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硕 士 学 位 论 文

苦楝果实中苦楝素的超临界二氧化碳萃取研究

Study on Extraction of Toosendanin from The Fruit of *Melia Toosendanin Linn* Using Supercritical Carbon Dioxide

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摘要

苦楝（*Melia azedarach Linn*）为我国一个乡土树种，广泛分布于我国南部地区。苦楝中含有多种杀虫活性成分，其中苦楝素（toosendanin）是最主要的活性成分, 它对多种害虫有毒杀作用, 主要表现为使虫忌避、拒食；抑制昆虫生长发育、触杀、内吸致毒作用等。苦楝素用以制备植物源农药，与化学农药相比, 具有安全、残效期短、高效、无毒、无污染、杀灭多种农业害虫等优越性，可以阻止害虫直接为害或抑制种群形成而达到对害虫的可持续控制，因而具有广泛的应用前景。

超临界 CO_2 萃取技术是一种新型高效洁净分离技术，与传统的溶剂萃取方法比较, 具有无溶剂残留、不破坏热敏性和易水解的物质等优点, 在天然产物的提取领域中有良好的应用前景。本文以苦楝果实为原料，对采用超临界二氧化碳萃取苦楝素工艺的可行性及其适宜条件进行了系统的研究。

采用有机溶剂提取法提取苦楝果实中的天然杀虫活性成分，考察了固液比、提取时间、提取次数对提取率的影响，同时成功地分离出萃取物中苦楝素，并将其纯化。采用差示扫描量热仪（DSC）测定苦楝素晶体基本的理化常数熔点。并通过红外吸收光谱（IR）、紫外吸收光谱（UV）、有机质谱（ESI–MS）对苦楝素晶体进行波谱分析，其分析结果和文献报道的苦楝素数据基本一致。

研究了以苦楝果实为原料的超临界二氧化碳萃取工艺。实验中采用自行设计的超临界萃取设备，重点考察了萃取温度、萃取压力对萃取率的影响，同时也考察了萃取时间、原料颗粒粒度、不同夹带剂及夹带剂用量对萃取率的影响。确定了适宜的萃取工艺条件。研究结果表明：萃取温度为 40 ℃，萃取压力为 20 MPa 时，选择 20 %（mol / mol）乙醇为夹带剂，苦楝素的萃取得率为 98 %。

在超临界CO_2萃取苦楝素过程中，利用收缩核模型描述溶质的浸出, 溶质首先从缩核边界层脱附分离出来, 再从固体颗粒的微孔扩散到颗粒表面, 最后跨过液膜, 进入流体主体。该模型结合固相和流体相的质量衡算，对整个萃取过程进行了模拟，采用差分法, 求得了模型的数值解，结果表明模型计算值与实验值基本上吻合。

关键词：苦楝；苦楝素；超临界二氧化碳萃取；收缩核模型

ABSTRACT

Melia azadarach Linn belonging to Meliaceae exists broadly in our country. Toosendanin, the main effective ingredient of the fruit of *Melia azadarach Linn,* is a kind of tetracyclic triterpene, which can be made into pesticide. Toosendanin could make pieris rapac antifeed and have stomachtoxic activities, and show high feeding inhibitory effects to Toxoptera aurantii. Toosendanin can be put into produce botanical insecticides. This botanical insecticide is a safe and effective and free of toxic residuals and adaptable to controlling pests over crops

With occurring the upsurge of savageness, nutrition and returning natural all over the world, supercritical fluid extraction technology holds prominent status in the domain of extraction of natural products as a kind of high effective and clean separation technology. This thesis mainly explores the feasibility and the optimal conditions of extracting toosendanin from the fruits of *Melia azadarach Linn* with supercritical carbon dioxide.

The process of extracting toosendanin from the fruits of *Melia azadarach Linn* using ethanol extraction and crystallization method was firstly studied. And effects of extraction time, erxtraction times and the ratio of liquid to solid on the yield of toosendanin were investigated. Two melting points of the crystal obtained were determined by using differential scanning calorimetric (DSC). Its structure was elucidated by spectroscopic means (ultra violet spectroscopy, electrospray ioniaztion mass spectroscopy and infrared spectroscopy). All the results of qualitative analysis agreed roughly well with the results in the related references.

The process of extracting toosendanin was further studied from the fruits of *Melia azadarach Linn* with supercritical CO_2 using the supercritical fluid extraction apparatus. which was made by ourselves. And effects of extraction temperature and pressure on the extraction yield were investigated. Effects of extraction time, the size of material and the concentration and dosage of modifiers on the extraction yield of toosendanin were studied. The optimal extraction conditions were obtained experimentally. It is found that the yield of toosendanin is up to 98% when using 20 % ethonal modifier at pressure 20 Mpa and temperature 40 ℃.

Moreover, the shrinking model was used to simulate the mass transfer process of toosendaninl in supercritical fluid. A simplex method was used to fit and optimize the model parameters. The numerical solution of yields vs. extraction time was obtained by differential method. The simulating results show that the model fits the experimental data well.

Key words: *Melia azadarach Linn*; Toosendanin; Supercritical carbon dioxide extraction; Shrinking core model

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