Subcritical processing of raw materials for the production of food additives

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Abstract. The article is devoted to the analysis of pectin diffusion extraction in the extraction process from plant raw materials in a solid-liquid system. The molecular diffusion process from the inside of the extractable substance particles to their outer surface is considered as the limiting stage. The aim of this work is to study the possibility of creating an efficient production of pectin from agricultural products by carrying out a more complete extraction using a period of mass isolation in the extraction process. As it is generally recognized all over the world, the rational use of natural resources is the dominant trend in the development of the economy, which assumes the fullest use of vegetable raw materials consumed by humans in the processing. To implement such approaches, a mathematical model of the discrete diffusion process in porous diffusionally isotropic particles has been developed. A conclusion is made about the effect of mass isolation on the extraction rate for a regular mode, while the regularities of the process remain unchanged.

1. Introduction
The processes of extraction of substances from plant materials using selective solvents (extraction) are widely used in industry. Particularly, modern research in the field of pectin production is associated with ultrasonic extraction as one of the most progressive and cost-effective production methods.

According to modern understanding of phenomena at the interface in the solid-liquid system, by the nature of the diffusion path, the extraction process can be divided into three main stages:

the first stage – a molecular diffusion of the outer surface of the particles of the extracted plant material from the inside (internal diffusion);

the second stage – molecular diffusion through the boundary layer (diffusion boundary layer or diffusion sublayer);

the third stage – convective transfer from the boundary layer to the moving solvent (external diffusion).

It was found that the process as a whole is limited by the first stage - diffusion of pectin inside the particle. Therefore, a theoretical study of the extraction process can be carried out by mathematical models describing its first stage with sufficient accuracy.
2. Problem statement

Currently, the process of plant raw materials extraction is carried out according to the principle of a stepless or multistage counterflow. The study of continuous diffusion that takes place in stepless processes has been described in detail by many authors. Based on the analysis of the known solutions of the boundary value problems, the regularities of the molecular microprocess underlying the first limiting stage of the process were studied, and some conclusions that have theoretical and practical significance were obtained, which show the direction of research in order to increase the productivity of extraction devices [1].

However, multistage extractors are more widely used in industry at present, in which the study of the extraction of plant raw materials is somewhat easier than that in stepless ones. Each stage in these devices can be considered as consisting two periods. The first, when the extracted material is irrigated with a solvent (and the pectin is removing from the inside of the particle into the surrounding extractant), and the second, the period of mass isolation, when there is no contact with the external solvent (and only redistribution of pectin inside the particle happens). In this case, during the period of mass isolation, the concentration of the extracted substance on the surface of the particle increases, which can result in an increase in the diffusion rate and the beginning of the next extraction stage. This technique is found in heterogeneous kinetics, in particular, in the system of absorption-desorption processes, where the period of mass isolation is called the "rest" of the particles. Thus, the described continuous extraction process is based on a physical diffusion process with a discrete removal of a substance (a stepwise process). An important task is to develop a mathematical model of diffusion with a discrete removal of a substance (multi-stage extraction) in order to evaluate and compare it with continuous diffusion (stepless extraction).

Some authors have changed the approach to the study of stepwise processes [2].

For example, for periodically operating reactors in the case when the extraction rate is determined by diffusion from the inside of the particle to its surface, the problem of extraction from solids was solved by J. Cranc and J. Pouchly. This problem for countercurrent extraction in a chain of reactors of ideal mix was solved by J. Skrzhivanek and Z. Krzhivski.

3. Model construction

To derive a mathematical model of the extraction process, they used the following definitions and assumptions: solid particles are porous spheres with a radius $R$; the resistance of the boundary layer on the particle surface can be neglected; the process of mass transfer from a particle is characterized by the Fick's second law; all reactors have the same volume.

The authors have solved the equation of nonstationary diffusion in partial derivatives in spherical coordinates:

$$\frac{\partial C}{\partial \tau} = D \left( \frac{\partial^2 C}{\partial r^2} + \frac{2}{r} \frac{\partial C}{\partial r} \right)$$

where $C = C(r, \tau)$ is the concentration of the extracted substance; $D$ is the internal diffusion coefficient; $r$ is the distance from the center of the particle; $\tau$ - time, under the following boundary conditions:

$$C = C_{in}, r = R, \tau > 0;$$
$$C = \varphi(r), 0 \leq r \leq R, \tau = 0;$$
$$C = \varphi(r), 0 \leq r \leq R, \tau = 0;$$

$\varphi(r)$ is the initial concentration distribution in the particle.

In this case, the average values of the distribution of the concentration of particles at the exit from the $k$-th reactor in a series of $i$ identical reactors (or stages) are set.

Taking into account the attempts made to study stepwise processes and above mentioned features of multistage extraction, a mathematical model that describes the intradiffusion area of this extraction process has been developed. The process of discrete diffusion (in other words, diffusion with intermittent removal of matter) is simulated in porous diffusionally isotropic particles of the basic geometric shape of a plate, a ball, a cylinder, which in the first approximation can be taken as actually
extractable e.g. algae leaf, grains, and granules.

Mathematically, the problem is formulated as follows: it is required to solve the differential diffusion equation:

$$\frac{\partial C(\xi, \tau)}{\partial \tau} = D \nabla^2 C(\xi, \tau),$$

where $\nabla^2 = \frac{\partial^2}{\partial \xi^2} + \nu - 1 \frac{\partial}{\partial \xi} - $ Laplacian

in the case of boundary conditions of the first kind:

$$C(R, \tau) = \varphi_1(\tau),$$
$$\frac{\partial C(0, \tau)}{\partial \xi} = 0$$
$$C(0, \tau) < \infty,$$

which corresponds to the first period of the process at the i-th stage (the material is in contact with the solvent and the diffusing substance is discharged into the environment), and in the case of boundary conditions of the second kind:

$$\frac{\partial C(R, \tau)}{\partial \xi} = 0$$
$$\frac{\partial C(0, \tau)}{\partial \xi} = 0$$
$$C(0, \tau) < \infty,$$

which corresponds to the second period of the process at this stage - the period of mass isolation, when only the alignment (redistribution) of the concentration field inside the material particle occurs. Here the following designations are introduced: $C(\xi, \tau)$ - concentration of the extracted substance; $D$ is the diffusion coefficient; $\xi$ is the current spatial coordinate equal to $x$ for an endless plate and $r$ for a ball and an endless cylinder; taking into account the symmetry $0 < \xi < R$ - the characteristic size of the body, equal for an endless plate to half of its thickness, and for a cylinder and a ball - their radius; $\tau$ is time; $\tau > 0$; $\nu$ is the number of final measurements, or the shape factor, equal to 1 for the plate, 2 for the cylinder, and 3 for the ball.

The concentration change occurs only in one direction, in the other two the concentration is unchanged, i.e. the task is one-dimensional.

The material being extracted is considered homogeneous. Therefore, the internal diffusion coefficient $D$ does not depend on the spatial coordinates. In the general case, the diffusion coefficient will be assumed to be dependent on the time of the process, but in such a way that at each period of the process at the stage it remains constant. Thus, the coefficient will change abruptly depending on the number and period of the step. Since the concentration of the pectin changes with the course of the extraction time and with the increase in the number of the stage, therefore, the internal diffusion coefficient will depend on the concentration of the extracted substance.

Boundary conditions of the second kind are the conditions of symmetry and physical limitation of the concentration values in the material.

Solutions of problems with boundary conditions of the first and second kind separately for each of the classical bodies of regular shape with arbitrary initial conditions are known [3-5]. They are obtained by various methods of mathematical physics.

In the problem in question (2), the distribution of the concentration of a substance in a porous body at the initial moment of time for the entire process ($\tau = 0$) is constant and equal to $C_0$, and the distribution of a substance in a material at the initial moment of each subsequent period will be equal to the distribution of a substance in the end of the previous period, i.e. in the general case, $C(\xi, \tau_0) = f(\xi)$, where $\tau_0$ is the beginning of the process on the period, i.e. the beginning of irrigation of the extracted material with a solvent (the moment the process starts in the first period) or the beginning of the mass isolation process (the moment the process starts in the second period); $f(\xi)$ is a function of the argument $\xi$ satisfying the Dirichlet conditions, that is, for all $\xi$ from the interval $[0, R]$ it is single-valued, finite, and integrable [6-8].
The boundary value problem with a uniform initial distribution of the concentration of the substance $C_0$, is solved for the particular case of the boundary condition, namely, for $C(R, \tau) = C_p = \text{const}$, (the material is in contact with the solvent of constant concentration). It should be noted that this formulation of the problem does not correspond to the principle of counterflow, but is a description of the limiting case of a sufficiently large volume of the solvent with a concentration $C_p (\beta \to 0)$ in comparison with the particle, provided that the extracted substance is rapidly removed from the surface of the particle into the surrounding extractant. However, setting (2) with $C(R, \tau) = C_p$ does not diminish the generality of studying the problem of diffusion with discrete removal of a substance during extraction and is sufficient for comparison with the continuous diffusion model obtained under similar conditions.

Taking into account the initial distribution of substance at each period of the process at the stage, the solutions of the diffusion equation for bodies of the basic geometric shape for each period separately were considered, and then by the method of mathematical induction the final solution of problem with the boundary conditions of both cases was obtained, which is the mathematical model describing the intradiffusion area of the multistage extraction process:

$$
\frac{\frac{C_{ik}(z, \tau) - C_p}{C_0 - C_p}}{\frac{\sum_{n_{i1}=1}^{\infty} \sum_{n_{i2}=1}^{\infty} \sum_{i=1}^{\infty} A_{n_{i1}}^{-2-k}(v) \exp \left(-\frac{D_{11} \mu_{n_{i1}, 1}^2 \tau_{ik} - \sum_{m=0}^{\infty} \xi_m}{R^2} \right) \prod_{m=0}^{i-1} C_m(v)}{\sum_{n_{i1}=1}^{\infty} \sum_{n_{i2}=1}^{\infty} \sum_{i=1}^{\infty} A_{n_{i2}}^{-2-k}(v) \exp \left(-\frac{D_{12} \mu_{n_{i2}, 2}^2 (\tau - \tau_{i1})}{R^2} \right) \prod_{m=0}^{i-1} C_m(v)}} = 2^i \sum_{n_{i1}=1}^{\infty} \sum_{n_{i2}=1}^{\infty} \sum_{i=1}^{\infty} A_{n_{i1}}^{-2-k}(v) \exp \left(-\frac{D_{11} \mu_{n_{i1}, 1}^2 \tau_{ik} - \sum_{m=0}^{\infty} \xi_m}{R^2} \right) \prod_{m=0}^{i-1} C_m(v), \quad (3)
$$

$$
\tau_{ik} = \begin{cases} 
\tau, k = 1, \\
\tau_{i1}, k = 2,
\end{cases}
$$

$$
\xi_m = D_{m+1, 1} \mu_{n_{m+1, 1}, 1}^2 \tau_{m2} - D_{m1} \mu_{n_{m1, 1}, 1}^2 \tau_{m1}; \quad \xi_0 = 0;
$$

$$
G_m(v) = \mu_{n_{m+1, 1}}^2 \left\{ \frac{\nu}{\mu_{n_{m1}} \mu_{n_{m1+1, 1}}} + 2 \sum_{m=2}^{\infty} \left( \mu_{n_{m1}} - \mu_{n_{m1+1, 1}} \right) \left( \mu_{n_{m2}} - \mu_{n_{m2+1, 1}} \right) \right\}; \quad C_0(v) = 1;
$$

$$
B_{n_{i2}}(v) = \frac{\nu}{\mu_{n_{i1}}} + 2 \sum_{n_{i2}=1}^{\infty} \frac{F_{n_{i2}}(v) \exp \left(-\frac{D_{12} \mu_{n_{i2}, 2}^2 (\tau - \tau_{i1})}{R^2} \right) \prod_{m=0}^{n_{i2}} C_m(v)}{\mu_{n_{i1}} \mu_{n_{i2}}}.
$$

$\tau_{ik}$ is the time interval from the beginning of the process ($\tau = 0$) to the end of the k-th period of the i-th stage.

The values of $A_{n_{i1}}$ and $F_{n_{i2}}$ and the characteristic equations from which the roots $\mu_{n_{ik}}$ are determined are given in Table 1. In the table, $J_0(z)$ and $J_1(z)$ are the Bessel function of the first kind from the real argument of the zero and the first order respectively [9].

| Geometric shape | Endless plate ($v = 1$) | Endless cylinder ($v = 1$) | Sphere ($v = 3$) |
|-----------------|------------------------|--------------------------|----------------|
| Characteristic equation | $k=1$ | $k=2$ | $k=1$ |
| | $cos \mu_{n_{i1}} = 0$; | $cos \mu_{n_{i1}} = 0$; | $sin \mu_{n_{i1}} = 0$; |
| | $\mu_{n_{i1}} = \frac{1}{2} (2n_{i1} - 1) \pi$; | $\mu_{n_{i2}} = \frac{1}{2} (2n_{i2} - 1) \pi$; | $\mu_{n_{i1}} = n_{i1} \pi$ |
| | $n_{i1} = 1, 2, 3, ...$ | $n_{i2} = 1, 2, 3, ...$ | $n_{i1} = 1, 2, 3, ...$ |
| $A_{n_{i1}}(v)$ | $(-1)^{n_{i1}+1} cos \left( \mu_{n_{i1}} \frac{\nu}{R} \right)$ | $1 \frac{J_0(\mu_{n_{i1}} \nu)}{\mu_{n_{i1}} J_0(\mu_{n_{i1}} \nu)}$ | $-1 \frac{R sin \left( \mu_{n_{i1}} \frac{\nu}{R} \right)}{R \mu_{n_{i1}}}$ |
| $F_{n_{i2}}(v)$ | $(-1)^{n_{i2}+1} cos \left( \mu_{n_{i2}} \frac{\nu}{R} \right)$ | $\frac{J_0(\mu_{n_{i2}} \nu)}{\mu_{n_{i2}} J_0(\mu_{n_{i2}} \nu)}$ | $\frac{R sin \left( \mu_{n_{i2}} \frac{\nu}{R} \right)}{R \mu_{n_{i2}}}$ |

| Table 1. Values of characteristic roots and solution coefficients. |
4. Results and Discussion
Analysis of the obtained solution to the boundary value problem of discrete diffusion shows that the absolute value of the sequential series of values of the corresponding characteristic roots $\mu_{nik}$ increases rapidly, i.e. $\mu_{1ik} < \mu_{2ik} < \cdots < \mu_{nik} < \cdots$, and, therefore, the exponential factors $\exp\left(-\mu_{nik}^2 \tau_{ik}\right)$, rapidly decrease, which implies the fast convergence of the infinite sums included in the solution. Therefore, with an accuracy sufficient for a fundamental analysis, we can restrict ourselves to only one of the first terms of the series and take as $n_{ik} = 1$.

For sufficiently large values of $n_{ik}$, we can not only take into account only the first terms of the infinite sums, but also assume that the extraction mode has passed to the regular one, i.e. described by a simple exponential law [10-12]. Calculations and analysis of the operation of industrial devices show that when extracting in a solid-liquid system, this situation occurs at a time $\tau_{ik} \geq 600-1200$ s. It should be noted that when analyzing the main solution (3), the total process time is important, which is equal to the product of the number of steps and the duration of the diffusion process per step. Let, in addition, all periods of the process be equal in time, i.e. $\tau_{m2} - \tau_{m1} = \frac{t}{2}$, where $t$ is the duration of the diffusion process at the stage in that case, and the diffusion coefficient $D_{ik} = D = \text{const}$. To simplify the study, let us take $C = 0$ (the material is in contact with a pure solvent). Then, taking into account the determination of the average concentration of the substance in the extracted body:

$$c_{ik}(\tau) = \frac{1}{R_0^2} \int_0^R \xi^{\nu-1}c_{ik}(\xi, \tau) d\xi;$$

and considering that:

$$\frac{c_{ik}(\tau)}{c_0} = \frac{q_{ik}}{q_0},$$

After simple transformations we finally get:

$$q_{i1}(\tau) = a^i c^{i-1} \exp\left\{-\frac{bD[\tau-(i-1)\frac{t}{2}]}{h^2}\right\}$$

for $(i-1)t = \tau_{i-1,2} < \tau = \tau_i \leq \tau_{i,1} = (i-1)t + \frac{t}{2}$.

Since during the period of mass isolation, the amount of the substance extracted from the particle does not change, then at $\tau = (i-1)t + \frac{t}{2}$ we obtain:

$$q_{i2} = q_{i1}(\tau_{i,1}) = a^i c^{i-1} \exp\left\{-\frac{i\beta dt}{2h^2}\right\} = \text{const}$$

for $\tau_{i,1} < \tau < \tau_{i,2}$.

In the formula (6), the mass of the substance remaining in the pectin-containing particle after a certain time $\tau_1 = \tau$, from the beginning of the process in the first period of the $i$-th stage; $\tau_i$ - (i-1) $t / 2$ - net outward diffusion time; $(i-1)\frac{t}{2}$ - time of body mass isolation; in formula (7), $q_{i2}$ ($\tau_1$) is the remainder of the substance in the particle during the entire second period of the $i$-th stage; $q_{i1}(\tau_{i,1})$ - the remainder of the substance in the particle at the end of the first period of the $i$-th stage; $q_0$ - initial content of matter in a particle $\tau = 0$.

The values of the parameters $a$ and $c$ specific to a body of a given geometric shape are determined as follows:

$$a = \frac{2\nu}{\mu_1^2};$$

$$c = 1 + d_0 \exp\left(-\frac{v\xi_0}{2}\right),$$

where, $d_0$ is the function of the shape coefficient $\nu$, equal to:

$$d_1 = \frac{2}{9};$$

$$d_2 = \frac{1}{(1-\mu^2)^2};$$

$$d_3 = \frac{1}{3(1-\mu^2)^2}.$$
\[ F_{0m} = \frac{D}{R^2} \] is a generalized variable, which can be called a discrete diffusion Fourier number, since \( t \) does not change continuously, but takes discrete values;

\[ \mu = \frac{\mu_1}{\mu_2}; \mu_1 \text{ and } \mu_2 \text{ is the first roots of the corresponding simplified characteristic equations, equal for the plate } \mu_1 = \frac{\pi}{2}; \mu_2 = \pi; \text{ for the cylinder } \mu_1 = 2,4048; \mu_2 = 3,8317, \text{ for the sphere } \mu_1 = \pi; \mu_2 = 4,4934. \]

The values of parameters \( a, b, c, \) and \( h \) are tabular data.

For \( i = 1 \) and in the case of a continuous diffusion process, solution (6) turns into the equality:

\[ \frac{q_i}{q_0} = a e^{\exp \left( -\frac{bD\tau}{2h^2} \right) \left[ 1 - \left( \frac{\mu_1}{\mu_2} \right)^{i-1} \right]} \tag{8} \]

used in [13-15] to study continuous diffusion, which is the basis of the stepless extraction process. Thus, a discrete process solution is more general than a continuous process solution.

The obtained basic solution (3) and simplified one (6) make it possible to calculate the ratio of the remainder of the substance in the particle \( q_{ik} \) after a certain period of time to the initial content of the substance \( q_0 \), depending on the size of the particle, its internal and external structure. The inverse problem can also be solved - determining the time required to obtain a given residue of a substance in a pectin-containing particle when extracting pectin in a multistage extractor. In this case, from equation (6) we obtain an expression for finding the desired time value:

\[ \tau = \frac{h^2}{bD} \ln \left[ \frac{q_i}{q_0} \right] + (i - 1) \frac{t}{2} \tag{9} \]

Let us note some works in which the ideas of stepwise processes are developed to one degree or another.

In [16-18], the authors, investigating discrete diffusion from the solid phase in a multistage countercurrent extraction process, take into account the increase in the concentration of the solvent at the steps and the degree of equalization of the concentration profile within the solid phase during the transition from step to step, and raise the question of the effect of the ratio of the periods of extraction - mass isolation on the depth extraction of extractive substances.

For the process of mass transfer in systems with a solid phase, a zone calculation method was used, which takes into account the non-uniform initial distribution of concentration in the solid phase, which takes place in the second and subsequent zones of the apparatus [19-20].

5. Conclusion

The paper considers the theoretical and practical aspects of calculating devices with a dispersed solid phase with a sequential change in the potential of a continuous medium with different conditions of heat transfer over the sections of the devices. Relationships are obtained that can be used in the analysis of stepwise mass transfer processes with constant values of the diffusion coefficient inside the material particles. The conclusion is drawn as follows: carrying out mass isolation, or equalization of concentration, promotes the acceleration of extraction for a regular regime, while the regularities of the process remain unchanged.

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