

**РАЗДЕЛЕНИЕ СМЕСИ ДИЭТИЛОВЫЙ ЭФИР + ГЕКСАН + ЭТИЛАЦЕТАТ + ЭТАНОЛ  
С ИСПОЛЬЗОВАНИЕМ ЭКСТРАКТИВНОЙ РЕКТИФИКАЦИИ****А.В. Фролкова, Д.С. Логачев, Т.Е. Ососкова**

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*Статья посвящена разработке схем разделения смеси растворителей производства дигидрокверцетина диэтиловый эфир + гексан + этилацетат + этанол на основе анализа структуры диаграммы парожидкостного равновесия. Фазовое равновесие в системе диэтиловый эфир + гексан + этилацетат + этанол исследовано с применением математического моделирования в программе AspenPlus V.10.0. Параметры уравнения Non Random Two Liquid позволяют воспроизводить данные парожидкостного равновесия и азеотропные свойства системы с относительной ошибкой, не превышающей 5%. Показано, что система характеризуется наличием трех бинарных азеотропов (седловидного типа), а диаграмма дистилляционных линий разбивается на три области дистилляции двумя сепаратрическими поверхностями, порожденными азеотропами этилацетат – этанол и этилацетат – гексан. N-метил-2-пирролидон выбран в качестве растворителя для процесса экстрактивной ректификации на основе анализа литературы. Рассчитана селективность разделяющего агента и показано, что выбранный растворитель в разной степени оказывает влияние на относительную летучесть компонентов (повышает летучесть гексана и этилацетата относительно этанола, а также летучесть гексана относительно этилацетата). Предложены две схемы ректификационного разделения смеси, содержащие колонну выделения диэтилового эфира и два комплекса экстрактивной ректификации (разделение тройной смеси гексан + этилацетат + этанол). Схемы отличаются последовательностью выделения компонентов: гексан, этилацетат и этанол. Подобраны параметры работы колонн (число тарелок, тарелка питания, флегмовое число, соотношение количества исходной смеси и разделяющего агента) отвечающие минимальным энергозатратам и обеспечивающие получение веществ товарного качества. Выбран энергоэффективный вариант разделения.*

**Ключевые слова:** дигидрокверцетин, разделение смеси растворителей, экстрактивная ректификация, азеотроп, схема разделения

**SEPARATION OF DIETHYL ETHER + HEXANE + ETHYL ACETATE + ETHANOL  
QUATERNARY SYSTEM VIA EXTRACTIVE DISTILLATION****A.V. Frolova, D.S. Logachev, T.E. Ososkova**

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*The paper is devoted to the development of diethyl ether + hexane + ethyl acetate + ethanol mixture (solvents of the dihydroquercetine production) separation flowsheets based on the analysis of the structure of the phase diagram. The phase equilibrium of the quaternary system was studied using mathematical modeling in the AspenPlus. The parameters of Non Random Two Liquid model allow reproducing vapor-liquid equilibrium data and azeotropes characteris-*

*tics with a relative error not exceeding 5%. It was shown that the system was characterized by the presence of three binary azeotropes and the phase diagram was divided into three distillation regions. Based on a literature analysis N-methyl-2-pyrrolidone was selected as a solvent for the extractive distillation. The selectivity of the separating agent was calculated and it was shown that N-methyl-2-pyrrolidone influenced the relative volatility of components to varying degrees. Two flowsheets containing a diethyl ether separation column and two extractive distillation complexes were proposed. The flowsheets differ in the sequence of separation of the components: hexane, ethyl acetate and ethanol. The distillation process was simulated and the operating column parameters (number of stages, feed stage, reflux ratio, solvent to feed volume ratio) were determined. Selected parameters meet the minimum energy consumption. An energy-efficient separation variant was chosen.*

**Key words:** dihydroquercetin, solvents mixture separation, extractive distillation, azeotrope, separation flowsheet

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## INTRODUCTION

Diethyl ether, hexane, ethyl acetate and ethanol are commonly used as organic solvents in chemical and pharmaceutical industries [1-3]. The quaternary system containing these components (40% mol. of diethyl ether, 16% mol. of hexane, 20% mol. of ethyl acetate, 24% mol. of ethanol) forms in the process of dihydroquercetin production and it needs to be separated and the components should be recycled in the extraction stages. However, this system contains three binary azeotropes and hence it cannot be separated using just conventional distillation. In order to obtain substances of commercial quality it is necessary to use special methods. One of the most effective methods for separating homogeneous azeotropic mixtures is extractive distillation [4-11]. The main task in this process is to find a solvent (extractive agent) that changes relative volatility of the original components. The same agent (depending on its amount) can have different effects on the relative volatility of components, which in turn can lead to polyvariance of the structure of the separation flowsheet.

The paper presents the results of the study of diethyl ether (DEE) + hexane (H) + ethyl acetate (EA) + ethanol (E) system phase diagram. Two flowsheets that differ in the sequence of component separation were proposed and the distillation columns operating parameters corresponding to the minimum energy consumption were determined.

## METHODS

Mathematical modelling of phase equilibrium of the diethyl ether + hexane + ethyl acetate + ethanol

quaternary system was based on the Non Random Two Liquid [12] equation using AspenPlus®.

$$\ln \gamma_i = \frac{\sum_j x_j \tau_{ji} G_{ji}}{\sum_k x_k G_{ki}} + \sum_j \frac{x_j G_{ij}}{\sum_k x_k G_{kj}} \left( \tau_{ij} - \frac{\sum_m x_m \tau_{mj} G_{mj}}{\sum_k x_k G_{kj}} \right)$$

where  $G_{ij} = \exp(-\alpha_{ij} \tau_{ij})$ ;

$$\tau_{ij} = a_{ij} + \frac{b_{ij}}{T} + e_{ij} \ln T + f_{ij} T;$$

$$\alpha_{ij} = c_{ij} + d_{ij}(T - 273.15K); \tau_{ii} = 0; G_{ii} = 1.$$

The parameters of NRTL model are given in table 1. The parameters  $a_{ij}$  ( $a_{ji}$ ) for binary constituents EA+E and DEE+E are -0.24 (-1.15) and -11.3 (11.7) respectively (for the other binary constituents they are zero). The relative uncertainties of vapor-liquid equilibrium description do not exceed 5%.

**Table 1**

**The parameters of NRTL model for DEE+H+EA+E system at 101.325 kPa**

**Таблица 1. Параметры модели NRTL для системы DEE+H+EA+E при 101,325 kPa**

System	$b_{ij}$	$b_{ji}$	$c_{ij}$
H+E	738.64	497.81	0.47
H+EA	228.01	120.16	0.3
EA+E	282.96	524.42	0.3
DEE+EA	211.08	-167.63	0.3
DEE+H	429.77	-223.22	0.3
DEE+E	3707.0	-3421.4	0.3

To check the adequacy of the mathematical modelling the experimental [13-15] and calculated azeotropic data (composition and boiling temperature)

have been compared (Table 2). The experimental data are: azeotrope H + E (58.45 °C;  $x_H = 66.25\%$  mol.); azeotrope H + EA (65.15 °C;  $x_H = 65.60\%$  mol.); azeotrope E + EA (71.80 °C;  $x_E = 45.25\%$  mol.).

Table 2

Calculated azeotropic data and relative uncertainties  
Таблица 2. Расчетные азеотропные данные и относительные ошибки

System 1-2	T, °C	$x_1$ , % mol.	$\Delta y$ , %	$\Delta T$ , %
H + E	58.35	66.18	0.17	0.11
H + EA	65.18	66.87	0.05	1.93
E + EA	71.81	44.67	0.01	1.28

The results obtained confirm the adequacy of the mathematical modelling and the possibility of using NRTL parameters (Table 1) for subsequent calculations.

RESULTS AND DISCUSSIONS

The phase diagram of DEE + H + EA + E quaternary mixture contains three binary azeotropes, two of which have an intermediate boiling point: EA + H and EA + E [16-17]. These azeotropes generate separate surfaces that divide composition tetrahedron into three distillation regions (Fig. 1 – the boiling points of the components are indicated in parentheses).

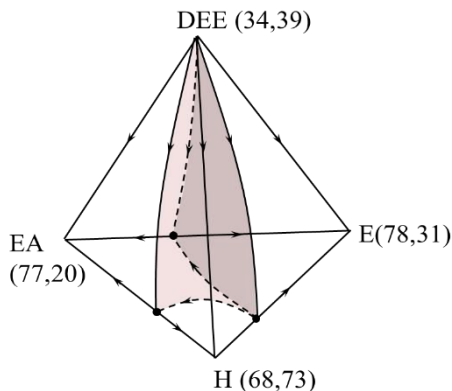


Fig. 1. Vapor-liquid equilibrium diagram of system DEE+H+EA+E

Рис. 1. Диаграмма парожидкостного равновесия системы DEE+H+EA+E

DEE does not form azeotropes with other components so it is possible to separate it using direct distillation in first column. Distillation was simulated with RadFrac routine of Aspen Plus. Sensitivity analysis was used to establish the operating conditions (number of stage (N), reflux ratio (R), mixture feed stage (NF)) for the column: N – 36; NF – 13; R = 2.7 (Feed (F) – 100 kmol/h). Theoretical stages were considered [18]. The purity of DEE (distillate flow) is 99.61% mol. Bottom flow contains mixture of the

next composition: 26.51% mol. of hexane, 39.89% mol. of ethyl acetate, 33.34% mol. of ethanol. The admixture of the diethyl ether in the mixture is 0.26% mol.

Extractive distillation was used to separate the mixture of H + EA + E. The analysis of the literature [6, 19-21] has shown N-methyl-2-pyrrolidone (NMP) can be used for this purpose. The missing parameters of NRTL model were taken from the AspenPlus database or were regressed using the UNIFAC model (for EA + NMP, DEE + NMP binary mixtures).

In order to predict the results of extractive distillation of H + EA + E ternary system with NMP the relative volatility of the three component pairs was calculated and the selectivity of the separating agent (SA) was determined by the formula:

$$S^{SA} = \frac{\alpha^{SA}}{\alpha_F}$$

where  $S^{SA}$  – the selectivity of SA;  $\alpha^{SA}$  ( $\alpha_F$ ) – the relative volatility of the components in the presence of SA (for the original ternary or binary mixture).

The selectivity of the NMP (solvent to feed volume ratio was 1/1) for component pairs H + E, H + EA and EA + E was 4.9, 3.6 and 1.4, respectively. It means that depending on the operating parameters of extractive distillation column it is possible to get H or binary mixture H + EA as a distillate product. Thus two flowsheets with different structure can be proposed (Fig. 2). Both flowsheets contain two extractive complexes: ED<sub>1</sub> – separation of H + EA + E ternary mixture and ED<sub>2</sub> – separation of EA + E (Fig. 2 (a)) or H + EA (Fig. 2 (b)) binary mixture. The selectivity of NMP is higher for H + EA than for EA + E (2.6 via 2.0).

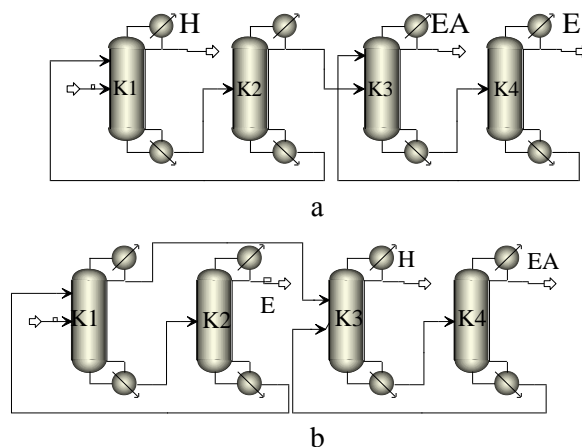


Fig. 2. Flowsheets of H+EA+E mixture separation: distillate of first column contains H (a) or binary mixture H+EA (b)  
Рис. 2. Схемы разделения смеси H+EA+E: поток дистиллята первой колонны содержит H (a) или смесь H+EA (b)

The distillation columns parameters of flowsheets proposed are given in Table 3 and Table 4 (the

components are listed in the following order: H, E, EA, DEE, NMP). The optimum parameters correspond to minimum energy consumption (Q). All columns work at atmospheric pressure.

Table 3

The distillation columns parameters for flowsheet presented in Fig. 2 (a)

Таблица 3. Параметры работы ректификационных колонн схемы, представленной на рис. 2 (а)

Column (SA/F)	N	N <sub>F</sub> (N <sub>SA</sub> )	R	Q, MWt
K1 (1/0.75)	29	22 (11)	0.9	0.36
K2	11	4	0.2	0.76
K3 (1/1.25)	30	19 (3)	2.0	0.75
K4	10	4	0.4	0.56
$\Sigma Q = 2.43 \text{ MWt}$				

Table 4

The distillation columns parameters for flowsheet presented in Fig. 2 (b)

Таблица 4. Параметры работы ректификационных колонн схемы, представленной на рис. 2 (б)

Column (EA/F)	N	N <sub>F</sub> (N <sub>EA</sub> )	R	Q, MWt
K1 (1/1)	30	19 (6)	1.0	0.87
K2	13	5	0.3	0.55
K3 (1/1)	29	21 (9)	0.6	0.32
K4	9	4	0.4	0.41
$\Sigma Q = 2.15 \text{ MWt}$				

The amount of separating agent depends on its selectivity: the higher the selectivity, the smaller the amount of solvent is required for mixture separation. The comparison of the energy costs for each

complex of extractive distillation (ED<sub>1</sub> and ED<sub>2</sub>) of flowsheets considered has shown that it is less energy-intensive to separate pure H than H + EA from the ternary mixture (ED<sub>1</sub>), but more energy-intensive extractive distillation of the binary mixture EA + E (compared to H + EA) (a greater amount of solvent is required and a higher reflux ratio of the first column is needed to reduce the NMP content in the distillate).

## CONCLUSIONS

Extractive distillation refers to energy-efficient methods for azeotropic mixtures separation. The disadvantage of the process is the addition of a new substance that can pollute the product streams. If all binary constituents of a homogeneous n-component mixture contain azeotropes, then (n-1) extractive distillation complexes are required to separate it. Each complex will contain two columns (an extractive distillation column and a separation agent regeneration column). If a single selective solvent, which affects the relative volatility of components in different ways, is used, several separation flowsheets of different structures (different sequence for the components isolation) can be proposed. It was illustrated on the example of separation of hexane + ethyl acetate + ethanol (after diethyl ether isolation) mixture by extractive distillation with N-methyl-2-pyrrolidone.

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