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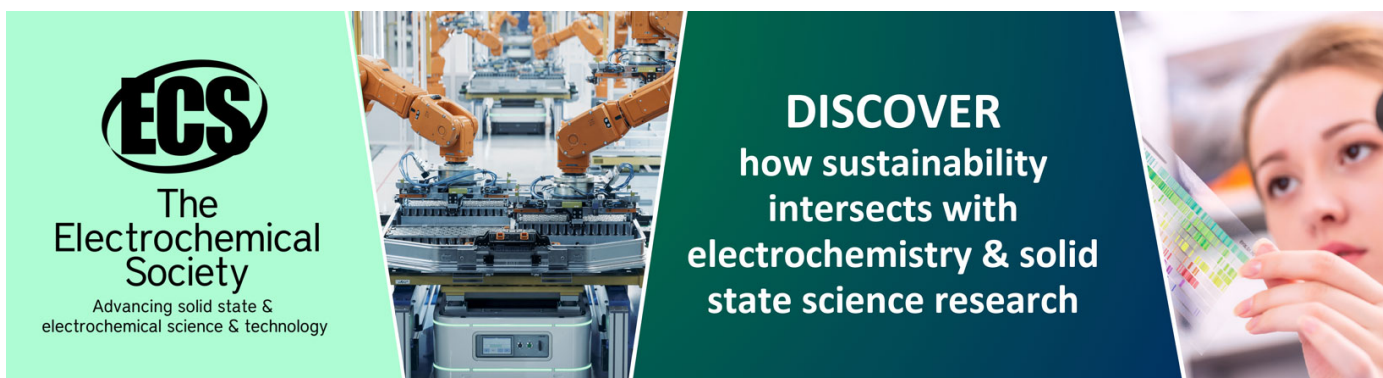
Experimental study of the extraction process of coniferous plants

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Experimental study of the extraction process of coniferous plants

Khayrullo Djuraev, Mamura Yodgorova, Akhtam Usmonov and Mukhammad Mizomov

Bukhara Engineering Technological Institute, Bukhara, Uzbekistan

E-mail: djuraev_xf@mail.ru

Abstract. The article presents the results of studying the influence of such factors as the contact surface of the phases, the degree of grinding of the material, the nature of the flow and the ratio of the two phases called the hydro-module. It has been experimentally proven that at a hydro-module of 1: 4.1: 5, soluble components diffuse to the surface of the material, mixing and dissolving almost completely. By choosing a more acceptable hydro-module, it is possible to obtain a large percentage yield of the extract with the most valuable components, as well as to intensify the extraction process.

1. Introduction

Over the past 15-20 years, the flora of Uzbekistan has become extremely rich and diverse. This may seem incredible, but in reality, compared to the neighboring regions of Central Asia. In the foothill and mountainous regions of Uzbekistan, the number of plants per unit area is many times greater.

The coniferous gardens of the HUMSON BULOQ sanatorium in the mountains of Uzbekistan are famous for their beauty and territory. The aroma of coniferous plants helps to cope with anxiety, insomnia, shortness of breath, lowers blood pressure, and also improves metabolism. The rich flora of the country has more than six thousand different plant species, among which there are conifers, which are environmentally friendly and are used in pharmacology, aromatherapy and perfumery.

In addition, conifers are included in embalming compositions. Even in ancient times, the Egyptian pharaohs knew about this. The healing properties of conifers are characterized by a number of unique anti-inflammatory, disinfecting, antispasmodic and hemostatic properties. They contain an impressive amount of essential oils, tannins, C vitamins, starch and carotene.

Improving the technology of extracting raw needles in order to increase the yield of biologically active substances while maintaining its quality is a necessary link in the development of scientific and technical approaches to the intensification of mass transfer processes. When determining innovative methods of conducting the extraction process, one should take into account the experience of using traditional methods and study the available non-traditional methods for extracting substances from materials of plant origin. To substantiate new technologies and extraction processes, it is necessary to conduct complex studies.

Analysis of the results of theoretical and experimental works devoted to the study of mass transfer processes under various conditions of interaction with the extracted material from the side of the extractant. In addition, external forces which are the degree of grinding, the ratio of the two phases, the temperature of the medium, mixing and patterns of flow [1].



In connection with the above, the study of the process of extracting biologically active substances from conifers by the extraction method is the most urgent.

2. Research methods

Taking into account the urgency of the problem, we carried out research on the extraction of useful components from conifers growing in Uzbekistan using the extraction method. On the basis of a deeper study of the process of obtaining soluble substances from conifers, new extraction methods have been developed.

At the same time, the proposed discrete-countercurrent two-phase flow makes it possible to accelerate the process of heat and mass transfer and complex processing of this raw material.

One of the main tasks for the extraction of biologically active substances from plant raw materials of needles is to study the factors influencing the extraction process. In this regard, the study of influencing factors aimed at obtaining the maximum amount of extract is the main factor in justifying the frequency and duration of the extraction process, the degree of grinding of raw materials, as well as the choice of solvent, which are the main goal of our study.

We have developed an experimental setup in which the process of extraction of coniferous plants was carried out in a multistage discrete countercurrent setup with an intermediate vessel.

This localized experimental extraction unit enables the direct transfer of target components into the solvent. Subjecting material to extraction moves sequentially from the first stage of the device to the last, and the solvent moves from the last stage to the first, while realizing countercurrent flow.

The movement of material and solvent within one stage is co-current. A significant multiplicity of the solvent turnover provides an increase in the speed of its movement. The speed of movement of the solvent provides the most turbulent mode possible, contributing to the smallest thickness of the diffusion layer, as well as maintaining a high concentration difference.

General regularities in mass exchange with the solid phase take into account the mass transfer of the substance being distributed in the solid material due to internal diffusion and the transfer of the substance in the liquid and solid phases due to external diffusion [2].

According to Fick's first law [3], in an isotropic medium, the amount of diffusing substance j , which passes per unit time through a unit of cross-sectional area, is proportional to the concentration gradient measured along the normal to this cross-section:

$$j = -D \text{grad}C \quad (1)$$

Where D - the diffusion coefficient; C - concentration of diffusing substance.

A consequence of Fick's law is the differential diffusion equation:

$$\frac{\partial C}{\partial \tau} = D \frac{\partial^2 C}{\partial x^2}, \quad (2)$$

Where τ – time, x - coordinate.

The concentration field, which describes the distribution of extractives in the volume of a particle, is calculated by the equation [12]:

$$\frac{\partial C}{\partial \tau} = D \left(\frac{\partial^2 C}{\partial x^2} + \frac{\partial^2 C}{\partial y^2} + \frac{\partial^2 C}{\partial z^2} \right) \quad (3)$$

Or

$$\frac{\partial C}{\partial \tau} = D \left(\frac{\partial^2 C}{\partial \xi^2} + \frac{G}{\xi} \frac{\partial C}{\partial \xi} \right) \quad (4)$$

Where ξ – generalized coordinate (for the plate $\xi = x$, for the sphere $\xi = r$, r – radius from the axis); G - constant value depending on the shape of the particles (for plate 0, for sphere 2).

Let us introduce the initial condition (concentration distribution inside the body at the initial moment of time) for equation (3):

$$C(x, y, z, 0) = C_0 \quad (5)$$

Where C_0 – initial concentration, kg/m³.

The boundary condition of the third kind has the form, according to [13], since we consider diffusional mass transfer, while the extracting flows around the outer surface of the body:

$$-D_m \left(\frac{\partial C}{\partial n} \right)_p = \alpha (C_p - C_1) \quad (6)$$

Where C_1 - concentration of solute in the environment, kg/m³, α - mass transfer coefficient, C_p - concentration on the surface, kg/m³.

Using equation (4) under boundary conditions (5) and (6), it is possible to determine the distribution of concentrations over the volume of the body in time [4]. The molecular diffusion coefficient depends on the structure of the solid and on the hydrodynamic conditions on the surface of the solid particles and the design of the apparatus.

The pore structure is largely determined by the extraction mechanism of the extracts and the rate of its flow. The sizes of the needles particles are much larger than the pore diameters, so they can be taken as isotropic porous bodies. The current concentration of extractives in the solid phase (with subsequent conversion in% dry matter) can be found using the material balance equation:

$$M_m(C_0 - C) = M_f C \quad (7)$$

Where M_m , M_f - mass of dry raw materials and extracting, g; C_0 - initial concentration of extractives in the solid phase, C- current concentration of extractives in the solid phase, C_1 - current concentration of extractives in the liquid phase,

3. Research results

The proposed extraction method makes it possible to investigate soluble substances in a wider range by selecting the required solvent and hydro module. The proposed method of extraction on all surfaces of the plant raw material improves the hydrodynamic structure of the flows, it takes time to reduce the thickness of the diffusion layer and accelerates the extraction process. It is known that the rate of dissolution of biologically active substances inside particles is characterized by the rate of mass transfer.

By providing a discrete countercurrent mode of flows, the process of mass transfer can be accelerated. The extraction rate depends on the washout ratio. Since the opened cells are quickly washed out by the extracting. Therefore, it can be noted that the degree of grinding and the coefficient of leaching are important technological characteristics of raw materials. The range of experimental studies for solid and liquid phases is shown in table 1.

Table 1. Experimental data range.

Name of raw materials	Solvent and solids movement	Hydro- module	Solvent	Particle size
Pine needles	Forward flow + counter flow	1:1-1:5	Ethanol	1-4 mm

The process of extracting biologically active substances from plant raw materials needles was experimentally investigated by the extraction method. The studies were carried out in the following ranges of variation of the process parameters: initial concentration $C = 30\%$, extraction time $\tau = 0 - 240 \text{ min}$ particle size $d = 0.5-4 \text{ mm}$ and hydro-module $HM = 1: 1-1: 5$.

The above limits of variation of the parameter values are selected based on the results of our preliminary experiments.

3.1. The experimental technique includes

- selection of the required hydronic module in two phases;
- ensuring countercurrent movement of flows by increasing the intensity of phase mixing;
- The process of increasing the contact of phases by grinding the extracted material.

To extract biologically active substances from plant raw materials needles, a number of experimental studies were carried out. Initially, the coniferous plant was washed with water, a set of particles 0.5–1 mm in size was prepared, then the initial moisture content and the initial concentration were determined according to the proposed methods [5,6].

During the experiment, a suspension weighing 2 liters was prepared, which consisted of a hydro-module in a ratio of 1: 1, 1: 2, 1: 3, 1: 4 and 1: solids and solvent. Each solids to solvent ratio was placed in a special container and the extraction process was carried out on the basis of a 240-minute intensive mixing system. The results of the experimental study are shown in figure 1.

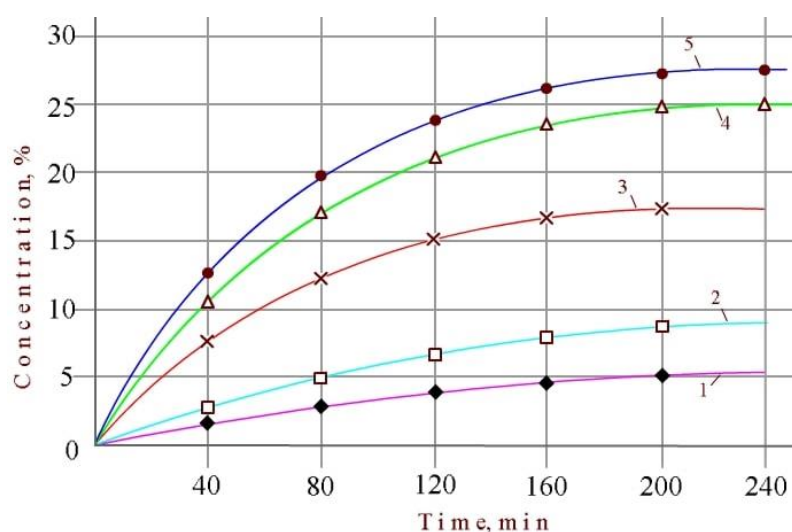


Figure 1. Change in the concentration of the extract over time when using 70% ethyl alcohol (hydro-module: 1-1: 1, 2-1: 2, 3-1: 3, 4-1.4, 5-1: 5).

As can be seen from the graph (figure 1), the duration of the complete extraction of biologically active substances from the particles of coniferous plants with a size of 0.5-1 mm is 240 minutes, and the optimal hydromodule has a ratio of 1: 4, 1: 5.

In order to obtain a mathematical expression reflecting the dependence of the extraction rate of influencing factors, the extraction process was investigated using the methods of mathematical calculation of a full factorial experiment [7-9].

An experimental study with the physical dimension of factors (HM, d, t) in dimensionless expression (x_1, x_2, x_3) is presented in table 2.

Table 2. Experimental indicators for different physical dimensions of factors.

№	Indicators	HM	x_1	d, mm	x_2	t,min	x_3
1	Maximum	5	+1	4	+1	240	+1
2	Minimum	1	-1	1	-1	30	-1
3	Average	3	0	2.5	0	135	0

The discrimination of the computer model was carried out in the following sequence:

- carried out a qualitative comparison of experimental functions with a set of theoretical data;
- quantitatively estimated the proximity of experimental and theoretical data using the methods of mathematical statistics;
- verification of the accepted hypothesis was carried out on the basis of the application of a number of statistical criteria (Kolmogorov, Romanovsky, Pearson, etc.) [10-12].

To check the adequacy of the mathematical model to the real process, the results of the calculations were compared with experimental data.

It should be noted that at present, most of the proposed methods, the main attention is paid to obtaining a mathematical description that adequately reflects the entire technological process.

For this purpose, for the extraction process, checking the adequacy of the results obtained is reduced to calculating the standard deviation of the experimental data from the theoretical. We have determined the absolute deviations of the experimental data taking into account the change in the concentration and extraction time from their calculated values.

The root-mean-square deviation of the experimental data from the calculated ones is:

$$\sigma = \sqrt{\frac{\sum \Delta C^2}{(C_{\max} - C_{\min})^2 \cdot n}} = 1.3\%; \quad \sigma_t = \sqrt{\frac{\sum \Delta t^2}{(t_{\max} - t_{\min})^2 \cdot n}} = 3.1\%. \quad (8)$$

Statistical processing of the obtained data was carried out using the MATLAB program. The verification of the adequacy of the mathematical model to the real process was carried out by comparing the calculation results with the experimental data given in table. 3.

Table 3. Comparison of experimental results with calculated data.

№	t, min	C _e , %	C _p	Δ, C	η ²	t _e , °C	t _p , °C	Δt	η ²
1	0	30	30	0	0	20	0	0	0
2	30	23	23.7	0.7	0.49	23	23.1	0.1	0.01
3	60	15	14.50	0.5	0.25	26	26.4	0.4	0.16
4	90	12	12.35	0.35	0.12	32	32.5	0.5	0.25
5	120	10	10.6	0.06	0.0036	39	39.3	0.3	0.09
6	150	8.67	8.69	0.02	0.0004	46	46.21	0.21	0.04
7	180	6.63	6.65	0.02	0.0004	50	50.15	0.15	0.02
8	210	3.63	3.62	0.01	0.0001	53	53	0	0
9	240	1.6	1.5	0.01	0.0001	55	56.5	1.5	2.25

4. Conclusion

The obtained results of experimental studies show that when extracting conifers (juniper, pine), crushed raw materials with a size of 0.5-1 mm should be used. The process should be carried out in a discrete-counter-current mode in a four-fold discrete-counter-current flow. The structure of the needles undergoing extraction has a significant effect on the internal mass transfer. From this point of view, it is required to identify the patterns of changes in internal mass transfer, which makes it possible to develop methods for calculating mass transfer processes.

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