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# PHYTOCHEMICAL STUDIES ON JASMINIUM MESNYI HEMSIL

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*Ceryl alcohol, ∝ -amyrin, B-sitosterol, Ursolic acid, mannitol, quercetin, flavonol rutin and bitter glucoside jasminin were isolated from the leaves of J. mesnyi Hemsil. Arachidic and arachidonic acids are the most prominent fatty acids present in the soap fraction of the petroleum-ether extract. The percentages of rutin as well as total flavonoids in the leaves were determined to be 2.58 and 2.95 g% W/W respection.*

Jasminium mesnyi Hemsil ( = Jasminium primulinum) is a climbing shrub belonging to F. Oleaceae [1]. Previous studies on J. mesnyi Hemsil reported that a bitter principle jasminin was isolated from its leaves [2]. A glycoside of unknown structure was isolated from J. mesnyi [3] and ursolic acid from F. Oleaceae [4] A monoterpenoid alkaloid have been isolated from J. gracile Andr. and J. linear R. Br. [5]. Many plants belonging to F.Oleaceae are used medicinally as laxative, diuretic, sedative, narcotic, bitter tonic, curing agent for wounds and antidote for cobra-venom (6-9).

## RESULTS AND DISCUSSION

Preliminary phytochemical examination indicated the presence of sterols, triterpenes, saponins, flavonoids, carbohydrates and alkaloids in the leaves and stems of J. mesnyi Hemsil.

On the basis of co-Chromatography, m.mp., physical properties, chemical tests and comparison of IR-spectra as well as preparation of certain derivatives, the following compounds were found to be present in the leaves: ceryl alcohol, ∝ -amyrin, B-sitosterol, Ursolic

acid and mannitol. The soap fraction of the petroleum-ether extract was methylated and examined by GLC. Arachidic and arachidonic acids were the most prominent fatty acids while palmitic, stearic, oleic and linoleic acids were detected as minor components.

By chromatographing the leaf alcoholic extract over cellulose and silica gel columns, two glycosides in large amounts were isolated and identified to be flavonol rutin and bitter glucoside Jasminin.

The percentages of rutin as well as total flavonoids calculated as rutin in the leaves were determined to be 2.58 and 2.95 g% W/W respectively.

## EXPERIMENTAL

### Plant material:

Collection from Experimental Station of the Faculty of Agriculture, Assiut University, Assiut, Egypt.

### Preliminary phytochemical examination:

Phytochemical screening for the characterization of the active principles in the leaves and stems (Table I) was carried out using Wall et al. method[10].

### Extraction and fractionation:

2 Kg. of the powdered leaves were successively extracted with pet.-ether, ether, acetone and alcohol 90%. All extracts were separately concentrated and fractionated as follows:

The pet.-ether extract was saponified with ½ N alcoholic KOH. The unsaponifiable matter (10 g .) was fractionated over alumina column (400 g) using eluents pet.-ether, then pet.-ether: benzene mixtures, yielded ceryl alcohol, ∝ -amyrin and B-sitosterol. The methyl esters of fatty acids mixture were prepared[11] and analysed by GLC on Reoplex 400 liquid phase. The following fatty acids were detected and estimated: arachidonic (51.5%), arachidic (33.25%), linoleic (0.35%), Oleic (0.23%), stearic (0.3%), unknown (3.8%), unknown (2.13%) and palmitic (2.89).

The dried ether extract was extracted with 2% alcoholic KOH. The alkaline extract was concentrated, acidified with HCL, Extracted with ether and the ether was distilled off. The residue was recrystallized from methanol to give ursolic acid.

When the acetone extract was concentrated, a white precipitate was separated. By recrystallization from methanol, white needles of mannitol were obtained.

About 25 g. of the concentrated alcoholic extract were fractionated over cellulose column (500 g.), eluted with a mixture of $CHCl_3$ : $CH_3OH$ : $H_2O$ (25 :12 : 2). Flavonol rutin and a mixture of 3 bitter glycosides were isolated. That mixture was again fractionated over silica gel column; yielded bitter glucoside Jasminin in a pure form.

Ceryl alcohol:

M.P. 80-82° ; TLC on silica gel G, solvent I, $R_f$ = 0.6. Ceryl acetate m.p 66-68°.

∝- amyrin:

m.p. 184-186°; TLC on silica gel G, solvent I, $R_f$= 0.47. ∝-amyrin acetate m.p. 225-227°.

B-sitosterol:

m.p. 135-137°; TLC on silica gel G, solvent I, $R_f$ =0.38. B-sitosterol acetate m.p. 125-127°.

Ursolic acid:

m .p. 277-279° {lit. (12) 277-279°}; m.mp. with auth. sample was undepressed; TLC on silica gel g, solvent II, $R_f$ 0.36.

Mannitol:

m.p. 167-169° {lit. (13) 166° } ; m.mp. with auth. sample was undepressed; $M^+$ 182; IR ($cm^{-1}$) 3200-3400 (OH).

Rutin:

m.p. 194-197° {lit. (14) 190-192° } ; TLC on cellulose, solvent III, $R_f$=0.28. UV (MeOH) 260,365 nm; + NaOAc 270, 380 nm; + NaOAc/$H_3BO_3$ 265,385 nm; + NaOMe 274,420 nm; +$AlCl_3$ 275, 430 nm. Hydrolysis in N HCl for 2 hours; yielded quercetin, rhamnose and glucose.

Quercetin;

m.p. 316-318° {Lit. (15) 317-319°} ; PC, solvent IV, $R_f$ =0.16 ; $M^+$ 302. UV (MeOH) 257,370 nm; + NaOAc 270,320,385 nm; + NaOAc/$H_3BO_3$ 265,390 nm; + $AlCl_3$ 270, 340, 460 nm. Quercetin pentaacetate m.p. 144-146° {Lit. (16) 146-148° }

Jasminin:

m.p. 159-160° {Lit. (2) 159-160°}; m.mp with auth. sample was undepressed; TLC on silica gel G, solvent V , $R_f$= 0.37. UV (MeOH) 238 nm. IR ($cm^{-1}$): 3400-3420 (OH); 1680 (C=O); 1730 (six membered lactone ring); 1080-1040 (-C-O-C-stretch); 720,765 and 775(adjacent hydrogen in aromatic ring). NMR-spectrum, $_{\delta}$methanol:1.0 (6H, dd, J=3.5 and 4 $H_z$, 2 secondary C-methyl groups) ; 1.80 (3H,d, J=9$H_z$, $CH_3$-CH = C< ); 3.3-3.9 (6H, m , glucose 6 protons) ; 5.0 (1H, anomeric proton of glucose); 5.95 (1H, $CH_3$-CH=C< ); 7.48 (1H, S , -O-CO-C =CH-O-).

Procedure for estimation of flavonoids:

A) Estimation of total flavonoids calculated as rutin :

5 g. of the defatted powdered leaves (P) were extracted with $CH_3OH$ and the extract was concentrated to 10 ml (V). 0.1 ml of the extract ($V_1$) was chromatographed on PC (3 mm) using solvent VI. Flavonoidal spots were cut, eluted with $CH_3OH$ and the eluate was concentrated to 10 ml (W). The absorbancy of the eluate was determined at wave length 365 nm and the corresponding concentration was calculated (C). Percentage of total flavonoids (X)was calculated:

$$X = \frac{W.C.V.100.100}{P.V_1.E.1000\ (100 - M)}$$

Where

M= moisture content in the leaves, was carried out according to the E.P. (1972) and was found to be 8.7%.

E= Percentage of elution of rutin from PC and was found to be 80.0%.

The percentage of total flavonoids calulated as rutin (X) was found to be 2.95 g% W/W

B) Estimation of rutin:

Followed the same procedure for estimation of total flavonoids. Here, we make elution only for the spot corresponding to rutin. The percentage of rutin in the leaves was found to be 2.58 g% W/W.

Table I: Preliminary phytochemical screening for the leaves and stems of J. mesnyi hemsil

| *Constituents* | *Leaves* | *Stems* |
|---|---|---|
| Sterols, triterpenes, flavonoids, saponins carbohydrates and alkaloids. | + | + |
| Coumarins, carderolides, volatile oils, anthraquinones, cyanogenetic glycosides and oxidase enzyme. | - | - |

Legend: + = positive ; - = nagative.

Table II: Solvent systems used

| *Solvents No.* | *Composition* |
|---|---|
| Solvent I | Chloroform: methanol (99.5 : 0.5 |
| Solvent II | Chloroform: petroleum-ether (1 : 1) |
| Solvent III | Chloroform: methanol : water (25:12:2) |
| Solvent IV | Chloroform: acetic acid : water (50:45:5) |
| Solvent V | Chloroform: methanol (85 : 15) |
| Solvent VI | n-butanol : acetic acid:water (4:1:2). |

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## دراسة فيتوكيميائية لنبات الياسمين الاصفر (جاسمينيم سناى هيمـــــــيل )

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أثبت المسح الكيميائى الاولى ان اوراق وسيقان نبات الياسمين الاصفر تحتوى على ستيرولات و/أو تربينات ثلاثية ،فلافونويدات ، قلويدات ، كربوهيدرات و/أو جليكوزيدات. اشتملت الدراسة الكيميائية على مايلى :

١ــ تمت دراسة المواد الدهنية فى الاوراق وقد درست :

أ ــ المواد الغير متصبنة للمواد الدهنية واسفرت عن فصل المواد الاتية :

بيتاسيتوستيرول ، درجة انصهارها ١٣٤ ــ ١٣٧°م .

الفا أميرين ، درجة انصهارها ١٨٤ ــ ١٨٦°م .

كحول سيريلى ، درجة انصهارها ٨٠ ــ ٨٤°م .

ب ــ أسفرت دراسة الاحماض الدهنية باستعمال كروماتوجرافيا الغاز عن التعرف على المكونات الاتية وتقديرها كميا وهى :

أحماض دهنية مشبعة : باليميتيك ، ستياريك ،أراشيديك

أحماض دهنية غير مشبعة : أوليك ،لينوليك ، اراشيدونيك .

٢ ــ تم فصل حمض الاورسوليك (درجة انصهاره ٢٧٧ ــ ٢٧٩°م ) من الخلاصة الاثيرية ومـــادة المانيتول (درجة انصهاره ١٦٧ ــ ١٦٩°م ) بكمية كبيرة وفى صورة نقية من الخلاصـــــة الاسيتونيـــــــة .

٣ ــ تمت تجزئة الخلاصة الكحولية باستعمال كروماتوجرافيا العمود مستخدما مادة السليلـــــوز وكذلك هلام السيلكا للحصول على مركبين فى صورة نقية . اثبت تركيبهم الكيميائى عن طريـــق دراسة صفاتهم الفيزيائية والتحليل الصيغى بالاشعة فوق البنفسجية والاشعة تحت الحمـــراء والرنين النووى المغناطيسى وايضا باستخدام مطياف الكتلة والحلماة المحمضية على انهم :

روتين ، درجة انصهاره ١٩٤ ــ ١٩٧°م .

جاسمينين درجة انصهاره ١٥٩ ــ ١٦٠°م .

٤ ــ تبين أن النسبة المئوية الكلية للمركبات الفلافونية فى الاوراق محسوبة على انها روتين هـــى ٢٫٩٥٪ بينما النسبة المئوية لمادة الروتين فى الاوراق تصل الى ٢٫٥٨٪ .