DOI: 10.6060/ivkkt.20236601.6670

УДК: 66.011:66.048.3

РАЗРАБОТКА ТЕХНОЛОГИИ РАЗДЕЛЕНИЯ СПИРТОВОЙ СМЕСИ РАСТВОРИТЕЛЕЙ

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Статья посвящена разработке технологии регенерации растворителей производства капролактама из смеси, содержащей н-бутанол, амилол, циклогексанон, циклогексанол, а также изоамилол и циклопентанон в примесных количествах. Для исследования фазового равновесия и процесса разделения выбран вычислительный эксперимент с использованием 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%. Разделение смеси обычной ректификацией затруднительно, что вызвано наличием трех бинарных азеотропов, а также трех пар компонентов, характеризующихся близкими летучестями. При уменьшении давления наблюдается усложнение структуры фазовой диаграммы из-за появления новых азеотропов. Предложена технологическая схема регенерации растворителей, основанная на использовании непрерывной ректификации. Схема содержит пять колонн. На первом этапе предполагается предварительное фракционирование смеси (режим промежуточного заданного разделения в первой колонне) с полным выделением в кубе циклогексанона и циклогексанола (для разделения данной пары используется ректификация при пониженном давлении, в дистилляте - смеси бутанола и амилола с примесными компонентами. Для очистки бутанола и амилола от примесей предложена экстрактивная ректификация с этиленгликолем. В присутствии последнего повышается летучесть бутанола по отношению к другим компонентам. Четвертая колонна предназначена для регенерации разделяющего агента. Отделение амилола предусмотрено в кубе пятой колонны. Определены статические параметры работы колонн (число тарелок, тарелка питания, флегмовое число, соотношение количеств исходной смеси и разделяющего агента), отвечающие минимальным энергозатратам и обеспечивающие получение веществ товарного качества.

Ключевые слова: смесь растворителей, бутанол, циклогексанон, циклогексанол, амилол, азеотроп, схема разделения, экстрактивная ректификация

DEVELOPMENT OF SOLVENTS ALCOHOL MIXTURE SEPARATION TECHNOLOGY

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The paper is devoted to the development of a technology for the regeneration of caprolactam solvents containing n-butanol, amyl alcohol, cyclohexanone, cyclohexanol, as well as isoamyl alcohol and cyclopentanone in impurity amounts. To study the phase equilibrium and the separation process, a computational experiment using AspenPlus V.10.0 was selected. The parameters of the Non Random Two Liquid equation allow reproducing the vapor-liquid equilibrium data and azeotropic properties of the system with a relative error not exceeding 5%. Separation of the mixture by conventional distillation is difficult, which is caused by the presence of three binary azeotropes,

as well as three pairs of components characterized by close volatility. The structure of the vapor-liquid equilibrium diagram becomes more complex due to the appearance of new azeotropes with a decrease in pressure. A technological flowsheet for solvent regeneration based on the use of continuous distillation was proposed. The flowsheet contains five columns. At the first stage, preliminary fractionation of the mixture is assumed (the mode of sharp distillation in the first column) with complete separation of cyclohexanone and cyclohexanol in the bottom (vacuum distillation is used to separate this pair) and of a mixture of butanol and amyl alcohol with impurity components in the distillate. Extractive distillation with ethylene glycol was proposed to purify butanol and amyl alcohol from impurities. In the presence of the latter, the volatility of butanol increases in relation to other components. The fourth column is designed for the regeneration of the separating agent. Amyl alcohol separation is provided on the bottom of fifth column. The column operation parameters (the number of stages, the feed stage, the reflux ratio, the ratio of the quantities of the initial mixture and the separating agent) that meet the minimum energy consumption and ensure the production of commercial-quality substances are determined.

Key words: solvents mixture, butanol, cyclohexanone, cyclohexanol, amyl alcohol azeotrope, separation flowsheet, extractive distillation

Для цитирования:

Фролкова А.В., Фролкова А.К. Разработка технологии разделения спиртовой смеси растворителей. *Изв. вузов. Химия и хим. технология*. 2023. Т. 66. Вып. 1. С. 93–98. DOI: 10.6060/ivkkt.20236601.6670.

For citation:

Frolkova A.V., Frolkova A.K. Development of solvents alcohol mixture separation technology. *ChemChemTech* [*Izv. Vyssh. Uchebn. Zaved. Khim. Khim. Tekhnol.*]. 2023. V. 66. N 1. P. 93–98. DOI: 10.6060/ivkkt.20236601.6770.

INTRODUCTION

Caprolactam is an important organic compound that is used for polyamide plastics, fibers production [1-6]. The global demand for caprolactam currently amounted to about 20 million tons. A multicomponent mixture of solvents, containing n-butanol, amyl alcohol, cyclohexanone, cyclohexanol and two components in impurity quantities (isoamylol and cyclopentanone) is formed in the process of caprolactam production. A flowsheet based on periodic distillation process was previously proposed to separate this mixture [7]. The first fraction was a mixture enriched with butanol (97.5% mass), a second one – a transition fraction (to remove impurity components), a third one – a mixture enriched with amyl alcohol. A mixture of cyclohexanone and cyclohexanol remained in the column bottom. To separate cyclohexanone from the third fraction, an extractive distillation process with ethylene glycol was proposed [7].

The composition of the mixture considered may vary depending on the operating conditions of the reaction unit [6]. The paper discusses the next composition: X_B = 0.0989; X_{CPON} = 0.0045; X_{IA} = 0.0116; X_A = 0.5191; X_{CHON} = 0.1504; X_{CHOL} = 0.2155 mass. frac. (Feed flow (F) – 100 kg/h)). A mixture separation flowsheet (continuous process) is proposed based on the study of phase equilibrium. Columns operation parameters ensuring the commercial quality products with minimal energy consumption are determined.

METHODS

Mathematical modelling of phase equilibrium of the n-butanol (B) + cyclopentanone (CPON) + isoamyl alcohol (IA) + amyl alcohol (A) + cyclohexanone (CHON) + cyclohexanol (CHOL) system was based on the Non Random Two Liquid (NRTL) equation using AspenPlus® [8].

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

where $G_{ij} = \exp(-\alpha_{ij}\tau_{il})$; $\tau_{ij} = a_{ij} + \frac{b_{ij}}{T} + e_{ij}\ln T + f_{ij}T$;

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

The system contains 15 binary constituents. There are experimental data on the vapor-liquid equilibrium (VLE) for each binary subsystem [9-10]. A complete set of NRTL equation parameters has been obtained based on the evaluation of these data (Table 1). The parameter c_{ij} is equal to 0.3 for all binary subsystems except for B+CHON (0.20102), IA+CPON (0.01334). The relative uncertainties of vapor-liquid equilibrium description do not exceed 5%. The results obtained confirm the adequacy of the mathematical modelling and the possibility of using NRTL parameters for subsequent calculations. The choice of the NTRL model is due to the higher quality of the phase equilibrium description in comparison with the Wilson and UNIQUAC models.

Table 1

The parameters of NRTL model for B+CPON+IA+A+CHON+CHOL system at 101.325 kPa

***Таблица 1.* Параметры модели NRTL для системы B+CPON+IA+A+CHON+CHOL) при 101,325 кПа**

Subsystem	a_{ij}	a_{ji}	b_{ij}	b_{ji}
B+CPON	1.373	0.05	-345.3	-72.21
B+A	184.0	0.45	184.01	-218.5
B+IA	-2.08	3.58	836.98	-1436
B+CHON	-1.14	-0.62	138.86	671.1
B+CHOL	0	0	674.31	-431.9
A+CPON	2.51	-0.80	-671.43	156.2
IA+A	0	0	-345.07	521.2
A+CHON	1.38	23.1	180.14	-10000
C+CHOL	-0.57	0.71	-2.983	4.515
IA+CPON	12.94	21.7	-7842.0	-5729
IA+CHON	-0.41	3.01	345.34	-1070
IA+CHOL	-1.27	1.19	47.668	364.34
CHON+CPON	-1.68	2.83	458.78	-810.0
CPON+CHOL	11.4	-3.5	-3842.5	1001.7
CHON+CHOL	0	0	368.3	-262.8

RESULTS AND DISCUSSIONS

Three binary azeotropes are modeled at 101.3 kPa in the six component system B (117.75 °C) + CPON (130.55 °C) + IA (131.01 °C) + A (137.80 °C) + + CHON (155.42 °C) + CHOL (160.84 °C), the boiling points of the pure components are indicated in parentheses: CPON + IA (X_{CPON}^{Az} = 0.6124 mol. frac.; T^{Az} = 129.03 °C); CPON + A (X_{CPON}^{Az} = 0.9665 mol. frac.; T^{Az} = 130.54 °C); IA + CHON (X_{IA}^{Az} = 0.9775 mol. frac.; T^{Az} = 131.0 °C). The components of CHON and CHOL, as well as IA and A are characterized by close volatility. There is a tangential azeotrope in the binary constituent of B + CPON. VLE diagrams of four ternary systems belong to class 3.1.0-1a, three – to class 3.1.0–2, two – to 3.2.0-2b, one – to 3.1.0–1b, the rest ten are zeotropic [11]. Butanol is represented by a point of the unstable node type in the VLE diagram, cyclohexanol is a stable node (Figure 1); all other singular points, including azeotropes, are saddles with a zero Poincare index relative to the total composition space [12]. There are two separatrix surfaces of second dimension in the system (highlighted in color in Fig. 1) and no separatrix manifolds of greater dimension. Despite the fact that there is one distillation region in the system, the use of the direct sequence separation at the first stage (the distillation process is considered) is not recommended due to the close volatility of the B and CPON in the vicinity of pure butanol (the maximum composition of butanol in the distillate will be 94% mass). The indirect sequence also has a restriction on the implementation, which is due to the above limitation in the binary constituent CHON + CHOL.

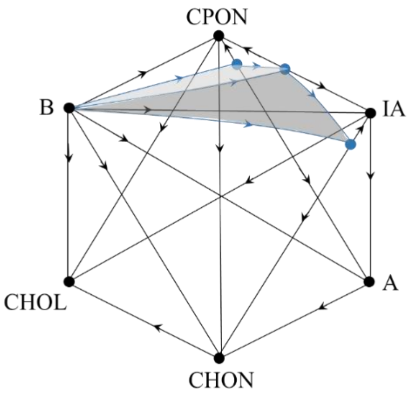

Fig. 1. Vapor-liquid equilibrium diagram of system B+CPON+IA+A+CHON+CHOL at 101.3 kPa
Рис. 1. Диаграмма парожидкостного равновесия системы B+CPON+IA+A+CHON+CHOL при давлении 101,3 кПа

However, these characteristics of the system favor the preliminary fractionation of the mixture (sharp distillation), when a mixture of B + CPON + IA + A will be separated in the distillate, and a binary mixture of CHON + CHOL in the bottom (Fig. 2). The values of the component distribution coefficients between the liquid and vapor phase in original mixture are 1.79, 1.67, 1.81, 1.16, 0.55, 0.52 for B, CPON, IA, A, CHON, CHOL respectively. According to [13-14], in such a situation, the preference should be given to sharp distillation, which confirms the hypothesis put forward above.

A decrease in pressure leads to a complication of the structure of the VLE diagram, since new azeotropes appear in the system (B+CPON, A+CHON).

Separation of CHON+CHOL mixture is possible in the second column operating at reduced pressure (there is an increase in the volatility of the CHON with respect CHOL from 1.20 to 2.04).

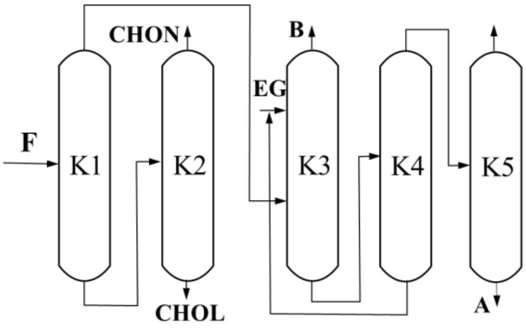

Fig. 2. Flowsheet of B+CPON+IA+A+CHON+CHOL mixture separation
Рис. 2. Схема разделения смеси B+CPON+IA+A+CHON+CHOL

Extractive distillation is used to purify butanol and amyl alcohol from impurities [15-19]. Ethylene glycol (EG) was selected as the separating agent. The change in the relative volatility of components in the presence of EG (the amount of EG / the amount of original mixture is 3/1) is given in Table 2.

Table 2
The relative volatility of components in the presence of EG
***Таблица 2.* Относительная летучесть компонентов в присутствии ЭГ**

Pair	Original mixture	In the EG presence
B+A	1.80	2.86
B+IA	1.59	8.90
A+IA	1.13	3.10
CPON+A	1.76	1.80
CPON+IA	1.55	5.68
B+CPON	1.02	1.60

The analysis of the data in Table 2 showed that it is possible to separate butanol as a distillate product in the column K3. K4 is an EG regeneration column. The column K5 is designed to remove impurity components and separation of amyl alcohol with a concentration of 98.0% mass. The distillate flow of K5 is increased to 4.3 kg/h for purification of amyl alcohol in the bottom.

Table 3
The flowsheet columns operation parameters
***Таблица 3.* Параметры работы колонн схемы**

Column (*SA*/*F*)	N	N_F (N_{EG})	R	Q, kW
K1	38	19	2.46	35.35
K2	38	14	7.48	12.42
K3	32	16 (12)	5.70	16.59
K4	14	6	4.1	38.09
K5	18	8	4.3	5.20
∑Q=107.65kW				

Distillation was simulated with RadFrac routine of Aspen Plus. Sensitivity analysis was used to find the operating conditions (number of theoretical stages (N), reflux ratio (R), mixture feed stage (N_F)). Theoretical stages were considered [20-21]. The distillation columns parameters of flowsheets proposed are given in Table 3. The optimum parameters correspond to minimum energy consumption (Q). Columns K1, K5 work at 101.3 kPa, K2 at 10.1 kPa, K3 at 120 kPa, K4 at 50 kPa (the pressure change in the columns is associated with the need to increase the volatility of certain components). The distillate flow of columns K1-K5 are equal to 63.41, 15.04, 9.89, 53.52 and 4.3 kg/h respectively. The purity of the CHOL, CHON, B and A is 99.8, 99.8, 99.7 and 98.0 % mass.

CONCLUSIONS

Thermodynamic limitations on the distillation process (azeotropes, components close volatility) were revealed, the expediency of using mixture preliminary fractionation at the first stage was substantiated based on the study of the phase equilibrium of the n-butanol, amyl alcohol, cyclohexanone, cyclohexanol, isoamyl alcohol and cyclopentanone system. A mixture separation flowsheet containing five distillation columns was proposed. Extractive distillation with ethylene glycol is used to purify butanol and amyl alcohol from impurity components (isoamyl alcohol and cyclopentanone). The column operation parameters corresponding minimum energy consumption were obtained. Products of the required purity were obtained. Cyclohexanone, cyclohexanol and butanol are separated in full, loss on amyl alcohol is about 5%. All the results were obtained using mathematical modeling methods and require further local field experiment to confirm some operating modes of the columns.

The work was carried out under support of Russian Science Foundation 19-19-00620 П.

Работа выполнена при поддержке РНФ 19-19-00620 П.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

The authors declare the absence a conflict of interest warranting disclosure in this article.

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Поступила в редакцию 15.05.2022
Принята к опубликованию 06.10.2022

Received 15.05.2022
Accepted 06.10.2022