



Ain Shams University
Faculty of Science
Chemistry Department

Studies on some plant extracts and their effective chemical constituents against some pests

Thesis

Presented for the Award of the Degree of Doctor of Philosophy
(Ph D)

By

Lobna Raies Amin Mohammed

B. Sc.(Chemistry), Faculty of Science-2000

M.Sc. Chemistry (physical chemistry)-2015

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Approval sheet

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2018



جامعة عين شمس
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قسم الكيمياء

دراسات على بعض المستخلصات النباتية و مكوناتها الكيميائية الفعالة ضد بعض الآفات

رسالة مقدمة إلى
كلية العلوم- جامعة عين شمس

للحصول على درجة دكتوراة الفلسفة في العلوم
(تخصص كيمياء فيزيائية)

من

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Dedication

To soul of My Father

Abstract

Peels of fresh navel orange (*citrus sinensis*) and lemon (*citrus limon*) fruits (Rutaceae) were extracted by absolute methanol (MeOH) and n-Hexane (Hex) as solvents of different polarities. The aim was to perform some chemical and physicochemical analysis on the peel extracts for potential use as botanicals against the cotton leafworm, *Spodoptera littoralis* (Boisd.) . The extraction solvent type was a principal factor in determining chemical composition, and consequently physicochemical properties of the citrus extracts. MeOH proved to be significantly ($P < 0.01$) more efficient than Hex to extract phytochemicals (proteins, carbohydrates, phenols, flavonoids and alkaloids) from lemon and orange. Also physicochemical characteristics, such as % yield and specific gravity of MeOH extracts were significantly higher than Hex extracts. However Hex extracts were more acidic than MeOH ones. Hex extracts were more toxic against of 4th larval instar the cotton leafworm, *S. littoralis*. It is probable that Hex more able to extract bioactive secondary metabolites than MeOH. Toxicity of citrus peel crude extracts against *S. littoralis* larvae was determined by the solvent used for extraction, citrus species and method of treatment. Generally, lemon extracted by Hex, used for repellency or contact action was the most efficient against the larvae.

Hex extracts were selected for identification of toxic bioactive secondary metabolites, since it is the first step for potential use as natural botanical ecofriendly products. Column chromatography and thin layer chromatography (TLC) as physical methods of separation were used to separate active secondary metabolites from Hex extracts of citrus peel. The extracts were fractionated and the components profile was checked by TLC. The eluted active fractions that showed toxicity against the 4th larval instar of the cotton leafworm by contact method were identified by gas chromatography combined to mass spectrometry (GC/MS). The results revealed that F₂ and F₃ fractions were the toxic active

fractions of both lemon and orange peel Hex extracts, but lemon fractions were more toxic than orange ones. The active fraction of both lemon and orange peel extracts contained d-limonene, α -pinene, linalool, citronellal and citral. Limonene oxide, geranial and terpineol were only detected in orange peel extracts. The results indicated that secondary metabolites quality and quantity differ according to the *citrus* spp., and the quantity of d-limonene is much lower in lemon ($\approx 15\%$) than orange ($\approx 86\%$) fractions in spite of lemon fractions were more toxic than the orange. However d-limonene was a major compound in both extracts. The results suggest that citrus extract contains many toxic compounds, other than limonene, and /or they could potentiate each other to give the observed toxicity.

Peel waste from native citrus species is promising as a natural biopesticide against the cotton leafworm, *S. littoralis* larvae. TLC developed showed remarkable separation and column chromatography yielded good amount of plant secondary metabolites. Further studies would be useful to identify the specific toxic constituents for each pest species, and to clarify measures of field use such as stability, methods of application and effect on non-target species.

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