APPLICATION OF HYBRID MICROWAVE THERMAL EXTRACTION TECHNIQUES FOR MULBERRY ROOT BARK

by

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Original scientific paper
DOI: 10.2298/TSCI1305311W

The main focus of this paper is the extraction of compounds from the mulberry root bark using a hybrid microwave thermal process. The shearing mechanism and an integrated circulation system, which increases the rate of contact between the solvent and extractive, are studied. The results are analyzed by the Taguchi method and verified by high performance liquid chromatography. Furthermore, the optimal operating parameters of the extraction of mulberry root bark are discussed. The results show that hybrid microwave thermal extraction can successfully extract the active ingredients from mulberry root bark. This is a reliable basis for further researches.

Key words: *microwave thermal extraction, Taguchi method, mulberry root bark*

Introduction

Microwave thermal extraction (MTE) is a new extraction process method [1]. It uses microwave energy to heat a solvent which is in contact with the solution from which the desired extract is held.

Microwave energy is an electromagnetic radiation with wavelengths between 1 mm to 1 m and frequencies in the range of 300 MHz to 300 GHz. This range is most used as the frequency for communications, in particular the radar, cell phones, television, and satellite applications, therefore the Federal Communications Commission agreement states that two frequencies of 0.915 and 2.45 GHz are specifically used for microwave heating in order to avoid interference with communications [2].

The application of microwave is heating sample in the early, microwaves can be used to heat water molecules. When microwaves stimulate molecules they promote molecular rotation and create kinetic energy which in turn releases heat or other forms of energy. As different materials have different dielectric constants, dielectric loss factors, heat and water content, and different reflection and absorption characteristics in terms of subjection to microwaves they can be selectively manipulated through microwave heating. These differential characteristics allow selective heating of the target material while allowing other components to be used as coolant during material processing. In microwave dielectric materials, the ability of ion polarization determines the size of the dielectric constant. When the material in a externally applied electric field, internal free charges produce the polarization phenomenon from electric field, and then affects the dielectric constant, quality, factor and frequency temperature coefficient. Materials response to the electric field are defined as dielectric properties [3].

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The way in which a solution reacts to microwaves can be divided into dipole rotation and ionic conductance. The main characteristics of microwave heating include fast heating, uniform energy distribution, and higher energy efficiency [4].

Mulberry root bark extracts (*ex:* resveratrol and myricetin) have been developed into drugs that treat diabetes, edema, and high blood pressure [5]. In this paper, hybrid microwave thermal extraction techniques are analyzed to extract the ingredients from mulberry root bark and also obtained the optimum operating parameters to increase the quantity of target extracts.

Experimental method

This experiment uses microwave energy to speed up the rotation and vibration of the polar molecules (water-soluble components) of a base material. This process further accelerates the solvent's ability to quickly dissolve in the solution. Together these properties increase the extraction rate. The process also involves an accurate temperature sensing control of the circulatory system in order to avoid destruction of active ingredients. The process is further enhanced by a mechanical shearing module which uses shear force to fracture the cell walls of plant fibers. It can speed up the dissolution of the active ingredients within the solution and increase the efficiency of the mixing of non-polar and polar components during the extraction process.

Description of experiment

This experiment applies Taguchi method [6] for the design of experiment and uses orthogonal arrays of L9(3^4). Also, the operating parameters are chosen and shown in tab. 1.

Table 1. Operating parameters of hybrid microwave thermal extraction of mulberry root bark

Symbol of factor	Extraction parameters	Level 1	Level 2	Level 3
A	Microwave power	193 W	139 W	46 W
B	Homogenizing speed	0 rpm	4000 rpm	5000 rpm
C	Ethyl alcohol concentration	40%	60%	80%
D	Extraction period time	5 minutes	10 minutes	15 minutes

Experimental material and equipment

The materials used in the experiment are 95% ethyl alcohol with edible level and mulberry root bark from Hunan, China. And then, the corresponding experimental equipment includes grinding, sieving, homogenizing and microwave thermal processes. After extraction procedures, the concentrations of different active ingredients are used as the base for the comparative analysis of high performance liquid chromatography (HPLC).

Structure of extraction system

This experiment used an extraction system of three main modules, including microwave module, shear module, and temperature sensing control module. The functions are:

– *Microwave module.* Includes a magnetron capable of producing microwaves of 2450 MHz at a maximum of 800 W. Through this process electrical energy produces microwaves in the magnetron which are then focus on the metal wall of the furnace. These microwaves are then reflected within the metal chamber and evenly heat the extract and solvents to the required temperature. Using glass vessels, that do not restrict microwave penetration, allows polar molecular rotation, vibration and molecular inter collision to further speed up the extraction effect.

- *Shear module.* It is comprised of an internal moving rotor, and a fixed external stator. The difference in speed between the fixed stator and spinning rotor create a great shear. In addition to making the solution more uniform it also generate tremendous effect on the flow. This further increases the extraction speed. An additional benefit is that the rotor generated flow field makes the microwave generated heat more evenly distributed throughout the solution. A uniform temperature throughout the mixture means a more accurate temperature control.
- *Temperature sensing control module.* This module consists of a back flow tube, a circulation pump and a temperature sensor. The temperature sensors on the tube transmit a signal to the computer control module. This information is then used to determine the requisite circulation pump flow speed. Signals to either increase or decrease the flow, so that the extract and solution maintain the required temperature, are then transmitted by the computer control module.

Process of experiment

First, a grinder is employed to grind the mulberry root bark into fine grains and filter them through a 40 mesh filter. Then, a solution of mulberry powder and ethyl alcohol is placed into the extract vessels and subjected to the microwave thermal extraction method. After being processed under the specific extraction condition, the extraction liquid is further filtered through a 0.45 μm filter paper.

The ingredients of resveratrol and myricetin are used as the base for the comparative analysis of HPLC. The integrity of the process and analysis is verified by using a known resveratrol and myricetin concentration from a standardized mulberry root bark sample. If these results are not correct, the experiment will be modified with adjustment to the operating conditions.

Finally, these results are shown and indicated the optimum values for extraction parameters including microwave power, shear rotational speed and integrated circulation system that will produce the greatest concentrations of the desired mulberry root bark compounds. The experimental process is shown in fig. 1.

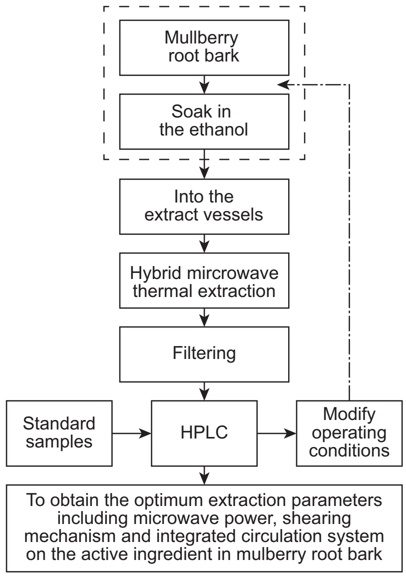

Figure 1. Process of experiment

This experiment uses orthogonal arrays of L9(3^4) and the results of the ingredients for resveratrol and myricetin are investigated with the comparative analysis by HPLC. The total flavonoids concentration is used as the base for the comparative analysis by spectrophotometer.

Results and discussion

The optimization analysis of resveratrol

Table 2 shows the effects of factors for resveratrol. The values of A1, B1, C1, and D1 are 4.19, 3.55, 5.16, and 3.64. The values of A2, B2, C2, and D2 are 4.22, 4.3, 4.06, and

3.98. The values of A3, B3, C3, and D3 are 3.66, 4.22, 2.86, and 4.45, the factor effects of A is 0.6, B is 0.8, C is 2.3, and D is 0.8; the best combination of parameters is A2B2C1D3.

The value of SS of total variations is 33.2, the DOF of total variations is 17, contribution of errors is 36.24%, so the influence of factor C is larger for experiment. The results are shown in tab. 3.

Table 2. Effects of factors for resveratrol

	A	B	C	D
Level 1	4.19	3.55	5.16	3.64
Level 2	4.22	4.30	4.06	3.98
Level 3	3.66	4.22	2.86	4.45
Effect	0.6	0.8	2.3	0.8

Table 3. ANOVA for resveratrol

Factor	SS	DOF	Contribution
A	1.188563	2	3.58%
B	2.026282	2	6.11%
C	15.93687	2	48.05%
D	1.996431	2	6.02%
Error	12.0202	9	36.24%
Total	33.16834	17	100.00%

Table 4. Effects of factors for myricetin

	A	B	C	D
Level 1	11.34	7.04	10.66	8.06
Level 2	8.12	12.22	11.29	7.18
Level 3	10.22	10.42	7.73	14.44
Effect	3.2	5.2	3.6	7.3

Table 5. ANOVA for myricetin

Factor	SS	DOF	Contribution
A	31.99356	2	6.57%
B	82.93239	2	17.02%
C	43.47381	2	8.92%
D	188.2781	2	38.64%
Error	140.5924	9	28.85%
Total	487.2703	17	100.00%

The optimization analysis of myricetin

Table 4 shows the effects of factors for myricetin. The values of A1, B1, C1, and D1 4 are 11.34, 7.04, 10.66, and 8.06. The values of A2, B2, C2, and D2 are 8.12, 12.22, 11.29, and 7.18. The values of A3, B3, C3, and D3 are 10.22, 10.42, 7.73, and 14.44, the factor effects of A is 3.2, B is 5.2, C is 3.6, and D is 7.3; the optimal parameters are A1B2C2D3.

The value of SS of total variations is 487.2, the value of DOF of total variations is 17, the contribution of errors is 28.85%, so the influence of D factor is larger for experiment. The results are shown in tab. 5.

Table 6. Effects of factor for total flavonoids

	A	B	C	D
Level 1	48.33	33.62	26.28	39.58
Level 2	44.29	45.15	53.66	36.49
Level 3	33.54	47.38	46.21	50.09
Effect	14.8	13.8	27.4	13.6

Table 7. ANOVA for total flavonoids

Factor	SS	DOF	Contribution
A	1051.52	2	13.11%
B	981.44	2	12.23%
C	3605.54	2	44.95%
D	609.63	2	7.60%
Error	1773.89	18	22.11%
Total	8022.01	26	100.00%

The optimization analysis of total flavonoids

Table 6 shows the effects of factor for total flavonoids. The values of A1, B1, C1, and D1 are 48.33, 33.62, 26.28, and 39.58. The values of A2, B2, C2, and D2 are 44.29, 45.15, 53.66, and

36.49. The values of A3, B3, C3, and D3 are 33.54, 47.38, 46.21, and 50.09, the factor effects of A is 14.8, B is 13.8, C is 27.4, and D is 13.6; the optimal parameters are A1B3C2D3. In tab. 7, the value of SS of total variations is 8022.01, the value of DOF of total variations is 26, the contribution of errors is 22.11%, so the influence of C factor is larger for experiment.

Conclusions

This study considered the effects of 4 variables on the extraction efficiency of resveratrol, myricetin, and flavonoids compounds from mulberry root bark using a hybrid microwave thermal extraction method. The optimal parameters for extracting different ingredients are obtained as follows:

- The best combination of parameters of the resveratrol is A2B2C1D3 from tab. 2.
- The best combination of parameters of the nyricetin is A1B2C2D3 from tab. 4.
- The best combination of parameters of the total flavonoids is A1B3C2D3 from tab. 6.

As a result, microwave thermal extraction of the active ingredients in mulberry root bark extracts showed that the potential application is indeed worthy of follow-up and more advanced research.

Acknowledgment

The financial support of this research was sponsored by National Science Council of the R.O.C., under Grant No. NSC 101-2622-E-269-010-CC3 and is greatly appreciated.

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Paper submitted: January 1, 2013
Paper revised: April 28, 2013
Paper accepted: May 1, 2013