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PHYTOCHEMICAL STUDIES ON SOME EGYPTIAN PLANTS RELATED TO FAMILIES COMPOSITAE AND RANUNCULACEAE

Submitted by

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For

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Title of [Ph.D.] Thesis:

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ABSTRACT

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This work has been carried out to investigate the "Chemical Constituents of three Egyptian Plants, Ambrosia maritima and Centaurea pallescens (Fam. compositae) and Nigella sativa (Fam. Ranunculaceae). Also the work has included synthesis of some new benzofuran derivatives from visnaginone.

Key words:

Ambrosia maritima, Centaurea pallescens, Nigella

sativa, Visnagin and Visnaginone.

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SUMMARY

SUMMARY

PHYTOCHEMICAL STUDIES ON SOME EGYPTIAN PLANTS RELATED TO FAMILIES COMPOSITAE AND RANUNCULACEAE

The work described in this thesis has been under taken with the objective of searching for naturally occurring substances of potential biological activities in Egyptian flora. Through this investigation a useful information were derived from the application of the modern technique of chromatography (column, paper, thin layer chromatography) and spectral measurements such as UV, IR, MS, ¹H-NMR and ¹³C-NMR.

The introductory part of this thesis comprises a brief account of the coumarins, their commoner structural types encountered in nature and some biological properties. Also, the introductory part comprises a brief account of the flavonoids, their commoner structures and methods of isolation and identification. The theoretical part of the thesis described the original work carried out and is summarised under the following tittle.

PART 1

CHAPTER 1

The Chemical Constituents Of The Aerial Parts Of Ambrosia Maritima L.

This work deals with the chemical constituents of the aerial parts of Ambrosia maritima (Fam. compositae) with the object of searching for some naturally occurring substances with expected biological properties which have been previously found in related compositae plants.

The aerial parts of *Ambrosia maritima* were exhaustively extracted with ethanol and the ethanolic extract was fractionated into an unsaponifiable fraction and coumarin fraction.

The unsaponifiable fraction.

By chromatographic fractionation on silica gel column, the unsaponifiable fraction was eluted with petroleum ether (40-60°C) and petroleum ether-benzene in ratio (1:1) afforded waxy hydrocarbons.

The waxy hydrocarbons were subjected to gas liquid chromatography.

A typical chromatogram of the extracted hydrocarbons revealed the presence of tetradecane, octadecane, eicosane, docosane, tricosane, tetracosane, pentacosane, hexacosane and octacosane. Pentacosane, tetracosane, tricosane and octacosane, representing the major content (46.40%, 22.4%, 12.82% and 10.25%), respectively.

Elution of the column with benzene alone gave a crystalline product, which was shown to be a triterpenoid alcohol, m.p. 197-98°C $[\alpha]_D$ +87° (chloroform), R_f 0.61, which was identified as β -amyrin (CCXV Chart 3). The identity of this compound was confirmed by direct comparison with an authentic sample of β -amyrin m.p. and mixed m.p. (undepressed).

Also, the identity of this product was confirmed by the preparation of the acetyl derivative and its comparison with an authentic sample of β -amyrin acetate, m.p. and mixed m.p. (undepressed).

Further elution of the column with benzene alone and benzene-methanol in ratios (99.5:0.5 and 99:1) afforded a mixture of sterols. This mixture was subjected to gas liquid chromatography (GLC). A typical chromatogram of the extracted sterols revealed the presence of squalene (CCXVI). Cholesterol (CCXVII), campesterol (CCXVIII) stigmasterol (CCIII) and lupulic acid (CCXIX), Cholesterol representing the major component.

The coumarin fraction:

The coumarin fraction of the aerial parts of Ambrosia maritima was also fractionated on a silica gel column. The early fraction eluted from the column with petroleum ether (40-60°C) and petroleum etherbenzene in ratio (1:1) gave a waxy hydrocarbons.

Further elution of the column with benzene alone and benzene-methanol in ratios (99.5:0.5; 99:1 and 95:5) afforded four coumarinic products, when examined on silica gel chromatoplates R_{fs} (0.43, 0.33, 0.18 and 0.09).

The first product, m.p. 112-4°C. This compound exhibited properties (physical and chemical) which were in full support of a methoxylated coumarin constitution, the identity of the compound was readily confirmed as herniarin (CCXX, Chart 5) by direct comparison and the action of mineral acid (to give umbelliferone) (II, Chart 1) and by synthesis.

The second product was obtained as colourless crystals (70 mg) m.p. 208-9°C, R_f (0.33), crystallised from methanol. It gave positive