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INVESTIGATION OF THE PRESS CAKE OF SUGAR CANE CULTIVATED IN EGYTP

A.A. Ali, A.M. El-Moghazy, M.A. El-Mottaleb, and M.S. Mansour
Department of Pharmacognosy , Faculty of Pharmacy, Assiut University, Assiut, Egytp.

Key Word Index: Press cake of sugar Cane(Graminae), Lipids, amino acids and Minerals.

The petroleum ether extract of the air-dried press cake resulting from Cane Sugar Industry was concentrated where ceryl alcohol precipitated out. The dried dark green mother liquor was saponified. Many long chain alkanes, long chain &lipatic, alcohols, and steroids were separated from the unsap. matter. The methyl esters of Fatty acids content and certain minerals of press cake were determined quantitataly.

Thousands of acres in most upper Egypt Governorates are cultivated with sugar cane(Saccharum officinarum. Family: Graminae) from which the lipid-protein heat coagulate,known as press cake,is produced in huge quantities as a by-product from sugar industry .

Many utilisations have been described for this press cake, the most promonent of which are : in animal feedings, production of sugar cane wax, as fertilizer etc. The wax was reported to be used as emulsifier, in fruit coating productions, in leather and floor polishes, etc.[1,2]

This paper deals with the investigation of certain contents of this press cake.

EXPERIMENTAL AND RESULTS

Material:

The press cake was obtained from Cane Sugar Factories in Nag-Hammadi and Abu-Kurkas in January and May (1977)and was air dried.

A- The determination of moisture (8.62%), ash (20.25%), acidinsoluble ash (6.75%), water-soluble ash (5.98%) and crude Fibres (5..75%) was performed[1].

B- Ash analysis:[1,3]

1 g. of the material was digested in HNO_3/ H_2SO_4/$HClO_4$ acid mixture (10 : 1 : 4).

a) Na (1.5%) and K (1.85%) content were measured by flame photometry.

b) P (0.26%) and Fe (0.27%) content were determined colorimetrically[3].

c) Ca (3.06%) and Mg (1.30%) content were assayed by titration with EDTA.
high calcium content is due to liming.

C- Investigation of Lipoid Matters:

Extraction:

5 kg. of material was continuously extracted with boiling petroleum ether (b.r. 60-80°C) to exhaustion. The dark green extract. was concentrated to 1 litre and left overnight at room temperature where a greeniah creamy wax precipitated. It was filtered off, purified by repeated crystallisation from petroleum then dissolution in boiling alc. where colourless wax (223 g) was obtained, m.p. 79-80°, corresponding to that of ceryl alc.[4]. Its identity with ceryl alc. was confirmed by mixed m.p., of its acetate deriv., identical I.R., mass and NMR spectra.

The combined petroleum ether left after removal of most of ceryl alcohol was evaporated. The residue(265 g) was saponified by refluxing with 3 litres of 0.5 N alc KOH for about 6 hr. The Unsaponified matter (85 g) was extracted with ether. The alkaline aqueous solution (soap) was then acidified with H_2SO_4. The liberated fatty acids (180 g) were extracted with ether. GLC of their methyl esters[5] revealed capric (12.44%), lauric (1.22%) myristic (2.00%), iso-palmitic (1.26%). palmitic (11.56%), palmitoleic (9.40%). stearic (12.22%). oleic (41.5%) and linolenic acids (8.30%).

Investigation Of Unsaponifiable Matter:

TLC investigation of the Unsaponifiable matter, using activated layers of silica gel G (F. Merck) and the solvent systems benzene/petroleum ether (3.1) system 1) and benzene/ethanol (9:1) (system II) revealed the presence of 12 spots(listed in Table 1).

Fractionation Of The Unsap. Matter:

The twelve components given in Table 1 were fractionated by C.C. using activated alumina (E. Merck) and the solvents petroleum ether, petroleum ether/benzene and finally benzene/ethanol.

The fractions showing a single spot were combined and evaporated and the residue purified by repeated cryst. from a suitable solvent (Table 2).

Identification Of The Separated Substances:

Eight out of these substances, detected as single spots were analysed by I. R., NMR and G.C. mass spectrometry. Table 3 compiles the results of G.C. mass spectra of six substances separated from the unsaponifiable matter.

Compound No. 10:

Steroidal in nature, contain OH and $-\overset{O}{\overset{\|}{C}}-$ groups at 3325 cm^{-1} and 1750 cm^{-1} respectively, M^+(430), gave intense blue colour with H_2SO_4, and due to its small quantity it was not investigated further.

Compound No. 12:

Steroidal in nature, contains -OH group at 3400 cm^{-1} M^+414. Although its molecular weight is identical to that of B.sitosterol yet it differs from the latter in both m.p. and R_f value. Its presence in trace quantities hindered its further investigation.

A thorough investigation of petroleum ether extract of press cake by G.C. mass spectrometry revealed the presence of long chain alkanes C_{25} - C_{31} (C_{27} and C_{29} being predominent).

According to the available literature, tricosane-3-one; dihydro-B-sitosterol, dihydro- ∝ -sitosterol and tetrahydro ∝ -sitosterol are reported for the 1st time.

The amino acids, of press cake [1,6]:

The proteins of each of two samples of press cake (sample A at January and sample B at Ma) in a total nitrogen content 1,127 g/100 g and 0.875 g /100 g respectively were h drolysed using 10 N HCl 23 amino acids were identified by using chromatostrip whatman NO 1., solvent system n-butanol/acetic acid/water (4:1:5) and amino acid analyser. The conc. of the different amino acids were calculated. They are listed in Table 4.

The study of proteins of press cake revealed the presence of 23 amino acids. The main components were ornithine, glutamic acid, arginine and glycine.

According to our knowledge, this paper represents the first report about the pre ence of systin, systin, ornithine, asparagine, glutamine and hydroxy proline.

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Many thanks to the members of Sugar Cane Factory, Abou-Kurkas for their help in giving us the material of press cake

Table 1: Thin-layer chromatography of the unsaponifiable matter(Fraction II) of the press cake of Sugar cane,

Spot	Colour reaction with sulphuric acid.	Colour reaction with antimony trichloride	R_f value on silica gel G. (E.Merck)		Reference
			System I	System II	
1	Brown	Brown	0.98	0.98	-
2	,,	Gray	0.90	-	-
3	Yellow	Yellow	0.40	0.94	-
4	Faint brown	Faint brown	0.36	0.92	-
5	,, ,,	,, ,,	0.29	0.88	-
6	Dark brown	Dark brown	0.20	0.83	Ceryl-alcohol
7	Faint brown	Faint brown	0.14	0.76	-
8	Dark violet	Violet	0.10	0.66	8-sitesterol
9	Faint brown	Yellow	0.00	0.43	-
10	Dark blue	Blue	0.00	0.33	-
11	Brown	Brown	0.00	0.24	-
12	Violet	,,	0.00	0.17	-

System I : Benzene-petroleum ether (9 : 1)
System II : Benzene-ethyl alcohol (9 : 1)

Table 2: Physical, Chemical and Instrumental Study of the Separated Substances:

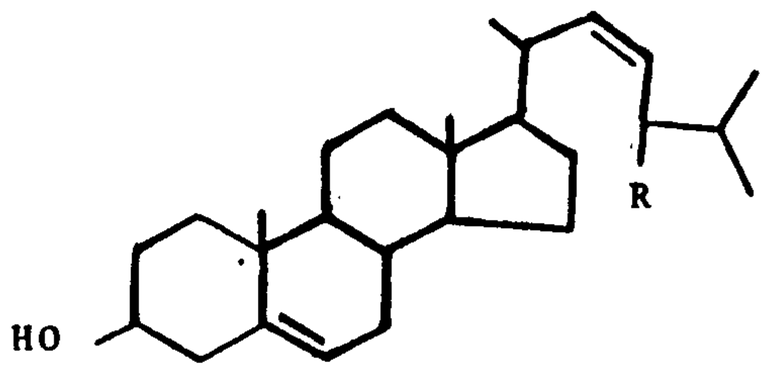
Sep. Sub.	Solvent of crystal.	Shape of crystals	Tests for sterols and/or triterpenes	I.R	N.M.R
I	acetone	flakes	(-) ve	no special functional groups.	Single peak at 3. 1.2 indicating a straight chain structure.
II	acetone	flakes	(-) ve	no special functional groups.	Single peak at 3. 1.2 indicating a straight chain structure.
III	acetone	needles	(+) ve	at 3400 cm^{-1}=oH at 1720 cm^{-1}= C ^{O}	Showing general pattern of N M R of sterols.
VI	methanol	flakes	(-) ve	at 3400 cm^{-1}= oH	Single peak at 3. 1.2 indicating a straight chain structure
VIII	methanol	needles	(+) ve	at 3420 cm^{-1}= oH	Showing general pattern of N M R of sterols.
X	acetone	granules	(+) ve	at 3325 cm^{-1}= oH at 1750 cm^{-1}= C=O	Showing general pattern of N M R of sterols
XI	ethyl acetate	needles	(+) ve	at 3400 cm^{-1}= oH	Showing general pattern of N M R of sterols.
XII	ethyl acetate	granules	(+) ve	at 3400 cm^{-1}= oH	Showing general pattern of N M R of sterols.

Table 3: G.C. Mass Spectral Analysis of the Six Substances Separated from the Unsap. Matter of the Press Cake of Sugar Cane:

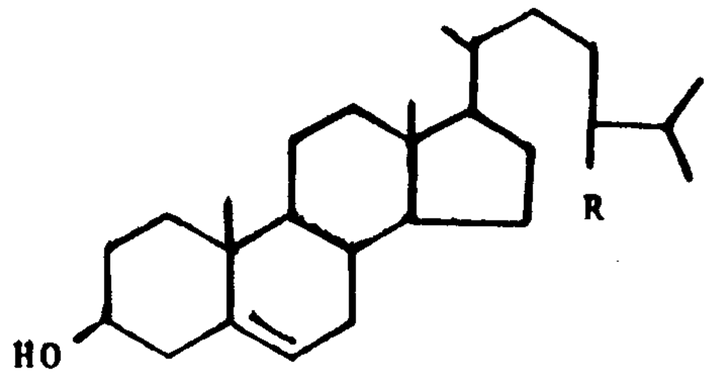
residue corresp. to spot No.	M+ molecular weight	Possible structure	% of each component	Possible to be
I	352	$CH_3(CH_2)_{23}CH_3$	4.10	pentacosane
	366	$CH_3(CH_2)_{24}CH_3$	4.60	hexacosane
	380	$CH_3(CH_2)_{25}CH_3$	35.26	heptacosane
	394	$CH_3(CH_2)_{26}CH_3$	2.70	octacosane
	408	$CH_3(CH_2)_{27}CH_3$	30.21	ninacosane
	436	$CH_3(CH_2)\ \ CH_3$	14.89	hentriacontane
II	352	$CH_3(CH_2)_{23}CH_3$	5.45	pentacosane
	366	$CH_3(CH_2)_{24}CH_3$	6.56	hexacosane
	380	$CH_3(CH_2)_{25}CH_3$	52.27	heptacosane
	408	$CH_3(CH_2)_{27}CH_3$	27.27	ninacosane
	436	$CH_3(CH_2)_{29}CH_3$	5.27	hentriacontane
	450	$CH_3(CH_2)_{30}CH_3$	3.59	dotriacontane
III	338	$CH_3CH_2C\overset{O}{}-(CH_2)_{19}CH_3$	69.00	tricosane-3-one
	398	outside this table in a separate figure	19.00	brassicasterol
	412		3.00	stigmasterol
	426		9.00	∝ -sitosterol
VI	382	$C_{26}H_{53}OH$		ceryl alcohol
VIII	414	outside this table in a separate figure	9.00	B-sitosterol
	412		27	stigmasterol
	400		64	campesterol
XI	416	outside this table in a separate figure	10.10	dihydro-B-sitosterol (stigmastanol)
	428		28.00	dihydro-∝-sitosterol
	430		61.00	tetrahydro-∝-sitosterol.

Table 4: Amino Acids Composition of Press Cake:

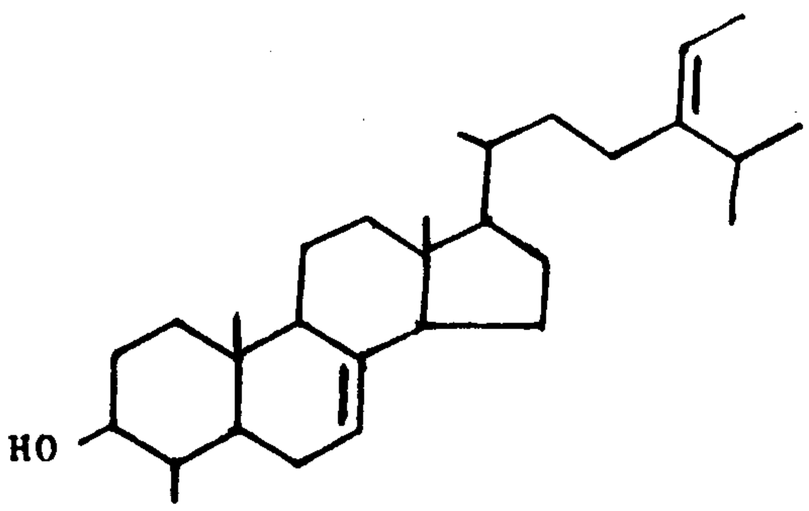
No	Amino acid	Sample A		Sample B	
		mg/100	g	mg/100	g
22	systein	0.16		0.78	
5	systein	0.43		0.31	
14	ornithine	1.63		0.39	
20	lysine	0.55		0.32	
15	histidine	0.50		0.68	
3	asparagine	0.54		0.38	
17	serine	0.58		0,16	
16	aspartic acid	0.25		0.26	
2	glutamine	0.50		0.27	
4	glycine	3.25		1.60	
11	arginine	1.35		1.05	
19	threonine	0.36		0.30	
1	glutamic acid	1.40		1.20	
9	alanine	0.68		0.56	
6	methionine	0.21		0.61	
12+13	prolino&OH proline	++		++	
21	tyrosine	0.09		0.12	
23	tryptophan	0.19		0.24	
8	valine	0.22		0.11	
18	phenyl alanins	0.70		0.54	
10	isoleucine	0.86		0.54	
7	leucine	0.67		0.75	



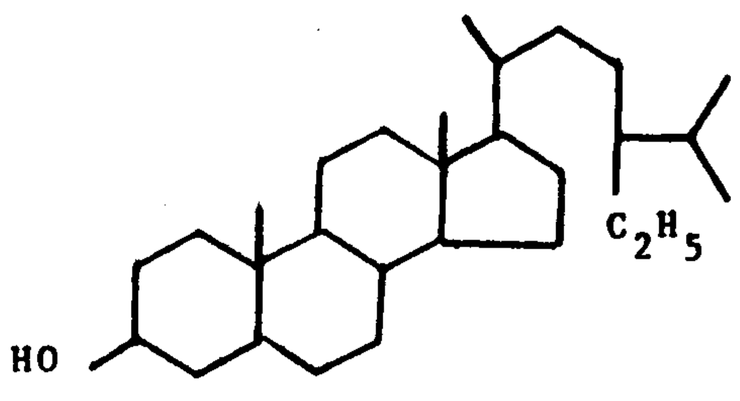
Brassicasterol (R = CH_3)
Stigmasterol (R = C_2H_5)



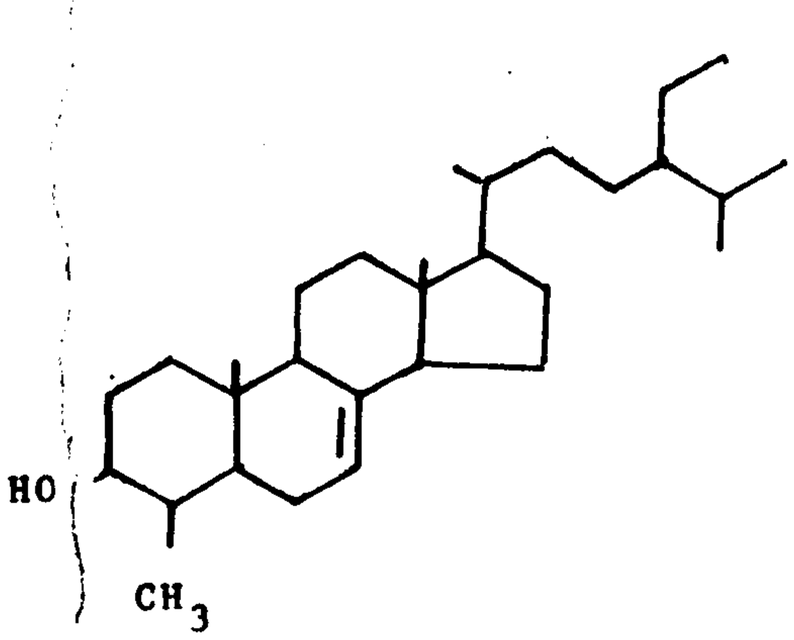
Compesterol (R = CH_3)
B-sitosterol (R = C_2H_5)



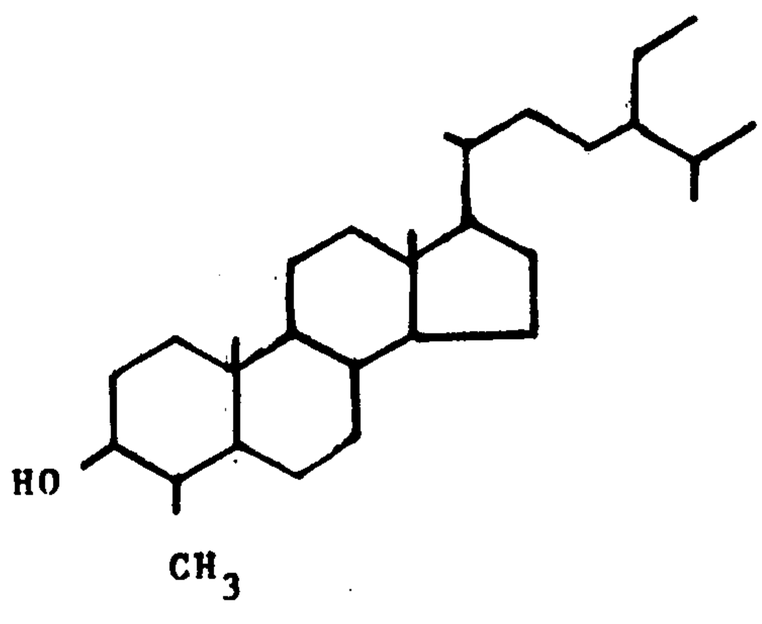
∝-sitosterol



Dihydro-B-sitosterol



Dihydro-∝- sitosterol



tetrahydro-∝-sitosterol

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دراسة طينة مرشحات قصب السكر المنزرع فى مصر

احمد عبدالرحمن على ـ احمد محمد المغازى ـ محمد عبدالمطلب ـ محمد منصور
قسم العقاقير ـ كلية الصيدلة ـ جامعة اسيوط
الهيئة القومية للابحاث والرقابة الدوائية ـ القاهرة

أشتملت الدراسة الكيميائية على مايلى :

١ـ عينت بعض الثوابت الدستورية (الرماد الكلى ـ والغير قابل للذوبان فى الحمض ونسبة الرطوبة).

٢ـ تم فصل المواد الدهنية باستخدام البترول الاثيرى الساخن وتبريده ترسبت مادة الكحول السيريلى .

٣ـ قد تم التعرف على الاحماض الدهنية بواسطة كروماتوجرافيا الغاز

٤ـ قد تم دراسة المواد الغير متصبنة بواسطة كروماتوجرافيا الغاز المقرون بمطياف الكتلة وامكن فصل والتعرف على العديد من الهيدروكربونات والكحوليات والكيتونات والاستيرودات.

٥ـ قد تم دراسة الاحماض الامينية وامكن التعرف على ٢٣ حمض امينى وتقدير نسبتهم.

٦ـ قد تم دراسة بعض العناصر المعدنية لطينة المرشحات وتقدير نسبتها المئوية.

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