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by Dhoni Hartanto

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Isopropyl Alcohol Purification through Extractive Distillation using Glycerol as an Entrainer: Technical Performances Simulation and Design

Dhoni Hartanto^{1,✉}, Prima Astuti Handayani¹, Akhmad Sutrisno¹, Viona Widya Anugrahani¹, Asalil Mustain², Ianatul Khoiroh³

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¹Department of Chemical Engineering, Faculty of Engineering, Universitas Negeri Semarang, Kampus Sekaran, Gunungpati, Semarang, 50229, Indonesia

²Department of Chemical Engineering, Politeknik Negeri Malang, Jl. Soekarno Hatta No. 9, Malang 65141, Indonesia

³Department of Chemical and Environmental Engineering, Faculty of Engineering, University of Nottingham Malaysia Campus, Jalan Broga, Semenyih, 43500 Selangor Darul Ehsan, Malaysia

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Abstract

Isopropyl alcohol is widely used as industrial chemical intermediates and common solvents in households, pharmaceuticals, food, cosmetics, and medical purposes. The high purity of isopropyl alcohol requires special separation from its impurity i.e. water due to isopropyl alcohol and water form an azeotropic point, which is difficult to separate using a conventional distillation method. The azeotropic point of this mixture is at isopropyl alcohol mole fraction of 0.68 and temperature of 353.4 K. One of the optimum methods to separate an azeotrope point is through the extractive distillation which use a third component as a solvent. Glycerol is one of the solvents which can be used as a potential entrainer in the extractive distillation. Glycerol is produced in the biodiesel production as a by-product. Moreover, glycerol is an eco-friendly chemical. In this work, the simulation of the extractive distillation of isopropyl alcohol/water system with glycerol as an entrainer was simulated using Aspen Plus. The Non-Random Two-Liquid (NRTL) model was used as thermodynamic model in the simulation. The effect of stage number, binary feed stage, entrainer feed stage, and reflux ratio to the purity of isopropyl alcohol, and reboiler-condenser duties were examined to achieve the optimum design for the extractive distillation column with its energy requirements. The simulation results showed that the optimum configurations in the extractive distillation column design are at 25 theoretical stages, binary feed stage (BFS) of 20, entrainer feed stage (EFS) of 2 and reflux ratio (RR) of 0.5 to produce isopropyl alcohol with the purity of 99.27%. The design and sizing of the extractive distillation column were also proposed in this work.

INTRODUCTION

Currently, isopropyl alcohol is one of most interest chemicals which used worldwide as an industrial chemical intermediates and solvent in the wide area such as households, pharmaceuticals, food, cosmetics, and medical purposes because it has disinfectant properties (US National Library of Medicine, 2020). Isopropyl alcohol mainly used in a high-purity property. As a

consequence of the isopropyl alcohol production, this solvent usually consists of water content in various proportions. Therefore, the purification through separation from water is an important part (Orchillés et al., 2017). The mixture of isopropyl alcohol/water has a minimum boiling point azeotrope at mole fraction of 0.68 and temperature of 353.4 K (Udovenko et al., 1973) which make the separation of isopropyl alcohol from water is difficult.

[✉] Corresponding author:
Department of Chemical Engineering, Faculty of Engineering,
Universitas Negeri Semarang, Sekaran, Gunungpati, Semarang, Indonesia
E-mail: dhoni.hartanto@mail.unnes.ac.id

Several methods can be used to separate the azeotropic point of alcohols with water such as azeotropic distillation (Pienaar et al., 2013), membrane pervaporation (Khosravi et al., 2012), liquid-liquid extraction (Cháfer et al., 2018), reactive extractive distillation (Zhigang et al., 2002), adsorptive-distillation (Megawati et al., 2018), and extractive distillation (Hartanto et al., 2016). Extractive distillation is one of the most effective methods to separate azeotropic point which used in the industry because it can reduce energy consumption and total annual cost (Li et al., 2019). Solvent as a third component is used as an entrainer in the extractive distillation to break azeotropic point for the mixture of isopropyl alcohol/water. Several entrainer was proposed to break the azeotropic point such as salt (Vora et al., 2013), ionic liquids (Pereiro et al., 2012), ethylene glycol (Kalla et al., 2016), and low transition temperature mixtures (Rodríguez & Kroon, 2015). The use of salt as an entrainer may cause corrosion in the plant equipment, while ionic liquids still have questionable issues as a green solvent due to its toxicity properties. Moreover, the price of ionic liquids is still high. Ethylene glycol is one of the most potential entrainer in the isopropyl alcohol/water separation, but it has disadvantages which is toxic for human and environmental. Low transition temperature mixtures also have great potential as entrainer, but the development of this chemical as entrainer is still rare. Thus, it requires other chemicals as potential green entrainer.

Glycerol can be one of the promising entrainer in the azeotropic mixture of isopropyl alcohol/water. Glycerol is a by-product from the biodiesel production with eco-friendly properties, sustainable, and low-cost material (Gu & Jérôme, 2013). The glycerol as a third component successfully breaks the azeotropic point in the ethanol/water mixture. The use of glycerol with the minimum mole fraction of 0.229 in the vapour-liquid equilibrium of isopropyl alcohol/water mixture can remove the azeotropic point from the system (Zhang et al., 2014). Moreover, it has low vapour pressure properties as ionic liquids, which it can be a promising entrainer in the future (Pla-Franco, Lladosa, Loras, & Montón, 2013).

The analysis of the technical performances of the extractive distillation is important to obtain the optimum conceptual design and configuration of the extractive distillation column through the simulation. There are some previous research

which simulated the extractive distillation for the isopropyl alcohol/water using several entrainer such as ethylene glycol with the optimum entrainer-binary feed ratio is 1, and optimum stage number is 42 (Kalla et al., 2016), and dimethyl sulfoxide with the optimum entrainer-binary feed ratio is 1 with the optimum stage number is 41 (Arifin & Chien, 2008). The simulation of the extractive distillation for separation of isopropyl alcohol from water using glycerol as an entrainer was reported previously with the hybrid extractive distillation using the pervaporation membrane. This study showed the performance of the extractive distillation in the total annual cost and energy aspects. The use of glycerol as an entrainer with the hybrid technology can save the total annual cost and energy up to 25% and 41%, respectively (Novita et al., 2018). But in this study, the detail technical performances of the extractive distillation column were not described. Therefore, this work examined the technical performances of the extractive distillation for the separation of isopropyl alcohol/water using glycerol as an entrainer. Moreover, the design and sizing of the extractive distillation column was also performed in this work.

PROBLEMS DEFINITION AND METHODOLOGY

The conceptual process design and simulation of extractive distillation for the separation of isopropyl alcohol/water using glycerol as an entrainer is showed in Figure 1. The glycerol and binary feed (isopropyl alcohol/water mixture) streams are fed into the extractive distillation column (EXT-DIST) or first column in the separated streams. The bottom product from the first column feeds to the glycerol recovery column (REC-COLM) or second column, where glycerol separated from water then recycled to the first column. Make-up entrainer stream is used because part of recycled entrainer is missing in the process. The glycerol is fed to one of the top stages of the first column continuously while the isopropyl alcohol/water mixture is fed to the middle part of the column. High purity of isopropyl alcohol (IPA) obtained as a top product of the column and the glycerol/water mixture removed from the bottom of the column then fed to the second column. Glycerol separated from

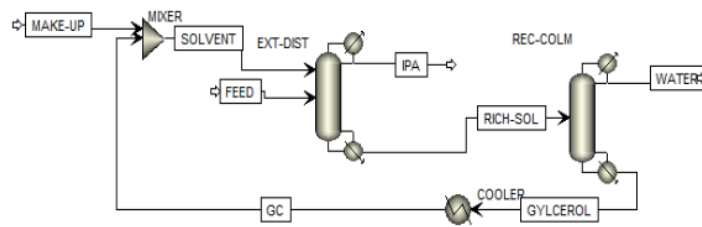


Figure 1. Extractive distillation conceptual process design and simulation for the isopropyl alcohol/water system with glycerol.

Table 1. Optimum binary interaction parameters of the NRTL model used in this study.

Components (i)	Components (j)	A_{ij}	b_{ij}/K	A_{ji}	b_{ji}/K	c_{ij}
Water	Isopropyl alcohol	5.3852	-1005.06	-2.5041	850.87	0.3
Water	Glycerol	0	617.62	0	-499.09	0.3
Isopropyl alcohol	Glycerol	0	259.42	0	402.30	0.3

$$\ln(P^s) = C_1 + \frac{C_2}{(T + C_3)} + C_4T + C_5 \ln T + C_6T^{C_7} \text{ for } C_8 < T < C_9 \quad (1)$$

where P^s is in kPa and T in K.

Table 2. Antoine parameters used in this study.

Parameters	Components		
	Isopropyl alcohol	Water	Glycerol
C_1	99.20707454	62.13607454	88.47307454
C_2	-9040	-7258.2	-13808
C_3	0	0	0
C_4	0	0	0
C_5	-12.676	-7.3037	-10.088
C_6	5.538×10^{-6}	4.1653×10^{-6}	3.5712×10^{-19}
C_7	2	2	6
C_8	-87.89	0.01	18.18
C_9	235.15	373.95	576.85

water in the second column to obtain high-purity glycerol, which is recycled to the first column.

The simulation was conducted using the Aspen Plus software package using rigorous methods. The NRTL thermodynamics model was used in this study to represent the nonideality of the liquid phase of the above-mentioned system while the vapour phase is assumed to be in an ideal condition. The optimum binary interaction parameters were taken from (Zhang et al., 2014). The detailed parameters provided in Table 1.

Total and partial pressures were calculated using the Antoine equation as shown in the Eq. (1). The Antoine parameters were taken from the Aspen Plus physical property databank, which is shown in Table 2.

The initial configuration of the simulation is shown in Table 3. The feed mole fraction of

isopropyl alcohol was adjusted to 0.6 because the mixture of isopropyl alcohol/water has a minimum boiling point azeotrope at a mole fraction of 0.68.

Table 3. Initial configuration of extractive distillation for the isopropyl alcohol/water system with glycerol.

Parameters	Value
Feed mole flow (kmol/h)	100
Distillate mole flow (kmol/h)	60
Feed temperature (°C)	25
Feed mole fraction of isopropyl alcohol	0.6
Feed mole fraction of water	0.4
Pressure (kPa)	100
Number of stage	30
Binary feed stage	18
Entrainment feed stage	3
Entrainment mole fraction	0.2

Table 4. Simulation results of extractive distillation for the ethanol/water system with ethylene glycol-glycerol mixture as entrainer in this work.

Stream	1	2	3	4	5	6	7	8	9	10
Flowrate (kmol/h)	100	100	86.8	93.2	13.3	79.9	79.9	79.9	0.1	80
Ethanol mole fraction	0.88	0.88	0.999	0.013	0.095	5×10^{-10}	5×10^{-10}	5×10^{-10}	0	0
Water mole fraction	0.12	0.12	0.0007	0.128	0.895	0.0003	0.0003	0.0004	0	0
Ethylene glycol mole fraction	0	0	5×10^{-5}	0.514	0.009	0.599	0.599	0.599	0.6	0.6
Glycerol	0	0	7×10^{-12}	0.343	3×10^{-9}	0.4	0.4	0.4	0.4	0.4
Temperature (°C)	20	78.15	78.3	156.2	49.8	163.9	164.05	60	60	60

Table 5. Simulation results of extractive distillation for the ethanol/water system with ethylene glycol-glycerol mixture as entrainer from literature (Gil et al., 2014).

Stream	1	2	3	4	5	6	7	8	9	10
Flowrate (kmol/h)	100	100	86.8	93.2	13.3	79.9	79.9	79.9	0.1	80
Ethanol mole fraction	0.88	0.88	0.999	0.013	0.091	0	0	0	0	0
Water mole fraction	0.12	0.12	0.0002	0.128	0.889	0.0004	0.0004	0.0004	0	0
Ethylene glycol mole fraction	0	0	0.0001	0.514	0.009	0.599	0.599	0.599	1	0.6
Glycerol	0	0	0	0.343	0	0.4	0.4	0.4	0	0.4
Temperature (°C)	20	78.1	78.3	156.7	49.8	163.9	164.05	60	60	60

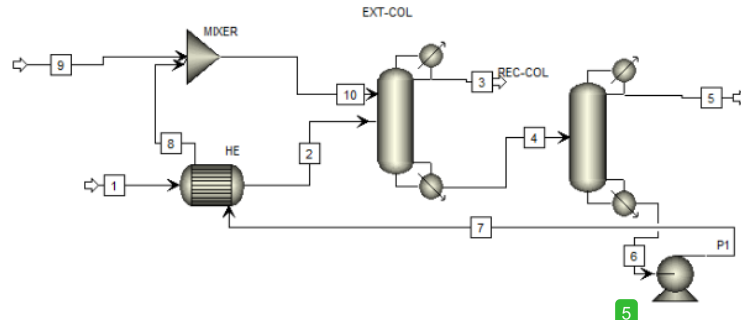


Figure 2. Extractive distillation conceptual process design and simulation for the ethanol/water system with mixed ethylene glycol-glycerol.

The several parameters were analyzed in this work, which affected the purity of the isopropyl alcohol as a desired product such as the number of stages, binary feed stages, entrainer feed stages, and reflux ratio. Sensitivity analysis was conducted to understand the behaviour of two factors to the purity of isopropyl alcohol and reboiler and condenser duties. The operating condition considered as effects of the above-mentioned factors were the purity of isopropyl alcohol and energy consumption.

RESULTS AND DISCUSSION

Preliminary Simulation

The preliminary simulation was conducted to validate the simulation step and method for extractive distillation. The azeotropic mixture of ethanol/water with mixed ethylene

glycol-glycerol as entrainer was used as a system. The process engineering flow diagram of the simulation provided in Figure 2. The preliminary simulation results were compared with the results from Gil et al. (2014). The preliminary simulation and literature results provided in Tables 4 and 5, respectively. The data shows that the preliminary simulation results from this work are similar with the results from the literature. Thus, it can be concluded that the simulation step and method are correct.

Simulation Of The Extractive Distillation Column

The Effect of Stage Number and Reflux Ratio to the Purity of Isopropyl Alcohol

The purity of isopropyl alcohol can be affected by the stage number and reflux ratio, which is shown in Figure 3. The reflux ratio of 0.5

- 0.7 can be used to obtain the purity of isopropyl alcohol more than 99%. The optimum reflux ratio was 0.5, which can produce the highest purity of isopropyl alcohol up to 99.2%. The simulation performed that an increase of reflux ratio causes an increase of the purity of isopropyl alcohol, especially at stage number 19 to 25. After stage 25, the purity remains constant for all reflux ratio except reflux ratio of 0.4 because it produced low purity results. The higher reflux ratio will produce more contact between vapour and liquid in the distillation column. Thus, the higher purity of isopropyl alcohol will be obtained from this condition.

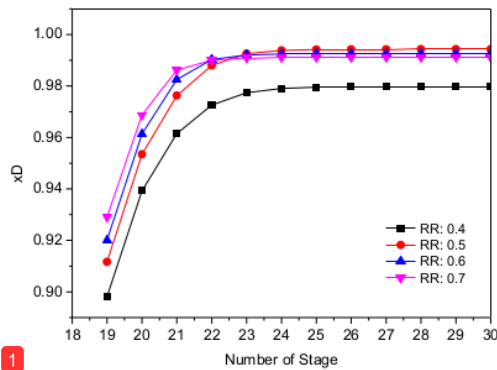


Figure 3. The Effect of Stage Number and the Reflux Ratio (RR) to the Purity of Isopropyl Alcohol (x_D).

The Effect of the Stage Number and the Reflux Ratio to the Reboiler and Condenser Duties

The effect of the stage number and the reflux ratio to the reboiler and condenser duties are shown in Figures 4 and 5. The stage number shows a slightly effect on the reboiler and condenser duty. On the other hand, reflux ratio gives a significant effect on the reboiler and condenser duty because a higher reflux ratio creates more amount of liquid and vapour composition followed by the increase of the reboiler and condenser duty. The increase of the reflux ratio means the load of the distillation column will increase, which caused the increase of the reboiler and the condenser duties. Thus, reflux ratio had an important direct relationship with the heating and cooling mechanism. According to Figures 3, 4, and 5, the best possible reflux ratio is 0.5 and 0.6 because the energy consumption is low, but the purity of the isopropyl alcohol can reach up to 99%.

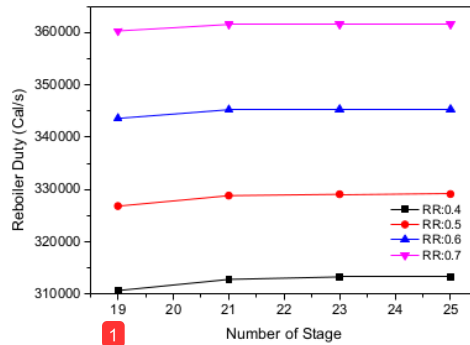


Figure 4. The Effect of Stage Number and Reflux Ratio to the Reboiler Duty.

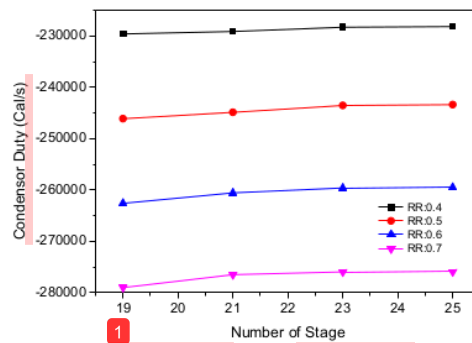


Figure 5. The Effect of Stage Number and Reflux Ratio to the Condenser Duty.

The Effect of the Binary Feed Stage and the Reflux Ratio to the Purity of Isopropyl Alcohol

The results analysis of the binary feed stage and the reflux ratio to the purity of isopropyl alcohol is provided in Figure 6. It shows that the highest purity of the distillate isopropyl alcohol compositions was obtained at the reflux ratio of 0.5, 0.6, and 0.7, with the purity of 99.1%, 99.3%, and 99.2%, respectively. The highest distillate concentration was performed at a binary feed stage of 20 due to the contact time between isopropyl alcohol/water mixture with glycerol is longer. Therefore, the purity of the isopropyl alcohol was increased.

The Effect of the Entrainer Feed Stage and the Reflux Ratio to the Purity of Isopropyl Alcohol

The entrainer feed stage and the reflux ratio on distillate isopropyl alcohol composition is shown in Figure 7. It is provided that the highest purity of isopropyl alcohol was achieved at the reflux ratio of 0.5, 0.6, and 0.7, with the purity of 99.2%, 99.2%, and 99.1%. The highest purity

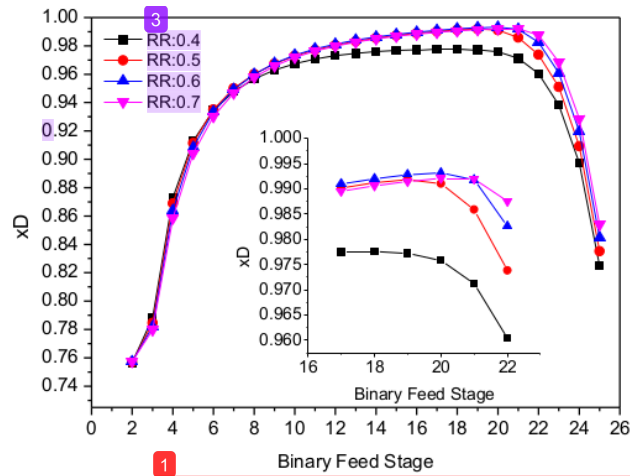


Figure 6. The effect of binary feed stage and reflux ratio to the purity of isopropyl alcohol.

obtained at the entrainer feed stage of 2. It is confirmed that the interaction between glycerol as entrainer and isopropyl alcohol/water as an azeotropic mixture mostly takes place when an entrainer in the liquid phase. The placed of an entrainer in the top stage of the extractive distillation column will ensure the entrainer in a liquid phase for all stages below the entrainer feed stage. From Figures 4-6, the optimum condition of the extractive distillation was the entrainer feed stage and the reflux ratio of 2 and 0.5, respectively, because the purity of the isopropyl alcohol was 99.2% and the energy consumption is low.

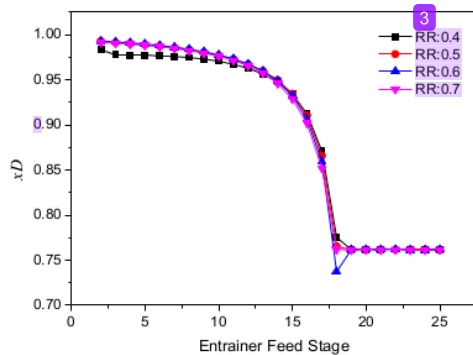


Figure 7. The effect of entrainer feed stage and reflux ratio to the purity of isopropyl alcohol.

The Effect of the Binary Feed Stage and the Reflux Ratio to the Reboiler and Condenser Duty

The energy consumption of the reboiler is higher than the condenser, as shown in Figure 8.

Binary feed stage did not affect the reboiler and condenser duties because the increasing of the binary feed stage produces constant reboiler and condenser duty. However, the reflux ratio plays an important role in the energy consumption in the extractive distillation column. The heating and cooling mechanism had a proportional relationship with the reflux ratio. The higher of reflux ratio, the higher of reboiler and condenser duties required. It also can be examined that the reboiler duty (Q_{reb}) is higher than condenser duty (Q_{con}) because the distillation with the close boiling or azeotropic mixture required higher energy in the reboiler.

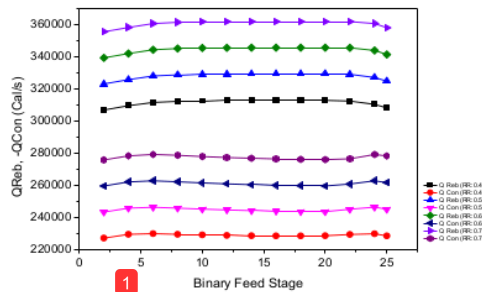


Figure 8. The effect of the binary feed stage and the reflux ratio to the reboiler and condenser duties.

The Effect of the Entrainer Feed Stage and the Reflux Ratio to the Reboiler and Condenser Duties

The performance of the reboiler and condenser duties were significantly influenced by the entrainer feed stage and the reflux ratio. Lower

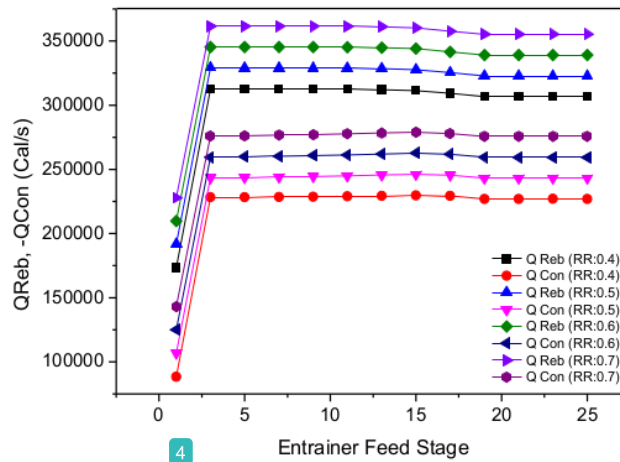


Figure 9. The effect of the entrainer feed stage and the reflux ratio to the reboiler and condenser duties.

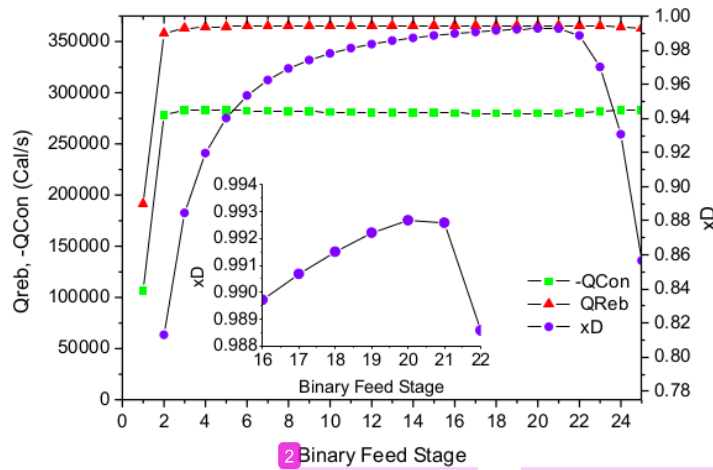


Figure 10. The effect of the binary feed stage on the distillate composition and energy duty at operating conditions: Stage numbers: 25, RR: 0.5, BFS: 20, EFS: 2.

energy consumption occurred when the entrainer feed stage was placed at the top of the extractive distillation column in the range of 1-3, as shown in Figure 9. The energy consumption was increased as an increase of the entrainer feed stage. However, the energy consumption was not change significantly at the stage of more than 3. It can be confirmed that the entrainer at the top of the column creates more interaction with the vapour phase (isopropyl alcohol), which caused the more liquid phase (water) flowed to the reboiler. Hence, the reboiler duty is lower, followed by low condenser duty. In case the entrainer is fed at the bottom of the extractive distillation column, the more water in the vapour phase will be produced

which caused less water in the liquid phase flow to the reboiler. Thus, reboiler and condenser duties will be higher. In addition, it can be observed that reboiler duty is greater than condenser duty at the different entrainer feed stage and the higher reflux ratio, the higher reboiler and condenser duties needed.

The Effect of the Binary Feed Stage on the Distillate Composition and Energy Duty

The analysis of the effect of the binary feed stage to the distillate composition and energy duty can be seen in Figure 10. The reflux ratio was fixed at 0.5. The binary feed stages did not give a significant change to reboiler and condenser

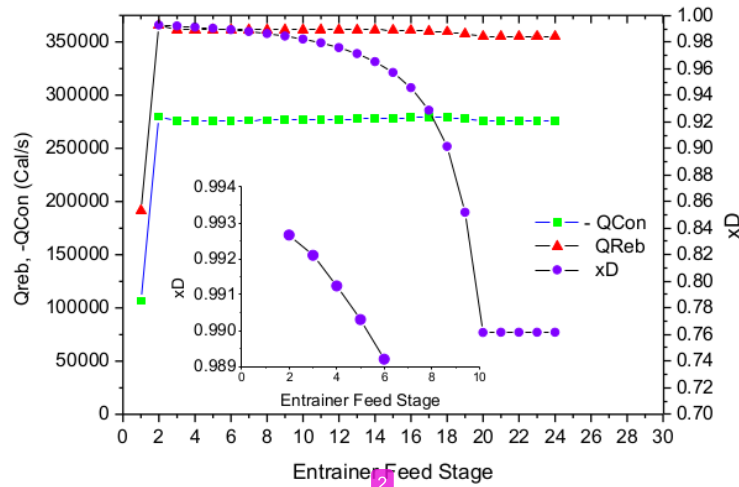


Figure 11. The effect of the entrainer feed stage on the distillate composition and energy duty at operating conditions: Stage numbers: 25, RR: 0.5, BFS: 20, EFS: 2.

Table 6. Optimum configuration of extractive distillation column

Parameter	Value
Feed mole flow (kmol/h)	100
Distillate mole flow (kmol/h)	60
Feed temperature (°C)	25
Feed mole fraction of isopropyl alcohol	0.6
Feed mole fraction of water	0.4
Pressure (kPa)	100
Number of stage	25
Binary feed stage	20
Entrainer feed stage	2
Reflux ratio (RR)	0.5
Entrainer mole fraction	0.2
Binary feed temperature (°C)	25
Entrainer feed temperature (°C)	25

Table 7. Optimum configuration of recovery column

Parameter	Value
Number of stage	30
Bottom product mole flow (kmol/h)	25
Feed stage	14
Reflux ratio	2

duties. The highest distillate composition was obtained at a stage number of 20.

The Effect of the Entrainer Feed Stage on the Distillate Composition and Energy Duty

The effect of the entrainer feed stage on the distillate composition and energy duty at certain operating conditions was provided in Figure 11. The highest isopropyl alcohol was obtained up to 99.2% at the operating condition of reflux ratio, binary feed stage, and entrainer feed stage of 0.5, 20, and 2, respectively.

Optimum Configuration

According to sensitivity analysis, the optimum configuration of the extractive distillation column, as well as the recovery column in the separation of azeotropic mixture of isopropyl alcohol/water, are provided in Tables 6 and 7. The minimum energy requirements for reboiler and condenser duties of the extractive distillation column were shown in Table 8.

The purity of the isopropyl alcohol obtained in this study was 99.27%, while the purity in the standard specification of ASTM is at grade

Table 8. Reboiler and condenser duty in the extractive distillation column

Parameter	Value
Reboiler (Cal/s)	365,701.2
Condenser (Cal/s)	-279,816.8

Table 9. Simulation results for the process engineering flow diagram presented in Figure 1.

Stream	T (°C)	Mole flow (kmol/h)			Mole fraction		
		Isopropyl alcohol	Water	Glycerol	Isopropyl alcohol	Water	Glycerol
MAKE-UP	25	0.000	0.000	1.3068	0.000	0.000	1
SOLVENT	25	0.000	0.000	26.3068	0.000	0.000	1
FEED	25	60	40	0.000	0.6	0.4	0.000
IPA	80.73	59.6278	0.3722	0.000	0.9927	0.0062	0.000
RICH-SOL	113.82	0.3722	39.6278	26.3068	0.0073	0.5976	0.3951
WATER	98.06	0.3722	39.6278	1.3068	0.009	0.9594	0.0326
GLYCEROL	283.16	0.000	0.000	25	0.000	0.000	1
GC	25	0.000	0.000	25	0.000	0.000	1

Table 10. Optimum design and sizing of the extractive distillation column

Parameter	Design and Sizing
Column type	Sieve Tray Tower
Binary feed stage	20
Entrainer feed stage	2
Tray Spacing	0.3 m
Column height	7.74 m
Column diameter	1.03 m
Downcomer width	0.16 m
Shell thickness	0.1875 in
Head thickness	0.1875 in
Material	Carbon Steel SA- 283 Grade C

99% (ASTM, 2019). Thus, the minimum purity has fulfilled the requirements.

Design of Extractive Distillation Column

The design and sizing for the optimum extractive distillation column was proposed to obtain the optimum extractive distillation. The proposed design and sizing are provided in Table 10.

CONCLUSION

The extractive distillation for the azeotropic mixture of isopropyl alcohol/water with glycerol as entrainer was simulated using Aspen Plus. The NRTL thermodynamic model was used as a thermodynamic package in the calculation. The preliminary simulation was performed then compared to the open literature to validate the step and method, which yielding satisfactory results. The sensitivity analysis, such as the effect of stage number, binary feed stage, entrainer feed stage, reflux ratio to the isopropyl alcohol purity, and reboiler-condenser duties was

studied to achieve the optimum design and configuration of the extractive distillation and recovery column. Optimum design and sizing were also proposed in this study to obtain the best extractive distillation. Glycerol can be one of the promising green-solvent which can be used as an entrainer in extractive distillation for the azeotropic mixture of isopropyl alcohol/water.

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