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RESEARCH ARTICLE

Extractive distillation of tetrahydrofuran–ethyl acetate–water mixture in schemes including columns with side sections and side draws

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Abstract

Objectives. The work set out to evaluate the energy efficiency of using schemes including columns with side sections and side draws in the extractive distillation of tetrahydrofuran–ethyl acetate–water mixture with dimethyl sulfoxide as an entrainer.

Methods. The main research method consisted of a computational experiment with the Aspen Plus v. 12 software package. The local composition UNIQUAC equation model was used for describing vapor–liquid equilibrium. Parametric optimization of initial scheme and schemes, including columns with side sections and side draws, was carried out according to the criterion of energy consumptions in distillation columns reboilers.

Results. Two variants of schemes including partially thermally coupled distillation columns and two variants of schemes including columns with side draws were synthesized on the basis of the conventional scheme of double extractive distillation consisting of two-withdrawal columns using the graph method. The optimal operating parameters of the conventional scheme and all schemes obtained on its basis were determined. The schemes, including columns with side draw, were modeled in two variants, namely, in the vapor phase with side draw, and in the liquid phase. The energy efficiency of the proposed schemes was evaluated in comparison with the conventional scheme.

Conclusions. The phase state of the side draw is shown to have little effect on the total energy consumption in column reboilers, the amount of liquid-phase side draw being 1.4–5.2 times greater than that of vapor-phase draw. Among the schemes including complex columns with a side section, the maximum reduction of energy consumption by 5.9% in relation to the scheme of two-withdrawal columns is provided by the scheme according to which the thermal coupling between the second extractive column and the regeneration column of the entrainer is realized. Thermal coupling of extractive columns provides a significantly lower energy saving (1.36%). Among the schemes including complex columns with side draw, the greatest energy efficiency (5.9%) is characterized by the scheme in which the draw in the vapor phase is taken from the second extractive column to the regeneration column.

Keywords

extractive distillation, side draws, side sections, tetrahydrofuran, ethyl acetate, water, energy saving

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НАУЧНАЯ СТАТЬЯ

Экстрактивная ректификация смеси тетрагидрофуран–этилацетат–вода в схемах, включающих колонны с боковыми секциями и отборами

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Аннотация

Цели. Провести оценку энергетической эффективности применения схем, включающих колонны с боковыми секциями и отборами, в процессе экстрактивной ректификации смеси тетрагидрофуран–этилацетат–вода с диметилсульфоксидом в качестве разделяющего агента.

Методы. Основным методом исследования являлся вычислительный эксперимент, реализуемый с применением программного комплекса Aspen Plus v. 12. Для моделирования парожидкостного равновесия было использовано уравнение локальных составов UNIQUAC. Параметрическая оптимизация всех рассмотренных в работе схем экстрактивной ректификации выполнялась по критерию суммарных энергетических затрат в кипятильниках колонн.

Результаты. С применением метода графов на основе базовой схемы двухступенчатой экстрактивной ректификации, состоящей из двухотборных колонн, синтезировано два варианта схем, включающих комплексы с частично связанными тепловыми и материальными потоками, и два варианта схем, включающих колонны с боковым отбором. Определены оптимальные рабочие параметры базовой схемы, а также всех полученных на ее основе схем. Схемы, включающие колонны с боковыми отборами, смоделированы в двух вариантах, а именно: с отбором бокового потока в паровой и в жидкой фазах. Проведена оценка энергоэффективности предложенных схем по сравнению с базовой схемой.

Выводы. Выявлено, что фазовое состояние бокового отбора мало влияет на суммарные энергозатраты в кипятильниках колонн, при этом количество жидкофазного бокового потока в 1.4–5.2 раза больше, чем парофазного. Установлено, что среди схем, включающих сложные колонны с боковой секцией, максимальное снижение энергозатрат на 5.9% относительно схемы из двухотборных колонн обеспечивает схема, в которой реализуется термическая связь между второй экстрактивной колонной и колонной регенерации разделяющего агента. Термическое связывание экстрактивных колонн дает существенно меньшую экономию энергозатрат (1.36%). Среди схем, включающих сложные колонны с боковым отбором, наибольшей энергоэффективностью (5.9%) характеризуется схема, в которой осуществляется отбор потока в паровой фазе из второй экстрактивной колонны в колонну регенерации.

Ключевые слова

экстрактивная ректификация, боковые отборы, боковые секции, тетрагидрофуран, этилацетат, вода, энергосбережение

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INTRODUCTION

Tetrahydrofuran (THF) and ethyl acetate (EAc) are widely used as solvents in the pharmaceutical industry. As a result of the technological process used to obtain these solvents, some pollutants get into wastewater [1]. Separation of THF–EAc–water mixture into pure components is difficult due to the presence of two binary azeotropes THF–water and EAc–water [2]. This mixture can be used to separated using extractive distillation (ED), which is the most widely used method for separating azeotropic mixtures and mixtures of components having relative volatility close to unity. However, like conventional distillation, ED has low thermodynamic efficiency, leading to high thermal energy consumption for its realization. This set of circumstances determines the relevance of searching for ways to improve the energy efficiency of the process [3–5].

One of the approaches to reduce energy consumption involves the transfer of heat removed from the strengthening section of the column to the distillation section of the same or another column, which is realized in schemes using heat pumps [6–8] and nonadiabatic distillation [9–11]. The same concept forms the basis for schemes using internal heat integration [11, 12] referred to as a heat integrated distillation column (HIDiC) system. However, despite their high energy efficiency, heat pump schemes are significant disadvantaged by the need to use expensive hot compressors.

Another approach is based on approximating the distillation process to a hypothetical thermodynamically reversible process using complexes with partially coupled heat and material flows, which are realized in practice in the form of complex columns with side sections [3, 13]. Another promising approach for reducing energy consumption involves the use of schemes including columns with side draws [14, 15]. The above-described methods can also be used in combination.

The authors [16] propose the use of complex columns with side draws to improve the energy efficiency of the scheme of two-stage ED of THF–EAc–water mixture with dimethyl sulfoxide (DMSO) as an entrainer (Fig. 1). Three schemes with side draws were considered: (1) a scheme with a side draw of the flow from the first extractive column (EC1) to the second; (2) with a side draw from the second extractive column (EC2) to the regeneration column (RC); (3) a scheme combining both previous options. In addition to this, a combination of schemes with side extraction and heat pump was studied. However, it should be noted that the authors considered all schemes with side flow draw exclusively in the liquid phase. Despite the block diagram of the optimization algorithm given in [16], it remains unclear according to what principle the transformation of the basic scheme into schemes with side draws was carried out, as well as on what basis the selection of initial conditions (number of the side draw plate and its number) in the optimization was made. The authors [16] also failed to take into account that DMSO begins to decompose at temperatures above 150°C [17] when selecting the working pressure in the columns. The top pressure of the DMSO RC was chosen to be 50 kPa at a temperature in the reboilers of 169°C. Thus, the results obtained by the authors of [16] require clarification and correction. Another interesting task consists in studying the energy efficiency of schemes with side flow draws in the vapor phase and comparing them with complexes having partially thermally coupled distillation columns (PTCDC).

Thus, the aim of the present study is to evaluate the energy efficiency of schemes including columns with side sections and columns with side flow draws in both vapor and liquid phases.

CALCULATIONS

Methods

Mathematical modeling and computational experiment were used as the main research method. In accordance with the recommendations of the authors [16], the UNIQUAC (UNIversal QUAsiChemical) equation was chosen as a model for describing the vapor–liquid equilibrium in the THF–EAc–water–DMSO system. For all binary components except for the THF–EAc pair, we used the equation parameters from the Aspen Properties v. 12.1 database integrated into the software package, while the parameters for the THF–EAc system were taken from the supplementary materials of the published work [16]. An analysis of the quality of description of experimental data on phase equilibrium [18–22] by UNIQUAC, NRTL (Non-Random Two Liquid) and UNIFAC (UNIQUAC Functional-group Activity Coefficients) models showed that the vapor phase composition and boiling point (or pressure in the case of isothermal data) of binary systems is described with the lowest error using the UNIQUAC model with the above parameters. The average relative errors in the description of vapor phase composition and boiling point (pressure in the case of isothermal data) do not exceed 3.0% and 0.5%, respectively.

The initial (basic) scheme of two-stage ED of THF–EAc–water mixture with DMSO as the entrainer is presented in Fig. 1 (Scheme A).

Calculations were carried out for 5900 kg/h of the initial mixture of the following composition: THF, 40.33 wt %; EAc, 49.28 wt %; water, 10.38 wt %. The feed flow was supplied at a temperature of 40°C and a pressure of 50 kPa. The concentrations of product flows were kept constant and equal for THF, 98.8 wt %; EAc, 99.5 wt %; water, 99.6 wt %; regenerated DMSO, 99.99 wt % [16].

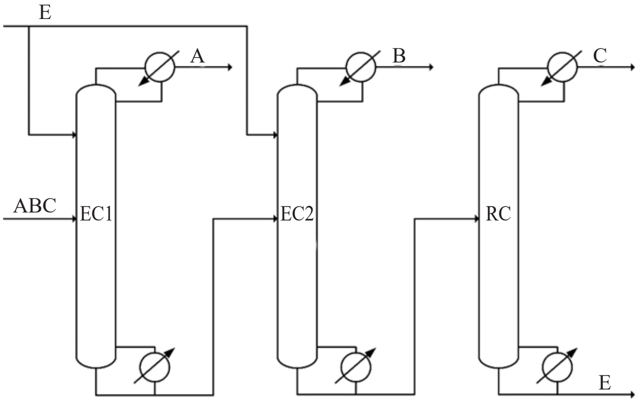


Fig. 1. Scheme of two-stage ED (Scheme A). EC1 and EC2 are extractive columns; RC is the entrainer regeneration column. Hereinafter: A is tetrahydrofuran, B is ethyl acetate, C is water, E is dimethyl sulfoxide

The pressure in the columns was chosen such that the temperature in the column bottoms did not exceed 150°C, since it is at this temperature that the decomposition of DMSO begins [17]. Thus, the pressure in columns EC1 and EC2 was set equal to 50 kPa, in RC—25 kPa. The optimized parameters were the number of theoretical plates in columns (N_{total}), temperature (T_{E}) and flow rate (F_{E}) of entrainer, position of feed plates (N_{F}) and entrainer (N_{E}) in columns. The total heat duty in the column reboilers was used as an optimization criterion ($\sum Q_{\text{reb}}$). The constraint on optimization consisted in the need to keep constant the concentrations of components in the product flow of the columns.

At the first stage, the influence of entrainer flow rate on the reflux ratio R in columns EC1 and EC2 at a fixed entrainer feed temperature (T_{E}) equal to 50°C was investigated. Based on the obtained dependence, the DMSO flow rate in EC1 equal to 4500 kg/h and in EC2 equal to 1150 kg/h was taken as an initial approximation. Then, the total number of plates (N_{total}) in the columns was determined at a fixed flow rate and entrainer temperature. After selecting N_{total}, the effect of T_{E} on the energy consumption in the ED column bottoms was investigated at a fixed total number of plates in the columns. This influence was found to be practically absent. Based on the top temperatures of columns EC1 and EC2, $T_{\text{E}} = 50$°C was chosen. At the next stage, the boundaries of variation of entrainer flow rates in each EC were determined (i.e., the values of the optimum and minimum flow rates). The final stage of optimization consisted in selecting the optimal amount of entrainer for the entire scheme. The optimization procedure was as follows:

1) setting values of N_{E} and N_{F} for EC1 and determining optimal values of entrainer flow rate for the entire scheme for all sets of N_{E} and N_{F} for EC2;
2) setting new values of N_{E} and N_{F} for EC1 and repeating step 1;
3) repeating steps 1–2 until reaching the minimum value of $\sum Q_{\text{reb}}$.

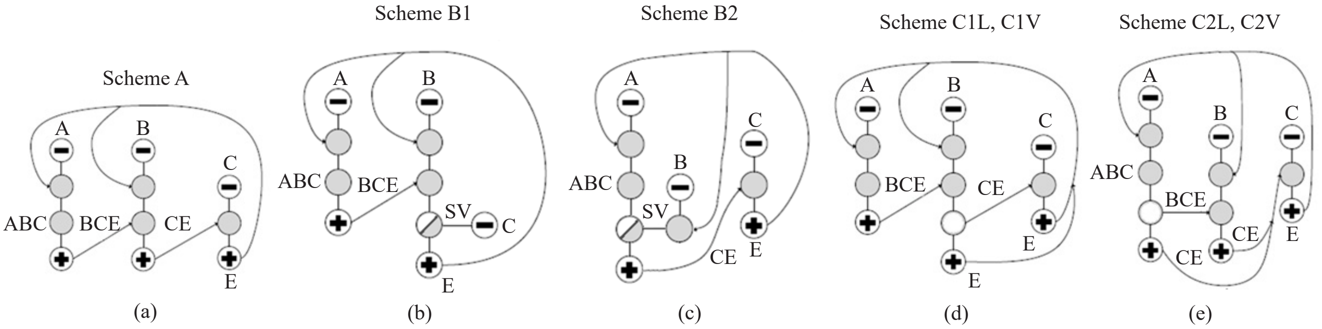
Optimal operating parameters of Scheme A are given in Table 1.

Synthesis of schemes including columns with side sections and side draws

The transformation of the graph of the basic two-stage ED scheme (Scheme A) into schemes including columns with side sections and side draws is shown in Fig. 2.

Table 1. Optimal operating parameters of Scheme A

Column	N_{total}	N_{E}	N_{F}	T_{E}, °C	F_{E}, kg/h	R	Q_{reb}, kW	$\sum Q_{\text{reb}}$, kW
EC1	46	10	16	50	6614	6.40	2417	3831
EC2	19	5	14	50	988	0.30	656	
RC	13	–	5	–	–	0.75	758	


Scheme A
Scheme B1
Scheme B2
Scheme C1L, C1V
Scheme C2L, C2V

(a) (b) (c) (d) (e)

Fig. 2. Transformation of the base scheme graph: (a) graph of Scheme A, (b) graph of Scheme B1, (c) graph of Scheme B2, (d) graphs of Schemes C1L and C1V, (e) graphs of Schemes C2L and C2V (letter L means side draw in the liquid phase, and letter V—in the vapor phase)

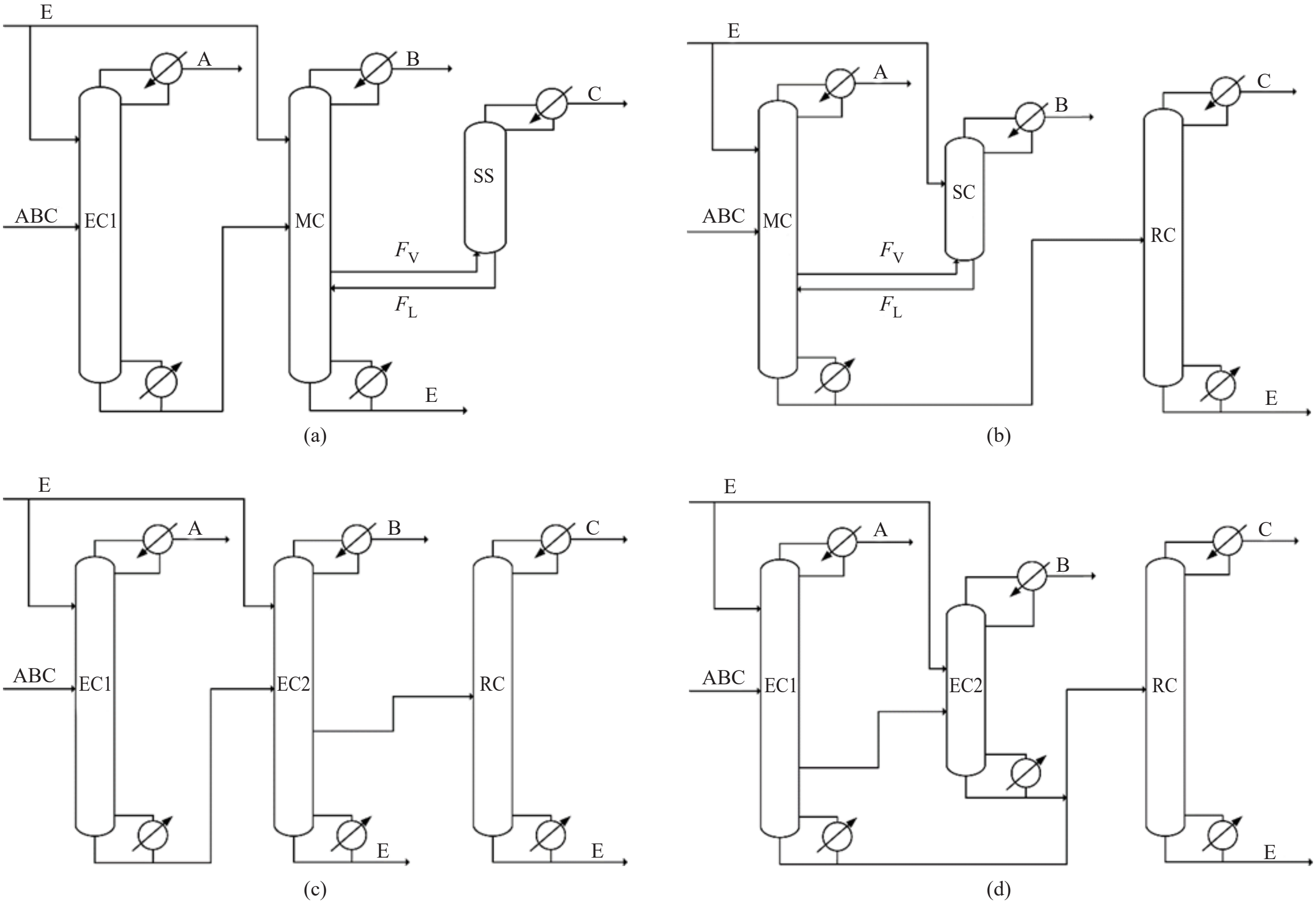
The initial scheme (Fig. 1) is transformed into a graph (Fig. 2a). In this graph, edges denote vapor and liquid flows within and between columns. The vertices are the cross sections bounding the column sections. By combining two vertices adjacent along the oriented edges CE and BCE, the graph can be simplified to the graphs shown in Figs. 2b and 2c, which depict schemes having partially coupled heat and material flows. Performing the simplification operation on the graphs (Figs. 2b and 2c) along the undirected edges of the side vapor (SV) extraction phase results in graphs (Figs. 2d and 2e) that display schemes involving complex columns with side extraction in both vapor (C1V and C2V) and liquid (C1L and C2L) phases. The side draws are located downstream of the extractive section in the respective columns. Thus, four different variants of schemes including complex columns were synthesized from the basic Scheme A (Fig. 3):

- Scheme B1, in which columns EC2 and RC are combined in a complex with PTCDC;
- Scheme B2, in which columns EC1 and EC2 are combined in a complex with PTCDC;
- Schemes C1L and C1V are obtained on the basis of Scheme B1, in them side flow draw in vapor phase (C1V) or in liquid phase (C1L) is organized from EC2 to RC;
- Schemes C2L and C2V are obtained on the basis of Scheme B2, in them from column EC1 to column EC2 the side flow draw in steam phase (C2V) or in liquid phase (C2L) is organized.

At the next stage, the optimal operating parameters of the synthesized variants of schemes were determined. Initial data and product flow quality was set the same as for Scheme A. Total heat duty in the column reboilers was used as an optimization criterion as follows.

Optimization of schemes including complexes with PTCDC

At transition to complexes with PTCDC, the pressure in the integrated apparatuses should be equalized. In Scheme B1, the pressure in column EC1 and the basic Scheme A is 50 kPa; in the main column of the PTCDC complex and side section, the pressure was set equal



(a) (b) (c) (d)

Fig. 3. Schemes involving columns with side sections and side draws: (a) Scheme B1, (b) Scheme B2, (c) Scheme C1L/C1V, (d) Scheme C2L/C2V. EC1 and EC2 are extractive columns; RC is the entrainer regeneration column; MC is the main column of the complex with partially thermally coupled distillation columns; SS is a side section; SC is a side column

to 25 kPa. In Scheme B2, the pressure in the main column and side section was set equal to 50 kPa; in the RC, as well as in the RC of the basic Scheme A, the corresponding pressure was 25 kPa. The optimized parameters were the amount of steam flow extraction into the side section (F_V) and the side draw plate location (N_{SD}). Other parameters such as temperature (T_E), entrainer flow rate (F_E), position of feed plates (N_F) in the columns, and entrainer inlet plates (N_E) were fixed according to their optimum values determined for Scheme A.

Since there were no changes in the mode of operation of columns EC1 (for Scheme B1) and CR (for Scheme B2), these parameters were not reoptimized.

Optimization of complexes with PTCDC was carried out according to the following algorithm:

1) setting the N_{SD};
2) setting the F_V;
3) calculating the value of heat duty in the reboilers of the main column Q_{reb}^{MC};
4) returning to step 2, setting a new F_V value, controlling the distillate quality of the side section (or side column) by changing the distillate flow rate F_D^{SS} (F_D^{SC}), until Q_{reb}^{MC} reaches the minimum value;
5) returning to step 1, setting a new N_{SD} value. Repeating steps 1–4 until the minimum value of Q_{reb}^{MC} is reached.

The optimization results of Schemes B1 and B2 are shown in Table 2.

Optimization of schemes including columns with side draw

Scheme C1 group. Since Schemes C1L and C1V are obtained by transforming the graph of Scheme B1 and differ only in the aggregate state of the side extraction flow, the procedure of their optimization is identical. Because the composition of the feed flow of this column in Schemes C1L and C1V differs from the composition in the basic Scheme A, the number of plates in the RC was selected at the first stage. The selection was based on an analysis of the dependence Q_{reb}^{RC} on the number of plates.

Additional optimized variables comprised the amount of side liquid extraction flow FL (Scheme C1L) or steam extraction F_V (Scheme C1V) from EC2 to the RC, as well as the location of the side extraction plate (N_{SD}) and the feed plate in the RC (N_F).

Other parameters, such as the number of plates in EC1 and EC2, entrainer temperature and flow rate, position of feed and extracting agent feed plates in EC1 and EC2, were set the same as in Scheme B1. The optimization criterion (target function) was the minimum of the total duty on the reboilers of columns EC2 (Q_{reb}^{EC2}) and CR (Q_{reb}^{RC}). The optimization procedure was as follows:

1) setting the N_{SD};
2) setting the amount of side flow F_L (or F_V);
3) determining the optimum feed plate in RC N_F^{RC};
4) repeating steps 2–3 until the minimum value of $\sum Q_{reb} = Q_{reb}^{EC2} + Q_{reb}^{RC}$ is reached;
5) returning to step 1;
6) repeating steps 1–5 before reaching the minimum value of $\sum Q_{reb}$.

The optimization results of Schemes C1L and C1V are shown in Table 3.

Scheme C2 group. Since Schemes C2L and C2V are obtained by transforming the graph of Scheme B2 and differ only in the aggregate state of the side flow draw,

Table 2. Optimal operating parameters of Schemes B1 and B2

Column	N_{total}	N_E	N_F	N_{SD}	F_E, kg/h	F_V, kg/h	R	Q_{reb}, kW	$\sum Q_{reb}$, kW
Scheme B1									
EC1	46	10	16	–	6614	–	6.40	2417	3604
Main column	27	5	14	19	988	972	0.25	1187	
Side section	5	–	–	–	–	–	0.25	–	
Scheme B2									
Main column	51	10	16	48	6614	3423	6.16	3021	3779
Side column	14	5	–	–	988	–	0.36	–	
RC	13	–	5	–	–	–	0.75	758	

Table 3. Optimal operating parameters of Schemes C1L and C1V

Column	N_{total}	N_{E}	N_{F}	N_{SD}	F_{E}, kg/h	F_{L} (F_{V}), kg/h	R	Q_{reb}, kW	$\sum Q_{\text{reb}}$, kW
Scheme C1L									
EC1	46	10	16	–	6614	–	6.40	2417	3663
EC2	27	5	14	19	988	4650	0.25	564	
RC	11	–	4	–	–	–	0.40	682	
Scheme C1V									
EC1	46	10	16	–	6614	–	6.40	2417	3604
EC2	27	5	14	19	988	899	0.25	1139	
RC	13	–	6	–	–	–	0.26	48	

the procedure of their optimization is identical. Because the composition of its feed differs from the composition in Scheme A, the number of plates in EC2 was selected at the first stage. The selection of the number of plates was performed at fixed entrainer flow rates in EC1 and EC2, as well as the position of the side extraction plate and its number. Further optimized variables were the amount of side liquid extraction flow F_{L} (Scheme C2L) or steam extraction F_{V} (Scheme C2V) from EC1 to EC2, the position of the side extraction plate (N_{SD}). As calculations have shown, the composition of the RC feed flow in Schemes C2L and C2V in comparison with Scheme A practically did not change; for this reason, optimization of parameters of this column was not carried out.

The further optimization procedure was as follows:

1) setting the N_{SD} in EC1;
2) setting the amount of side flow F_{L} (or F_{V});
3) variating N_{SD} and N_{F} in EC2 until reaching the minimum value of $Q_{\text{reb}}^{\text{EC2}}$;
4) specifying the position of N_{F} in the RC;
5) calculating $\sum Q_{\text{reb}}$ for the entire scheme;
6) returning to step 2;
7) repeating steps 2–6 until reaching the minimum value of $\sum Q_{\text{reb}}$;
8) returning to step 1;
9) repeating steps 1–8 until reaching the minimum value of $\sum Q_{\text{reb}}$.

The optimization results of Schemes C2L and C2V are shown in Table 4.

Table 4. Optimal operating parameters of Schemes C2L and C2V

Column	N_{total}	N_{E}	N_{F}	N_{SD}	F_{E}, kg/h	F_{L} (F_{V}), kg/h	R	Q_{reb}, kW	$\sum Q_{\text{reb}}$, kW
Scheme C2L									
EC1	51	10	16	48	6614	4250	6.20	2549	3800
EC2	15	4	12	–	988	–	0.36	491	
RC	13	–	5	–	–	–	0.77	760	
Scheme C2V									
EC1	51	10	16	47	6614	3008	7.62	3357	4192
EC2	16	4	10	–	988	–	0.29	73	
RC	13	–	5	–	–	–	0.78	762	

RESULTS AND DISCUSSION

Six different variants of the schemes of ED of THF–EAc–water mixture, including columns with side sections and side draws, were considered in the course of the conducted studies. Comparison of the proposed scheme variants with the prototype scheme (Scheme A) by the criterion of total energy consumption is presented in Table 5. Reduction of energy consumption ΔQ_{reb} was calculated by the formula:

$$\Delta Q_{\text{reb}} = \left(\sum Q_{\text{reb}}^{\text{A}} - \sum Q_{\text{reb}}^{i}\right) / \sum Q_{\text{reb}}^{\text{A}} \times 100\%, \qquad (1)$$

where $\sum Q_{\text{reb}}^{\text{A}}$ is the total energy consumption in the column reboilers of Scheme A, $\sum Q_{\text{reb}}^{i}$ is the total energy consumption in the reboilers of columns of the scheme including a complex with PTCDC or a column with side extraction.

Among the schemes including complexes with PTCDC, it can be seen that the maximum reduction of energy consumption by 5.9% relative to the prototype Scheme A is provided by Scheme B1, in which the complex with PTCDC is obtained by thermal bonding of columns EC2 and RC of Scheme A. Thermal bonding of columns EC1 and EC2 in Scheme B2 gives significantly lower energy savings (1.36%). The different energy efficiency of Schemes B1 and B2 is explained by different reflux ratios in columns RC and EC2 of Scheme A, which are prototypes of the side section (side column) in Schemes B1 and B2, respectively: $R^{\text{RC}} = 0.75$ and $R^{\text{EC2}} = 0.3$. Thus, the obtained results are consistent with the criterion for evaluating the energy efficiency of the application of complexes with PTCDC in the ED.[1] It should also be noted that the reflux ratio in the side section of Scheme B1 is three times lower than the reflux ratio in the RC of Scheme A. This can be explained by the fact that the concentration of water in the steam flow entering the side section is 9 times higher than in the feed flow of the RC of Scheme A. The reflux ratio in the side column of Scheme B2 has a slightly higher value as compared to the reflux ratio in EC2 of Scheme A. This is explained by the fact that about three times more THF falls in the side section than in the bottom flow of EC1; thus, to obtain EAc of a given quality in the distillate of the side column, a slightly higher reflux ratio is required.

Scheme C1V is characterized by the same energy efficiency as Scheme B1.

In Scheme C1V, the reduction of total energy consumption occurs mainly due to a significant reduction in the duty on the reboilers of the RC (by 94%, compared to the RC of Scheme A). This is due to several reasons. First, the amount of RC feed flow in Scheme C1V is less than in Scheme A due to most of the DMSO in Scheme C1V being released in the column bottom with side extraction. Secondly, the water concentration in the RC feed flow in Scheme C1V is about 70 wt %, while in Scheme A it is 8 wt %. Thirdly, the Scheme C1V RC feed is supplied in the vapor phase, while the Scheme A RC feed is supplied in the liquid phase.

The energy efficiency of Scheme C1L is less than that of Scheme C1V. This is due to the amount of liquid side draw being 5.2 times greater than that of vapor side draw, while the concentration of DMSO in the liquid-phase side flow is already 86 wt % (instead of 30 wt % for the Scheme C1V). Thus, the RC feed flow in Scheme C1L is substantially greater than in Scheme C1V. In addition, the feed to the RC of Scheme C1L is supplied in the liquid phase, so there is no additional source of steam in this column.

The insignificant total reduction of energy consumption (0.81%) in Scheme C2L can be explained by the small reduction in energy consumption in the EC2 reboilers due to the heat contributed by the entrance into it of the withdrawal flow from EC1, which also leads to an increase in the concentration of DMSO in the column. However, energy costs increase by about the same amount in the reboilers of EC1 due to the side extraction flow carrying a certain amount of heat with it, which reduces the concentration of DMSO. Thus, the total positive energy effect is absent in this case due to a redistribution of duty on the reboilers of columns EC1 and EC2.

In Scheme C2P, total energy consumption increases by 9.4% as compared to the basic Scheme A. This significant

Table 5. Comparison of total energy consumption of the considered schemes

Parameter	Scheme A	Scheme B1	Scheme B2	Scheme C1L	Scheme C1V	Scheme C2L	Scheme C2V
$\sum Q_{\text{reb}}$, kW	3831	3604	3779	3663	3604	3800	4192
ΔQ_{reb}, %	–	5.9	1.36	4.4	5.9	0.8	−9.4

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[1] Anokhina E.A. Extractive distillation in complexes with partially coupled heat and material flows. Dr. Sci. Thesis (Eng.). Moscow; 2020. 549 p. (in Russ.).]

increase in energy consumption is explained by the flow of side extraction in the vapor phase reflux ratio $R = 6.40$ (as in EC1 of Schemes A and C1V) containing such a quantity of THF that prevents the extraction of EAc of a given quality in EC2. Thus, in order to maintain the required concentration of EAc in EC2, it is necessary to increase the reflux ratio (up to 7.62) in EC1, which leads to a significant increase in the duty on its reboilers. In addition, the steam flow of the side extraction carries a significant amount of heat, which requires replenishing.

CONCLUSIONS

Thus, it can be concluded that the organization of side draw from one EC to another in two-stage ED THF–EAc–water mixture schemes does not lead to a reduction of total energy consumption. Conversely, the organization of side draw from EC to CR can reduce the total energy consumption in the column reboilers by 4.4% at side draw in the liquid phase and by 5.9% at draw in the vapor phase. It should be noted that Scheme B1, which includes a complex with PTCDC obtained by thermal bonding of EC and RC, as well as Scheme C1V, in which draw in the vapor phase from EC to RC is carried out, are characterized by the same energy efficiency (5.9%). The final choice of the variant for practical implementation can be made after modeling the dynamic behavior of these schemes.

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Authors' contribution

D.G. Rudakov—planning and conducting research, analyzing research materials, writing the manuscript.

S.M. Kharlamov—conducting research, analyzing research materials.

P.S. Klauzner—analyzing research materials, writing the manuscript.

E.A. Anokhina—management and scientific consulting, analyzing research materials.

A.V. Timoshenko—formulation of the scientific concept, general management.

The authors declare no conflicts of interest.

REFERENCES

1. Wittmann G., Karg E., Mühl A., Bodamer O.A., Túri S. Comparison of tetrahydrofuran and ethyl acetate as extraction solvents for urinary organic acid analysis. *J. Inherit. Metab. Dis.* 2008;31(1):73–80. https://doi.org/10.1007/s10545-007-0767-8
2. Yang A., Su Y., Shi T., *et al.* Energy-efficient recovery of tetrahydrofuran and ethyl acetate by triple-column extractive distillation: entrainer design and process optimization. *Front. Chem. Sci. Eng.* 2022;16(5):303–315. https://doi.org/10.1007/s11705-021-2044-z
3. Timoshenko A.V., Anokhina E.A., Morgunov A.V., Rudakov D.G. Application of the partially thermally coupled distillation flowsheets for the extractive distillation of ternary azeotropic mixtures. Chem. *Eng. Res. Des.* 2015;104(3):139–155. https://doi.org/10.1016/j.cherd.2015.07.007
4. Gerbaud V., Rodriguez-Donis I., Hegely L., Lang P., Denes F., You X.-Q. Review of extractive distillation. Process design, operation, optimization and control. *Chem. Eng. Res. Des.* 2019;141:229–271. https://doi.org/10.1016/j.cherd.2018.09.020
5. Valdez J.A., Patino-Herrera R., Martinez A.A., Perez E. Separation of the cyclohexane-benzene mixture by the extractive distillation process using ethylene glycol as a solvent. *Chem. Eng. Process.* 2024;196:109686. https://doi.org/10.1016/j.cep.2024.109686
6. Rudakov D.G., Klausner P.S., Ramochnikov D.A., Anokhina E.A., Timoshenko A.V. Efficiency of heat pumps in extractive rectification of the allyl alcohol–allyl acetate mixture with different compositions of the feed: Part 1. Application of heat pumps in schemes with two-selection columns. *Theor. Found. Chem. Eng.* 2023;57(4):770–778. https://doi.org/10.1134/S0040579523040231

СПИСОК ЛИТЕРАТУРЫ

1. Wittmann G., Karg E., Mühl A., Bodamer O.A., Túri S. Comparison of tetrahydrofuran and ethyl acetate as extraction solvents for urinary organic acid analysis. *J. Inherit. Metab. Dis.* 2008;31(1):73–80. https://doi.org/10.1007/s10545-007-0767-8
2. Yang A., Su Y., Shi T., *et al.* Energy-efficient recovery of tetrahydrofuran and ethyl acetate by triple-column extractive distillation: entrainer design and process optimization. *Front. Chem. Sci. Eng.* 2022;16(5):303–315. https://doi.org/10.1007/s11705-021-2044-z
3. Timoshenko A.V., Anokhina E.A., Morgunov A.V., Rudakov D.G. Application of the partially thermally coupled distillation flowsheets for the extractive distillation of ternary azeotropic mixtures. *Chem. Eng. Res. Des.* 2015;104(3):139–155. https://doi.org/10.1016/j.cherd.2015.07.007
4. Gerbaud V., Rodriguez-Donis I., Hegely L., Lang P., Denes F., You X.-Q. Review of extractive distillation. Process design, operation, optimization and control. *Chem. Eng. Res. Des.* 2019;141:229–271. https://doi.org/10.1016/j.cherd.2018.09.020
5. Valdez J.A., Patino-Herrera R., Martinez A.A., Perez E. Separation of the cyclohexane-benzene mixture by the extractive distillation process using ethylene glycol as a solvent. *Chem. Eng. Process.* 2024;196:109686. https://doi.org/10.1016/j.cep.2024.109686
6. Rudakov D.G., Klausner P.S., Ramochnikov D.A., Anokhina E.A., Timoshenko A.V. Efficiency of heat pumps in extractive rectification of the allyl alcohol–allyl acetate mixture with different compositions of the feed: Part 1. Application of heat pumps in schemes with two-selection columns. *Theor. Found. Chem. Eng.* 2023;57(4):770–778. https://doi.org/10.1134/S0040579523040231

7. Ma J., Wang X., Zhou Y., Du Y., Wang B. Energy-saving process development for the purification of propylene glycol based on MVR heat pump distillation combined with thermally coupled technology. *Sep. Purif. Technol*. 2024;329:125064. https://doi.org/10.1016/j.seppur.2023.125064
8. Wu T., Wang C., Liu J., Zhuang Y., Du J. Design and 4E analysis of heat pump-assisted extractive distillation processes with preconcentration for recovering ethyl-acetate and ethanol from wastewater. *Chem. Eng. Res. Des*. 2024;201:510–522. https://doi.org/10.1016/j.cherd.2023.12.011
9. Klauzner P.S., Rudakov D.G., Anokhina E.A., Timoshenko A.V. Energy efficiency of diabatic distillation schemes for an acetone–toluene–*n*-butanol mixture with an entrainer in the first column. *Tonk. Khem. Tekhnol. = Fine Chem. Technol.* 2023;18(1):7–20. https://doi.org/10.32362/2410-6593-2023-18-1-7-20
10. Voinov N.A., Zemtsov D.A., Deryagina N.V., Bogatkova A.V., Zhukova O.P. A study of diabatic distillation in a column with a low pressure drop. *Chem. Eng. Res. Des.* 2022;185:1–13. https://doi.org/10.1016/j.cherd.2022.06.033
11. Fang J., Li Z., Huang G., Li H., Li C. Externally heat-integrated multiple diabatic distillation columns (EHIm$_x$DC): Basic concept and general characteristics. *Ind. Eng. Chem. Res.* 2020;59(4):1668–1681. https://doi.org/10.1021/acs.iecr.9b05841
12. Gutiérrez-Guerra R., Segovia-Hernández J.G. Novel approach to design and optimize heat-integrated distillation columns using Aspen Plus and an optimization algorithm. *Chem. Eng. Res. Des.* 2023;196:13–27. https://doi.org/10.1016/j.cherd.2023.06.015
13. Pan C., Qiu X., Guo J., Liu Y., Feng B., Li G., Gai H., Song H., Xiao M., Huang T., Zhu Q. Efficient purification of *n*-butanol by thermally coupled extractive distillation with mixed entrainer. *Sep. Purif. Technol*. 2024;336:126365. https://doi.org/10.1016/j.seppur.2024.126365
14. Anokhina E.A., Yakutin R.I., Timoshenko A.V. Purifying benzene from thiophene by extractive distillation using columns with side withdrawal in the vapor phase. *Theor. Found. Chem. Eng.* 2021;55(5):880–887. https://doi.org/10.1134/S0040579521040205 [Original Russian Text: Anokhina E.A., Yakutin R.I., Timoshenko A.V. Purifying benzene from thiophene by extractive distillation using columns with side withdrawal in the vapor phase. *Teoreticheskie osnovy khimicheskoi tekhnologii*. 2021;55(5):578–586 (in Russ.). https://doi.org/10.31857/S0040357121040011]
15. Sun S., Fu L., Yang A., Shen W. An intensified energy-saving architecture for side-stream extractive distillation of four-azeotrope mixtures considering economic, environmental and safety criteria simultaneously. *Sep. Purif. Technol*. 2023;310:123132. https://doi.org/10.1016/j.seppur.2023.123132
16. Yang A., Kong Z.Y., Sunarso J., Su Y., Wang G., Zhu S. Insights on sustainable separation of ternary azeotropic mixture tetrahydrofuran/ethyl acetate/water using hybrid vapor recompression assisted side-stream extractive distillation. *Sep. Purif. Technol*. 2022;290:120884. https://doi.org/10.1016/j.seppur.2022.120884
17. Head D.L., McCart C.G. The thermal decomposition of DMSO. *Tetrahedron Lett*. 1973;14(16):1405–1408. https://doi.org/10.1016/S0040-4039(01)95955-6
18. Yang J., Pan X., Yu M., Cui P., Ma Y., Wang Y., Gao J. Vapor-liquid equilibrium for binary and ternary systems of tetrahydrofuran, ethyl acetate and *N*-methyl pyrrolidone at pressure 101.3 kPa. *J. Mol. Liq.* 2018;268:19–25. https://doi.org/10.1016/j.molliq.2018.07.038
19. Xie Q., Wan H., Guan G.F. Isobaric vapor-liquid equilibrium for tetrahydrofuran-water-*n*-hexane system. *Chem. Eng.* 2010;38(4):61–72.

7. Ma J., Wang X., Zhou Y., Du Y., Wang B. Energy-saving process development for the purification of propylene glycol based on MVR heat pump distillation combined with thermally coupled technology. *Sep. Purif. Technol*. 2024;329:125064. https://doi.org/10.1016/j.seppur.2023.125064
8. Wu T., Wang C., Liu J., Zhuang Y., Du J. Design and 4E analysis of heat pump-assisted extractive distillation processes with preconcentration for recovering ethyl-acetate and ethanol from wastewater. *Chem. Eng. Res. Des*. 2024;201:510–522. https://doi.org/10.1016/j.cherd.2023.12.011
9. Клаузнер П.С., Рудаков Д.Г., Анохина Е.А., Тимошенко А.В. Оценка энергетической эффективности схем неадиабатической ректификации смеси ацетон–толуол–*н*-бутанол с использованием экстрактивного агента в первой колонне. *Тонкие химические технологии*. 2023;18(1):7–20. https://doi.org/10.32362/2410-6593-2023-18-1-7-20
10. Voinov N.A., Zemtsov D.A., Deryagina N.V., Bogatkova A.V., Zhukova O.P. A study of diabatic distillation in a column with a low pressure drop. *Chem. Eng. Res. Des.* 2022;185:1–13. https://doi.org/10.1016/j.cherd.2022.06.033
11. Fang J., Li Z., Huang G., Li H., Li C. Externally heat-integrated multiple diabatic distillation columns (EHIm$_x$DC): Basic concept and general characteristics. *Ind. Eng. Chem. Res.* 2020;59(4):1668–1681. https://doi.org/10.1021/acs.iecr.9b05841
12. Gutiérrez-Guerra R., Segovia-Hernández J.G. Novel approach to design and optimize heat-integrated distillation columns using Aspen Plus and an optimization algorithm. *Chem. Eng. Res. Des.* 2023;196:13–27. https://doi.org/10.1016/j.cherd.2023.06.015
13. Pan C., Qiu X., Guo J., Liu Y., Feng B., Li G., Gai H., Song H., Xiao M., Huang T., Zhu Q. Efficient purification of *n*-butanol by thermally coupled extractive distillation with mixed entrainer. *Sep. Purif. Technol*. 2024;336:126365. https://doi.org/10.1016/j.seppur.2024.126365
14. Анохина Е.А., Якутин Р.И., Тимошенко А.В. Очистка бензола от тиофена экстрактивной ректификацией с применением колонн с боковым отбором в паровой фазе. *Теоретические основы химической технологии*. 2021;55(5):578–586. https://doi.org/10.31857/S0040357121040011
15. Sun S., Fu L., Yang A., Shen W. An intensified energy-saving architecture for side-stream extractive distillation of four-azeotrope mixtures considering economic, environmental and safety criteria simultaneously. *Sep. Purif. Technol*. 2023;310:123132. https://doi.org/10.1016/j.seppur.2023.123132
16. Yang A., Kong Z.Y., Sunarso J., Su Y., Wang G., Zhu S. Insights on sustainable separation of ternary azeotropic mixture tetrahydrofuran/ethyl acetate/water using hybrid vapor recompression assisted side-stream extractive distillation. *Sep. Purif. Technol*. 2022;290:120884. https://doi.org/10.1016/j.seppur.2022.120884
17. Head D.L., McCart C.G. The thermal decomposition of DMSO. *Tetrahedron Lett*. 1973;14(16):1405–1408. https://doi.org/10.1016/S0040-4039(01)95955-6
18. Yang J., Pan X., Yu M., Cui P., Ma Y., Wang Y., Gao J. Vapor-liquid equilibrium for binary and ternary systems of tetrahydrofuran, ethyl acetate and *N*-methyl pyrrolidone at pressure 101.3 kPa. *J. Mol. Liq.* 2018;268:19–25. https://doi.org/10.1016/j.molliq.2018.07.038
19. Xie Q., Wan H., Guan G.F. Isobaric vapor-liquid equilibrium for tetrahydrofuran-water-*n*-hexane system. *Chem. Eng.* 2010;38(4):61–72.

20. Rajedran M., Renganarayanan S., Srinivasan D. Salt effect in phase equilibria and heat of mixing: effect of dissolved inorganic salts on the liquid-liquid equilibria of ethyl acetate-2-propanol-water system and the vapor-liquid equilibria and heat of mixing of its constituent binaries. *Fluid Ph. Equilib.* 1991;70(1):65–106. https://doi.org/10.1016/0378-3812(91)85005-F
21. Sassa Y., Konishi R., Katayama T. Isothermal vapor-liquid equilibrium data of DMSO [dimethyl sulfoxide] solutions by total pressure method. DMSO-acetone, DMSO-tetrahydrofuran, and DMSO-ethyl acetate systems. *J. Chem. Eng. Data.* 1974;19(1):44–48. https://doi.org/10.1021/je60060a004
22. Zhang Z., Lv M., Huang D., Jia P., Sun D., Li W. Isobaric Vapor Liquid Equilibrium for the Extractive Distillation of Acetonitrile + Water Mixtures Using Dimethyl Sulfoxide at 101.3 kPa. *J. Chem. Eng. Data*. 2013;58(12):3364–3369. https://doi.org/10.1021/je400531a

20. Rajedran M., Renganarayanan S., Srinivasan D. Salt effect in phase equilibria and heat of mixing: effect of dissolved inorganic salts on the liquid-liquid equilibria of ethyl acetate-2-propanol-water system and the vapor-liquid equilibria and heat of mixing of its constituent binaries. *Fluid Ph. Equilib.* 1991;70(1):65–106. https://doi.org/10.1016/0378-3812(91)85005-F
21. Sassa Y., Konishi R., Katayama T. Isothermal vapor-liquid equilibrium data of DMSO [dimethyl sulfoxide] solutions by total pressure method. DMSO-acetone, DMSO-tetrahydrofuran, and DMSO-ethyl acetate systems. *J. Chem. Eng. Data*. 1974;19(1):44–48. https://doi.org/10.1021/je60060a004
22. Zhang Z., Lv M., Huang D., Jia P., Sun D., Li W. Isobaric Vapor Liquid Equilibrium for the Extractive Distillation of Acetonitrile + Water Mixtures Using Dimethyl Sulfoxide at 101.3 kPa. *J. Chem. Eng. Data*. 2013;58(12):3364–3369. https://doi.org/10.1021/je400531a

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