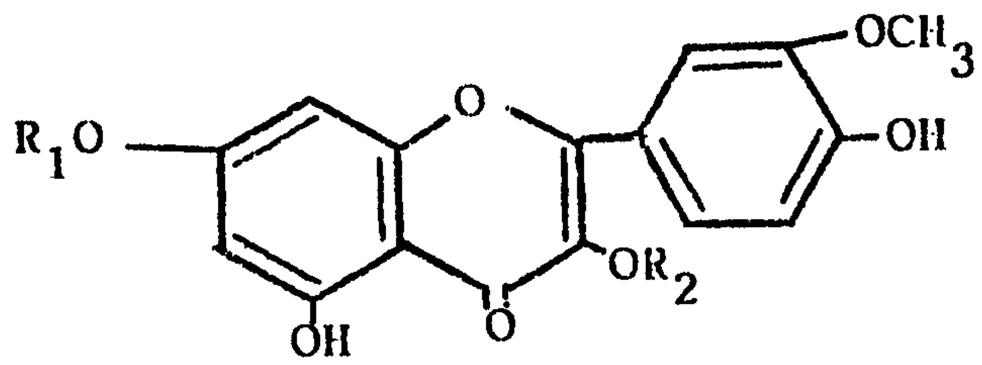
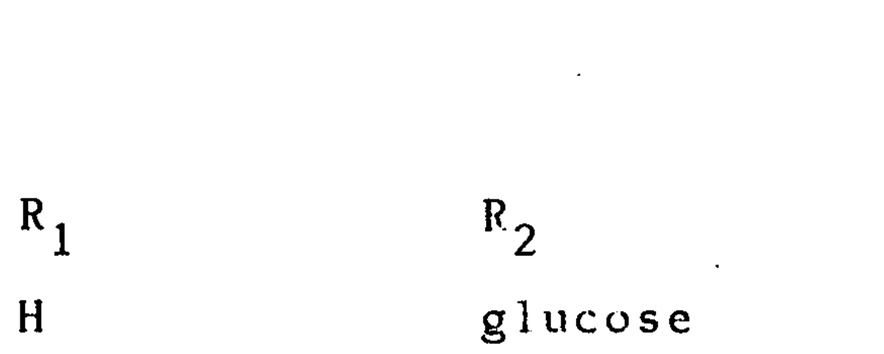


Compound	R_1	R_2	
3	H	H	kaempferol
7	H	glucose	kaempfero-3-O-glucoside
8	rutinoside	H	kaempferol-7-O-rutinoside.
9	H	rutinoside	kaempferol-3-O-rutinoside



Compound	R_1	R_2	
5	H	glucose	isorhamnetin-3-O-glucoside
6	glucose	H	isorhamnetin-7-O-glucoside
10	H	rutinoside	isorhamnetin-3-O-rutinoside

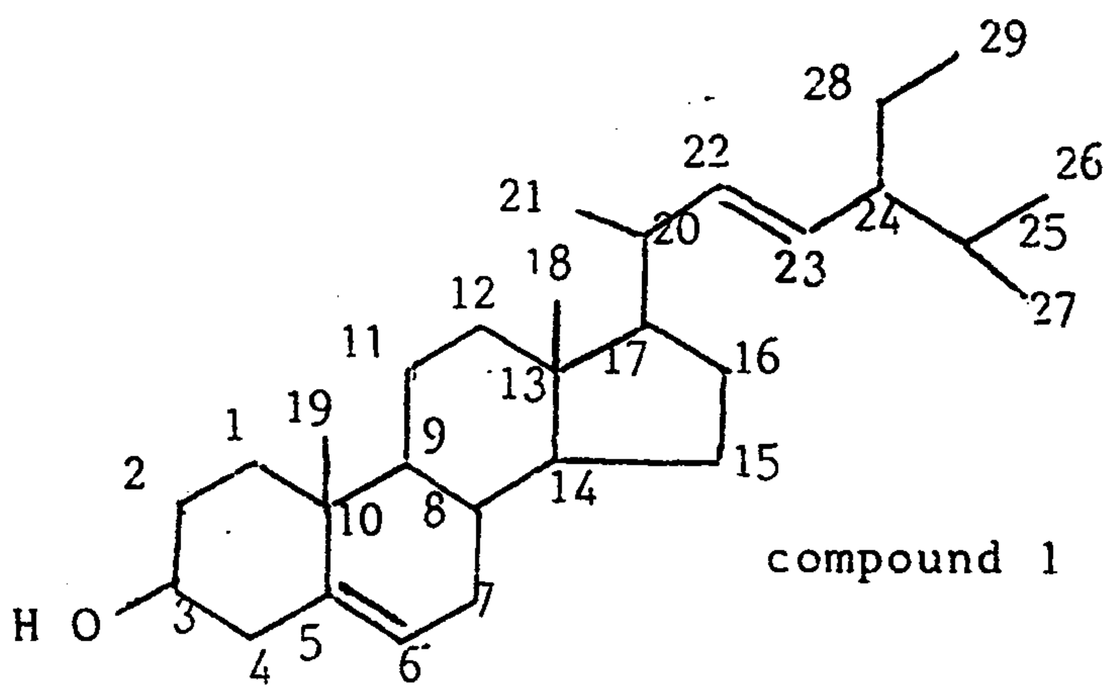


Compound 4; quercetin.

Table 1: Characters of the isolated steroids.

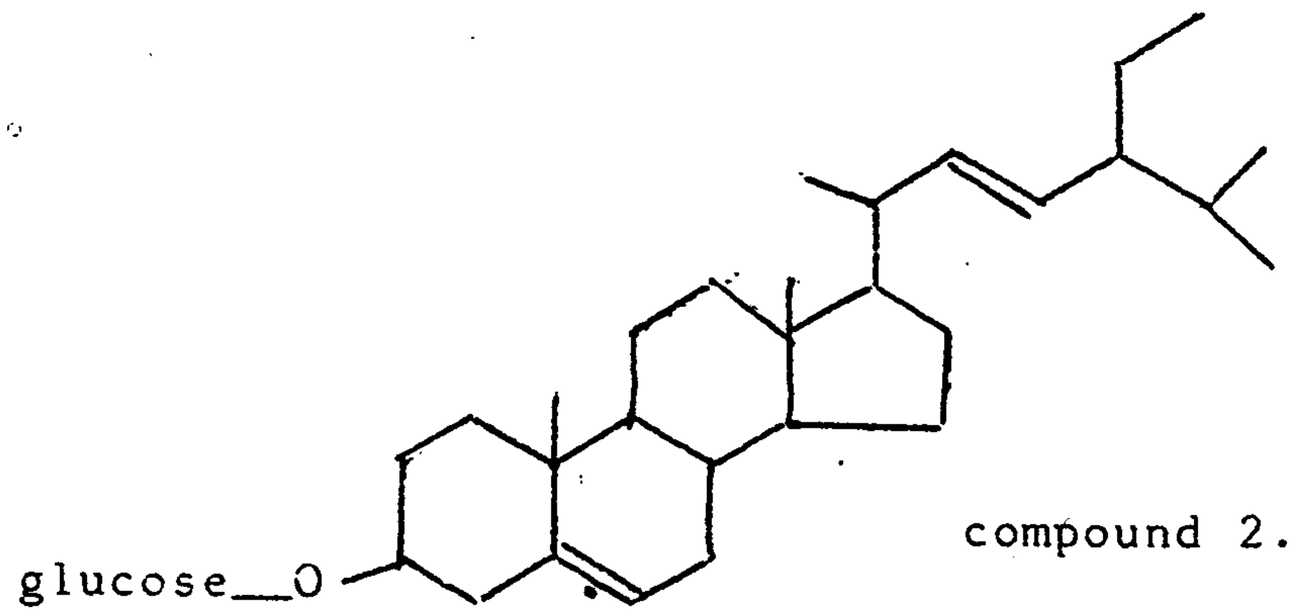
No.	R_f	solvent system	Occurrence	colour with Vanillin-H_2SO_4 and heating at 110° C	crystal forms and m.p.	Wt. and percentage yield.
1	0.14	I	L., S. &fl. (ether fract.)	violet colour	white needles m.p.160-64 C°	50 mg, 0.001%
2	0.05	VI	L, S, fl &Fr. (ether fract.)	deep violet colour	white amorphous m.p.233-35 C°	7 mg, ,0.00014%

L=leaves ; S=stems; fl.=flowers and fr.=fruits.



compound 1

ethylcholesta-5,22-diene-3B-OI
(stigmasterol).



compound 2.

stigmasterol-3-O-glucoside

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Table 2: ^{1}H-NMR (300 MH) of compounds 1 &2

Proton	Chemical shift (δ - ppm)	
	Compound 1	Compound 2
H-6	5.35(dd) J=7.5 & 2.5 Hz	5.35 (dd) J=7.5 & 2.5 Hz
H-22	5.15(dd) J=15.0 ,8.5 Hz	5.20
H-23	5.00(dd) J=15.0,8.5 Hz	5.3
H-3	3.52(dddd) J=10,10,4.5,4.5	3.95 (m)
18-CH_3	0.70(s)	0.64 (s)
19-CH_3	1.03 (s)	0.85 (s)
21-CH_3	1.02 (d) J=6.5 Hz	0.98 (d) J=6.5 Hz
26-CH_3	0.84 (d) J=6.5 Hz	0.90 (d) J=6.5 Hz
27-CH_3	0.79 (d) J=6.5 Hz	0.84 (d) J=6.5 Hz
29-CH_3	0.80 (t) J=7.5 Hz	0.85 (t)
CH_2 and CH	1.0 -2.3	1.0-2.8
H-$\bar{1}$		5.07 (d) J=7.5 Hz
H-$\bar{2}$		4.07 (t) J=8.0 Hz
H-$\bar{3}$		4.30 (t) J=8.5 Hz
H-$\bar{4}$		4.32 (t) J=8.5 Hz
H-$\bar{5}$		3.98 (m)
H-$\bar{6}$-a		4.58 (dd) J=11.5,2.5 Hz
H-$\bar{6}$-b		4.42 (dd) J=11.0,5.5 Hz

Table 3: ^{13}C-NMR spectrum of compound 1

Carbon atom	Chemical shift (δ-ppm) Isolated	Authentic	Reported*
C-18	12.0	12.0	12.1
C-29	12.3	12.3	12.5
C-19	19.0	19.0	19.2
C-26	19.4	19.4	19.6
C-27	21.1	21.1	21.3
C-21	21.1	21.1	21.4
C-11	21.2	21.2	21.5
C-15	24.4	24.4	24.6
C-16	25.4	25.4	25.7
C-28	28.9	28.9	29.3
C-7	31.6	31.6	32.0
C-2	31.9	31.9	32.3
C-8	31.9	31.9	32.3
C-25	31.9	31.9	32.3
C-10	36.5	36,5	36.9
C-1	37.2	37.2	37.8
C-4	39.7	39.7	39.9
C-20	40.5	40.5	40.8
C-13	42.2	42.2	42.4
C-12	42.3	42.3	42.2
C-9	50.1	50.1	50.5
C-24	51.2	51.2	51.5
C-14	55.9	55.9	56.1
C-17	56.9	56.8	57.0
C-3	71.8	71.8	71.2
C-23	121.7	121.6	121.2
C-22	129.2	129.2	129.5
C-6	138.2	138.2	138.8
C-5	140.6	140.7	142.0

*Measured in pyridine. (Reference [8,9])

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Table 4:Flavonoids Isolated from Cassia didymobotrya Fres.

Comp.No.	R_f	Solvent system	Colour UV/with 1%sol. of $AlCl_3$	Presence	Forms and m.p.	Wt.and % percentage
3	0.35	II	yell.fluore.	L,S&fr. ether fra.	pale yellow powder,282°C	20 mg 0.0004%
4	0.25	II	yell.fluore.	L,S,Fl&fr. ether fra.	yell.green 315-17°C	15 mg 0.0003%
5	0.70	I	yell.fluore.	L,S &Fl. ether fra.	yell.powder 165-66°C	7 mg 0.0001%
6	0.70	II	yell.fluore	L,S&Fl. ether fra.	yell.powder 218-20°C	10 mg 0.0002%
7	0.06	II	yell.fluore.	L,S,Fl&Fr. ethyl acet.	yell.powder .242-44°C	20 mg 0.0004%
8	0.29	I	yell.fluore.	L,S,Fl&Fr. ethyl acet.	yell.powder .250-52°C	12 mg 0.00024%
9	0.31	VII	yell.fluore.	L,S,Fl7Fr. ethyl acet.	yell.powder .260-62°C	30 mg 0.0006%
10	0.34	III	yell.fluore.	L,S,& Fl. n-butanol	yell.powder 128-31°C	10 mg 0.0002%
11	0.20	VIII	yell.fluore.	L,S.&Fl.	yell.amorph.	4 mg 0.00008%

L=leaves;S=stems;Fl=flowers and Fr=fruits.

Table 5:UV spectroscopic data of the isolated flavonoids .

Compound	MeOH max, nm	NaOMe max, nm	$AlCl_3$ max, nm	$AlCl_3$/HCl max, nm	NaOAc max, nm	NaOAc/H_3BO_3 max, nm
3	262,295,320,362	270,310,410	262,345,422	262,345,422	267,320,380	262,310 362
4	253,268,300,365	253,270,322,405	268,300,340,440	262,355,425	253,288,372	255,288,378
5	253,262,295,353	268,323,407	265,300,365,400	265,300,355,400	268,320,366	253,262,305,353
6	250,295,365	265,320,405	260,350,425	260,350,425	250,295,365	250,295,365
7	262,300,346	273,322,395	272,300,348,390	274,300,348,390	270,300,358	262,300,346
8	265,290,320,365	275,320,405	265,300,345,425	265,300,345,425	265,290,320,365	265,290,320,365
9	262,300,346	270,320,396	270,300,350	270,300,345,395	263,300,345	262,300,346
10	252,260,353	265,322,410	263,300,400	263,300,400	270,320,367	252,260,353
11	252,260,353	262,325,415	263 300,400,	263,300,395	265,300,370,	252,260,353.

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Table 6: ^{1}H-NMR data (300 MHz) of the isolated flavonoids.

Compound	3	4	5	6
	6.17(d,J=2 Hz,H-6)	6.04(d,J=2.5 Hz,H-6)	3.94(s,-OCH_3)	3.95(s,-OCH_3)
	6.38(d,J=2 Hz,H-8)	6.20(d,J=2.5 Hz,H-8)	6.0(d,J=2.5 Hz,H-6)	6.02(d,J=2.5 Hz,H-6)
	6.88(dd,J=8.2,5 Hz,H-3,5)	6.85(d,J=8.5 Hz,H-5')	6.13(d,J=2.5 Hz,H-8)	6.16(d,J=2.5 Hz,H-8)
	8.08(dd,J=8,2.5 Hz,H-2,6)	7.70(d,J=2.5 Hz,H-2')	6.86(d,J=8.6 Hz,H-5')	6.9(d,J=8.5 Hz,H-5')
		7.60(dd,J=8.5,2.5 Hz,H-6')	7.88(d,J=2.5 Hz H-2')	7.9(d,J=2.5 Hz,H-2')
			7.58(dd,J=8.5.2.5 Hz,H-6')	7.8(dd,J=8.5,2.5 Hz,H-6')
			5.18(dd,J=7.5,2.5 Hz,H-1'')	3.6-3.8=sugar protons.
			3.7(dd,J=11.5,2.5 Hz,H-6a)	
			3.56(dd,J=11.5,5.0 Hz,H-6b)	
			3.46(dd,J=7.0,2.0 Hz,H-2'')	
			3.34(dd,J=6.0,2.5 Hz,H-4'')	
			3.24(t,J=8.5 Hz)	
			3.20(ddd,J=2.0,6.0,10, Hz,H-5'')	

Compound	7	8	9	10	11
	.21(d,J=2 Hz,H-6)	1.13(d,J=6 Hz,CH_3-rha)	1.14(d,J=6 Hz,CH_3-rha)	3.95(s,-OCH_3)	3.95(s,-OCH_3)
	6.41(d,J=2 Hz,H-8)	6.2(d,J=2 Hz,H-6)	6.21(d,J=2 Hz,H-6)	6.02(d,J=2.5 Hz,H-6)	5.96(d,J=2.5 Hz,H-6)
	6.89(dd,J=8,2 Hz,	6.4(d,J=2 Hz,H-8)	6.41(d,J=2 Hz,H-8)	6.16(d,J=2.5 Hz,h-8)	6.00(d,J=2.5 Hz,H-8)
	H-3'-5')	4.53(d,J=1.5 Hz,H-1'''	5.13(d,J=7.5 Hz,H-1')	6.85(d,J=8.5 Hz,H-5')	6.68(d,J=8.5 Hz,H-5')
	8.06(dd,J=8,2 Hz,	rha.)	4.5(d,J=1.5 Hz,H-1''')	7.93(d,J=2.5 Hz,H-2')	7.59(dd,J=2.5,8.5 Hz,H-6')
	H-2',6')	6.89(dd,J=8.8 Hz,H-3',5')	6.89(dd,J=8,2 Hz,H-3',5')	5.03(d,J=7.5 Hz,H-1'')	7.90(d,J=2.5 Hz,H-2')
	5.25(d,J=7.5 Hz,H-1'')	8.07(dd,J=8.8 Hz,H-2',6')	8.06(dd,J=8,2 Hz,h-2',6')	4.49(d,J=2.0 Hz,H-1''')	4.89(d,J=6.0 Hz,H-1'')
	3.66(dd,J=10,2.5 Hz,	5.11(br.,H-1'' gluco.)	3.66(dd,J=10,2.5 Hz,H-6'' a)	.13(d,J=6.5 Hz.CH_3)	4.81(d,J=6.0 Hz,H-1''')
	H-6'' a)	3.2-3.8(sugar protons).	3.38(t,H-3'')	7.62(dd,J=2.5.8.5 Hz,H-6')	4.47(d,J=2.0 Hz,H-1'')
	3.44(dd,J=8,2 Hz,H-2'')		3.44(dd,J=8,2 Hz,H-2'')	3.81(dd,J=10,2.0 Hz,H-6''a)	1.14(d,J=6.5 Hz,CH_3)
	3.38(t,H-3'')		3.33(dd,J=10,2 Hz,H-4'')	3.50(dd,J=10,3.5 Hz,H-6''b)	3.5-3.8(sugar protons)
	3.33(dd,J=10,2 Hz,H-4'')		3.17(ddd,J=3,6.5,10 Hz,H-5'')	3.48(dd,J=3.5.2.0 Hz,H-2'').	
	3.17(ddd,J=3,6,5,10 Hz,H-5'')				
	3.66(dd,J=10,2.5 Hz,H-6'' a)				

Table 7 : ^{13}C-NMR spectrum of compounds 7 and 9

C-atom	Chemical shifts (δ-ppm)		Reported Data	
	Compound 7	Compound 9	Comp.7[13]	Comp.9[14]
C-2	157.17	158.22	156.30	158.70
C-3	134.96	134.74	133.00	135.50
C-4 1	178.02	177.71	177.40	179.40
C-5	159.09	162.07	161.10	163.03
C-6	103.25	103.53	98.70	100.02
C-7	162.50	164.17	164.10	166.21
C-8	97.12	97.42	93.60	94.95
C-9	157.71	159.15	156.30	159.41
C-10	100.60	102.17	104.10	105.63
C-1	122.32	121.28	121.00	122.76
C-2	131.96	132.07	130.70	132.38
C-3	116.31	116.94	115.00	116.15
C-4	159.09	162.03	159.80	161.52
C-5	116.31	116.94	115.00	116.15
C-6	131.96	132.07	130.70	132.80
C-7	105.20	105.93	101.40	104.06
C-2	75.66	75.66	74.20	75.76
C-3	78.25	78.29	76.50	78.14
C-4	71.22	72.17	70.10	71.45
C-5	78.14	77.09	77.20	77.22
C-6	62.58	68.72	61.00	68.57
C-1		102.45		102.60
C-2		72.01		72.10
C-3		71.36		72.30
C-4		73.92		73.39
C-5		69.70		69.73
C-6		18.01		17.93

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دراســـة كيميائية لنبات الكاسيا ديديموبوتريا فرس المنـــزرع فى مصـــر

٢ـ الجزء الثانى : دراسة الفلافونيدات والاستيرولات

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قسم العقاقـــير ـ كلية الصـــيدلة ـ جامعـــة أســـيوط
معهـــد الصيدلة ـ جامعة بـــون ـ المانيا الغربيـــة

تشمل هذه الدراسة فصل والتعرف على الفلافونيدات والاستيـــرولات الموجودة فى أوراق ، سيقان ، أزهار وثمار نبات الكاسيا ديديموبريافرس .

وقد تم فصل والتعرف على الفلافونيدات الموجودة فى النبات فـــى صورة نقية من خلال دراستها فيزيقيا وكيميائيا وطيفيا باستعمال الرنيـــن النووى البروتونى والكربونى ومطياف الكتله كذلك الاشعة تحت الحمراء وفوق البنفسجية والفلافونيدات المفصولة والمتعرف عليها هى كالاتى : ـ
أيرورامنتين ـ ٣ ـ جلوكوز ، ايزورامنتين ـ ٣ جلوكوزـرامنوز ، ايزورامنتين ـ ٧ ـ جلوكوز ، كامبيفرول ـ ٧ ـ جلوكوزرامنوز ، كامبيفرول ، كوراستين كامبيفرول ـ ٣ ـ جلوكوز وكامبيفرول ـ ٣ ـ جلوكوز رامنوز بالاضافة الـــى فلافونيد آخر مازال تحت البحث .

ومن الاستيرولات تم فصل والتعرف على ستيجما ستيرول وستيجما ستيـــــرول ٣ ـ جلوكـــوز .

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