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MASTER DEGREE THESIS

TWO-STEP EXTRACTION OF PYRETHRINS FROM CHRYSANTHEMUM

除虫菊花中的除虫菊酯的两步萃取研究

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Major: Chemical Engineering

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厦门大学博硕士学位论文摘要

TWO-STEP EXTRACTION OF PYRETHRINS FROM CHRYSANTHEMUM Name: IBRAHIM ABDUL - RAUF Supervisor: Li Jun Xiamen University

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I. CERTIFICATION

I, Professor LI JUN, hereby certify that I have read this manuscript and recommend for acceptance by the Xiamen University; a dissertation entitled “TWO-STEP EXTRACTION OF PYRETHRINS FROM CHRYSANTHEMUM” in fulfillment of degree of Master of Engineering at Xiamen University, People’s Republic of China.

Signed.....

Supervisor

Date: July, 2010

Department of Chemical and Biochemical Engineering

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July, 2010

II. ORIGINAL STATEMENT

The research described in this Master of Engineering thesis was conducted under the supervision of Professor LI JUN at the Department of Biochemical and Chemical Engineering, Xiamen University. I hereby declare that the work submitted is my own and that appropriate credit has been given where reference has been made to the work of others. I also confirm that it has not been previously or concurrently submitted for any other degree, diploma or any other qualifications at Xiamen University, P.R China or other institutions.

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IBRAHIM ABDUL - RAUF

July, 2010

IV. DEDICATION

I dedicate this work to my dear wife, Sawdatu; a loving helpmate and a Caring mother; and to our lovely children – Kifayatul-Llah, Tun-Taiya, Neina and Zul-Khair. It is also dedicated to all my brothers and sisters and in memory of my parents Al-Haji Yibram Bla and Mma Marta of blessed memory.

July, 2010

V. ACKNOWLEDGEMENTS

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May ALLAH, in His infinite mercy shower HIS blessings on all those who in diverse ways contributed to my success.

July, 2010

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2010年7月

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VII. ABSTRACT

The experimental determination of the concentration and yield of pyrethrins from chrysanthemum (pyrethrum flower) is usually carried out with chromatographic techniques. A lot of methods about this have been reported over the years [Wang et al, (1997)]. These include high performance liquid chromatography (HPLC) [Todd et al, (2003); Essig and Zhao, (2001b)], gas chromatography (GC) [Essig and Zhao, (2001a)] and supercritical fluid chromatography (SFC) [Wenclawiak et al, (2000)]. GC was chosen as a convenient method in this study.

The first extraction using n-hexane as a first solvent to extract the pyrethrins from the solid sample (grounded and unsieved, particles size of about 30 meshes), and then the second extraction using supercritical carbon dioxide (as a second solvent) with a self built supercritical fluid extraction (SFE) unit to obtain the pyrethrins from the crude hexane extract (CHE) were done in this study.

The hexane extractions (100g sample size) in a water bath at controlled temperatures and vigorous stirring yielded pyrethrins concentrations varying from 69.85 - 95.50mg/ml and yield of 0.85 - 3.76% of the dry weight. Extraction efficiencies under several conditions were investigated and the optimum extraction conditions were about 40⁰C in 4hrs. Compared with the pyrethrins product from the factory, several undesirable components exist in the CHE.

The SFE was carried out in an extraction vessel (125ml) with a sample size of 40ml CHE. The SFE extract yielded concentrations varying from 57.25 - 93.79mg/ml and the yield range (after the second extraction) of 0.99 - 2.15%. The optimum conditions here were 35⁰C at a pressure of 20MPa in 2hrs. Compared with the pyrethrins product from the factory, two undesirable components are evidently eliminated in the extract from SFE. The results show that the two-step extraction technique is appropriate for the effective extraction of pyrethrins.

Key words: Hexane, supercritical carbon dioxide, pyrethrins, two-step extraction

VIII. 摘要

本文采用色谱法对除虫菊中的除虫菊酯萃取物进行定量分析。近些年除虫菊酯的定量分析大多使用色谱法，如高效液相色谱法、气相色谱法、超临界流体色谱法。除虫菊酯是指菊科植物除虫菊花中所含有效杀虫成分的总称，它主要由 6 个结构极相似的化合物组成，但由于结构相似，要同时制备 6 种化合物的标准品非常困难，通常只需测定这 6 个组分含量总和，故本论文选择较方便的气相色谱法。

本研究采用两步萃取研究从除虫菊花中获取除虫菊酯。首先使用第一溶剂正己烷对除虫菊进行溶剂提取，然后采用自制的超临界流体萃取装置用第二溶剂超临界二氧化碳对正己烷粗提物进一步纯化。

于一定水浴控温和强烈搅拌下，研究了正己烷萃取。获得的萃取物中除虫菊酯的浓度为 69.85 - 95.50mg/ml，其得率为 0.85%-3.76%。优化后的提取条件约为 40°C、4 小时。相对于工厂样品，一次萃取的萃取物中明显含有若干杂质成份。

用超临界流体萃取装置（125ml）对约 40ml 的一次萃取物进行二次萃取（提纯）研究，超临界二氧化碳萃取得到的提取物中除虫菊酯的浓度为 57.25 - 93.79mg/ml，二次萃取后得率为 0.99 - 2.15%。优化后的提取条件约为 35°C、20MPa、2 小时。相对于样品，二次萃取的萃取物中有两种杂质成份明显消失。因此，研究表明两步萃取法效果较好。

关键词：正己烷，超临界二氧化碳，除虫菊酯，两步萃取

IX. ABBREVIATIONS AND SYMBOLS

GC	gas chromatography
HPLC	high performance liquid chromatography
SFC	supercritical fluid chromatography
SCF	supercritical fluid
SCFs	supercritical fluids
SFE	supercritical fluid extraction
SC-CO ₂	supercritical carbon dioxide
SC	supercritical
CO ₂	carbon dioxide
et al	et alii (and others)
pp	page
%	percentage
CHE	crude hexane extracts
BC	before christ
PY	pyrethrin
PYI	pyrethrin 1
PYII	pyrethrin 2
C1	cinerin 1
C2	cinerin 2
J1	jasmolin 1

J2	jasmolin 2
P1	pyrethrin 1
P2	pyrethrin 2
A1	area of pyrethrins 1
A2	area of pyrethrins 2
WHO	world health organization
Fig	figure
P _c	critical pressure
T _c	critical temperature
C _p	critical point
Cm	centimeters
~	approximately
°C	degree centigrade
MPa	mega pascal
FID	flame ionization detector
n-hexane	normal hexane
mL	milliliters
/min	per minute
Soc.	society
Eds	editions
Sci.	science

m	meters
mm	millimeters
t_R	retention time
k'	retention factor
R^2	pearson correlation coefficient
LOQ	limit of quantification
LOL	limit of linearity
LOD	limit of detection
BOD	beyond limit of detection
IS	internal standard
k	response factor
f	relative response factor
μm	micro meters
μL	micro liters
rpm	revolutions per minute
cp	component
T _{tl}	total
St	standard
No.	number
Ext	extract

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