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Optimization Methodology for Supercritical Co_2 Extraction of Resveratlor From Mulberry Leaves

Berdieva Zulfiya Mukhiddinovna
Senior Lecturer,
Bukhara Engineering and Technology Institute,
Republic of Uzbekistan, Bukhara
Mukhamadiev Bakhodir Temurovich
Docent,
Bukhara Engineering and Technology Institute,
Republic of Uzbekistan, Bukhara

Abstract: Mulberry berries (white, red and black) contain a large number of substances useful for the human body: simple sugars (glucose and fructose), organic acids, essential oils, vitamins (water- and diro-soluble). Carotenes. A whole range of micro and macro elements. The content in the fruits of the strongest near-bottom antioxidants - carotenes, vitamins C.E. resveratrol and selenium - relieves the aging body of many diseases and has a rejuvenating property

Keywords: Mulberry berries, SC-CO_2

Mulberry berries (white, red and black) contain a large number of substances useful for the human body: simple sugars (glucose and fructose), organic acids, essential oils, vitamins (water- and diro-soluble). Carotenes. A whole range of micro and macro elements. The content in the fruits of the strongest near-bottom antioxidants - carotenes, vitamins C.E. resveratrol and selenium - relieves the aging body of many diseases and has a rejuvenating property.

The purpose of this study was to analyze the chemical composition of mulberry leaves, as well as the isolation of individual ingredients, in particular resveratrol (RT), using extraction using supercritical carbon dioxide (CO_2).

The advantages of the SC-CO_2 extraction method, compared to traditional methods, are already known (4). The SC-extract does not contain a solvent, the extraction process can be carried out very quickly and, finally, there is no need for further purification of the SC-extract from the extractant.

The extraction was carried out in ASTELL (CK) autoclave bottles, the extracts were collected in vessels to determine their total content. Samples were stored in a refrigerator (at -5°C) for RT analysis by high resolution liquid chromatography (HPTLC, ELGAIng.-2000 Techm., UK).

The influence of the duration of the SC - CO_2 extraction was studied at 32°C and 16 MPa. Samples of the extracts were collected after different extraction times, which corresponded to the specific flow rate of the SC fluid. The extract yield was compared and used for subsequent analysis of the effect of operating conditions.

RSV and CCRD were used to study the influence of pressure, temperature and duration of extraction (SA consumption - CO_2), as well as their interactions. A similar procedure was used earlier

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[1,3,8,11]. However, instead of comparing the yield of diosgenin (DG) SA - CO_2 by extraction determined by the use of "overlapping" parameters (the yield of DG obtained by SA - CO_2 was compared with the yield according to the method [2,6,9,13]), as a dependent value [1] , yield of total extract per gram of prepared isolate, and DH content per 100 g of total isolate or per 100 g of dry plant were used in this work. The studied parameters were pressure (denoted as x_1, MPa, temperature (x_2, °C) and the amount of CO_2 consumed (x_3, mCO_2 /mHA)). The real and coded variables used in the design of the experiment were determined on the basis of preliminary experiments carried out for different densities of SA - CO_2. The central design of the experiment presents 3 variables and all 20 experiments, including 9 factorial, 5 vertices and 6 central points for RSM and CCRD analysis and used as shown in Table 1. A 2nd order polynomial equation was considered to predict the RT output as a function of independent (I = 3) pressure (x_1), temperature (x2) and CO_2 amount (x_3) as coded quantities.

$$\gamma = \sum \beta_o + \sum \beta_1 x_1 + \sum \beta_1 j x_1^2 + \sum \sum \beta_{1j} x_1 x_j \qquad (1)$$

Table 1.
The composition of mulberry leaves per 100 g of fruit

Organic acids 1.2 g	Organic acids 1.2 g
Mono- and disaccharides 8-12 g	Mono- and disaccharides 8-12 g
Vitamin B1 (thiamine) 0.04 mg	Vitamin B1 (thiamine) 0.04 mg
Vitamin B2 (riboflavin) 0.01 mg	Vitamin B2 (riboflavin) 0.01 mg
Vitamin PP (niacin) 0.80 mg	Vitamin PP (niacin) 0.80 mg
Vitamin C (ascorbic acid) 30 mg	Vitamin C (ascorbic acid) 30 mg
Vitamin B4 (choline) 12 mg	Vitamin B4 (choline) 12 mg
Vitamin A (retinol) 6 mcg	Vitamin A (retinol) 6 mcg
Carotene (provitamin of vitamin A) 0.02 mcg	Carotene (provitamin of vitamin A) 0.02 mcg
K1 (phylloquinone) 8 mcg	K1 (phylloquinone) 8 mcg
Lutein 130 mcg	Lutein 130 mcg
Anthocyanins 20 mcg	Anthocyanins 20 mcg
Resveratrol 40 mcg	Resveratrol 40 mcg

Matlab 2014 version was used to apply RSM and analyze experimental data with 3D surface dependence and contour map of independent variables and their interactions [5,7,10,12].

Results and its discussion

In table.1. Data on the composition of mulberry leaves according to the USDA Nutrient Database are presented.

A comparative study of amino acid extracts of white, black and red mulberry leaves showed that their total content in black mulberry is 13.10%, white 10.60% and red mulberry 9.4%.

The first part of the research is devoted to the study of the effect of different densities of SA - CO_2 on the total yield of the extract. Based on the preliminary data obtained, as well as tests at various pressures, temperatures and flow rates of SC - CO_2, an attempt was made to establish the

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optimal operating conditions for the isolation of RT by SC - CO_2 extraction. Studies concerning the influence of pressure, temperature and density of SC - CO_2 on the output of RT, as well as the interaction of these parameters during the process, have not been carried out so far. It turns out that the selectivity of SC - CO_2 extraction can be achieved using different pressures, temperatures and flow rates of SC - CO_2 (3).

Resveratrol belongs to polyphenols, solubility in water is 0.003 g/100 ml. The content in the material is 0.2-5.8 mg/l.

Chemical structure of resveratrol

The rotatable scheme was applied for the above independent variables (pressure, temperature, CO_2 flow), which was carried out 20 times. Influence of SC-CO_2 density and time.

It turned out that the total yield of the extract could8 be increased by almost 2 times if the time of SC-CO_2 extraction was increased from 80 min (CO_2 consumption = 12 g_(〖CO_2)/g_cm to 150 min (CO_2) It is interesting that if a further increase in the extraction time continues and ends after 180 min, then only an additional (10%) increase in the extract yield is achieved. This fact was the main reason for using 150 min. 〖CO_2)/g_cm) at which the yield of the total extract was analyzed for various densities of 690-780 kg/m^3).

For the used density of SC-CO_2 above 780 kg/m^3, the extract yield was more than 12%, and about 780 kg/m^3 the maximum yield (15%) was reached. While the relationship between total extract yield and SC-CO_2 density indicates that some maximum yield could be at around 780 kg/m^3, it is more realistic to assume that the yield could be higher for densities above 780 kg/m3 and, that as the density increases above 780 kg/m^3, the yield of the initial total extract should be between 12 and 15% 9 (gewp/100 gcm).

The linear and quadratic effects of the variables, as well as their interactions and coefficients, on the value of the variables obtained from ANOVA were calculated and a probable equation was derived that represents the empirical interaction between the RT output and the independent variables.

Since the value of R2 (0.9088) is greater than 0.8, this indicates that the model confirms the results well.

Both linear and quadratic terms for SC-CO_2 flow rate had higher reliability ($p<0.05$), as did linear and quadratic pressure terms. Interactions between pressure, temperature and CO_2 were not significant. The analysis carried out by RSM proved that the initial results and expectations, and that the pressure and flow of SC-CO_2, denoted in that case as x_1, x_3, x_1^2, x_3^2 terms, were significant parameters of the model, which means that these independent variables (pressure and flow of CO_2) could be used to determine the yield of RT in SC-CO_2 extraction.

The effect of temperature on SC-CO_2 was observed through two possible effects on RT extraction, although, as indicated by statistical analyses, the effect of temperature on RT yield was not significant. Knowing that SCFE is also referred to as a “destruction” process, which means a combination of extraction and distillation, it is obvious that high pressure of SC-CO_2 was not favorable for extraction (high density), while high temperature increases the vapor pressure of various compounds. However, in accordance with the molecular weight of RT, as well as other compounds that could be extracted from licorice, one would expect a very small effect of temperature

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on the yield of RT by extraction. Namely, RT apparently has a low vapor pressure at the temperatures used in this study (30-50°C) and RSM analysis proved it. This is a state where temperature (as a coded value x_2) is a practically insignificant parameter that determines the output of RT GFR by extraction (equation 1). Moreover, an increase in temperature at constant pressure leads to a decrease in the density of CO_2 and thus reduces the strength of the solvent. Thus, an increase in the RT yield with increasing temperature is also analyzed, but no noticeable effect is found.

Keeping in mind that the more important independent variables with significant values and influence on the RT output are the pressure and flow of SC-CO_2, the following and simplified equation could be used to calculate the release of RT from mulberries:

$$\gamma = 0.70 + 0.32x_1 + 0.073x_3 - 0.22x_1^2 - 0.064x_3^2 \qquad (3)$$

This equation can be used for the output (γ) RT at pressures from 16.9 to 32.2 MPa (x1 coded value from -1.662 to + 1.668) and for the SC-CO_2 flow rate from 11.08 to 28.84 (also used as x2 coded value from -1.688 to + 1.668).

Comparison of the allocated amount of RT γ calculated using equations (2) and (3) and experimentally found values showed only a small difference in the correlation coefficient between the calculated and experimentally determined values of γ (r = 0.987 and using equation (2), r = 0.989) also applies to show the results of the RSM-CCRD analysis.

It has been shown that the amount of extract that can be obtained from licorice roots ranges from 8.17 to 16.80%. These results can be compared with those literature data, where extraction was combined with microwave treatment and they amounted to 8.7% - 16.8%. Some other literature data refer to SC-CO_2 extraction [4]. However, these authors use a term defined as the ratio of SC-CO_2 yield to that of classical extraction methods. They found that SC-CO_2 extraction gave a yield of 28-85%, while in the classical method the yield ranged from 11-43%.

Conclusions:

1. To describe and predict the yield of resveratrol (RT) during extraction with supercritical (SC) CO_2 from mulberries under various conditions, the RSM (response surface methodology) method in combination with the CCRD (central composite rot a table design) method is adequately used. It has been established that the RT yield mainly depends on the pressure and the amount of SC-CO_2 used for extraction. It turned out. That there is a significant dependence for linear and quadratic terms of the relationship between the output of the RT and these parameters. There was no noticeable interaction between the three process parameters (pressure, temperature and amounts of SC-CO_2).

2. Optimal conditions are determined: 15.8 MPa. 30.5°C and 20.08 gCO_2/gf.m. consumption of CO_2 for the extraction of resveratrol (RT) from mulberries using supercritical (SC)CO_2. The maximum yield of RT was 158 mg from 1 g of dry material (about 0.5% of the total extract) can be achieved under certain optimal extraction conditions, it will be possible to obtain the maximum yield of RT at a density of SC-CO_2 of approximately 725 kg/m^3.

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