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РАЗДЕЛЕНИЕ СМЕСИ ЭТИЛОВОГО СПИРТА С ВОДОЙ ПУТЕМ ЭКСТРАКТИВНОЙ ДИСТИЛЛЯЦИИ: МОДЕЛИРОВАНИЕ С ИСПОЛЬЗОВАНИЕМ ASPEN PLUS™ И ЭКСПЕРИМЕНТАЛЬНОЕ ПОДТВЕРЖДЕНИЕ

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Разбавленный этиловый спирт (этанол, EtOH) получают методом брожения (< 20 масс. %), но он требует дальнейшей очистки, поскольку имеет большое содержание воды. EtOH образует азеотроп с водой, и для разделения этого азеотропа используются усовершенствованные процессы разделения, такие как дистилляция с переменным давлением (PSD), азеотропная дистилляция (AD) и экстрактивная дистилляция (ED). В данной работе этиленгликоль (ЭГ) использовался в качестве азеотропного агента при экстрактивной дистилляции, а данные парожидкостного равновесия (VLE) использовались для проверки выбранной строгой термодинамической модели. Для определения рабочего диапазона рабочих параметров был проведен анализ чувствительности с целью определения количества ступеней (N), ступени питания (N_F), скорости дистиллята (DR) и флегмового числа (RR). Моделирование показывает, что использование точных рабочих параметров и правильного азеосорбента может помочь получить практически чистый EtOH (99,89 моль. %, 99,92 масс. %). Для проверки теоретических выводов моделирования были проведены экспериментальные исследования в форме периодической и полупериодической дистилляции. Из этих экспериментов было видно, что действительно можно получить EtOH высокой чистоты. Также было подтверждено, что ЭГ можно отделять и возвращать обратно с высокой чистотой и очень высокой степенью извлечения.

Ключевые слова: этиловый спирт (этанол), экстрактивная дистилляция (ED), этиленгликоль (ЭГ), моделирование, анализ чувствительности (SA), циклические перегонки, полупериодические перегонки

ETHYL ALCOHOL - WATER SEPARATION BY EXTRACTIVE DISTILLATION: SIMULATION USING ASPEN PLUS™ AND EXPERIMENTAL VALIDATION

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Dilute ethyl alcohol (ethanol, EtOH) is produced using fermentation (< 20 mass%), but it needs to be further purified because it contains large water content. EtOH forms an azeotrope with water and to separate this azeotrope, advanced separation processes like pressure swing distillation (PSD), azeotropic distillation (AD), and extractive distillation (ED) are employed. In this work, ethylene glycol (EG) was used as an entrainer in extractive distillation. Vapour liquid equilibrium (VLE) data was used to validate the chosen rigorous thermodynamic model. To find a workable range of operating parameters, sensitivity analysis was performed to find out the number of stages (N), feed stage (N_F), distillate rate (DR), and reflux ratio (RR). Simulation shows that use of accurate operating parameters and correct entrainer can help to obtain almost pure EtOH (99.89 mole %, 99.92 mass %). In order to validate the theoretical findings from the simulation, experimental investigations in the form of batch and semi - batch distillation runs were conducted. It was seen from these runs that it is indeed possible to obtain EtOH with high purity. It was also verified that EG can be separated and recycled back with high purity with a very high recovery.

Key words: ethyl alcohol (ethanol), extractive distillation (ED), ethylene glycol (EG), simulation, sensitivity analysis (SA), batch distillation runs, semi - batch distillation runs

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INTRODUCTION

Separation of azeotropic mixtures is a topic of great practical and industrial interest. Many liquid mixtures of organic components form non - ideal systems. Azeotropes frequently occur when certain specific groups, especially polar groups (oxygen, nitrogen, chlorine, and fluorine), are present. Process industry deals with a lot of mixtures emerging out of operations like reaction and separation. Many of these compounds are expensive and hence recovery is essential. Environmental regulations prohibit the passage of these chemicals through atmospheric bodies including air, water and soil. Azeotropes are mixtures of two or more liquids in a ratio such that their composition cannot be altered by simple distillation. Often, advanced distillation techniques are used to successfully separate azeotropic mixtures. In many cases, azeotropic mixtures can be effectively separated by distillation by using a third element (entrainer / solvent, Knapp and Doherty 1992).

Alternative fuels like bio fuels, hydrogen and natural gas help to reduce crude oil consumption. One of the most useful chemicals is ethanol (ethyl alcohol, EtOH). It is useful as a solvent, beverage, antifreeze, fuel, depressant, and notably as a chemical intermediary for other organic molecules (Sanap et al. 2021, 2023). Three significant industrial processes are used to make ethanol: fermentation of biomass (sugarcane, corn), chemical processes like ethane hydration and ethanol production from biomass utilizing bacteria (Gnansounou and Dauriat 2005). Although fermentation is the most dependable and straightforward of these processes, it yields maximun 20 mass % of ethanol (Adeleye et al. 2020). Pure EtOH (at least 99.5%) is required for industrial use or for use as a fuel (Karimi et al. 2019). After fermentation, traditional distillation is employed to obtain pure ethanol; however, this method is constrained by the existence of a minimum boiling azeotrope (95.6 mass %, 89.6 mole % EtOH at 350.81 K, Gmehling and Onken 1991). Azeotropic distillation (AD), extractive distillation (ED), pressure swing distillation (PSD) and other methods were developed in the past to change the relative volatility (α) of chemicals in order to improve the purity, via distillation. In the past, azeotropic distillation was used by Chianese. and Zinnamosca 1990 with benzene as the entrainer, iso - octane was used by Gomis et al. 2007. Mulia - Soto and Flores - Tlacuahuac 2011 used PSD. However, ED has been widely used because of its effectiveness.

In order to improve the relative volatility of the desired components, a separating agent (additional solvent) is added in the ED process, which is the most popular and commonly used method for separating azeotropic mixtures (Sanap et al., 2021; Frolkova et al. 2020 and Ma et al., 2017). ED offers advantages like low energy consumption but selection of suitable solvent is important. Solvents mostly used in ED are glycols, glycerols, deep eutectic solvents (DES) and ionic liquids (ILs) (Dai et al. 2014, Shang et al. 2019, Fadia et al. 2022). Glycerols and glycols are most commonly employed organic solvents due to advantages like suitable boiling points and their ability to obtain high purity product. DESs and ILs are shown to offer almost equal separation of ethanol as glycerols, posses the ability to be easily recycled and offer salting out effect. But, their commercial applications are yet to be showcased.

Thorough literature survey was performed and although researchers presented important work, all the aspects covered in the present work are not presented. One of the earlier pioneer work by Stabnikov, 1969 presented experimental data on distillation/rectification. Batista et al. 1997 used thermal integration for this system although experimental work is not presented. Ravagnani et al. 2010 compared EG with tetraethylene glycol (TEG) on the basis of energy consumption and safety hazards like toxicity. Korotkova and Konstantinov 2013 used azeotropic distillation to obtain absolute EtOH. Gutierrez 2013 performed ED with ionic liquids, but did not use EG. Their experimental work is limited to laboratory/bench scale operation. Recently Klinov et al. 2023 designed a pilot plant for use of EG in ED. Design calculations and analysis of the ED column is presented. Lei et al. 2003 presented work on this important separation through a review. Sanap and Mahajan 2023 reviewed EtOH-water separation and purification alternatives. All the above cited researchers have done important work for the problem at hand. However, most of their work is limited by their chosen scope. In most cases, VLE modeling is not presented, sensitivity analysis is not done and also EG recovery is not put forth. The present work has used ED to separate the azeotrope between EtOH and water using EG as an extractive solvent. For this, the tasks as detailed in the next section were performed.

PROCESS MODELING AND SIMULATION

For the ED simulation, RADFRAC module in Aspen Plus environment was used. For the effective use of RADFRAC module, it is important to choose correct thermodynamic model. Vapor-liquid equilibrium (VLE) data was obtained from literature (Gmehling and Onken 1991, 2000). Thermodynamic models available in Aspen Plus were used to predict binaries of the system (EtOH - Water, EG – EtOH and EG - Water, Fig. 1). The aim of the present work is to provide a window for the range of parameters like reflux ratio, distillate rate, number of stages, feed stage, solvent stage and rate. For this, sensitivity analysis was carried out.

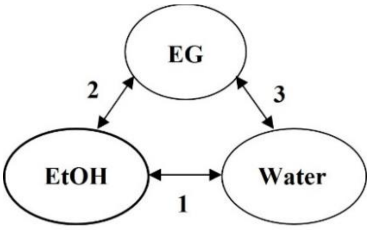


Fig. 1. Possible binaries in the system
Рис. 1. Возможные двойные составы в системе

Aim of this work was to separate the azeotropic mixture of EtOH - water using EG as a solvent, to recover the solvent and to minimize waste. Following steps were carried out while developing the distillation system to obtain pure EtOH:

A. Solvent/Entrainer selection.

B. Selection of thermodynamic model and simulation using RADFRAC module.

C. Sensitivity analysis and proposing a complete scheme for the separation of EtOH - water azeotrope and recovery of solvent.

D. Validation of the results through experiments.

RESULTS AND DISCUSSION:

A. Selection of entrainer:

In order to select better entrainer, literature survey was carried out. Researchers used many solvents while considering EtOH – water azeotropic mixture. Ma et al. 2017 used low transition temperature mixtures (Glycolic acid – Choline chloride) for simulating ED in Aspen plus, NRTL property method was used and they obtained significant saving in terms of total annual cost (TAC). Gil et al. 2008 used EG and calcium chloride for ED and obtained 99.66 mass % pure EtOH. Frolkova and Frolkova 2022 used EG to purify butanol. Other solvents (tetra ethylene – glycol, paraffin oil), deep eutectic solvent (DES) and ionic liquid (ILs) were also used. The present work has chosen ethylene glycol because of its beneficial properties to separate ethanol – water, availability and cost.

B. Selection of thermodynamic model:

It is important to select proper thermodynamic model. VLE prediction was performed for all binaries in the system. Thermodynamic models (NRTL, Wilson, UNIQUAC and UNIFAC) were chosen to predict the VLE data from those available, based on heuristics and rules of thumb (Carlson, 1996). The prediction was compared with experimental data (Gmehling and Onken 1991, 2000) and error was calculated (eq. 1).

$$\%\,\mathrm{Error} = \mathrm{Abs}\left(\frac{Y_{\mathrm{expt.}} - Y_{\mathrm{Sim.}}}{Y_{\mathrm{expt.}}}\right) \cdot 100 \qquad (1)$$

Table 1 shows the errors for all three binaries of the system. From this table, it is seen that the NRTL model offered least error for all three binaries. Hence, it was selected for further simulation work with EG as the entrainer. Table 2 shows the binary interaction parameters from Aspen Plus for the system using NRTL.

Table 1

Error (%) for the binaries in the system at 1 atm

***Таблица 1.* Ошибка (%) для двойных составов в системе при 1 атм.**

Binary	Thermodynamic Model			
	NRTL	Wilson	UNIQUAC	UNIFAC
EtOH-Water	0.52	1.06	0.58	0.98
EtOH-Ethylene Glycol	1.53	1.52	1.58	2.40
Water- Ethylene Glycol	0.91	8.80	0.99	4.42
Average	0.99	3.79	1.05	2.60

Table 2

Binary interaction parameters for EtOH-water system using NRTL

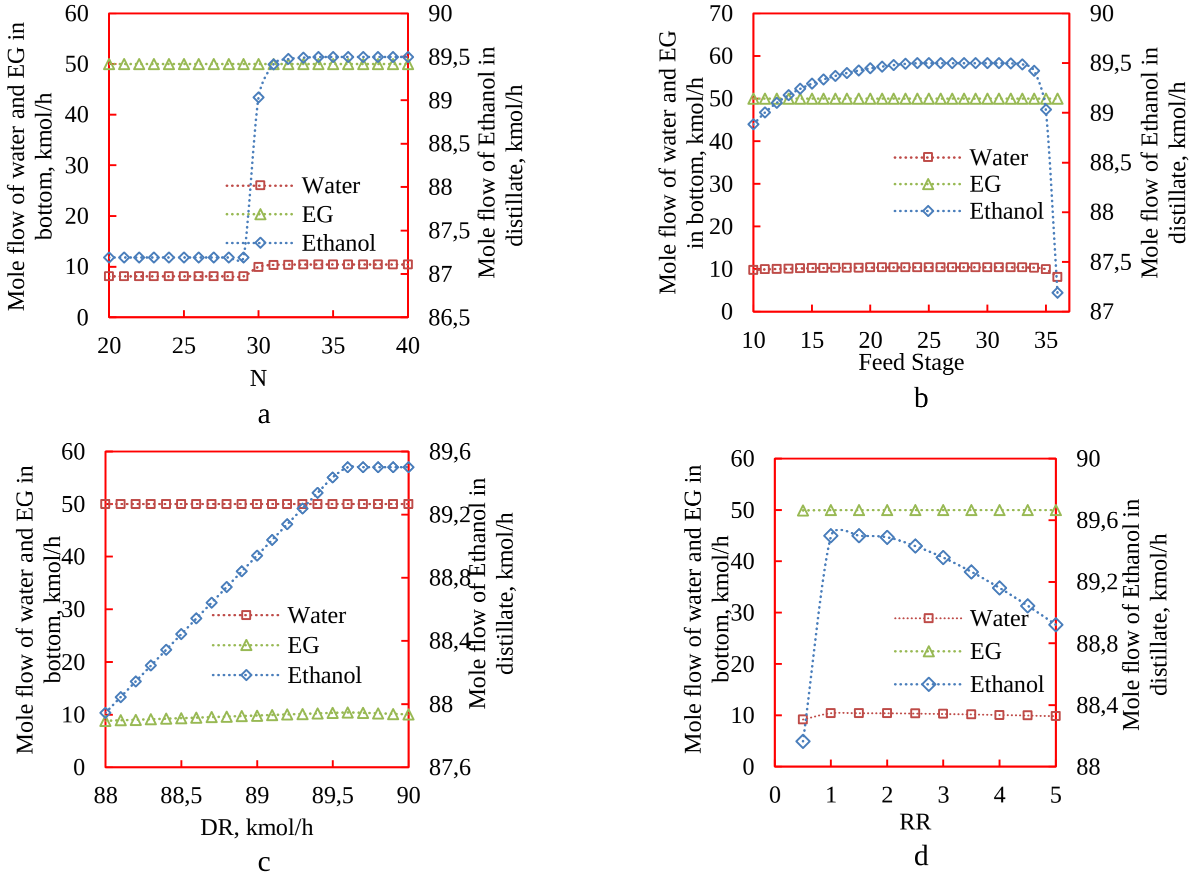
***Таблица 2.* Параметры бинарного взаимодействия для системы этанол-вода**

Component i	Component j	Aij	Aji	Bij	Bji
EtOH	Water	-0.8009	3.4578	246.18	-586.081
EtOH	EG	14.8422	-0.1115	-4664.41	157.594
Water	EG	0.3479	-0.0567	34.8234	-147.137

C. Sensitivity Analysis and Simulation

100 kmol/h of feed with the azeotropic composition (89.5 mole % of EtOH and 10.5 mole % of water) was subjected to separation using distillation in Aspen Plus. Feed was sent to the first distillation column. As previously stated, sensitivity analysis was employed to determine parameters like number of stages, feed stage, distillate rate, reflux ratio, solvent rate and stage.

Column I: Fig. 2a shows the effect of the number of stages (N) on mole flows of EtOH, water and ethylene glycol. N was varied from 20 to 40, but it is seen that the range of 34 to 40 produces best results and hence 36 stages were selected. Feed stage (N_F) was varied from 10 to 36, and it was discovered that the 25-31 window produces satisfactory results. 29th stage was chosen as the N_F (Fig. 2b). Further, distillate rate (DR) was varied from 88-90 kmol/h (Fig. 2c). Satisfactory mole flow was obtained within the range of 89.6 to 89.9 kmol/h. RR was selected as 1 (Fig. 2d). Effect of solvent rate and solvent feed stage was also checked and final configuration for column I is: N: 36, feed stage: 29, DR: 89.6 kmol/h, RR: 1, solvent rate: 50 kmol/h and solvent stage: 3. Pure EtOH (99.89 mole %, 99.92 mass %) was obtained in the distillate with almost 100% recovery. Bottom stream contained EG and water (83 mole % and 17 mole % respectively) which needs separation.



a

b

c

d

Fig. 2. a) Effect of number of stages on mole flow of water and EG in bottom and EtOH in distillate of column I; b) Effect of feed stage on mole flow of water and EG in bottom and EtOH in distillate of column I; c) Effect of DR on mole flow of water and EG in bottom and EtOH in distillate of column I; d) Effect of RR on mole flow of water and EG in bottom and EtOH in distillate of column I

Рис. 2. а) Влияние количества ступеней на мольный расход воды и ЭГ в кубе и этанола в дистилляте колонны I; b) Влияние стадии питания на мольный расход воды и ЭГ в кубе и этанола в дистилляте колонны I; в) Влияние ДР на мольный расход воды и ЭГ в кубе и этанола в дистилляте колонны I; d) Влияние RR на мольный расход воды и ЭГ в кубе и этанола в дистилляте колонны I

Column II: Bottom stream from column I was sent to column II for further separation of EG and water. Fig. 3a shows that 12-20 stages offer satisfactory results. Feed stage between 4-10 offers good results as between 2-3 and after 10th feed stage, the mole % of EG decreases (Fig 3b). Feed stage was chosen to be 10th and total number of stages were chosen to be 13. RR was varied between 0.5 to 3 and a value of 1.5 was chosen. DR was chosen by considering highest mole % of EG in bottom. Final configurations for column II are: N: 13, N_F: 10, DR: 10.45 kmol/h and RR: 1.5. Almost pure EG was obtained in the bottom of column II with 99.94% recovery. Fig. 4 shows the overall separation sequence.

Fig. 5 and 6 depict the concentration and temperature curves for column I (azeotrope separation) and column II (entrainer recovery). EtOH with a purity of 99. 89 mole % (99.92 mass %) was obtained in the distillate of column I with almost 100 % recovery. EG extracted all the water and accumulated in the bottom of column I which is further sent to column II for recovery of EG. Pure EG (99.94 mole %, 99.98 mass %) was obtained from the bottom of column II. Recovered EG can be recycled back to column I. The optimized column I and II configurations are indicated in Table 3. It is demonstrated that with EG as the entrainer, it is possible to break the EtOH – water azeotrope and to obtain high purity EtOH.

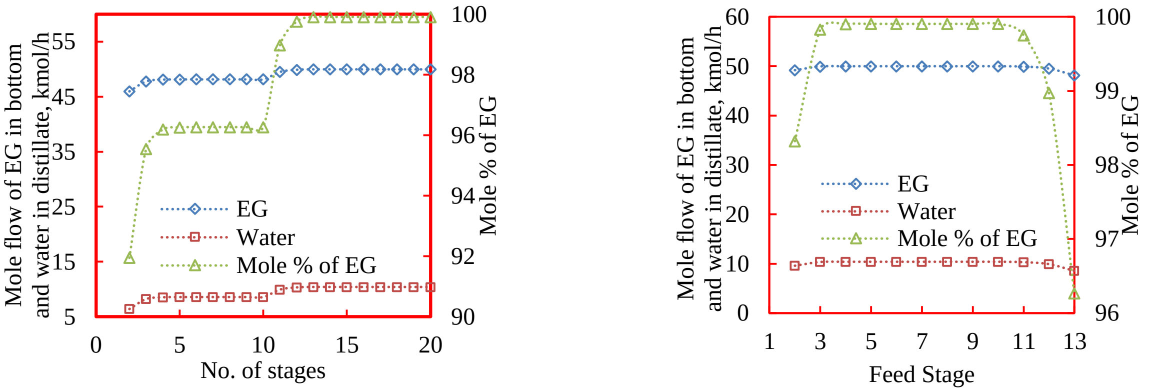


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Fig. 3. a) Effect of no. of stages on mole flow of EG in bottom and water in distillate of column II; b) Effect of feed stages on mole flow of EG in bottom and water in distillate of column II

Рис. 3. а) Эффект №. стадий по мольному расходу ЭГ в кубе и воды в дистилляте колонны II; b) Влияние стадий подачи на мольный расход ЭГ в кубе и воды в дистилляте колонны II

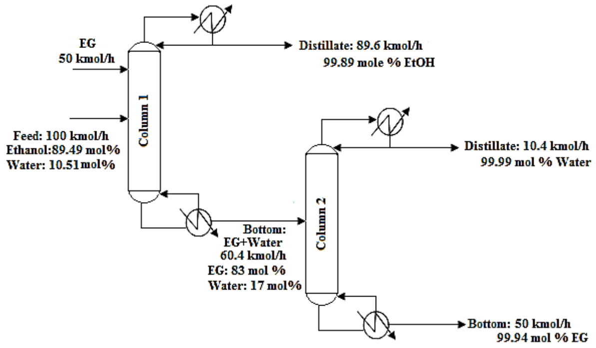

Fig. 4. Overall flow sheet of separation of azeotrope and recovery of solvent

Рис. 4. Общая технологическая схема разделения азеотропа и регенерации растворителя

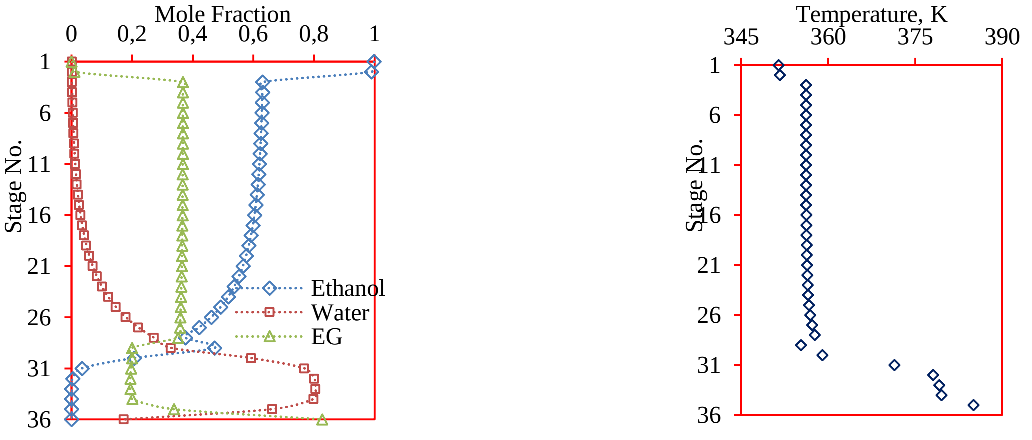

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Fig. 5. a) Composition profile (column I); b) Temperature profile (column I)

Рис. 5. а) Профиль состава (колонна I); б) Температурный профиль (колонна I)

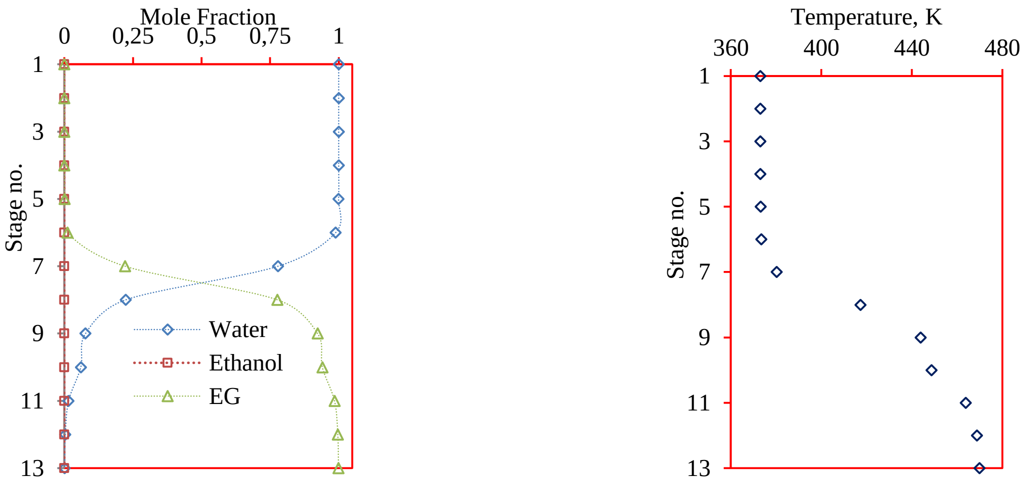

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Fig. 6. a) Composition profile (column II); b) Temperature profile (column II)
Рис. 6. а) Профиль состава (колонна II); b) Температурный профиль (колонна II)

Table 3

Configurations for column I and II
***Таблица 3.* Конфигурации колон I и II**

	Column I	Column II
Number of stages	36	13
Feed Rate, kmol/h	100 (EtOH-Water) + 50 (EG)	60.4
Feed stage	29	10
Molar reflux ratio	1	1.5
Distillate rate, kmol/h	89.6	10.45
Solvent rate, kmol/h	50	-
Solvent stage	3	-
Purity and Recovery of components	**EtOH:** 99.89 mole % (99.92 mass %) 100 % recovery	**EG:** 99.94 mole % (99.98 mass %) 99.94 % recovery

EXPERIMENTATION

Simulation and sensitivity analysis indicated that EG can be used effectively in a ED sequence. High purity EtOH and almost complete recovery of EG were indicated. In order to validate this work, experimental investigations were performed in the batch and semi - batch mode distillation. Distillation runs were performed in a laboratory scale (51 mm ID, 1 m long column, reboiler - 3.8 L capacity - fitted with 2 kW heater, teflon disks with provision for sample collection, feed input and temperature indication, fitted with Dean and Stark assembly (800 mL capacity, fitted with coiled condenser). Schematic of the setup is shown in Fig. 7. The column, made up of high quality glass was insulated with glass wool and aluminum tape; the valves are made up of SS 316 L. Hyflux distillation packings made up of SS 316 L, knitted wire mesh packings with higher mass transfer rate, known for better separation efficiency (HETP: 0.075 m, NTU: 10-13/min and large surface area ~1200 m^2/m^3, Web reference 1) were used. Following runs were performed:

Batch and semi - batch distillation

A batch of EtOH - water mixture (90 mass % EtOH - rest water) was taken in the reboiler and heating was started (Run 1). At pseudo - equilibrium, azeotropic composition of EtOH - water was obtained as the distillate (95.6 mass% EtOH - 4.4 mass % water). In the next run, azeotropic composition (2000 g) was taken in the reboiler and EG (1 mL/min) was introduced at top of the column (Run 2, Fig. 7). The vapors condensed, filled the Dean and Stark assembly and the reflux to the column started and then the composition and temperature stabilized. Following observations were made.

a) As time progresses, EG concentration in the reboiler increases and EtOH concentration decreases.

b) Distillate conc. of EtOH stabilizes and after about one hour, it becomes ≥ 99.5 mass % indicating that indeed high purity EtOH can be obtained. Results of this run are shown in Table 4. It is seen that the distillate composition is not affected and the reboiler composition stabilizes rapidly. The mode of this run was semi - batch in which EG was continuously fed but distillate and bottom streams were not continuous. As per simulation, it is evident that almost pure EtOH (~ 99.95 mass %) EtOH is obtained as the distillate and a mixture of EG and water as bottom stream.

c) In the next run (Run 3), 2000 g of EG - water (86 and 14 mass % of EG and water) were charged to the reboiler and batch distillation was conducted. Results of this run are shown in Table 4. Almost pure EG and water streams emerged.

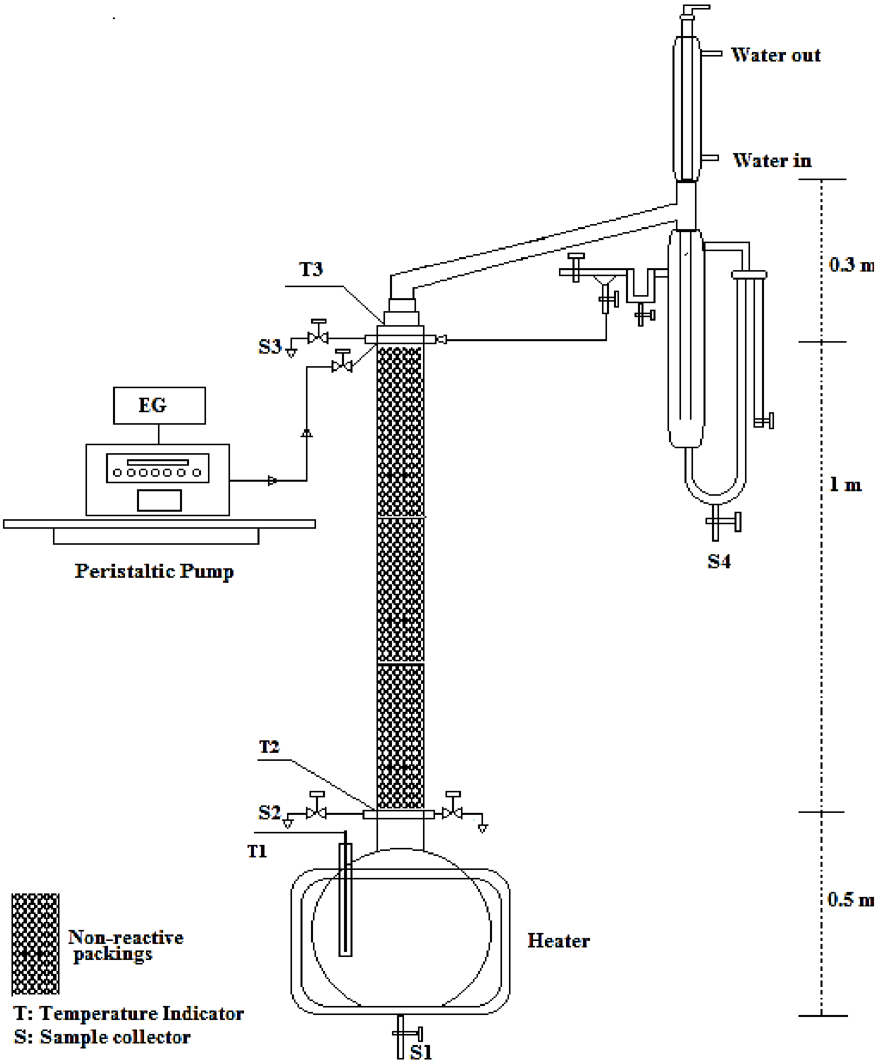

Fig. 7. Schematic of distillation setup
Рис. 7. Схема перегонной установки

Table 4
Composition of components from the distillation runs (mass %)
***Таблица 4.* Состав компонентов дистилляционных партий (массовые %)**

	Component	Run 2	Run 3
Distillate	Ethanol	99.95	-
	Water	-	99.99
Reboiler	Water	25.58	-
	Ethylene Glycol	74.42	99.97

It is thus seen that it is possible to separate EtOH-water azeotropic mixture. EtOH can be obtained with very high purity and the solvent EG can be almost completely recovered. This work has thus demonstrated by way of simulation and by experimentation that it is possible to separate and purify EtOH from its aqueous azeotropic mixture by using EG in a ED process. Simulation was performed in a continuous mode whereas experimentation was done in the batch / semi - batch mode. The results of experimentation indicate that if conducive conditions like sufficient separation stages and time are produced, then it is possible to obtain the results indicated by simulation.

Thus, this work has successfully demonstrated the following:

1. A methodology to minimize error while predicting VLE and especially to predict azeotrope. This way, a validated thermodynamic model is used.
2. Through simulation, sensitivity analysis was performed which offered a workable range for variables / parameters.
3. Qualitative validation of the RadFrac model predictions was showcased.
4. A complete flow sheet of EtOH purification from its aqueous solution was demonstrated which also includes EG recovery.

CONCLUSION

Separation of EtOH - water azeotrope was performed using extractive distillation in this work. Choice of the thermodynamic model was done by validation based on experimental VLE data. Sensitivity analysis was carried to suggest a possible range of operating parameters in Aspen Plus™. Afterwards, simulation was carried out using RadFrac module. For separation of the azeotrope and recovery of the entrainer, two distillation columns were utilized. Approximately 99.89 mole % (99.92 mass %) pure EtOH was obtained as distillate of first column with almost 100% recovery. EG was purified in column II, where approximately 99.94 mole % (99.98 mass %) pure was obtained from the bottom of the column II with 99.94 % recovery. In order to validate these findings, batch and semi - batch distillation runs were performed wherein almost 99.95 mass % purity of EtOH was obtained. Trends indicate that it is possible to obtain EG with high purity (99.97 mass %) and high recovery (99.94 %).

NOMENCLATURE

AD	-	Azeotropic distillation
DES	-	Deep Eutectic solvents
DR	-	Distillate rate
ED	-	Extractive distillation
EG	-	Ethyl glycol
EtOH	-	Ethanol, Ethyl alcohol
ILs	-	Ionic liquids
N	-	Number of stages
PSD	-	Pressure swing distillation
RR	-	Reflux ratio
SA	-	Sensitivity analysis
TAC	-	Total annual cost
VLE	-	Vapour liquid equilibrium

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

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

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