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Research Article

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Supercritical CO_2 extraction of Codonopsis

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ABSTRACT

In the present study, Radix Codonopsis was extracted by supercritical CO_2 extraction. As Ointment collecting rate and the yield of stigmasterol are index, Various reaction parameters were changed to attain the optimal conditions. The appropriate process conditions are size 40 - 60Mesh, 30 Mpa pressure, temperature 65 ℃, 120 min, entrainer Alcohol dynamic extraction (entrainer pump flow rate of 1 ml/min, the CO_2 flow rate 2L /min.

Key words: Supercritical CO_2 extraction; Radix Codonopsis; Chinese medichine

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INTRODUCTION

Codonopsis is Campanulaceae plants, which is biennial root dry. It can aid digestion, clear hot detoxify, strong lung, nourishing body [1]. At present, the extraction method of effective components of Radix Codonopsis is mainly traditional organic solvent extraction that has high extraction efficiency [2]. However, none can afford to neglect that organic solvent extraction easy to destroying the Codonopsis effective components and bring the existence of residual solvent, solvent consumption that result in environmental pollution.

As a new green method, supercritical CO_2 extraction assures the extraction and separation to accomplish at once and can retain active ingredient retains intact and no solvent residue [3-4]. Therefore, supercritical CO_2 extraction has been the widely used in the fields of food and pharmaceutical [5].

In the present study, the effective components of Radix Codonopsis were extracted by supercritical CO_2 extraction. Ointment collecting rate and the yield of stigmasterol as the study objective were investigated by using the single factor experiment in order to optimize extraction process.

EXPERIMENTAL SECTION

2.1 Materials and equipment.

The chromatographically pure Dangshen were purchased from Milli states yinpian co., LTD. Speed SFE supercritical fluid extraction equiments (Applied ─ Seperation Company), LML-1 wet type gas flow meter (Changchun instrument manufacturer) , J03 9504 air compressors (Shanghai yue jin generator manufacturer), FE80 omnipotent disintegratorn (qinjiaw manufacturer), 6890-5973NGas Chromatograph-Mass Spectrometer (Agilen Technologies).

2.2 Stigmasterol spectrophotometric analytical method

Precision weighing stigmasterol 10mg, dissolved and diluted with chloroform to scale in the 25ml volumetric flask. From the full spectrum scan, it is found that the maximum absorption wavelength at 542nm, which will be as the detection wavelength.

Take reference substance solution 1 ml and diluted with chloroform to scale in the 50ml volumetric flask.1, 2, 3, 4, and 5 ml were taken in five test tubes and boiled away in 100 ℃ water bath. After that, added 0. 4 ml vanillin solution and 1 ml perchloric acid solution into the test tubes respectively, the test tubes were placed into the water Bath at 60 ℃ and color 15 min, finally add 5 ml glacial acetic acid to dilute. After shaking, the absorbance can be determined directly under 542 nm absorbance. From the data, the standard curve can draw that the the bean steroid Mg as dependent variable while the absorbance as independent variable.

Codonopsis pilosula plates were extracted by supercritical CO_2 extraction, and extraction liquid were weighing 100mg, and take over 2.2.2 to prepare solutions that measured the absorbance. The data substituted in the standard curve, multiplied by the dilution times, the content of each sample of stigmasterol can be obtained.

RESULTS AND DISCUSSION

As Ointment collecting rate and the yield of stigmasterol are index, the effects of particle size, pressure, temperature, time and solvent were investigated in the supercritical CO2 extraction process.

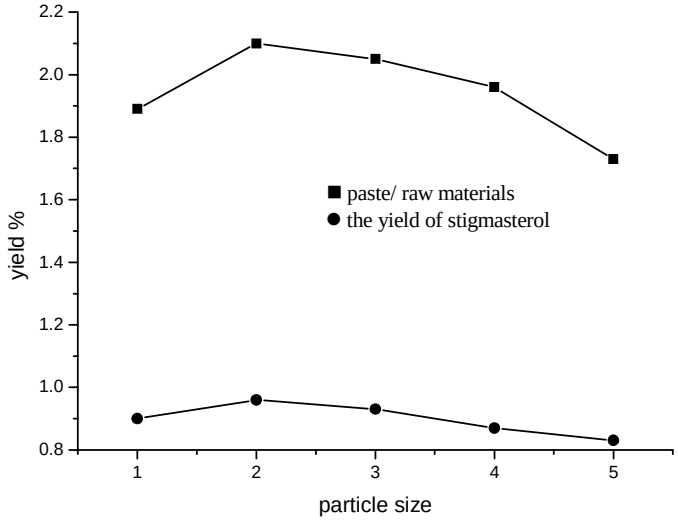

Fig. 1 The impact of particle size on the extraction yield

The crushed materials were divided into 5 groups: 20-40 mesh（１）; 40 -60 mesh（２）; 60- 80 mesh（３）; 80 - 100 mesh（４）, over 100 mesh（５）. The influence of raw material particle size on the ointment collecting rate and stigmasterol yield were investigated under the conditions of the extraction temperature is 65℃, the pressure for the 30MPa. It is found that ointment collecting rate and stigmasterol yield were highest when the crushed materials are 40 -60 mesh as shown in Fig.1.

The reason for the above result is that internal diffusion is often key step in the practical application of supercritical extraction. With the raw material particles become small, supercritical fluid flows more easily, penetrate into the particle interior, mass transfer resistance decreases, the extraction efficiency increases[7]. While as the particles become smaller, the raw material is compacted more closely which is not convenient for loading and unloading of raw materials. The other reason for the choice of the 40 -60 mesh size in the later experiment is that the 40 -60 mesh accounted for 56% of the total under the 30MPa crush strength.

The effect of temperature on the ointment collecting rate and stigmasterol yield in Fig. 2 with conditions: CO_2 flow rate of 2L/min, extraction pressure of 30 mpa, the partical size of 40 ~ 60 mesh. With the temperature rise, extraction efficiency is also increased that due to elevated temperature increased flow diffusion rate which is beneficial to the mass transfer. Nevertheless, high extraction temperature, the ability of CO_2 binding with the material and carrying capacity decreased, which makes the material qualitative dissolution is reduced, so the experiment selected extraction temperature is 65 ℃.

The effect of pressure on the ointment collecting rate and stigmasterol yield in Fig. 3 with conditions: CO_2 flow rate of 2L/min, temperature is 65℃, the partical size of 40 - 60 mesh. When the pressure is at 15MPa, the ointment collecting

rate and stigmasterol yield is low; when above 30MPa, with the increase of pressure, the yield increased slightly. The reason is described as followed: when the pressure increases, the carbon dioxide density and the ability of dissolve also increased, therefore the extraction rate increases with pressure. Considering the cost, the selection pressure is 30MPa.

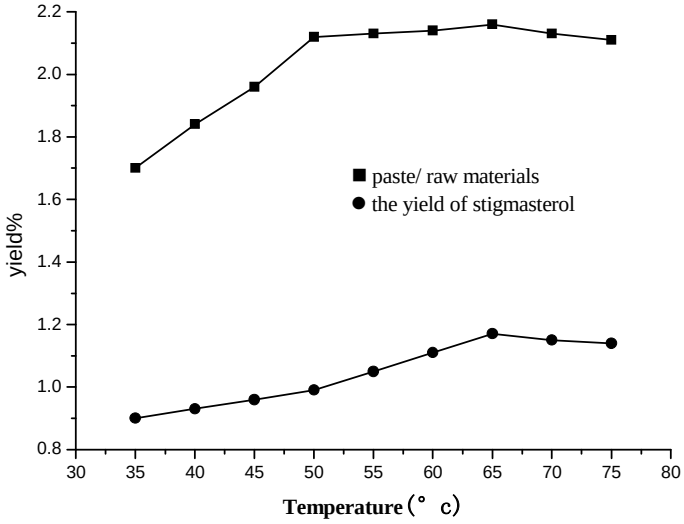

Fig. 2 The impact of temperature on the extraction yield

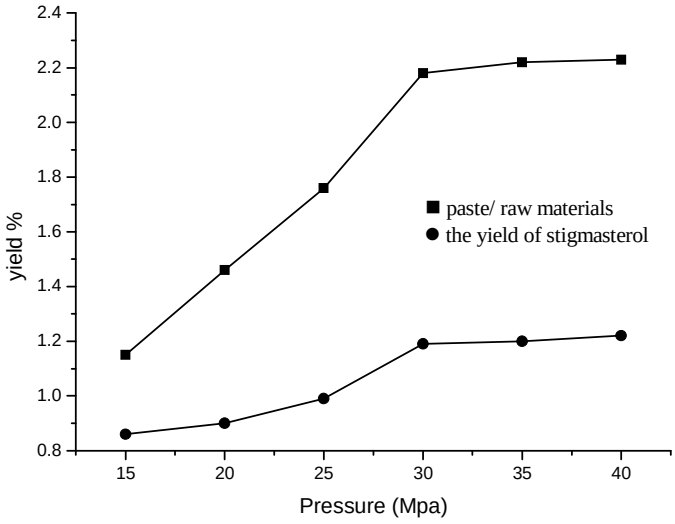

Fig. 3 The impact of pressure on the extraction yield

The effect of extraction time on the reaction was investigated with other conditions unchanged. From the Fig.4, it can be obtained that the before 30 min, the extraction efficiency is rapid increased with time, from 30 to 100min, the extraction efficiency slow growth, the extraction efficiency remained stable after 100min. The extraction time is setting 80min that take capital appreciation of the extraction efficiency and cost into account.

The effect of cosolvent on the extraction efficiency was investigated with other conditions unchanged. Entrainer enhancing technology can divide into static extraction and dynamic extraction. In the process of static extraction, the cosolvent is ethanol(1), ethyl acetate(2) and 80% ethanol(3) respectively. In the process of dynamic extraction, the entrainer is ethanol. One condition of dynamic extraction is ethanol flow rate:0.15ml/min, CO_2 flow rate：2L/min(4); the other condition of dynamic extraction is ethanol flow rate:1 ml/min, CO_2 flow rate： 2L/min(5). The result is as shown in Fig . 5.

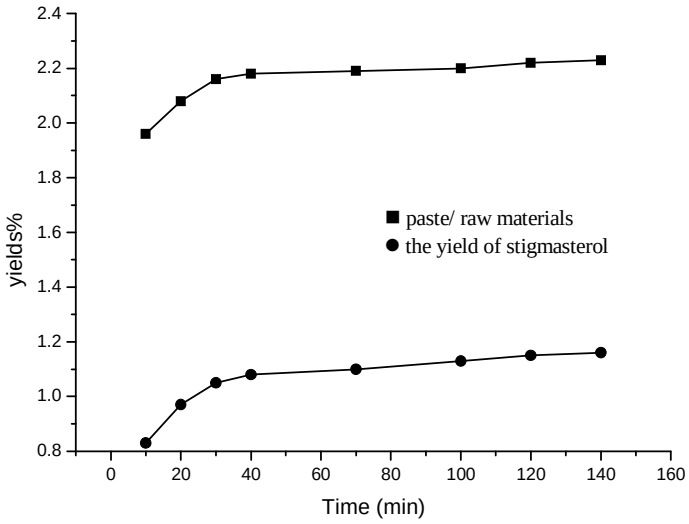

Fig. 4 The impact of time on the extraction yield

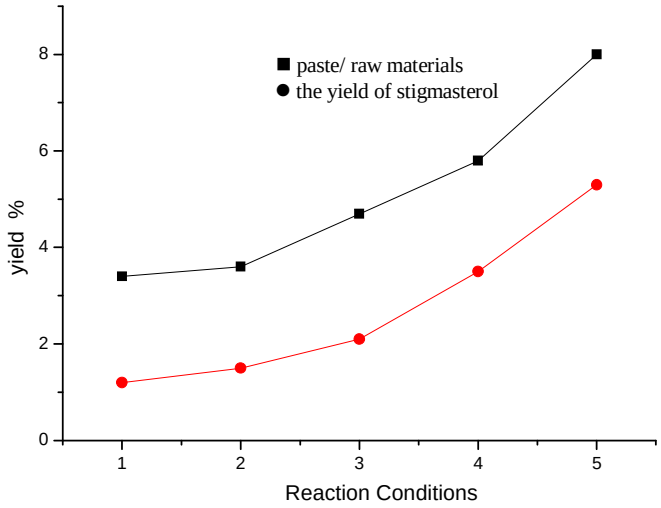

Fig.5 The impact of cosolvent and extraction fashion on the extraction yield

From the Fig. 5, when the cosolvent is ethanol, the extraction efficiency is highest in the process of static extraction. While in the process of dynamic extraction, the extraction efficiency is in proportion to ethanol flow rate. In the extraction process, material and supercritical fluid required to maintain a certain apparent contact time, static extraction can improve the supercritical fluid and cytoplast contact that enhance mutual dissolution efficiency. Dynamic state extraction time duration show great effects on extraction rate at the fixed static extraction time.Dynamic Extraction can compensate fresh CO_2 constantly and separate the component dissolved in fluid with air circulation timely, which cause the concentration of effective components in the fluid extractor always low in favor of extraction. Meanwhile, dynamic extraction can make CO_2 recycle.

CONCLUSION

Traditional Chinese medicine (TCM) has complex components, which efficient and inefficient components coexist. Hence effective constituents and enrichment of effective parts will be key for drug development. In order to improve the therapeutic effect of medicine, reduce toxic side effects, and improve the Chinese native medicine preparation, selection of reasonable the extraction technology is very important. Through the single factor experiment, granularity, extraction pressure, extraction temperature, extraction time, entrainer for super critical carbon dioxide extraction of effective components on Codonopsis pilosula extraction yield of shadow Ring were investigated. It is

concluded that the factors of appropriate process conditions are size 40 - 60Mesh, 30 mpa pressure, temperature 65 ℃, 120 min, entrainer Alcohol dynamic extraction (entrainer pump flow rate of 1 ml/min, the CO_2 flow rate 2L /min).

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