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Analysis of the Three-Step Cyclic Process of Countercurrent Extraction

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Abstract—A mathematical model of the three-step cyclic process of countercurrent extraction has been developed. Each cycle consists of two half periods, i.e., the half period of the motion of the flow of the first solvent phase and the half period of the motion of the flow of the second solvent phase. The first half period is composed of two steps, i.e., the step of feed supply (first solvent containing an initial mixture of components) and the step of the supply of the first solvent (without the initial mixture). The analytical dependences that describe the concentration profiles in a cascade of equilibrium stages at all three steps of the process for each cycle and a change in the concentrations in the output flows of the raffinate and extract phases were obtained. An analysis of the influence of operating parameters on the efficiency of the process of separation of a binary mixture was performed.

Keywords: liquid extraction, liquid chromatography, cyclic mode of counterflow mass transfer, separation of liquid mixtures

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INTRODUCTION

New versions of the cyclic process of extraction separation of liquid mixtures [1–11], which represent a combination of liquid chromatography without solid support [12–16] and countercurrent liquid extraction [8, 14, 17–19], are actively developed and investigated in recent years. These processes are characterized by high efficiency and can be used both for analytical purposes and in the commercial production of high-purity substances [8, 14]. Previously, an analysis of the cyclic process of the countercurrent liquid extraction in a cascade of equilibrium stages, each cycle of which consists of two steps, i.e., the motion of the flow of the initial solution (raffinate phase) and the motion of the flow of extractant (extract phase) [20].

The purpose of this work is to analyze the process, each cycle of which consists of the following three steps:

1. the motion of the flow of the initial solution, which consists of the first solvent and a mixture of components to be separated;

2) the motion of the flow of pure (without a mixture of components) first solvent;

3) the motion of the flow of the second solvent (extractant).

Thus, the time of the motion of the first solvent phase is divided into two intervals that differ in the presence and absence of a mixture of components that are separated in the flow of the first solvent phase. These conditions for performing cyclic countercurrent extraction are recommended when separating binary liquid mixtures.

MATHEMATICAL MODEL OF THE THREE-STEP CYCLIC PROCESS OF COUNTERCURRENT EXTRACTION

The scheme of the model of the three-step cyclic process of countercurrent extraction in the cascade of ideal mixing steps is shown in Fig. 1. Each cycle consists of two half periods, i.e., the half period of the motion of the flow of the first solvent phase with a duration of τ_{1x} and the half period of the motion of the flow of the second solvent phase with a duration of τ_{2y}. The first half period is composed of two steps. In the first step, the feed flow (first solvent containing the extractable substance) is supplied within a certain period of time τ_f and, in the second step, pure first solvent is supplied for the remaining time of the first half period $\tau_{1x} - \tau_f$. To simplify the mathematical manipulations, we assume that the equilibrium distribution of the transferring component between phases is achieved at each step, and the distribution coefficient is independent of the concentration.

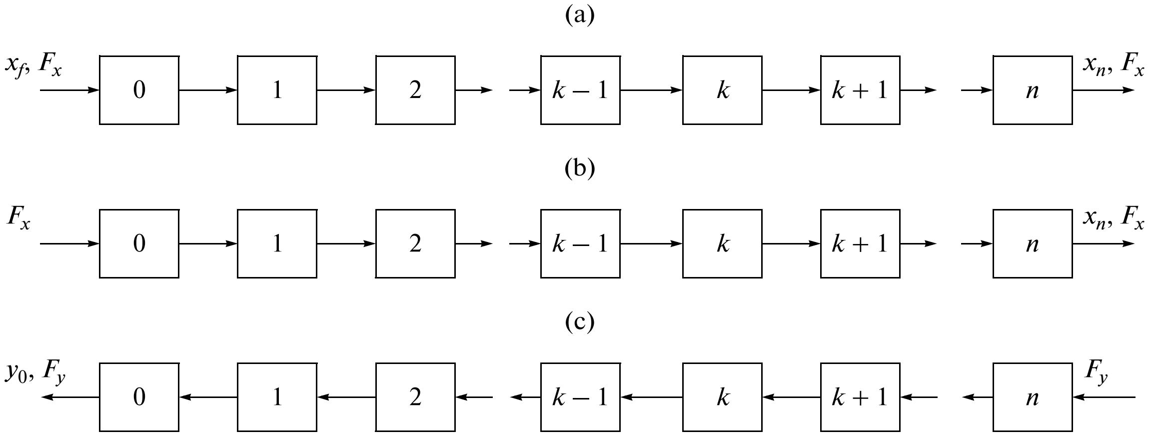

Fig. 1. Scheme of the model of the three-step cyclic process of countercurrent extraction: (a) step of the motion of the flow of the initial solution, which is composed of a first solvent and a mixture of components being separated; (b) step of the motion of the flow of pure (without a mixture of components) first solvent; (c) step of the motion of the second solvent flow.

The mathematical model of the process can be represented by the following system of equations with regard to made assumptions and according to Fig. 1.

The half period of the motion of the flow of the first solvent phase (Figs. 1a, 1b) is as follows:

At the step of the feed supply,

$$0 \le \tau \le \tau_f$$

$$\frac{\mathrm{v}_x}{N}\frac{dx_0}{d\tau}+\frac{\mathrm{v}_y}{N}\frac{dy_0}{d\tau}=\frac{(\mathrm{v}_x+m\mathrm{v}_y)}{N}\frac{dx_0}{d\tau}=F_xx_f-F_xx_0, \tag{1}$$

$$\frac{\mathrm{v}_x}{N}\frac{dx_k}{d\tau}+\frac{\mathrm{v}_y}{N}\frac{dy_k}{d\tau}=\frac{(\mathrm{v}_x+m\mathrm{v}_y)}{N}\frac{dx_k}{d\tau}=F_xx_{k-1}-F_xx_k, \tag{2}$$
$$k=1,2,\ldots,n.$$

At the step of the pure solvent supply,

$$\tau \ge \tau_f$$

$$\frac{(\mathrm{v}_x+m\mathrm{v}_y)}{N}\frac{dx_0}{d\tau}=-F_xx_0, \tag{3}$$

$$\frac{(\mathrm{v}_x+m\mathrm{v}_y)}{N}\frac{dx_k}{d\tau}=F_xx_{k-1}-F_xx_k,\ \ k=1,2,\ldots,n. \tag{4}$$

The half period of the motion of the flow of the second solvent phase (Fig. 1c) is as shown below:

$$\frac{\mathrm{v}_x}{N}\frac{dx_n}{d\tau}+\frac{\mathrm{v}_y}{N}\frac{dy_n}{d\tau}=\frac{(\mathrm{v}_x/m+\mathrm{v}_y)}{N}\frac{dy_n}{d\tau}=-F_yy_n, \tag{5}$$

$$\frac{\mathrm{v}_x}{N}\frac{dx_k}{d\tau}+\frac{\mathrm{v}_y}{N}\frac{dy_k}{d\tau}=\frac{(\mathrm{v}_x/m+\mathrm{v}_y)}{N}\frac{dy_k}{d\tau}=F_yy_{k+1}-F_yy_k, \tag{6}$$
$$k=n-1,\ n-2,\ldots,2,1,0.$$

In Eqs. (1)–(6), the following designations are assumed: x is the concentration in the first solvent phase, x_f is the concentration in the feed flow, y is the concentration in the second solvent phase, m is the distribution coefficient, $m = y/x$, k is a number of the step (numbering starts from the side of the inlet of the feed flow into the system, the step of the inlet of feed flow has the number of zero, the step of the exit of raffinate from the system is n), $N = 1 + n$ is the total number of steps in the system (apparatus), v_x and v_y is a volume occupied by the corresponding phase in the apparatus, F_x and F_y are volumetric flow rates of phases, and τ is time.

We assume that each half period of the cyclic process starts from $\tau = 0$. Let us introduce the following dimensionless variables:

$t_x = \frac{\tau_x F_x}{\mathrm{v}}$ is dimensionless time in the half period of the motion of the first solvent phase ($\mathrm{v} = \mathrm{v}_x + \mathrm{v}_y$ is the total volume of liquids in the system), $t_y = \frac{\tau_y F_y}{\mathrm{v}}$ is dimensionless time in the half period of the motion of the second solvent phase, $t_f = \frac{\tau_f F_x}{\mathrm{v}}$ is the duration of the feed supply in dimensionless units of time, and $X = \frac{x}{x_f}$ and $Y = \frac{y}{x_f}$ are the dimensionless concentrations in phases.

Let us rewrite system of equations (1)–(6) with new variables.

For the half period of the motion of the flow of the first solvent phase, the step of the feed supply is

$$0 \le t_x \le t_f \quad \frac{1}{a}\frac{dX_0}{dt_x}=1-X_0, \tag{7}$$

$$\frac{1}{a}\frac{dX_k}{dt_x}=X_{k-1}-X_k,\ \ k=1,\ 2,\ldots,\ n; \tag{8}$$

and the step of the solvent supply is

$$t_x > t_f \quad \frac{1}{a}\frac{dX_0}{dt_x}=-X_0, \tag{9}$$

$$\frac{1}{a}\frac{dX_k}{dt_x}=X_{k-1}-X_k,\ \ k=1,\ 2,\ldots,\ n. \tag{10}$$

The step of the motion of the flow of the second solvent phase is

$$\frac{1}{am}\frac{dY_n}{dt_y} = -Y_n, \tag{11}$$

$$\frac{1}{am}\frac{dY_k}{dt_y} = Y_{k+1} - Y_k;\ \ k = n-1, n-2, \ldots, 2, 1, 0, \tag{12}$$

where

$$a = \frac{N}{1 - S + Sm};\ \ S = \frac{\mathrm{v}_y}{\mathrm{v}_y + \mathrm{v}_x} = \frac{\mathrm{v}_y}{\mathrm{v}}.$$

We assume that the process starts from the supply of the feed flow.

Using the results from [16], the solution of the system of equations (7)–(10) for the first cycle of the process can be represented as follows:

$$0 \le t_x \le t_f \quad X_{1,1}(t_x, k) = 1 - e^{-at_x}\sum_0^k \frac{(at_x)^i}{i!}; \tag{13}$$

$$t_x > t_f$$

$$X_{1,1}(t_x, k) = e^{-a(t_x - t_f)}\sum_0^k \frac{[a(t_x - t_f)]^{k-i}}{(k-i)!} X_{1,1}(t_f, i). \tag{14}$$

The $X_{1,1}(t_f, i)$ function on the right side of Eq. (14) describes the distribution of concentrations established in the cascade of steps at the end of the feed step in the first half period of the first cycle; it is determined by expression (13) at $t_x = t_f$.

The dependences for the concentration in the raffinate when it leaves the apparatus in the first cycle can be obtained by substituting the value $k = n$ into equations (13)–(14) as follows:

$$0 \le t_x \le t_f \quad X_{1,1}(t_x, n) = 1 - e^{-at_x}\sum_0^n \frac{(at_x)^i}{i!}; \tag{15}$$

$$t_x > t_f$$

$$X_{1,1}(t_x, n) = e^{-a(t_x - t_f)}\sum_0^n \frac{[a(t_x - t_f)]^{n-i}}{(n-i)!} X_{1,1}(t_f, i). \tag{16}$$

Let us designate the duration of the first and second half periods of each cycle in dimensionless time units, i.e., t_{1x} and t_{2y}, respectively:

$$t_{1x} = \frac{\tau_{1x} F_x}{\mathrm{v}},\ \ t_{2y} = \frac{\tau_{2y} F_y}{\mathrm{v}}.$$

The initial conditions for the second half period of the first cycle (for equations (11)–(12)) can be written as follows:

$$t_y = 0\colon Y_{1,2}(0, k) = mX_{1,1}(t_{1x}, k). \tag{17}$$

Here, $X_{1,1}(t_{1x}, k)$ is the distribution of concentrations established in the cascade of steps at the end of the first half period of the first cycle; it is determined by the relationship (14) at $t_x = t_{1x}$. The solution of Eqs. (11)–(12) with initial conditions (17) has the following form:

$$Y_{1,2}(t_y, k) = me^{-mat_y}\sum_{i=k}^n \frac{(mat_y)^{i-k}}{(i-k)!} X_{1,1}(t_{1x}, i). \tag{18}$$

The dependence for the concentration in the extract flow that leaves the system at the first cycle can be obtained by substituting the value $k = 0$ into Eq. (18) as follows:

$$Y_1(t_y, 0) = me^{-mat_y}\sum_{i=0}^n \frac{(mat_y)^i}{i!} X_{1,1}(t_{1x}, i). \tag{19}$$

The solution of the system of equations (7)–(10) for the second cycle of the process was obtained in the following form:

$$\begin{gathered} 0 \le t_x \le t_f \\ X_{2,1}(t_x, k) \\ = 1 - e^{-at_x}\sum_0^k \frac{(at_x)^i}{i!} + e^{-at_x}\sum_0^k \frac{(at_x)^{k-i}}{(k-i)!} X_{1,2}(t_{2y}, i); \end{gathered} \tag{20}$$

$$t_x > t_f$$

$$X_{2,1}(t_x, k) = e^{-a(t_x - t_f)}\sum_0^k \frac{[a(t_x - t_f)]^{k-i}}{(k-i)!} X_{2,1}(t_f, i), \tag{21}$$

where $X_{1,2}(t_{2y}, k) = Y_{1,2}(t_{2y}, k)/m$ is the distribution of concentrations established in the cascade of steps at the end of the second half period of the first cycle; it is determined by the relationship (18) when substituting $t_y = t_{2y}$. The $X_{2,1}(t_f, i)$ function in Eq. (21) is determined by expression (20) at $t_x = t_f$.

The output concentrations in the raffinate in the second cycle can be calculated from equations (20)–(21) by substituting the value $k = n$ into them.

The solution of the system of equations (7)–(12) of the mathematical model for each cycle j ($j = 2, 3, 4, \ldots$) can be written in the following general form.

The half period of the motion of the flow of the first solvent phase based on Eqs. (7)–(10) is described as follows:

at the step of the feed supply,

$$\begin{gathered} 0 \le t_x \le t_f \\ X_{j,1}(t_x, k) \\ = 1 - e^{-at_x}\sum_0^k \frac{(at_x)^i}{i!} + e^{-at_x}\sum_0^k \frac{(at_x)^{k-i}}{(k-i)!} X_{j-1,2}(t_{2y}, i), \\ X_{j-1,2}(t_{2y}, k) = Y_{j-1,2}(t_{2y}, k)/m; \end{gathered} \tag{22}$$

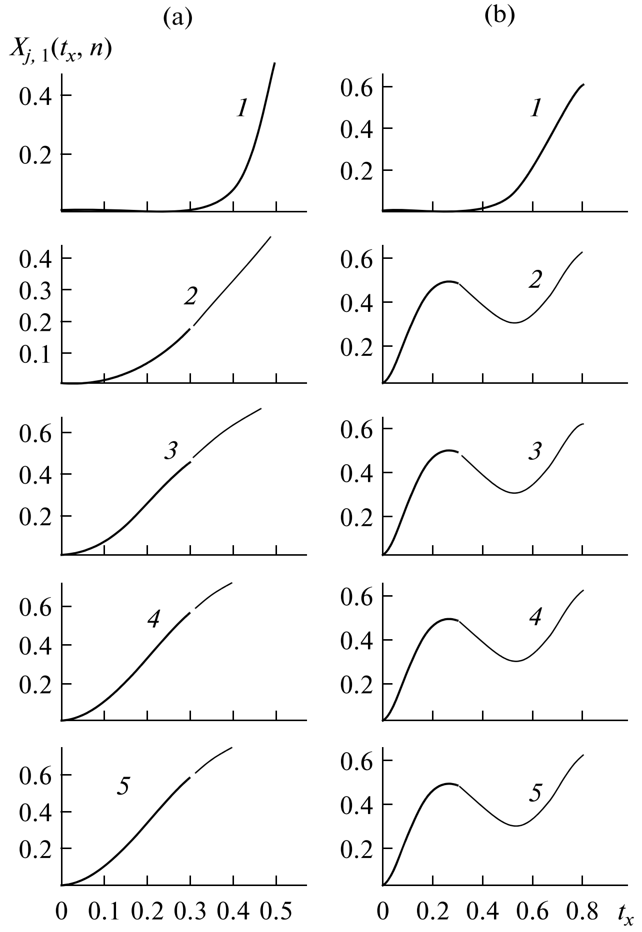

Fig. 2. Output profiles of concentrations in the raffinate for first five cycles of the process (j = 1–5). Bold line corresponds to the step of feed supply. Process parameters: (a) $N = 20$; $m = 0.5$; $t_f = 0.3$; $t_{1x} = 0.5$; $t_{2y} = 0.5$: $S = 0.5$; (b) $N = 20$; $m = 0.5$; $t_f = 0.3$; $t_{1x} = 0.8$; $t_{2y} = 0.3$: $S = 0.5$.

and, at the step of the solvent supply,

$$t_x > t_f:$$

$$X_{j,1}(t_x,k) = e^{-a(t_x-t_f)}\sum_{0}^{k}\frac{[a(t_x-t_f)]^{k-i}}{(k-i)!}X_{j,1}(t_f,i). \tag{23}$$

The step of the motion of the flow of the second solvent phase as shown in Eqs. (11)–(12) is

$$Y_{j,2}(t_y,k) = me^{-mat_y}\sum_{i=k}^{n}\frac{(mat_y)^{i-k}}{(i-k)!}X_{j,1}(t_{1x},i). \tag{24}$$

When $t_x = t_{1x}$ and $t_y = t_{2y}$, Eqs. (23) and (24) describe the distribution of concentrations established in the cascade of steps at the end of first and second half periods of the cycle j.

The output concentrations in the raffinate and extract in the cycle j (j = 2, 3, 4, ...) can be calculated from Eqs. (22)–(24), correspondingly, by substituting the value $k = n$ into Eqs. (22), (23), and $k = 0$ into Eq. (24).

In the above equations, the indices at concentrations represent the following: first index is the number of cycle and the second index is the number of the step (1 is the step of the motion of the first solvent phase, 2 is the step of the motion of the second solvent phase).

Equations (13)–(16) and (18)–(24) represent a complete analytical solution of the mathematical model of the cyclic process of countercurrent extraction with an intermittent feed supply (mixture being separated).

ANALYSIS OF THE MATHEMATICAL MODEL

Let us perform an analysis of the mathematical model of the three-step cyclic process of countercurrent extraction using the obtained dependences. The dimensionless parameters of the model are the distri-

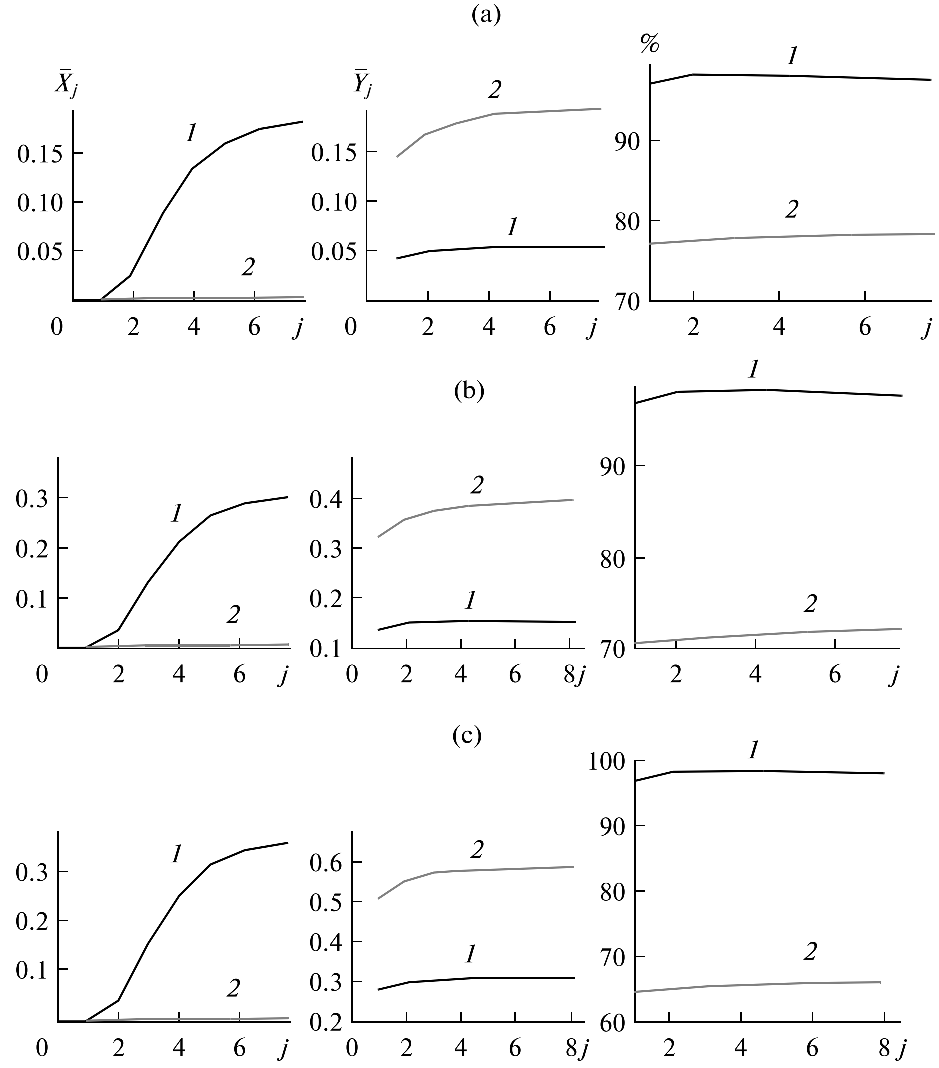

Fig. 3. Influence of the duration of the feed supply (t_f) on the values of average concentrations of components 1 ($m_1 = 0.5$) and 2 ($m_2 = 1$) in the leaving raffinate ($\bar{X}_j$) and extract ($\bar{Y}_j$) fractions, and the percentage composition of fractions (%); the composition of the initial mixture was 50/50%: *1.* component 1 in the raffinate fractions; *2.* component 2 in the extract fractions. Process parameters: $N = 20$; $t_{1x} = 0.4$; $t_{2y} = 0.5$: $S = 0.5$: (a) $t_f = 0.1$; (b) $t_f = 0.2$; (c) $t_f = 0.3$.

bution coefficient of the component m; the number of equilibrium steps N; and the duration of the step of feed supply t_f, the duration (t_{1x} and t_{2y}) and number (j) of cycles, and a complex parameter $a = \dfrac{N}{1 - S + Sm}$, which includes N, m, and the volume ratio of phases in the apparatus S. The parameters S, t_f, t_{1x}, and t_{2y} are operating (controlled) parameters. The concentration profiles in the output flows of phases change from cycle to cycle at the initial stage of the process. Depending on the values of operating parameters, they may have a quite complicated form, as, e.g., in Fig. 2, where the output profiles in the phases for first five cycles of the process calculated from Eqs. (15), (16) and (22), (23) are shown.

However, after a certain number of cycles, a quasi-stationary mode in which the concentration profiles become constant and repeat in subsequent cycles is achieved (Fig. 2).

The average concentrations in the fractions of phases that leave any cycle j

$$\bar{x}_j = \frac{F_x}{F_x \tau_{1x}} \left[\int_0^{\tau_f} x_{j,1}(\tau_x, n) d\tau_x + \int_{\tau_f}^{\tau_{1x}} x_{j,1}(\tau_x, n) d\tau_x \right],$$

$$\bar{y}_j = \frac{F_y}{F_y \tau_{2y}} \int_0^{\tau_{2y}} y_{j,1}(\tau_y, 0) d\tau_y$$

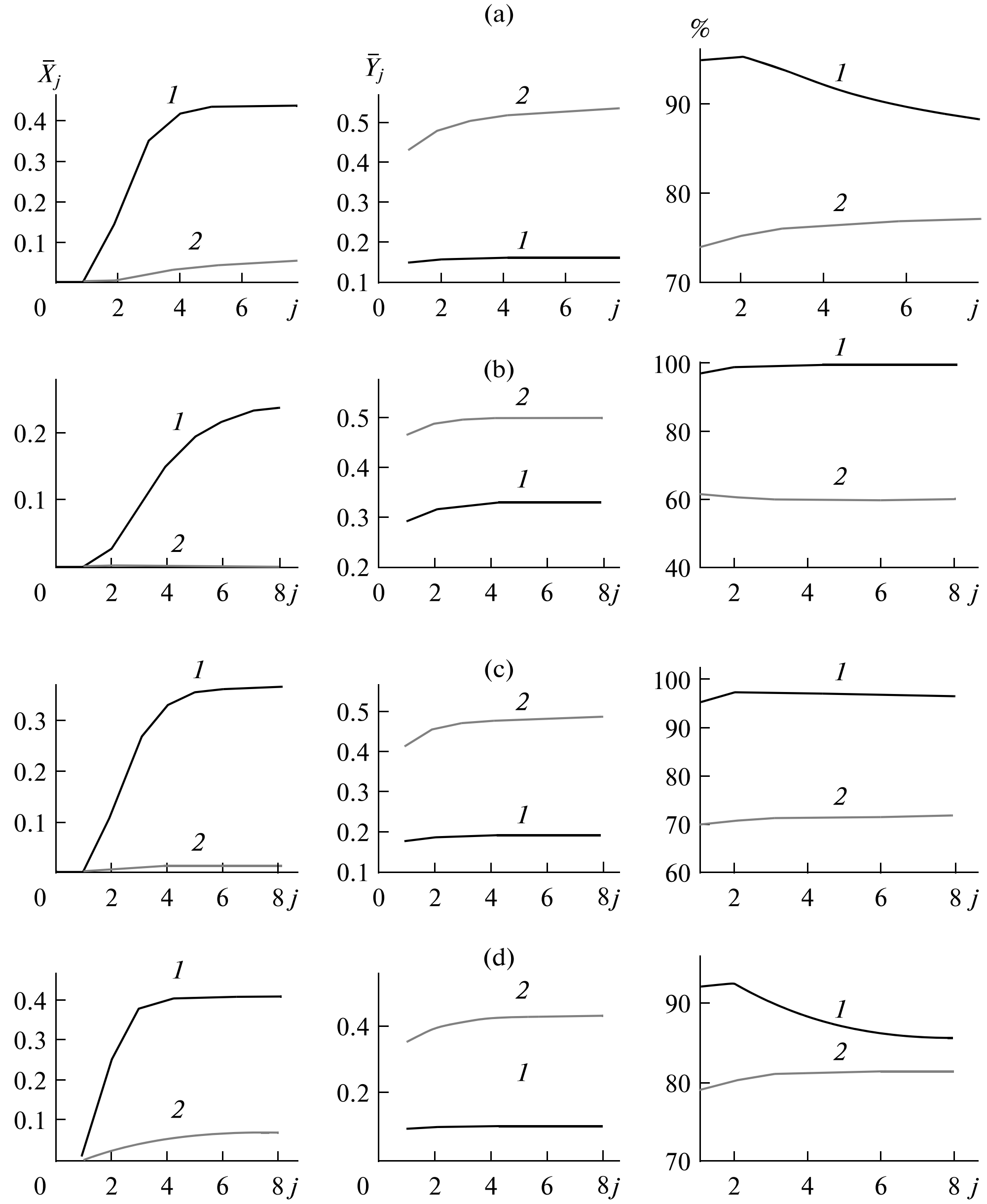

Fig. 4. Influence of the duration of the solvent supply on the process of separating a binary mixture. For other designations, see Fig. 3. Process parameters: $N = 20$; $S = 0.5$; $t_f = 0.3$: (a) $t_{1x} = 0.5$, $t_{2y} = 0.5$; (b) $t_{1x} = 0.4$, $t_{2y} = 0.6$; (c) $t_{1x} = 0.5$, $t_{2y} = 0.6$; (d) $t_{1x} = 0.6$, $t_{2y} = 0.6$.

can be determined from the material balance equations for the first and second half periods of the cycle j as follows:

$$\tau_{1x}F_x\bar{x}_j = \frac{\mathrm{v}_x + m\mathrm{v}_y}{N} \times \left[\sum_{i=0}^{n} x_{j-1,2}(\tau_{2y}, i) - \sum_{i=0}^{n} x_{j,1}(\tau_{1x}, i)\right] + \tau_f F_x x_f,$$

$$\tau_{2y}F_y\bar{y}_j = \frac{\mathrm{v}_x + m\mathrm{v}_y}{N}\left[\sum_{i=0}^{n} x_{j,1}(\tau_{1xy}, i) - \sum_{i=0}^{n} x_{j,2}(\tau_{2y}, i)\right].$$

Where

$$\bar{X}_j = \frac{\bar{x}_j}{x_f} = \frac{1}{at_{1x}}\left[\sum_{i=0}^{n} X_{j-1,2}(t_{2y}, i) - \sum_{i=0}^{n} X_{j,1}(t_{1x}, i)\right] + \frac{t_f}{t_{1x}}, \tag{25}$$

$$\bar{Y}_j = \frac{\bar{y}_j}{x_f} = \frac{1}{at_{2y}}\left[\sum_{i=0}^{n} X_{j,1}(t_{1x}, i) - \sum_{i=0}^{n} X_{j,2}(t_{2y}, i)\right]. \tag{26}$$

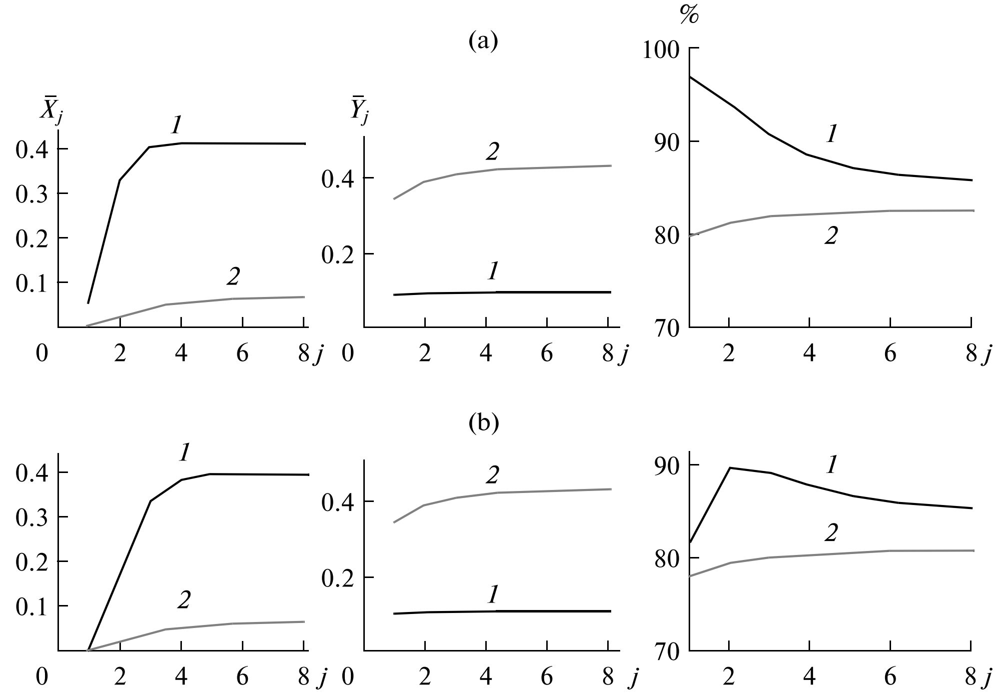

Fig. 5. Influence of the volume ratio of phases S on the process of separation of a binary mixture. For other designations, see Fig. 3. Process parameters: $N = 20$; $t_f = 0.3$; $t_{1x} = 0.6$, $t_{2y} = 0.6$: (a) $S = 0.3$; (b) $S = 0.7$.

In the steady state, the values of average concentrations in the portions (volumes) of raffinate $\bar{X}_j$ and extract $\bar{Y}_j$ that leave each cycle become constant.

The influence of the process parameters, such as the duration of the feed supply period (Fig. 3) and the periods of solvent supply (Fig. 4) and the volume ratios of the phases (Fig. 5) on the separation of a binary mixture of components is shown in Figs. 3–5.

Based on an analysis of the results presented in Figs. 3–5, the following conclusions can be drawn:

As follows from Fig. 3, the values of average concentrations in the portions of raffinate $\bar{X}_j$ and extract $\bar{Y}_j$ that leave in the cyclic mode rise with an increase in the duration of feed supply t_f; however, at the same time, the separation of the components gets a little worse.

The separation factor and concentrations of components in the raffinate and extract can be adjusted by selecting the duration of the periods of solvent supply t_{1x} and t_{2y} (Fig. 4).

As can be seen from Figs. 4 and 5, changes in the volume ratio of phases S have no significant effect on the separation process.

CONCLUSIONS

A mathematical model of the three-step cyclic process of countercurrent extraction has been developed. An analysis of the influence of operating parameters, i.e., the duration of the step of feed supply, the duration and number of cycles, and the volume ratio of phases in the apparatus, on the process of separation of a binary mixture was performed. It has been found that an increase in the duration of the feed supply leads to a rise in concentrations in the portions of raffinate and extract that leave in the cyclic mode; however, at the same time, the separation of the components gets a little worse. A change in the volume ratio of phases has no significant effect on the separation process. It has been shown that the separation factor and concentrations of components in the raffinate and extract can be adjusted by selecting the duration of the periods of solvent supply.

The results of the present study may be useful when developing new highly efficient processes of separation of rare-earth metals and processes for preparing high-purity substances.

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NOTATION

F_x—volumetric flow rate of the first solvent, m^3/s;

F_y—volumetric flow rate of the second solvent, m^3/s;

m—distribution coefficient;

N—total number of steps in the system (apparatus);

S—volume ratio of phases in the apparatus;

t_f—dimensionless duration of the feed supply;

t_x—dimensionless time in a half period of the motion of the first solvent flow;

t_y—dimensionless time in a half period of the motion of the second solvent flow;

t_{1x}—dimensionless duration of a half period of the motion of the first solvent phase;

t_{2y}—dimensionless duration of a half period of the motion of the second solvent phase;

v_x—volume of the first solvent phase in the system, m^3;

v_y—volume of the second solvent phase in the system, m^3;

X—dimensionless concentration in the first solvent phase;

x—concentration in the first solvent phase, mol/m^3;

Y—dimensionless concentration in the second solvent phase;

y—concentration in the second solvent phase, mol/m^3;

τ—time, s.

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