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Statistical and Mathematical Model of the Process of Extracting Pumpkin Seeds by CO₂-Extraction

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Abstract. Uzbekistan is a major producer of fruits, grapes and melons. The seeds and pits of these crops contain a valuable oil used in the pharmaceutical, perfume and food industries. The development of methods of energy-saving technologies that allow obtaining new high-quality products in the pharmaceutical, perfumery and food industries is due to the acute social need for high-quality medicines and foodstuffs, as well as in environmentally friendly industries. One of the solutions to this problem is the use of carbon dioxide in pre- and supercritical states as extractants. In this work, using the method of full-factor planning of experiments, the process of extraction of seeds of a local variety of pumpkin with supercritical carbon dioxide was studied, a statistical-mathematical model of the process was obtained, and on the basis of this model a rational mode of the process was determined.

INTRODUCTION

The Republic of Uzbekistan is a major producer of agricultural products and therefore special attention is paid to their deep processing.

According to the Decree of the President of the Republic of Uzbekistan No. PP-4406 dated July 29, 2019 "On additional measures for the deep processing of agricultural products and the further development of the food industry", the development of the food industry is provided, in particular, the deep processing of raw materials with the involvement of cooperatives, dekhkan (peasant) farms, farmers and small businesses [1]. At the same time, the most important task of processing vegetable raw materials is to increase the yield of finished products while preserving useful substances.

Every year the production of vegetables, grapes, fruits and berries, melons is increasing. The total volume of their production is up to approximately 17,000 thousand tons per year. [2]. The seeds of these crops make up from 2 to 10% of the mass of processed raw materials. The kernel of their seeds contains a valuable oil used in the pharmaceutical, perfume and food industries. The main method for obtaining oil (from apricot kernels, grapes, and melon seeds) is cold pressing, i.e. pressing carried out at a temperature below 600C. Such oil immediately moves from the category of ordinary table oils to the category of medicinal ones.

Currently, in Uzbekistan, the processing of apricot kernels, grape seeds and gourds in order to obtain high-quality vegetable oil is carried out on low-tonnage technological lines by cold pressing. etc. using the so-called "cold" pressing method. But this method of processing raw materials does not provide the maximum yield of valuable vegetable oil and useful substances [3,4,5]. It is not practiced to use extraction as a method for obtaining these substances.

Now 90% of all supercritical fluid technologies are focused on supercritical carbon dioxide (SC- CO₂). Its widespread use is associated with a number of properties that make carbon dioxide a unique solvent, namely [3,6,7,8,9,10]: critical temperature close to room temperature (31.30C) and low critical pressure (7.39 MPa); environmental safety and extremely low toxicity, which makes it indispensable in the food and pharmaceutical industries; explosion and fire safety; low cost and general availability; at atmospheric pressure is a gas, which ensures complete separation of the extract by depressurization.

Supercritical carbon dioxide effectively extracts various oils from plant materials: mono-, di-, and triglycerides and fatty acid esters [8–10]. Technologies have been developed for extracting valuable ingredients from plant raw materials [11], antioxidants from marjoram [12], carotenoids, tocopherols, and sitosterols from tomato industrial processing waste [13], etc.

Currently, extraction using sub- and supercritical CO₂ as a solvent produces vegetable oil from pistachio seeds [14], from cumbara [15], from juniper leaves [16], from grape seeds [17], from hemp seeds [18], from pine grains [19], from mint [20], from corn germs [21], etc. Valuable essential oil is extracted from Chilean sunflower [22],

saffron [23], a plant from the labiate family *Dracocephalum kotschy* Boiss [24,25], lemon peel (medicinal lemon balm) [26], turmeric [27], wild bergamot [28], sage [29], hops [30], basil, mint, oregano [31], etc.

Features of the structure of vegetable raw materials in our region - fruit stones, seeds of grapes, pumpkins, melons, etc., suggest the development of technological modes of extraction using liquid and supercritical carbon dioxide, the kinetics and dynamics of the extraction process, determining the effect of the extraction process on the yield and quality of the resulting product [32].

Based on this, **the purpose of this study** is to experimentally identify the dependence of the yield of pumpkin seed oil on the influencing factors during CO₂ extraction.

The objectives of the study are: to determine the boundary values of the factors influencing the CO₂-extraction process, to develop an experimental plan, to obtain a statistical and mathematical model of the process.

MATERIALS AND METHODS

Seeds of local pumpkin varieties “Oshkovok” and “Nos-kovok” were used for research. After grinding, as a result of the calculation, the weighted average size of the crushed seeds was 0.3-0.4 mm. CO₂ was used as an extractant in the supercritical state.

The study of the process of extracting ingredients from local plant materials with supercritical carbon dioxide was carried out on a laboratory installation, the schematic diagram of which is shown in Figure 1 [33,34].

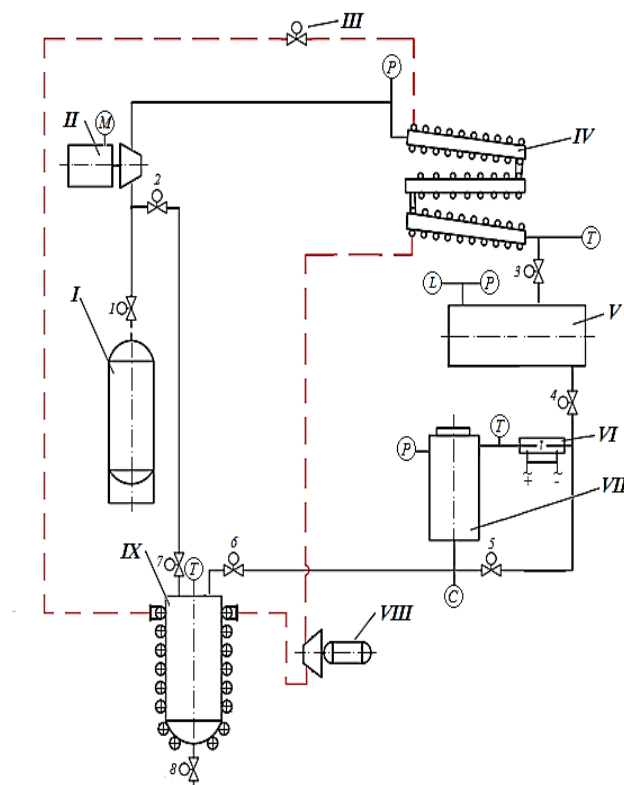


FIGURE 1. Schematic diagram of a laboratory setup for studying the process of CO₂ extraction of ingredients from plant materials [34]. I-cylinder with CO₂ gas; II-compressor; III-throttle valve; IV-capacitor; V-capacity for liquefied CO₂; VI-heat exchanger; VII-extractor; VIII-heat pump compressor; IX-separator-evaporator.

The laboratory installation consists of the following main elements: a high-pressure extractor with a cassette for placing a sample of plant raw materials, an extractant supply system, a product collection system, a condensation system, a heat pump system, a system of instrumentation and automation.

By changing the temperature or pressure of the fluid, one can change its properties in a wide range. Thus, it is possible to obtain a fluid whose properties are close to either a liquid or a gas. The dissolving power of the fluid increases with increasing density (at constant temperature). Since the density increases with increasing pressure,

changing the pressure can affect the dissolving power of the fluid (at a constant temperature). In the case of temperature, the dependence of fluid properties is somewhat more complicated - at a constant density, the dissolving power of the fluid also increases, however, near the critical point, a slight increase in temperature can lead to a sharp drop in density, and, accordingly, dissolving power. Therefore, the pressure and temperature of the extractant (SC-CO₂) were chosen as influencing factors. The limits of factor variation are determined on the basis of preliminary experiments.

The levels of variation of the two factors are as follows:

Upper level (+):

- pressure 8.5 MPa, temperature 33⁰C.

Lower level (-):

- pressure 7.5 MPa, temperature 31⁰C.

Conducted coding factors (table 1).

TABLE 1. Factor coding.

Factors	Upper level x^+	Lower level x^-	Center	Interval variations λ	Dependence of the coded variable on the natural one
x_1	8,5	7,5	8	0,5	$(z_1-8)/0,5$
x_2	33	31	32	1	$(z_2-32)/1$

Since we have two influencing factors and they change at two levels, we get the planning of experiments for 2². Thus, it is necessary to conduct 4 experiments.

Results and its discussion. The plan matrix and the results of the experiment are shown in Table 2.

TABLE 2. Plan matrix and experiment results for 2².

Experiment number	Factors		Interaction effects of factors	Experimental results			Average results \bar{y}_j
	x_1	x_2		x_1x_2	y_1	y_2	
1	-1	-1	1	5,2	5	4,6	4,933
2	1	-1	-1	4,2	3,8	4	4,000
3	-1	1	-1	3,5	3,7	3,3	3,500
4	1	1	1	2,8	3,2	3	3,000
						$\Sigma \bar{y}_j$	15,433

The regression equation in this case has the following form:

$$y = b_0 + b_1x_1 + b_2x_2 + b_{12}x_1x_2 \quad (1)$$

Regression coefficients are calculated using the formulas:

$$b_0 = \frac{\sum_{i=1}^N y_i}{N}$$

$$b_1 = \frac{\sum_{i=1}^N \bar{y}_i x_{kod}}{N} \quad i. e. \quad (2)$$

The calculation results are shown in Table 3.

TABLE 3. The results of calculating the values of regression coefficients

Odds	b_0	b_1	b_2	b_{12}
Values	3,8583	-0,3583	-0,6083	0,1083

We determine the significance of these coefficients by the Student's criterion From the Student's distribution tables [35,36] by the number of degrees of freedom $n(m-1)=4 \cdot 2=8$ at a significance level $\alpha = 0.05$ we find $t_{cr.} = 1.86$.

Estimated value of Student's criterion $|b_j| = t_{cr.} \cdot S_{coef} = 1,86 \cdot 0,0667 = 0,124$.

Comparing the obtained value of 0.124 with the coefficients of the regression equation, we find that all coefficients except b_{12} are greater in absolute value $|b_j|$. Therefore, all coefficients except b_{12} are significant. Thus, this coefficient is excluded from the regression equation.

The regression equation (1) has the form:

$$y = 3,8583 - 0,3583x_1 - 0,6083x_2 \quad (3)$$

The adequacy of the resulting regression equation was tested using the Fisher criterion [35].

For our case $S_{rem}^2 = 0,14083$. At a significance level of $\alpha = 0.05$ and degrees of freedom $k_1 = n - r = 4 - 3 = 1$ и $k_2 = n(m - 1) = 8$ $F_{table} = 5,32$.

This means that the resulting model (3) adequately describes the process.

In model (3), the coefficients b_1 and b_2 have a negative sign, which means that with an increase in the values of the factors x_1 and x_2 , the value of the residual oil content of the meal in %.

Let us transform equation (3) from the coded values of the influencing factors to natural values, substituting instead of x_i their expressions through z_i , which are calculated according to the formulas given in Table 1. Having carried out arithmetic operations, we obtain the equation in natural values:

$$Y = 14,50 - 0,72z_1 - 0,12z_2 \quad (4)$$

According to the obtained equation (4), we plot the dependence of the residual oil content of pumpkin seeds (meal) on the influencing factors of pressure and temperature using the MathCAD program (Figure 2).

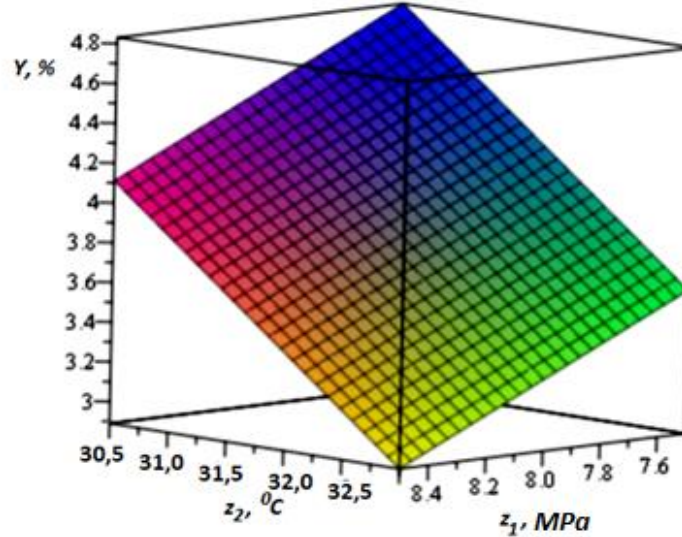


FIGURE 2. Dependence of the residual oil content of pumpkin seeds (meal) Y on the influencing factors of pressure z_1 and temperature z_2 .

CONCLUSION

According to the graph (Figure 2), it can be seen that with an increase in pressure and temperature, the oil content of the meal decreases. According to Figure 2, the rational values of the influencing factors are the pressure of the extractant $P=8.4$ MPa, the temperature $t=31.5^{\circ}C$. With this extraction mode, the residual oil content of the meal is 3.03%. For the production of pumpkin seed oil using CO_2 extraction, this extraction mode is recommended.

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