THREE-PHASE DISTILLATION OF ETHYL ACETATE/WATER/ETHANOL: SEPARATION FEASIBILITY AND CONCEPTUAL DESIGN

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Abstract. Continuous production of ethyl acetate involves various separation challenges due to multiple azeotropes. In this study, three-phase advanced distillation method is applied through diverse purification scenarios for ternary separation of ethyl acetate system (ethyl acetate/water/ethanol). This highly non-ideal mixture contains four azeotropes and three distillation regions. To select the best distillation region, the separation feasibility and conceptual design of ethyl acetate three-phase distillation unit are comprehensively investigated by the extended boundary value method for various feed locations and numerous product recoveries. It was found that the region in which ethanol is a stable component was the most suitable region for the distillation process. Further, the conceptual design of the three-phase column is optimized by variation of reflux ratio and operating pressure. Ultimately, based on the conceptual design results, rigorous simulation of the process is accomplished and ethanol is separated with 99.25 mol % purity.

Keywords: three-phase distillation, ethyl acetate process, separation feasibility, conceptual design, rigorous simulation.

1. Introduction

Distillation process is the most important thermal separation method for liquid mixtures in chemical and petrochemical industries. High energy consumption for boiling and vaporization of the mixture is the main drawback of this method. Therefore, application of this method is limited for separation of mixtures with high boiling point components or heat sensitive, unstable, and chemically active mixtures. The conventional technique for eliminating this shortcoming is lowering the operating pressure to diminish the boiling temperature of the mixture. Implementing this technique for processes with high volume flow rates and high total pressure drop is not possible.[1,2]

The three-phase distillation method is proper for ruling out the mentioned obstacles. In this method, the gas phase is in contact and equilibrium with the two liquid phases in the sieve tray or packed bed distillation column. This distillation method occurs when at least one of the components in the liquid mixture has partial solubility or is insoluble in other components.[3,4] Thus, a highly non-ideal heteroazeotropic mixture is achieved. The boiling temperature in heteroazeotropic mixtures is always lower than that of its pure constituents. In fact, this physical principle is employed in the three-phase distillation operation to perform the separation process at low temperatures.[1,5]

Identifying the mixture characteristics and the type of azeotropic points with respect to homogeneity and heterogeneity plays a key role in choosing the proper strategy for three-phase distillation operation. Classification of azeotropic mixtures in three-phase distillation is illustrated in Fig. 1. It goes without saying that the higher number of azeotropic points due to creating limiting distillation boundaries makes the three-phase separation more challenging and reduces the likelihood of achieving high-purity products. Given the existence of heterogeneity in the liquid phase, the three-phase distillation column constantly works with decanter. In the decanter, the two liquid/liquid (LL) phases are

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separated and recovered.[1] Based on the type of heterogeneity, the three-phase distillation is carried out in two forms:

1. Operation with one decanter. In case one of the components is insoluble in another component, the separation process is conducted with one decanter, and considering that the heterogeneity in the liquid phase is on the top or bottom of the column, the decanter is placed on the top or bottom of the column.

2. Operation with two decanters. If one of the constituents is insoluble in other two constituents, the separation operation is carried out with two decanters. The methods of three-phase distillation with one and two decanters are presented in Fig. 2.
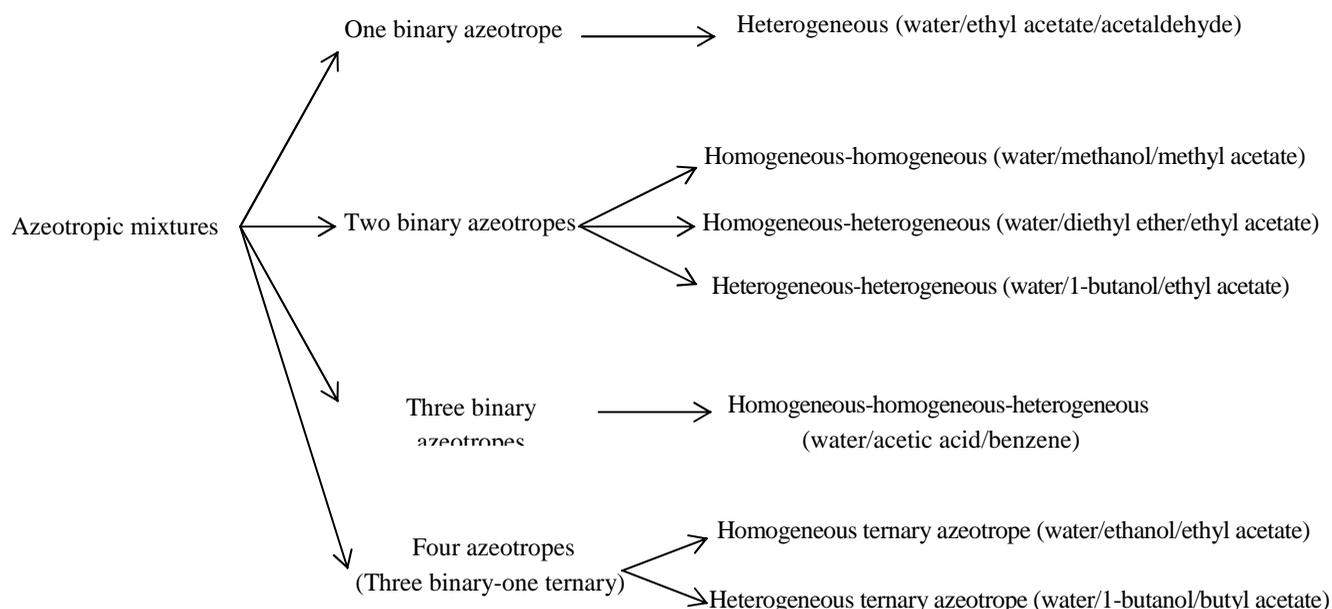

Fig. 1. Classification of ternary azeotropic mixtures in three-phase distillation
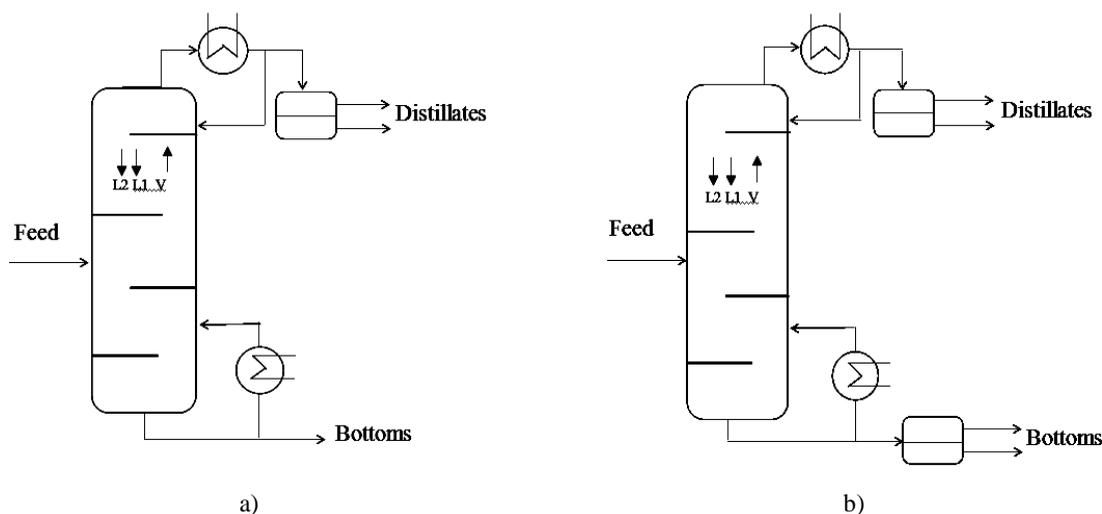

Fig. 2. The methods of three-phase distillation: separation with one decanter (a) and two decanters (b)

As mentioned above, the three-phase distillation is an attractive and effective process for separation of highly non-ideal mixtures, which is currently required by numerous chemical industries. Here we evaluate the three-phase distillation of ethyl acetate process, because continuous production of ethyl acetate is involving with

various separation challenges due to multiple azeotropes as well as heterogeneous liquid-liquid phase split.

Ethyl acetate is distinguished as one of the most important chemicals, which is widely used as solvent in production of inks, adhesives, paints, and coatings. In addition, it is the main ingredient in different fragrances, flavors, and pharmaceuticals.[6-12]

The industrial production of ethyl acetate is primarily based on the classical Fischer esterification process of acetic acid with excessive ethanol as EtOH + AcOH ↔ EtAc + H$_2$O, in the presence of acid catalyst.[6,11,13] The conversion of this reaction is limited by chemical equilibrium.[10] Therefore, to increase conversion percent, the excess amount of ethanol is applied. Consequently, the resultant ethyl acetate contains produced water and the residual ethanol, which forms an acetic acid free ternary stream. The separation of this ternary mixture is complicated because of the formation of binary and ternary azeotropes. Therefore, a feasible separation scheme and its conceptual design are required for processing of this complex system.

Almost all studies conducted in the field of the separation of this sophisticated mixture have been either on reactive distillation[7-10,14,15] or on pervaporation.[6,11,12]

Reactive distillation is a quite complex process. This complexity is due to strong interactions of chemical reaction with heat and mass transfer and high sensitivity to column continuous variables such as reflux ratio.[7,8] On the other hand, applying the reactive distillation is somewhat restricted by constraints such as operating conditions (temperature and pressure) for distillation and reaction and difficulties in controllability and providing suitable residence time characteristics.[8] Another disadvantage of reactive distillation is suffering from problems associated with the use of homogeneous acid or base catalysts, leading to serious economic and environmental consequences. Moreover, these catalysts are expensive and require severe considerations to maintain their long thermal stability.[7,10] Also, catalysts are poisoned through basic impurities that are likely to be present in the feed.[10]

Pervaporation suffers from problems associated with the strong competition among the mentioned mixture components to interact with the membrane. Furthermore, there is also the possibility of the co-permeation of the components in studied mixture, which would direly affect the separation performance of the membrane in this process.[6,11,12]

The main contribution of the current study is comprehensive evaluation of the separation feasibility and conceptual design of ethyl acetate unit based on principles of three-phase distillation method that leads to

significant energy saving. To the best of our knowledge, the proposed method is innovative for ethyl acetate process, and has not been reported in open literature sources.

The objective function of the present work is considered achieving high purity products with the minimum capital and operating costs. To achieve this goal, the effect of various distillation regions, feed location, recovery percent of products, reflux ratio, and operating pressure is investigated on the conceptual design of three-phase separation column. Thereafter, based on the obtained results from the conceptual design, rigorous simulation of the separation process of the azeotropic mixture is carried out. Finally, the effect of variation of the bottoms flow rate and reflux ratio is evaluated by rigorous design.

2. Experimental

2.1. Conceptual Design

Design of three-phase distillation columns is taken into account as an industrial challenge due to the presence of heterogeneity in liquid phase.[5] Applying the conceptual design principles can be an efficient strategy to eliminate these challenges.

The shortcut methods applied in the conceptual design are essential tools for determining the characteristics of these non-ideal separation units. These methods evaluate the separation feasibility and determine the minimum required parameters for design (*e.g.*, the number of stages, feed stage, reflux ratio, and energy duty). The capital and operating costs of separation unit can be estimated with these parameters. Various shortcut methods either rely on applying pinch points (at minimum reflux) or rigorous concentration profile (tray-to-tray) calculations to estimate the column performance. Some of the most important of these methods are listed in Table 1.

Table 1. The shortcut methods for conceptual design of non-ideal separation units

Pinch-based calculations	Tray-to-tray calculations
Pitchfork boundary method	Boundary value method (BVM)
Minimum angle & zero-volume criterion	
Feed angle method	Shortest stripping line method (SSLM)
Rectification body method	
Petlyuk's methodology	

The pinch-based methods have weak performance in confronting with strongly non-ideal mixtures such as our case study in this paper. Contrary to pinch calculation

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methods, the shortcut methods relying on concentration profile calculations such as the BVM and the SSLM are very accurate and result in an exact column profile except when the distribution of trace impurities exists in the product specifications. Both the BVM and the SSLM are actually the optimization problems at which the lowest energy occurs when the concentration profiles of both column sections intersect.[16,17] To our knowledge, there was no report on systematic conceptual design of separation unit of ethyl acetate/water/ethanol mixture using an efficient shortcut method in different distillation regions.

In the current study, extended BVM was used to predict the hard splits in our strongly non-ideal mixture. This extended method (implemented in the environment of Aspen Plus software (version 8.8)) has overcome the only mentioned drawback of conventional BVM, hence it is a powerful tool to analyze some important challenges and gaps in the design of ethyl acetate three-phase distillation column. Using this method, the column design is carried out based on purity or recovery of products, reflux ratio, and operating pressure for a specific feed and separation process performance is evaluated based on the required number of stages and calculated energy duty.

2.2. Process Thermodynamics

Prediction of equilibrium and thermodynamic behavior of the mixture is of great significance in design and simulation of the process. Therefore, the selection of appropriate thermodynamic model is very essential. Since the ethyl acetate unit contains polar components and has highly non-ideal behavior, activity models such as NRTL, UNIFAC and UNIQUAC were employed on Aspen Plus software to analyze the process thermodynamic. Given that Wilson activity model is not able to predict liquid-liquid equilibrium; this model was not used for ethyl acetate/water/ethanol mixture that forms two liquid phases.

To choose the most fitting thermodynamic equation, the predictions of the mentioned models in the form of binary equilibrium curves (*Txy* and *xy*) were compared with the experimental equilibrium data derived from literature. Needless to say, lower error percentage indicates more proper performance of the thermodynamic model in evaluation of the mixture behavior.

Water-ethanol mixture. In Fig. 3, *xy* and *Txy* curves for water/ethanol mixture are drawn using thermodynamic equations of NRTL, UNIFAC and UNIQUAC along with experimental equilibrium data extracted from Beebe Jr. *et al.*[18] and Kgima *et al.*[19] This mixture has a minimum boiling homogeneous azeotrope at 89.52 mol % of ethanol. As can be observed, all the three equations have relatively similar performance in prediction of equilibrium and are closely comparable with experimental data. Sum of squared errors (SSE) of each of the employed models are indicated in Table 2.
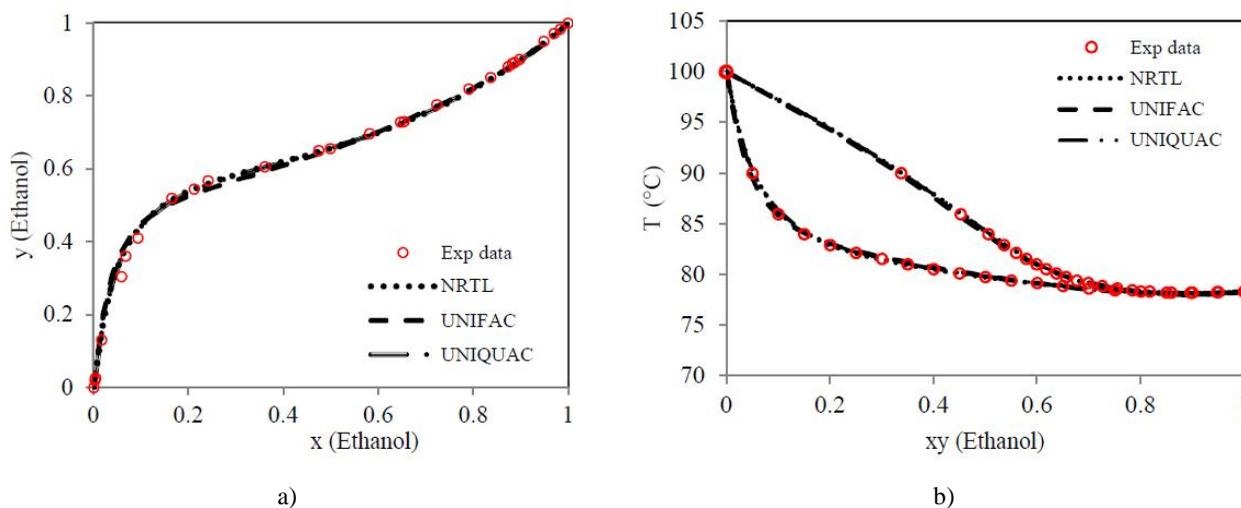

a)   b)

Fig. 3. Equilibrium curves for water-ethanol mixture: *xy* (a) and *Txy* (b)

Water-ethyl acetate mixture. Xy and *Txy* equilibrium curves for water/ethyl acetate mixture through the NRTL, UNIFAC, and UNIQUAC thermodynamic equations, along with experimental data derived from Ellis and Garbett,[20] are shown in Fig. 4. The mixture has a minimum boiling homogeneous azeotrope at 67.34 mol % of ethyl acetate. This time the prediction of all three equations has almost the same trend and is very well

adapted to the experimental data. In Table 2, the SSE value is expressed for each of the applied models. The water/ethyl acetate mixture has partial miscibility in each other, thus forming two liquid-liquid phases (in the *Txy* graph of Fig. 4, the two-phase liquid-liquid region is not shown).

Ethanol-ethyl acetate mixture. In Fig. 5, the comparison of the predictions of the NRTL, UNIFAC and UNIQUAC thermodynamic equations was performed with the experimental equilibrium data

extracted from Griswold *et al.*[21] for ethanol/ethyl acetate mixture through the *xy* and *Txy* curves. The mixture has a minimum boiling homogeneous azeotrope at 55.33 mol % of ethyl acetate. As can be seen, in the *xy* curve, all three equations have almost the same performance in predicting the equilibrium, but in the *Txy* curve, the UNIFAC model shows a higher error rate than the other models. The SSE value of each of the used models is demonstrated in Table 2.
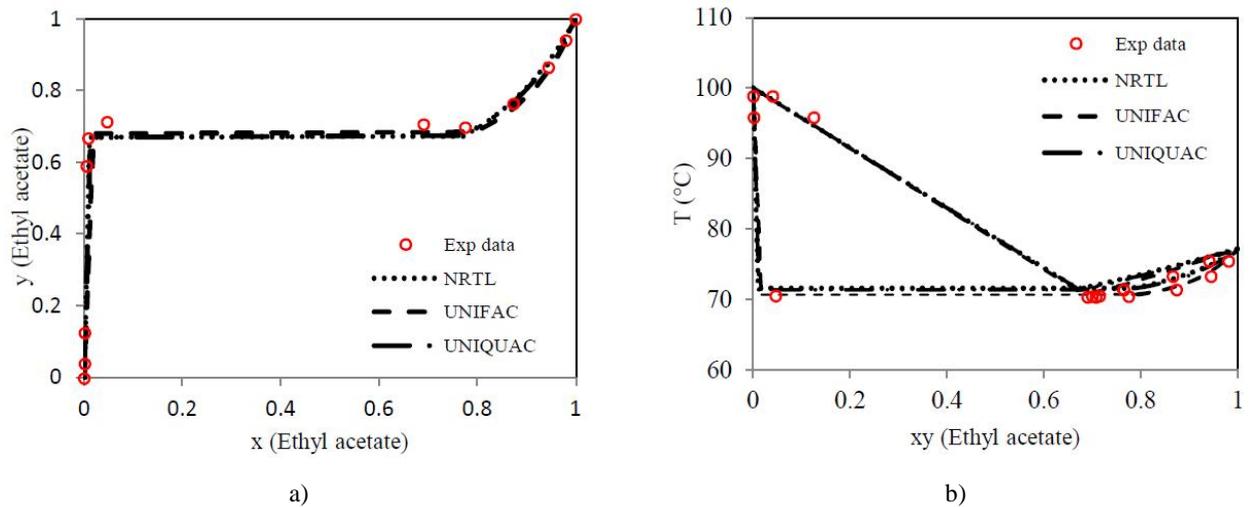

a)	b)

Fig. 4. Equilibrium curves for water-ethyl acetate mixture: *xy* (a) and *Txy* (b)
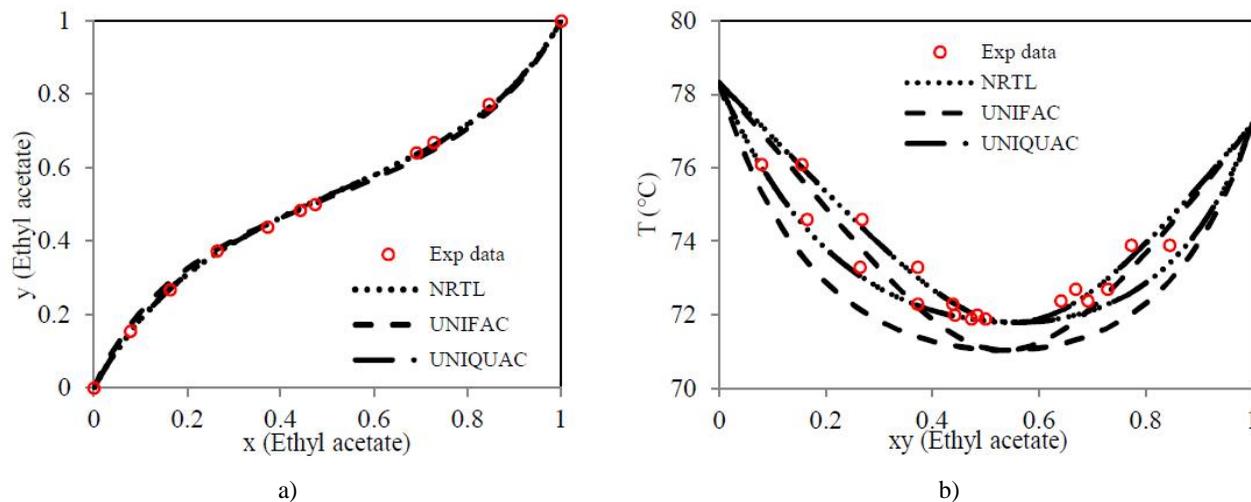

a)	b)

Fig. 5. Equilibrium curves for ethanol-ethyl acetate mixture: *xy* (a) and *Txy* (b)

According to the comparison of the results obtained in Figs. 3 and 5, as well as the SSE values reported in Table 2, the NRTL and UNIQUAC equations have the same potential in the thermodynamic analysis of the process and have less error than the UNIFAC

equation in predicting the system equilibrium. Consequently, the NRTL and UNIQUAC equations can be used for predicting the complex phase equilibriums of the studied mixture with high accuracy. In this study, the NRTL equation was employed to analyze the non-ideal

vapor–liquid equilibrium (VLE) and possible vapor–liquid–liquid equilibrium (VLLE) of the system. Also, the vapor phase was modelled by Hayden-O'Connell (HOC) equation of state at higher operating pressures.[5]

Table 2. The SSE values obtained from prediction of NRTL, UNIQUAC and UNIFAC models in equilibrium behavior analysis using *xy* and *Txy* curves for water/ethanol/ethyl acetate system

Equilibrium curve	NRTL	UNIQUAC	UNIFAC
xy water and ethanol	0.0002	0.0003	0.001
Txy water and ethanol	0.032	0.0266	0.052
xy water and ethyl acetate	0.0034	0.0033	0.0019
Txy water and ethyl acetate	0.9228	0.928	0.923
xy ethanol and ethyl acetate	0.0003	0.0003	0.0021
Txy ethanol and ethyl acetate	0. 0113	0.0115	0.11
SSE_{Sum}	0.97	0.97	1.09

3. Results and Discussion

3.1. Conceptual Design

In Fig. 6, a ternary diagram of the water/ethanol/ethyl acetate mixture, along with the boundaries and distillation regions, is shown to evaluate the feasible separation flowsheets. As it is obvious, significant two-liquid (LL) phase envelope is observed in this diagram.

The minimum-boiling homogeneous ternary azeotrope (with composition of 28.17 mol % of water, 18.12 mol % of ethanol, and 53.71 mol % of ethyl acetate) lies closely to the boundary of LL envelope. As shown in the diagram, the LL tie lines slop toward pure water node and, consequently, nearly pure water could be recovered from the LL separation. This implies that the operation with one decanter (according to Fig. 2) is desirable to remove water in ethyl acetate separation unit.
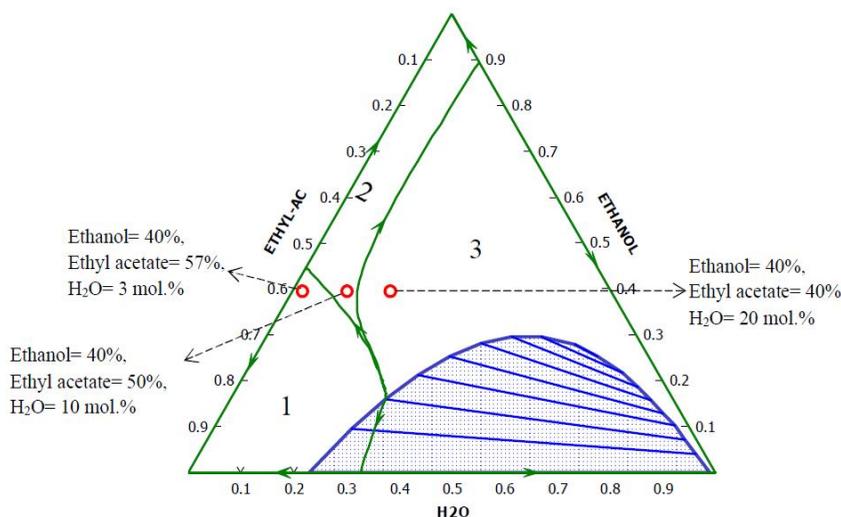

Fig. 6. Three different distillation regions for entering the feed with a constant molar percentage of ethanol in a ternary diagram of water/ethanol/ethyl acetate mixture

With regard to the distillation boundaries shown in Fig. 6, three distillation regions are visible. In the first step, the conceptual design of the separation column of the water/ethanol/ethyl acetate mixture was performed separately in three distillation regions to determine the best region for the separation process. Design operations were performed in each of the three regions for 100 kmol/h feeds with a constant mole fraction of ethanol (40 mol %) at the reflux ratio of 1.5 and atmospheric pressure to provide the correct comparison.

Conceptual design in triple distillation regions. Based on Fig. 6, in the first distillation region, ethyl acetate is the stable node; therefore, it is considered as the main component in the bottom product. In this

region, the feed contains 40 mol % of ethanol, 57 mol % of ethyl acetate, and 3 mol % of water. Conceptual design was carried out in different recovery amounts of ethyl acetate at the bottom of the column, and the separation feasibility was investigated in each case. It is worth mentioning that in identical recoveries of ethyl acetate, the case was selected, which provides the minimum number of required theoretical stages and duty in the condenser and the reboiler and the maximum purity of the products. Fig. 7 illustrates the obtained values from the conceptual designs (the number of stages and energy duty) in various ethyl acetate recoveries at the bottom of the column. By increasing the recovery percent of ethyl acetate, the number of equilibrium stages elevates, and energy duty first decreases to 40% recovery of ethyl acetate and then rises. It should be noted that there was no significant change in the purity of the products, which is why results for products purity were not reported.

According to the conceptual design results in Fig. 7, the optimal mode for distillation operation in terms of the number of stages and energy duty was considered in recovery of 35 % ethyl acetate. In this recovery amount, the number of required stages is 10 and duty is 3223.06 kW.

The ternary diagram of optimum mode of design with feed stream and concentration profiles of the components in the rectifying and stripping sections of the heterogeneous distillation column is depicted in Fig. 8. As already mentioned, the design is feasible if the concentration profiles of the components in the rectifying and stripping sections intersect each other. Since the feed has entered the first region, the products were also received at the same region.
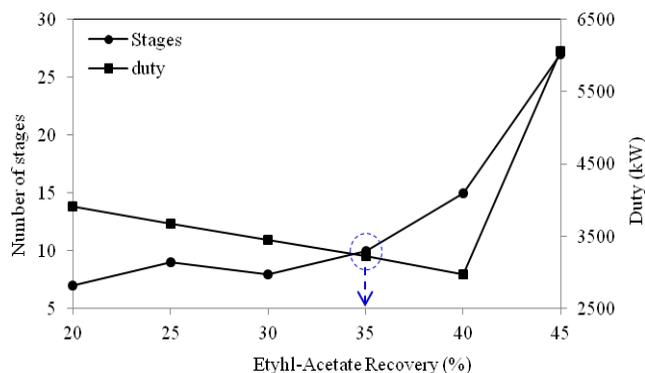
As Fig. 6 reflects, in the second distillation region, ethanol is the stable node, so it is considered to be the main component in the bottom product. In this region, the feed contains 40 mol % of ethanol, 50 mol % of ethyl acetate, and 10 mol % of water. Conceptual design was carried out in different ethanol recoveries at the bottom of the column, and in each case, the feasibility of separation was investigated. It should be noted that in similar recoveries of ethanol, a mode was selected in which the number of equilibrium stages and energy duty in the condenser and the reboiler were minimal and the purity of the products was at the maximum level. In Fig. 9, the values obtained from conceptual designs (the number of equilibrium stages, energy duty, and purity of products) are demonstrated in different recoveries of ethanol at the bottom of the column. As can be observed, with increasing ethanol recovery percent, the number of equilibrium stages has a nonlinear trend, the minimum value of which was in 45 % recovery of ethanol and energy duty had a decreasing trend of about 34 %. Ethanol purity also shows a decreasing trend from 59.2 to 47 mol %, which is equivalent to a decrease of about 21 %.

By comparing the results of the conceptual design in Fig. 9, the optimum design mode was considered in recovering 45 % of ethanol, because the number of stages and energy duty in this recovery were minimum, that is, 3 and 2812.9 kW, respectively. Although the purity of ethanol in the mentioned recovery amount was at its minimum, its variations are much lower compared to the changes in the number of trays and duty, thus, it is of lower significance degree.
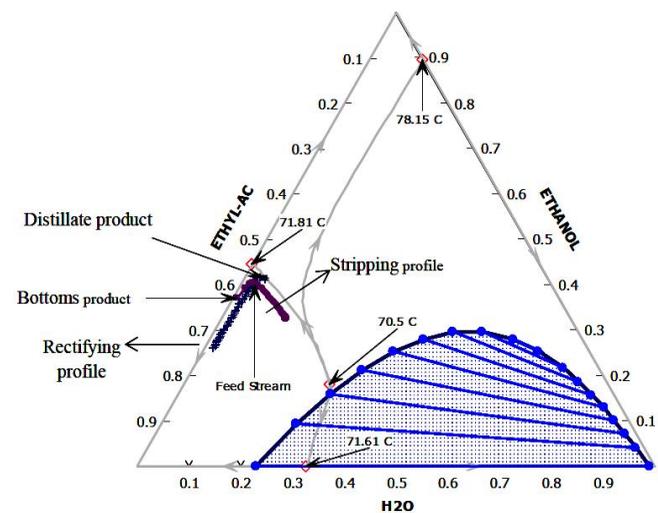

Fig. 7. The number of required stages and energy duty in various ethyl acetate recoveries at the bottom of the column for feed containing 40 mol % of ethanol, 57 mol % of ethyl acetate and 3 mol % of water

Fig. 8. Ternary diagram for a feed containing 40 mol % of ethanol, 57 mol % of ethyl acetate, and 3 mol % of water in recovery amount of 35 % ethyl acetate
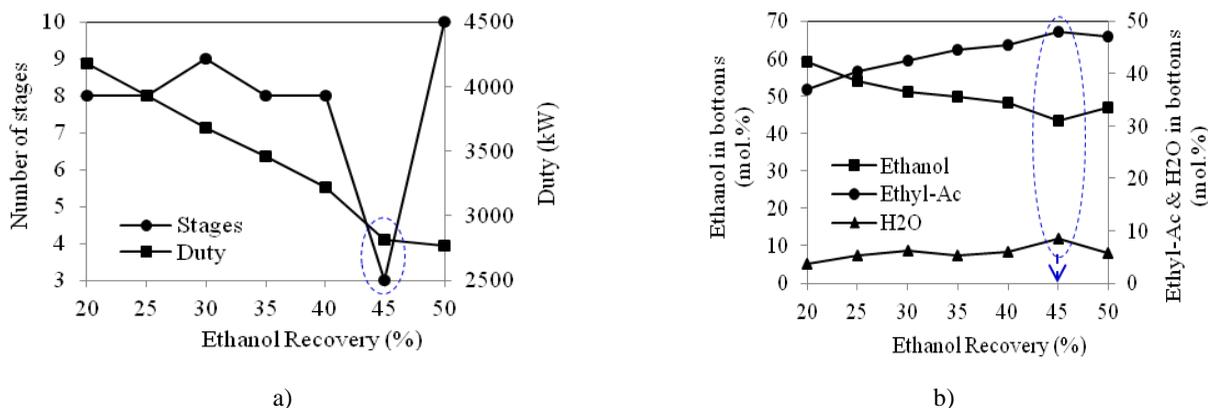
Fig. 9. The number of stages and energy duty (a) and purity of products at the bottom of the column (b) in different recoveries of ethanol for feed containing 40 mol % of ethanol, 50 mol % of ethyl acetate and 10 mol % of water

In Fig. 10, the ternary diagram of the optimal conceptual design mode, along with feed stream and component concentration profiles in the rectifying and stripping sections of the three-phase tower, is presented. Obviously, the intersection of the concentration profiles of the rectifying and stripping sections and the location receiving the products took place in the second region.

Regarding Fig. 6, in the third distillation region, water is the stable node, and therefore the main component is considered in the bottoms product. In this region, the feed contains 40 mol % of ethanol, 40 mol % of ethyl acetate, and 20 mol % of water. Similar to the first and second regions, conceptual design was carried out in different water recoveries at the bottom of the column, and the feasibility of separation was investigated in each case. It should be noted that in similar recoveries of water, the mode was chosen in which the number of theoretical stages and energy duty were the minimum and the purity of the products was at the maximum level.

The values obtained from conceptual designs (the number of stages, energy duty, and the purity of products) in various water recoveries at the bottom of the column are provided in Fig. 11. It is known that by increasing water recovery percent, the number of equilibrium stages has approximately increased trend, but energy duty until 35 % recovery had a decreasing trend, followed by a fluctuating trend. Water purity also exhibits an alternating trend, ranging from 28.5 to 38.4 mol %, an increase of about 35 %. With the analysis of conceptual design results, Fig. 11 shows that in 35% recovering of water, the number of stages and energy duty are minimized, likewise, water purity in this amount of recovery will reach its minimum. Therefore, the optimal design was considered in 40 % water recovery. In this amount of recovery, the number of required stages is 10 and duty is 3565.55 kW.
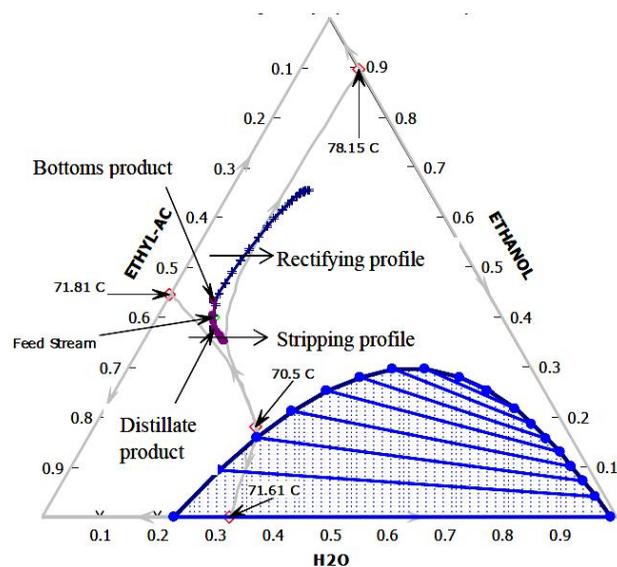

Fig. 10. Ternary diagram for a feed containing 40 mol % of ethanol, 50 mol % of ethyl acetate and 10 mol % of water in recovery amount of 45 % ethanol

The ternary diagram of the optimal design mode with the feed stream and the concentration profiles of the components in the rectifying and stripping sections of the heterogeneous distillation tower are shown in Fig. 12. As expected, the intersection of the concentration profiles of the rectifying and stripping sections is located in the third region.

In Table 3, the comparison of the results obtained from conceptual designs of all the three distillation regions is presented in the optimal recovery value from the key component (stable component in each region). It is clear that the implementation of the three-phase distillation process in terms of the number of stages and energy duty in the second region is more satisfactory than in the other regions.
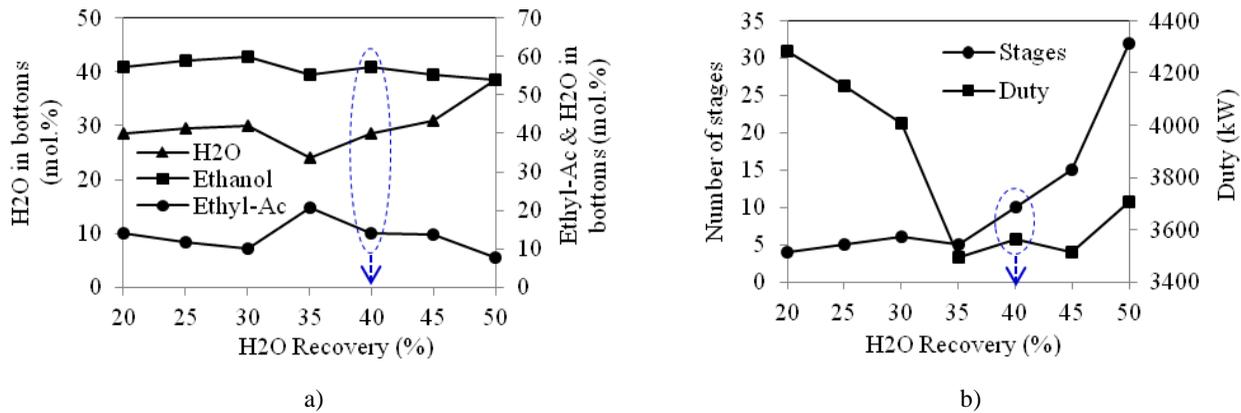
a) b)

Fig. 11. Number of required stages and energy duty (a) and purity of products at the bottom of the column (b) in different water recoveries for feed containing 40 mol % of ethanol, 40 mol % of ethyl acetate and 20 mol % of water

The presented designs were carried out at constant R, thus, in order to ensure the achieved results, in the next step, R analysis on conceptual design was performed in all three distillation regions.

Fig. 12. Ternary diagram for a feed containing 40 mol % of ethanol, 40 mol % of ethyl acetate and 20 mol % of water in water recovery 40 %

Table 3. Comparison of the obtained results from conceptual designs of all three distillation regions with constant mole fraction of ethanol at constant R

Distillation region	The number of equilibrium stages	Energy duty, kW
First region	10	3223.06
Second region	3	2812.9
Third region	10	3565.55

Analysis of reflux ratio in the triple distillation regions. At this stage, the effect of R on the conceptual design of the three-phase tower was evaluated for each distillation region to determine the optimum R in each region. This process was carried out in the optimal recovery amount obtained from the previous step for the key component in each region and on the same feeds containing the same mole fraction of ethanol. It should be noted that in this situation, due to change in reflux ratio, there was no significant change in the purity of the products.
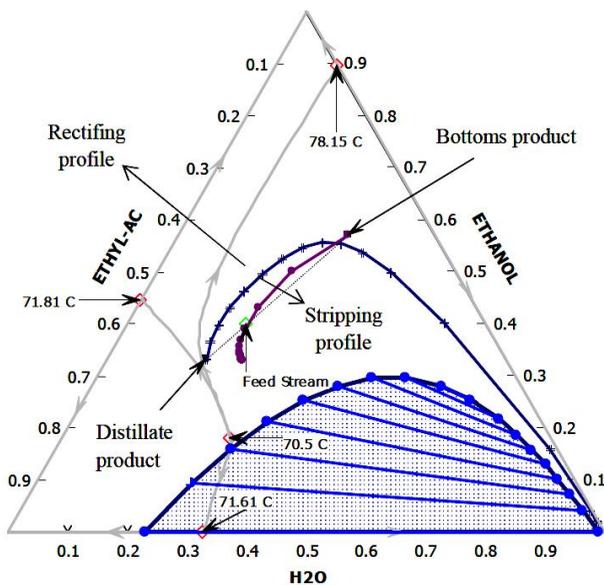
In the first distillation region, the conceptual design of the separation tower was accomplished by changing the R amount in 35 % recovery of ethyl acetate (optimal amount). The results and its effects on the number of theoretical stages and energy duty are presented in Fig. 13. According to the results shown in Fig. 13, the minimum number of stages required for the separation was 7 stages, which is achieved at $R = 3.5$. The minimum reflux ratio (R_{min}) in which separation was feasible was equal to 1. Therefore, the optimal reflux ratio (R_{opt}) was considered to be 1.5 times the minimum value according to general principle in distillation processes. The results of the conceptual design in $R_{opt} = 1.5$ are provided in Table 4.

The effect of R on the conceptual design of the three-phase tower in the second distillation region was investigated in 45 % ethanol recovery (optimal amount). Fig. 14 shows the number of stages and energy duty based on R. It is clear from Fig. 14 that the minimum number of stages required for the separation is 3 stages obtained at $R = 1$. The value of R_{min}, in which separation

is feasible, is equal to 0.2. The same as before, R_{opt} was 1.5 times its minimum value. The results of conceptual design in $R_{opt} = 0.3$ are reported in Table 4.

The conceptual design of the tower in the third distillation region was performed by changing R in 40 % water recovery (optimal amount). Its results and impact on the number of stages and energy duty are illustrated
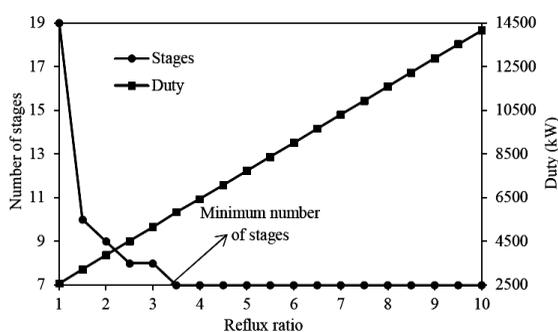
in Fig. 15. According to the results presented in Fig. 15, the minimum number of stages required for the separation was 9, which is achieved in $R = 1.7$. The value of R_{min}, where the separation was feasible, was 0.8. Similar to the other regions, the R_{opt} was 1.5 times its minimum value. The results of conceptual design at $R_{opt} = 1.1$ are given in Table 4.
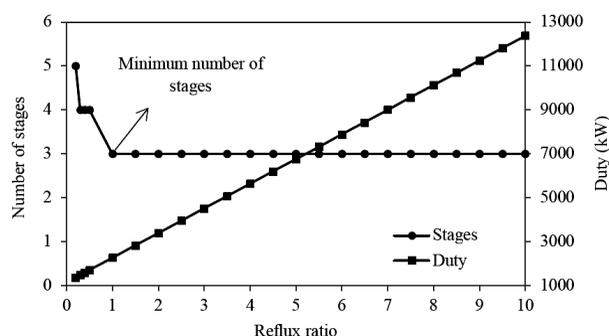
Fig. 13. The effect of R on the number of theoretical stages and energy duty in the first distillation region by 35 % ethyl acetate recovery at the bottom of the column

Fig. 14. The effect of R on the number of theoretical stages and energy duty in the second distillation region by 45 % ethanol recovery at the bottom of the column
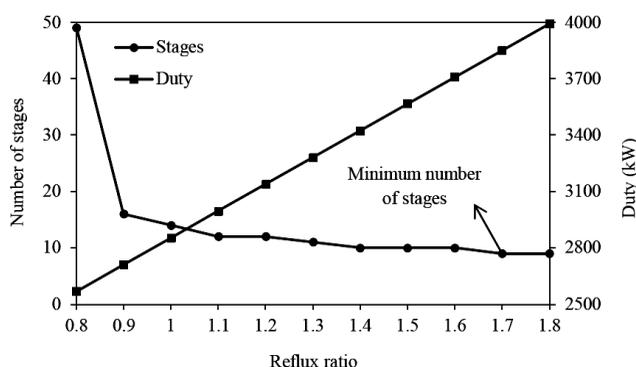
Fig. 15. The effect of R on the number of theoretical stages and energy duty in the third distillation region with 40 % water recovery at the bottom of the column

Table 4. The results of the conceptual design of the three-phase tower in the triple distillation regions in R_{opt}

Distillation region	R	The number of equilibrium stages	Energy duty, kW	Composition at bottom of the column, mol %		
				Ethyl acetate	Water	Ethanol
First region	1.5	10	3223.6	61.86	0.93	37.21
Second region	0.3	4	1456.077	48.19	8.44	43.37
Third region	1.1	12	2995.66	14.29	28.57	57.14

By comparing energy duty, the number of stages, and the composition of the products in the three distillation regions, it is clear that the second distillation region is the best region for accomplishing the separation process of this non-ideal mixture. Another advantage of the second region is that, by separating ethanol from the bottom of the tower, the overhead product mainly

includes ethyl acetate and water, which can be easily separated in the decanter due to the low miscibility of these two components in each other. The only disadvantage of the region is its small expanse, which makes the region less flexible for the separation process. Of course, this shortcoming can be easily overcome by slight modifications of the process operating pressure.

It should also be noted that in the first region, by separating ethyl acetate from the bottom of the column, in addition to the high energy consumption, the overhead product contains more water and ethanol (a miscible azeotropic mixture) that cannot be easily separated. In the third distillation region, in addition to the high energy consumption and the number of stages to separate water from the bottom of the column, the overhead product contains ethanol and ethyl acetate (a miscible azeotropic mixture) that are not easily separable. As of yet, it has been determined that the optimal range of performance of a three-phase distillation unit for feeds with constant mole fraction from one of the components in different R_s is the second distillation region. In order to provide a more comprehensive assessment and to ensure the optimum region for distillation, the conceptual design of the tower for various feeds was carried out on the mass balance lines leading to the stable node in each region.

Conceptual design on mass balance lines in triple distillation regions. As mentioned above, in the first step of conceptual design, selected feeds with a constant mole fraction of ethanol in each region were investigated. In this step, in order to arrive at a decisive conclusion to identify the best distillation region, conceptual design for different feeds was performed on the mass balance lines leading to the stable node in each region. It should be noted that, for proper comparison, all the designs were carried out at operating pressure of 1 atm and reflux ratio of 1.5 for 100 kmol/h feed.

Fig. 16 shows the location of feeds on the mass balance line leading to the stable node of the first region. Conceptual design was carried out in different recoveries of the stable component for four specified feeds, and the separation feasibility was evaluated. The results of the conceptual design for the feeds included in the first region in the optimal recovery value of the stable component at the bottom of the tower are given in Table 5.

As shown in Table 5, for feeds containing 60, 65, 75, and 90 mol % of ethyl acetate, the highest purities of ethyl acetate at the bottom of the tower were 84.7 % (in 30 % ethyl acetate recovery), 91.2 % (in 45% ethyl acetate recovery), 95.7 % (in 60 % ethyl acetate recovery), and 98.9 % (in 75 % ethyl acetate recovery), respectively.

The location of the feeds considered on the mass balance line leading to the stable node of the second region is shown in Fig. 17. As before, for the 4 feeds shown, the separation tower was designed in different amounts of recovery from the stable component, and the

separation feasibility was investigated. The results of the conceptual design for the included feeds in the second region in the optimal recovery value of the stable component at the bottom of the column are presented in Table 6.
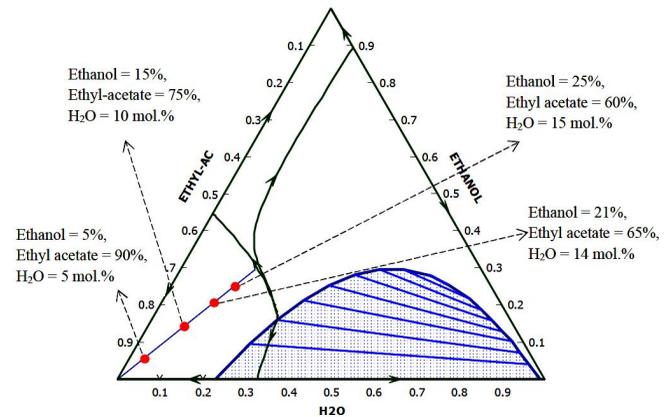

Fig. 16. Composition of various feeds on the mass balance line leading to the stable node of the first distillation region

Table 5. The results of conceptual design for different feeds on the mass balance line in the first region

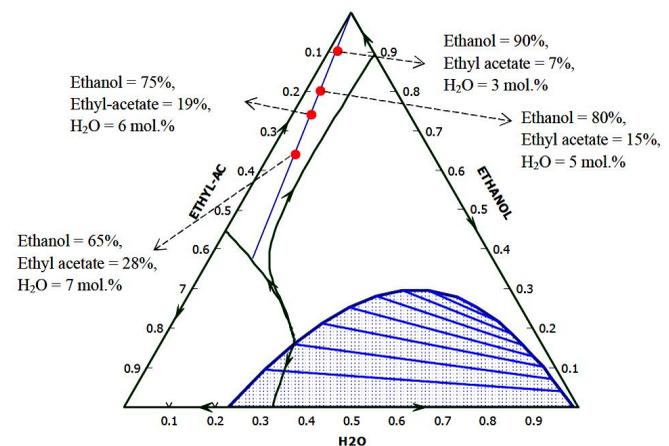| Mole percent of ethyl acetate in the feed	Ethyl acetate purity at the bottom of the column, mol %	The number of stages	Duty, kW
60	84.7	10	3703.67
65	91.2	17	3184.59
75	95.7	11	2446.59
90	98.9	13	1467.22

Fig. 17. Composition of various feeds on the mass balance line leading to the stable node of the second distillation region

Table 6. Results from conceptual design for different feeds on the mass balance line in the second region

Mole percent of ethanol in the feed	Ethanol purity at the bottom of the column, mol %	The number of stages	Duty, kW
65	91	17	2926.58
75	92.5	11	2243.25
80	93.5	7	3024.21
90	96.6	8	2378.86

Table 6 shows that for feeds containing 65, 75, 80, and 90 mol % of ethanol, the highest purities of ethanol at the bottom product were 91 % (in 60% ethanol recovery), 92.5 % (in 70 % ethanol recovery), 93.5 % (in 50 % ethanol recovery), and 96.6 % (in 65 % ethanol recovery), respectively.

Fig. 18 shows the location of the considered feed on the mass balance line leading to the stable node of the third region. Conceptual design was carried out in different recoveries of the stable component for the five specified feeds and the separation feasibility was evaluated. The conceptual design carried out in this region revealed the infeasibility of separation for most of the used feeds. Separation was feasible only for a feed containing 50 % mole of water, and the highest purity for water at the bottom of the column was 82.8 % (in 65 % water recovery). In this case, the required number of stages is 4 and energy duty is 2906.67 kW. Therefore, the third region has a small flexibility for the separation of the desired three-phase mixture.
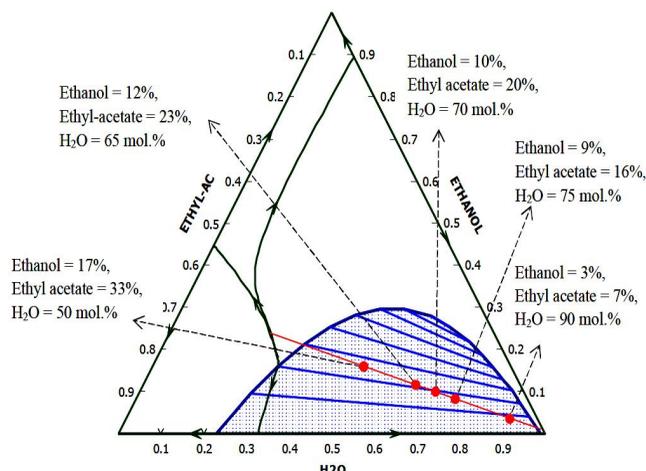
Fig. 18. Composition of different feeds on the mass balance line leading to the stable node of the third distillation region

By comparing the results of conceptual design in the three distillation regions, it should be mentioned that the second region, in terms of the number of required stages and energy duty, has a better status than the other regions, which reflects the simplicity of the separation operations in this region. Although the purity of the product in this region is slightly lower than that of the first region, it can be easily mitigated by slight changes in the operating pressure of the process, which will be appraised in the next section.

Finally, based on the three indices characterizing purity, the number of stages and energy duty, it can be concluded that the second region is the best for performing the three-phase distillation of the desired mixture. Therefore, in the following, the main focus of this research was set on the second distillation region and the effect of pressure on the separation flexibility was investigated for feeds with concentration less than 80 mol % of the stable component (ethanol). Feeds with concentration above 80 mol % of the stable component were not considered as the evaluation criterion due to high purity from the beginning.

Effect of operating pressure. Since ethyl acetate and water have partial miscibility in each other, therefore, LL phase separation would only be possible at low ethanol concentrations in distillate, and a higher ethanol content prevents phase splitting. Consequently, at this stage, in order to separate the maximum ethanol in the bottom product, the three-phase distillation process was evaluated under different operating pressures in the second region. In this regard, the conceptual design of the azeotropic tower was carried out at pressures of 0.8, 1, 2, 3, 6, and 8 atm to separate the various feeds containing 65 mol% of the stable component (ethanol), and energy consumption, the number of stages, and products purity were determined. The reflux ratio was fixed and equal to 1.5. The distillation boundaries along with the location and composition of the feeds in each pressure are specified in Fig. 19.

As shown in Fig. 19, with a change in pressure, the azeotropic points and distillation boundaries are displaced in the ternary diagram. At lower pressures, the second distillation region becomes narrower and longer and becomes wider and shorter at higher pressures. Due to excessive shrinkage of the second distillation region, it is not possible to perform three-phase distillation operations at pressures higher than 8 atm.
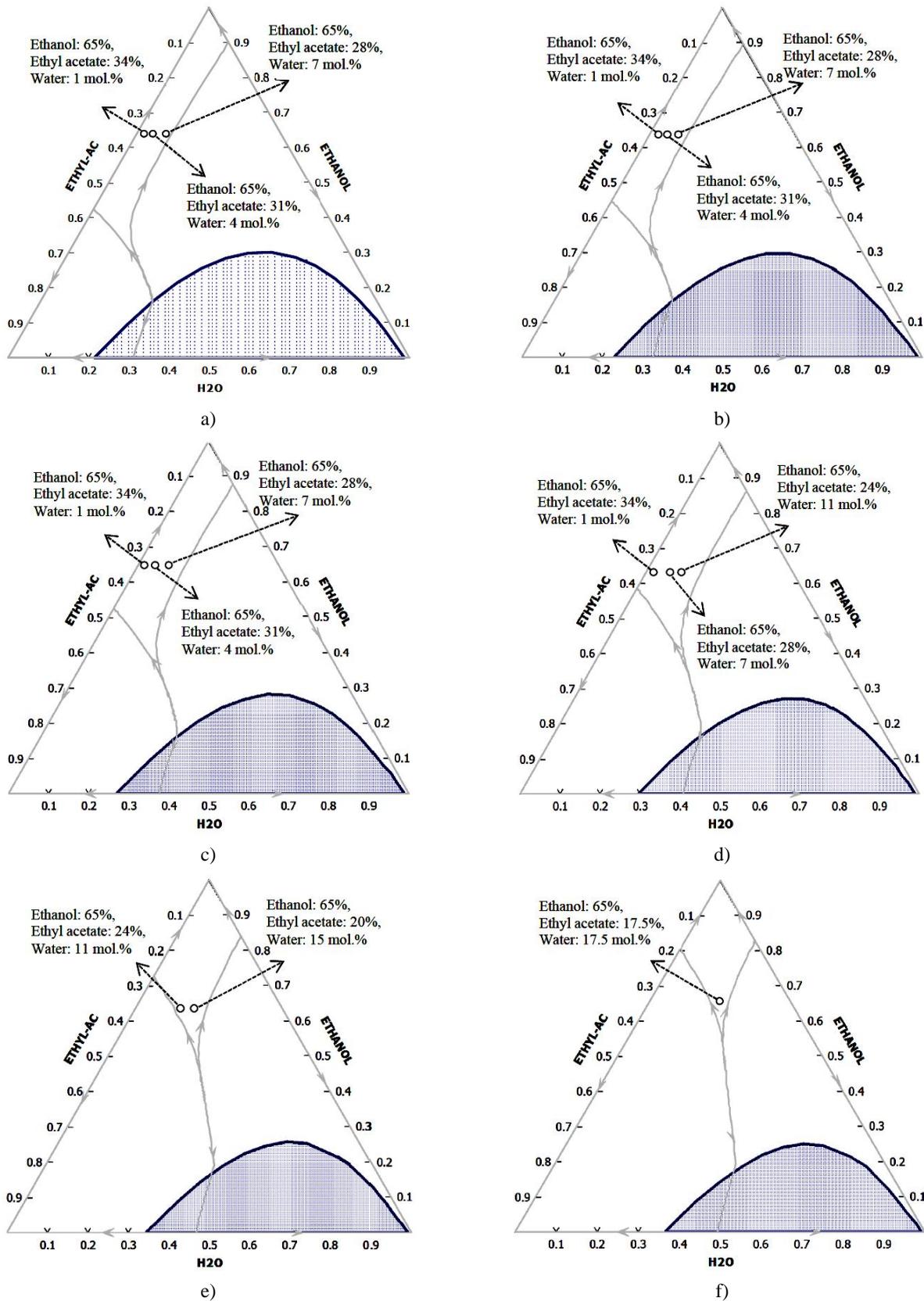
Fig. 19. The distillation boundaries and compositions of feeds included in the second distillation region at different pressures: 0.8 atm (a), 1 atm (b), 2 atm (c), 3 atm (d), 6 atm (e), and 8 atm (f)

The results of conceptual design at various pressures in the optimal recovery value of the stable component at the bottom of the column for each feed are reported in Table 7. It should be noted that the reported purities at 0.8 and 1 atm pressures were obtained at optimal recovery values of 45 and 60 % of ethanol at the bottom of the column. At 2 atm pressure, the optimal recovery value of ethanol at the bottom of the column is 45 % for feeds containing 28 and 31 mol % of ethyl acetate, and 40 % for a feed containing 34 mol % of ethyl acetate, which results in product with the given purities in Table 7. At 3 atm pressure, the reported purities for feeds containing 24 and 28 mol % of ethyl acetate were achieved at the optimal recovery value of 40 % ethanol and for a feed containing 34 mol % of ethyl acetate – at the optimal recovery value of 20 % of ethanol at the bottom of the column. At 6 atm pressure, due to the shrinkage of the second distillation region, only two feeds were considered in this region. For feed containing 20 mol % of ethyl acetate, no conceptual design was possible at any recovery amount. However, for a feed containing 24 mol % of ethyl acetate, the highest purity was achieved at 30 % of ethanol recovery at the bottom of the column. At 8 atm pressure, the extent of the second distillation region reached its lowest limit, so only a feed containing 17.5 mol % of ethanol could be considered. For the mentioned feed, conceptual design was not possible at any recovery amount. At 0.8 atm pressure (and lower pressures), in addition to the cost of creating vacuum conditions, the lack of design flexibility due to the narrowing of the second distillation region is one of the disadvantages of using these operating conditions. At pressures higher than 3 atm, the high-pressure costs on the one side and the shrinkage of the second distillation region, which leads to the achievement of products with not so high purity, on the other hand, does not justify the application of such operating conditions. It should also be noted that with increasing pressure, energy duty increases due to the rising temperature of the bubble and dew points of the mixture.

Table 7. Obtained results from conceptual design at various pressures

Operating pressure, atm	Feed composition, mol %	Ethanol purity at the bottom of the column, mol %	Number of stages	Duty, kW
0.8	65 % ethanol, 28 % ethyl acetate, 7 % water	84.7	6	2304.43
	65 % ethanol, 31 % ethyl acetate, 4 % water	91.4	8	2424.92
	65 % ethanol, 34 % ethyl acetate, 1 % water	93.7	8	2449.86
1	65 % ethanol, 28 % ethyl acetate, 7 % water	91	17	2926.5
	65 % ethanol, 31 % ethyl acetate, 4 % water	93.8	33	2976.19
	65 % ethanol, 34 % ethyl acetate, 1 % water	91.3	21	2880.16
2	65 % ethanol, 28 % ethyl acetate, 7 % water	92.2	24	3471.02
	65 % ethanol, 31 % ethyl acetate, 4 % water	94.3	44	3485.75
	65 % ethanol, 34 % ethyl acetate, 1 % water	93.6	27	3630.17
3	65 % ethanol, 24 % ethyl acetate, 11 % water	88.4	16	3582.22
	65 % ethanol, 28 % ethyl acetate, 7 % water	93.6	42	3629.68
	65 % ethanol, 34 % ethyl acetate, 1 % water	88.1	15	4196.99
6	65 % ethanol, 20 % ethyl acetate, 15 % water	–	–	–
	65 % ethanol, 24 % ethyl acetate, 11 % water	78.4	29	3626.35
8	65 % ethanol, 17.5 % ethyl acetate, 17.5 % water	–	–	–

Comparing the results presented in Table 7, it is clear that the highest purity (94.3 mol % of ethanol) was achieved at the 2 atm operating pressure for the feed containing 65 mol % of ethanol, 31 mol % of ethyl acetate, and 4 mol % of water. Conceptual design for other feeds also resulted in purities higher than 90 mol %. The reason for this is the greater extent of the second distillation region at this pressure compared to other operating pressures, which increase the separation process flexibility of the aforementioned azeotropic mixture. As a result, the pressure of 2 atm is introduced as the optimal operating pressure for a three-phase distillation process. Although the number of stages required in this operating pressure is somewhat high, it is easy to overcome this problem by changing the reflux ratio (discussed in the following section).

The impact of the reflux ratio at the optimal pressure. In this section, the effect of *R* on conceptual

design at optimal operating pressure has been investigated. Fig. 20 exhibits the effect of R on the number of equilibrium stages and energy duty at the pressure of 2 atm. Obviously, with increasing R, the number of equilibrium stages decreases, and energy duty elevates. Initially, with an increase in R from 1.4 to 2.5, a sharp decrease occurs in the number of equilibrium stages, reaching from 57 to 23; then, the decreasing trend of the number of stages occurs with a gentle slope and eventually becomes fixed (17 stages). This is why energy duty raises linearly from the beginning.

It is clear from Fig. 20 that the minimum R value obtained at the pressure of 2 atm, which leads to feasible design, is 1.4. Regarding the general principle of $R_{opt} = (1.2–2) \cdot R_{min}$ and the presented profiles in Fig. 20, a value should be considered as optimal R, in which the number of required stages and duty are minimal. Therefore, the reflux ratio of 2.5 is persuasive. The results of conceptual design in the optimum R value are given in

Table 8. It should be noted that by changing the reflux ratio, there was no significant change in the purity of the products.
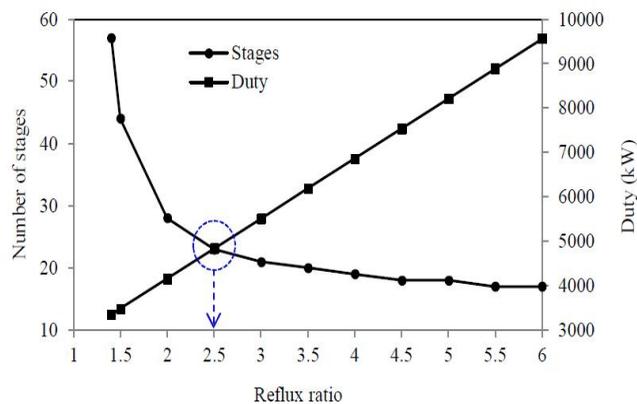

Fig. 20. Effect of R on the number of equilibrium stages and duty for feed containing 65 mol % of ethanol, 31 mol % of ethyl acetate, and 4 mol % of water at the pressure of 2 atm

Table 8. The results of conceptual design in optimal R for a feed containing 65 mol % of ethanol, 31 mol % of ethyl acetate, and 4 mol % of water at 2 atm

Optimal R	Number of stages	Feed stage	Duty, kW	Composition at the bottom of the column, mol %		
				Ethanol	Ethyl acetate	Water
2.5	23	4	4836.37	95	4.3	0.6

Relying on the results of this section, it can be argued that in complex systems with numerous azeotropic points, it is possible to achieve optimal purities and costs by implementing a three-phase distillation in the correct distillation region. Based on the results presented in Table 8, the rigorous design of the three-phase distillation column is carried out, which is discussed in the next section.

3.2. Rigorous Design

The rigorous simulation and design of the three-phase columns has always been a challenging issue due to the difficulty of convergence caused by the highly non-ideal system. This strife can be overcome using the correct implementation of conceptual design model as the base platform for the rigorous design of the non-ideal column. Consequently, in this section, the results of the conceptual design are used for rigorous design of the three-phase distillation column so that logical simulation can be implemented. For this purpose, *Radfrac* tower was used in the environment of Aspen Plus software

with 23 equilibrium stages. This type of towers has the ability to perform simulation based on both thermodynamic equilibrium (equilibrium base) and mass transfer (rate base). Nevertheless, since the rate base model is only implemented for two-phase vapor-liquid processes, in this study, simulation was conducted based on thermodynamic equilibrium.

In the first step of the rigorous design, R is considered a constant value and equal to the optimal value obtained from the conceptual design (*i.e.*, 2.5), and by changing the bottoms flow rate, which mostly contains the key component of ethanol (the stable component in the second distillation region), energy duty and composition of the bottoms product were evaluated. In the next step, the bottoms flow rate of the column was fixed and equal to the value obtained from the conceptual design (*i.e.*, 31 kmol/h), and the effect of R on energy duty and composition of the bottoms product were investigated. Eventually, in the final step, a rigorous design was made in the optimal values of bottoms flow rate and R (as the required 2 degrees of freedom).

The effect of bottoms flow rate. The impact of bottoms flow rate on energy duty and composition of the bottoms product in $R = 2.5$ are shown in Fig. 21. As previously mentioned, the entering feed was 100 kmol/h of the non-ideal ternary mixture containing 65 kmol/h of ethanol. Therefore, the maximum flow rate for the bottom of the column is considered to be 65 kmol/h. It is clear that as the bottoms flow rate increases, the energy duty decreases linearly. In fact, with increasing bottoms flow rate, the boil-up ratio diminishes and therefore the reboiler duty is reduced. On the other hand, with an increase in the flow rate from 28 kmol/h, a drastic drop in the purity of ethanol at the bottom of the column occurs, resulting in a significant increase in the amount of ethyl acetate at the bottom of the column. Consequently, to reach a high purity product (at least 98 mol % of ethanol), the optimum bottoms flow rate was considered to be 28 kmol/h, in which duty is 4806.89 kW.
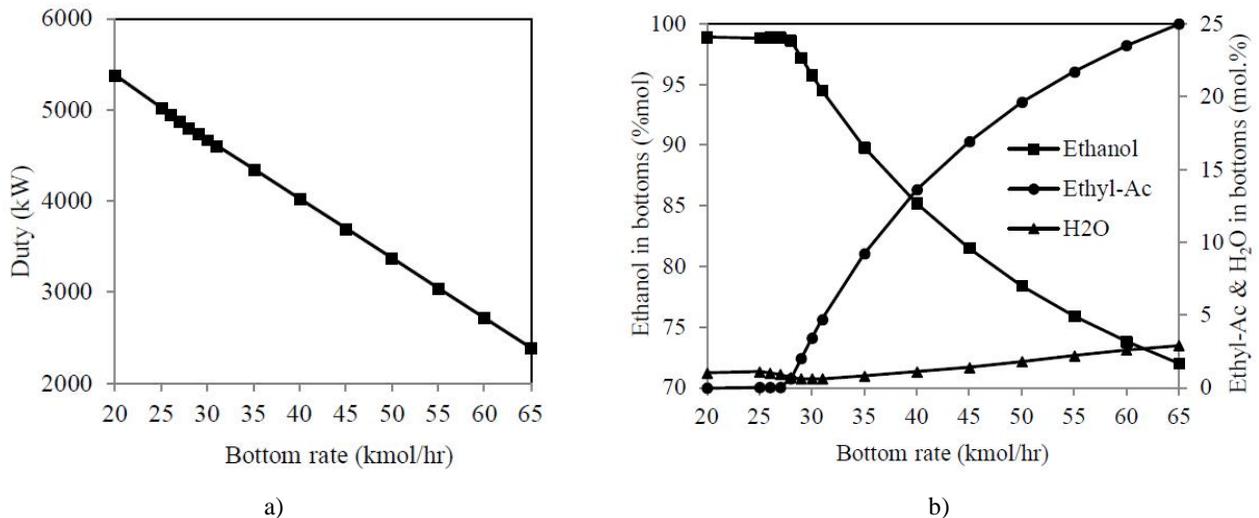

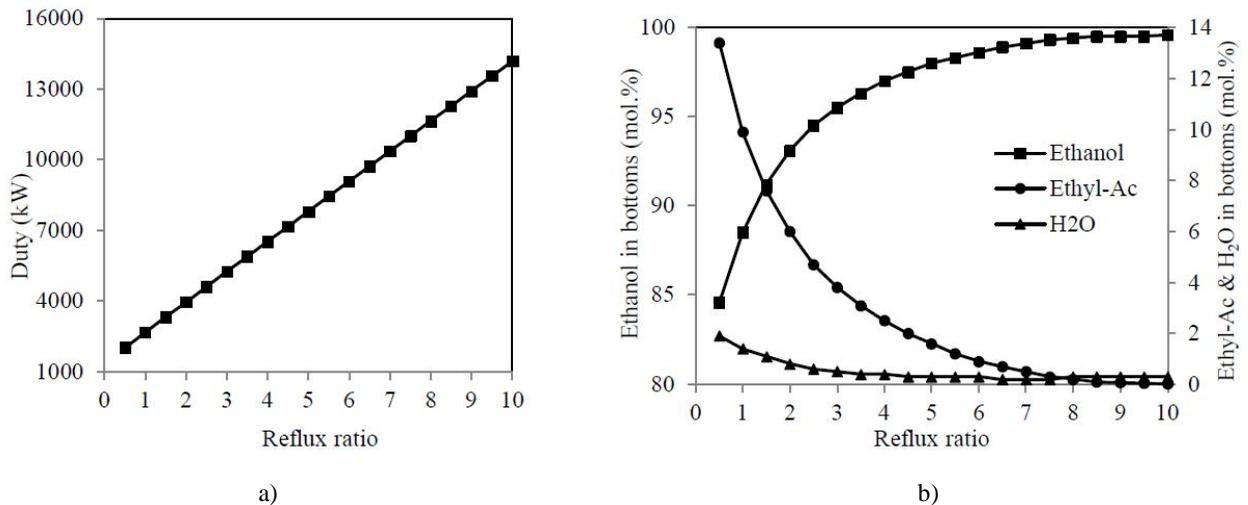**Fig. 21.** Effect of bottoms flow rate on energy duty (a) and bottoms product composition (b) at $R = 2.5$

Fig. 22. Effect of R on energy duty (a) and bottoms product composition (b) at bottoms flow rate of 31 kmol/h

The effect of reflux ratio. The effect of R on energy duty and composition of the product at constant bottoms flow rate (31 kmol/h) is depicted in Fig. 22.

As can be observed, with raising R, energy duty increases linearly and ethanol purity increases nonlinearly. Clearly, at $R > 7$, there is no marked change in the purity of ethanol, and only energy duty is enhanced. Here, in order to reach 98 mol % of ethanol and prevent excessive energy consumption, the optimum value of R was considered 5. Energy duty in this R is 7807 kW. In

the next step, the final rigorous simulation was performed at optimum values of bottoms flow rate and *R*.

Final simulation. At this stage, a final simulation of the separation unit of the three-phase mixture of ethanol/ethyl acetate/water was performed at the optimum values of bottoms flow rate and *R*, that is, 28 kmol/h and 5, respectively. The obtained results are presented in Table 9.

Table 9. Final results obtained from the rigorous simulation

Operating pressure, atm	Optimum *R*	Number of stages	Energy duty, kW	Composition at the bottom of the column, mol %			Composition at the top of the column, mol %		
				Ethanol	Ethyl acetate	Water	Ethanol	Ethyl acetate	Water
2	5	23	8163.72	99.25	trace	0.75	0.96	88.2	10.84

In order to be used as a raw material in the chemical industry and a fuel in the engines, ethanol must have the purity equal or higher than 98.7 mol % (99.5 wt %), according to the international standards (EN 15376, ASTM D 4806) [13, 22, 23]. As indicated in Table 9, the implementation of rigorous simulation and design based on the conceptual design principles resulted in the achievement of ethanol with purity (99.25 mol %) higher than defined standards with the minimum number of stages and energy duty. Also, the overhead product components, which mainly include ethyl acetate and water, can be easily separated in a decanter with high purity (due to immiscibility).

In the end, it should be noted that the proper strategies for performing rigorous designs could be achieved by employing effective methods in conceptual design of non-ideal units. In so doing, three-phase distillation units can be designed and launched at industrial scales with minimal capital and operating costs.

4. Conclusions

Three-phase distillation is an attractive process for the separation of highly non-ideal and heat-sensitive mixtures. This research focused on the conceptual and rigorous design of the three-phase distillation unit of ethyl acetate complex system as an innovative assessment to select the best separation strategy.

In this regard, the thermodynamic behavior of the mixture was first analyzed using various activity models, and finally, the NRTL model has shown more appropriate performance than the others, and it was used to predict the phase equilibrium trend. Then, the separation feasibility and conceptual design of the three-phase column in the triple distillation regions were performed for different feed locations, reflux ratios and product recoveries using the extended BVM. By comparing the obtained results, it was found that the optimal region of performance of the three-phase distillation unit based on the three indices of products purity, the number of stages, and energy duty is the second distillation region with ethanol as the stable component. Subsequently, by expanding the second distillation region and increasing the flexibility of separation through altering operating pressure, the conceptual design of three-phase distillation column was optimized at 2 atm pressure with 23 required stages.

Based on the conceptual design results, rigorous simulation of the three-phase distillation unit was easily accomplished, and the optimal values of bottoms flow rate and reflux ratio were obtained 28 kmol/h and 5, respectively. In these circumstances, ethanol with the purity of 99.25 mol % was achieved by energy consumption of 8163.72 kW. The achievements of this research can pave the way for choosing the right strategies for the separation of ethyl acetate complex systems in pilot and industrial scales with maximum energy saving.

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ТРИФАЗНА ДИСТИЛЯЦІЯ ЕТИЛАЦЕТАТУ/ВОДИ/ЕТАНОЛУ: МОЖЛИВІСТЬ РОЗДІЛЕННЯ ТА КОНЦЕПТУАЛЬНЕ ПРОЕКТУВАННЯ

Анотація. Показано, що при безперервному виробництві етилацетату виникають проблеми розділення через значну кількість азеотропів. Застосовано вдосконалений трифазний метод дистиляції за допомогою різних сценаріїв очищення для потрійного розділення етилацетатної системи (етилацетат/вода/етанол). Встановлено, що вказана неідеальна суміш містить чотири азеотропи та три області дистиляції. Для вибору найкращої області дистиляції досліджена можливість розділення та розроблений концептуальний проект установки за допомогою методу розширеної граничної величини для різних варіантів подачі сировини та вилучення продукту. Визначено, що область, в якій етанол є стабільним компонентом, є найбільш придатною для процесу дистиляції. Концептуальна конструкція трифазної колони оптимізована внаслідок зміни флегмового числа та робочого тиску. На основі результатів концептуального проектування проведено точне моделювання процесу, і визначено, що чистота відділення етанолу становить 99,25 % мол.

Ключові слова: трифазна дистиляція, етилацетатний процес, доцільність розділення, концептуальне проектування, суворе моделювання.