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Comparative Study of Fluid Package on Extractive Distillation of Ethanol-water Mixture Using Pure Glycerol and Ethylene Glycol as Extracting Solvents

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Authors' contributions

This work was carried out in collaboration between all authors. Author OJO designed the study, supervised the simulation and wrote the manuscript from the first draft to the final draft accepted for publication. Authors ORO and FJA carried out the simulation to generate the data used in the study. All the authors read and approved the final manuscript.

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ABSTRACT

The comparative study of Peng Robinson Strygek Vera (PRSV) and Non-Random Two Liquid (NRTL) fluid packages on the extractive distillation of ethanol-water mixture was done using pure glycerol and ethylene glycol as extracting agents. The process was simulated using HYSYS V. 8.4. Pure glycerol and ethylene glycol-glycerol mixture were used as separating agents. The results showed the highest molar composition of ethanol in the distillate at the solvent feed rate of 80 kmol/hr when pure glycerol was used with NRTL fluid package. With the PRSV fluid package the highest ethanol in the distillate was obtained at pure glycerol feed rate of 50 kmol/hr. The ethanol fraction in the distillate was decreasing with increasing ethylene glycol and decreasing glycerol in the ethylene glycol-glycerol mixture. The comparative study of the base case and alternative designs showed high ethanol composition in distillate in the range of 99.46 - 99.96% and 99.96 - 99.67% for NRTL and PRSV fluid package, respectively. It was concluded from the study that the PRSV fluid package could save cost of operation because of lower solvent requirement and energy consumption in the reboiler for both the base case and alternative designs.

Keywords: Extractive distillation; fluid package; glycerol; ethylene glycol; solvent feed rate.

1. INTRODUCTION

Ethanol is a widely used raw material in industrial processes. For example it is used in the synthesis of esters and ethers, as solvent in the production of paint, cosmetics, sprays. perfumery, medicine, food and as fuels. In order to produce absolute ethanol, several methods of separating the azeotropic ethanol-water mixture have been proposed and used over time. Extractive distillation is one of them, others are: chemical dehydration, dehydration by vacuum distillation, azeotropic distillation, membrane processes, adsorption processes and diffusion distillation [1]. Extractive distillation is a method of separating two components of very similar boiling point from a mixture. The principle driving extractive distillation is based on the introduction of a selective solvent that interacts differently with each of the components of the original mixture and generally shows a strong affinity for one of the key components [2,3].

Quite a number of researchers have worked on the simulation of the extractive distillation process. García-Herreros et al. [4] carried out extractive distillation study for the production of fuel grade ethanol using glycerol as entrainer due to many factors such as availability, economical, and advantages over other solvents and distillation techniques. Gil et al. [5] carried out the simulation of ethanol extractive distillation using mixed glycols as separating agent. In their research, Aspen Plus was used with Non-Random Two Liquid (NRTL) model as the fluid package. The most adequate composition of the glycols mixture and the most efficient operating conditions for the columns were established. In the work by Igbal and Ahmad [6], a simulation study of pressure swing distillation of azeotrope mixture was done using Aspen plus. The author asserted that among the available activity coefficient models, the WILSON RK model with binary parameters predicted by the Aspen Plus simulator was shown to be the most accurate to correlate the experimental vapour/liquid equilibrium (VLE) data available for the ethanolwater system.

Equation of state is a thermodynamic equation describing the state of matter under a given set of physical conditions. It is a constitutive equation which provides a mathematical relationship between two or more state functions associated with the matter, such as the temperature, pressure, volume, or internal energy. Equations of state play an important role in chemical engineering design, and they have assumed an expanding role in the study of the phase equilibria of fluids and fluid mixtures. They are useful in describing the properties of fluids, mixtures of fluids and solids [7]. Originally, equations of state were used mainly for pure components. When first applied to mixtures, they were used only for mixtures that were nonpolar [8,9] and slightly polar compounds [10,11,12]. Subsequently, equations of state have developed rapidly for the calculation of phase equilibria in nonpolar and polar mixtures.

The van der Waals equation of state was the first equation to predict vapor-liquid coexistence. Later, the Redlich-Kwong equation of state [13] improved the accuracy of the van der Waals equation by introducing temperature-dependence for the attractive term. Soave [8], Peng and Robinson [9] proposed additional modifications to predict the vapor pressure, liquid density, and equilibria ratios of vapour-liquid system more accurately. The modification of the Peng-Robinson equation of state proposed by Stryjek and Vera [14] called Peng Robinson Stryjek Vera (PRSV), was said to reproduce well the vapor pressure data of pure compounds [15,16]. Also, it was of particular advantage for Vapour Liquid Equilibrium (VLE) calculations [17]. However, since there is dearth of information on the application of PRSV equation of state to the separation of ethanol-water mixture, this research is aimed at investigating the effect of using PRSV and NRTL equations of state on the degree separation and energy consumption in the reboiler and condenser for the extractive distillation of ethanol-water mixture using pure glycerol and ethylene glycol-glycerol mixture as separating agents.

2. MATERIALS AND METHODS

2.1 Materials

The major process equipment for the extractive distillation of the ethanol-water mixture includes (a) dehydration column (C-01) where water in the feed are removed using the selected separating agent. (b) recovery distillation column (C-02) in which the used separating agent is recovered for reuse. (c) shell and tube heat exchanger (H-01) for preheating the feed using the heat recovered from the process before it is fed to the extractive

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distillation column. Other equipment includes the pump (P-01) for pumping the recycled separating agent for reuse, the mixer used for mixing the recycled separating agent and the makeup separating agent before the mixture is used in the extractive distillation column, the splitter used to divide the separating agent mixture into two streams which are fed to the extractive distillation column at different stages and the storage tank used for storing the produced ethanol withdrawn as the overhead distillate product of the extractive distillation column. The process was simulated using HYSYS version 8.4. The solvents used are pure glycerol and mixture of glycerol and ethylene glycol. The schematic diagram of the setup used for the simulation is presented in Fig. 1. The initial columns input data are presented in Table 1.

2.2 Determination of Relative Volatility of Feed at Various Feed Temperature

The Antoine equation used for the determination of vapour liquid ratio for an ideal solution from the estimated P^{sat} for the ethanol and water is given by Equation (1).

$$\ln P^{sat}(kPa) = A - \frac{B}{C + T(^{\circ}C)}$$
(1)

Table 1. Initial input data for the process

Initial input data for extraction column				
Process parameter and operating condition	Condition			
Feed temperature	30°C			
Extracting solvent temperature	30°C			
Pressure	1 atm (14.7 psig)			
Number of theoretical stage	20			
Feed flowrate	100 kmol/hr (80%-ethanol; 20%-water)			
Solvent flowrate	10 kmol/hr			
Feed plate number	12			
Reflux ratio	0.45			
Initial input data for recovery column				
Feed plate number	4			
Feed Pressure	1 atm (14.69 psig)			
Column operating pressure	0.2 bar			
Reflux ratio	0.92			
Number of theoretical stage	8			



Fig. 1. Process diagram of extractive distillation of ethanol-water azeotropic mixture

where A, B and C are Antoine constants for vapour pressure estimation at a given temperature range [17]; T is the temperature of the mixture (°C). For ethanol: A, B and C is 16.8958, 3795.17 and 230.918, respectively. For water: A, B and C is 16.3872, 3885.7 and 230.17, respectively. The solvent selectivity (relative volatility) is evaluated from the ratio of activity coefficient (K) of the more volatile component (i) to the less volatile component (j) of the binary mixtures using Equations (2)-(3b) [18].

$$\alpha_{i,j} = \frac{K_i}{K_j} \tag{2}$$

where:

$$K_i = \frac{P_i^{sat}}{P}$$
(3a)

$$K_j = \frac{P_j^{sat}}{P}$$
(3b)

2.2 Process Costing

The process costing was done using the economics solver in Aspen HYSYS v. 8.4 software. The following constraints were employed for the heat transfer equipment:

- i. Minimum Logarithm Mean Temperature Difference (LMTD_{min}) correction factor = 0.8
- ii. Maximum area per shell $(m^2) = 500$
- iii. Exchanger Minimum Approach Temperature (EMAT) (°C) = 10

Following the equipment sizing based on the process parameters using the software, the equipment cost from Aspen HYSYS was used as the purchased cost of equipment (PCE) for the cost estimation. Physical plant cost (PPC) and fixed capital cost (FCC) were estimated using Equations (4) and (5), respectively [19]:

$$PPC = PCE \times \left(1 + \sum f_i\right) \tag{4}$$

$$FCC = PPC \times \left(1 + \sum f_i\right) \tag{5}$$

The detailed factor, f used in the factorial method for capital cost estimation was provided in literature [19]. The capital cost annualization factor, A^{F} was calculated as:

$$A^{F} = \left[\frac{m\left(1+m\right)^{N}}{\left(1+m\right)^{N}-1}\right]$$
(6)

Therefore, the annualized fixed capital cost, ${\cal A}^{\rm FCC}$, was calculated as:

$$A^{FCC} = FCC \times A^F \tag{7}$$

The operating life of the plant, N of 5 years and the rate of return on investment, m of 10% were adopted. The plant operation hours per year was taken to be 8766 hours.

3. RESULTS AND ANALYSIS

The Simulation of extractive distillation of ethanol-water mixture was done for PRSV and NRTL fluid packages. The results obtained when pure glycerol and mixed glycol were used as separating agent are presented in Figs 2 (a) and (b), respectively.





Fig. 2. Simulation of extractive distillation of ethanol using (a) pure glycerol (b) mixed glycols as solvents for NRTL and PRSV fluid packages.

3.1 Effects of Solvent Feed Rate on Ethanol Recovery When Pure Glycerol is Used as Solvent for NRTL Fluid Package

The results of the effects of solvent on ethanol in the distillate, reboiler energy as well as condenser energy consumption while varying pure glycerol feed rate from 10 to 100 kmol/hr using NRTL fluid package are presented in Fig. 3. The ethanol-water feed rate was kept constant at 100 kmol/hr. It could be observed from the figure that sufficiently high ethanol recovery in the distillate was attained when the solvent feed was 80 kmol/hr. However, increasing the quantity of solvent above this value did not make any significant improvement in the composition of ethanol recovery in the distillate. It could therefore be inferred from this result that the optimum solvent flow rate for the cost effective operation of this process in terms of solvent consumption is 80 kmol/hr. As presented in Figure 4, the increase in the ethanol recovery is however at the expense of increase in energy consumed in the reboiler while energy used in the condenser is not affected. The reboiler duty increases with the net weight of the solvent. Heat being an extensive property, an increase in the solvent feed rate requires more heat from the reboiler to strip off lighter component.



Fig. 3. Effect of solvent feed rate on composition of distillate for NRTL fluid package



Fig. 4. Effect of solvent feed rate on energy of the reboiler and condenser for NRTL fluid package

3.2 Effects of Solvent Feed Rate on Ethanol Recovery When Pure Glycerol is Used as Solvent with PRSV as Fluid Package

The result of ethanol recovery and energy consumption in the reboiler and condenser for increasing solvent feed rate when PRSV fluid package was used is presented in Fig. 5. Similar to the results obtained for NRTL fluid package, there is an increase in the recovered ethanol in the distillate with increasing solvent feed rate. The highest recovery of ethanol was observed when the solvent feed rate was 50 kmol/hr. The result showed that when PRSV fluid package was used, the economical solvent feed rate for total recovery of ethanol from the ethanol-water mixture is 50 kmol/hr. As presented in Fig. 6, the reboiler energy increases with increasing solvent in the feed while the condenser energy consumption is unaffected.

3.3 Effects of Mixed Solvents (Ethylene Glycol and Glycerol) on Ethanol Recovery and Energy Consumption at Constant Ethanol-Water Mixture Feed Rate

3.3.1 With NRTL fluid package

The result showing effects of solvent mixing ratio when 80 kmol/hr of glycerol is used as basis for

solvent mixing at constant ethanol-water mixture feed rate of 100 kmol/h is presented in Table 2. It could be observed from the table that the ethanol fraction in the distillate was decreasing with increasing ethylene glycol and decreasing glycerol in the ethylene glycol-glycerol mixture. The observed trend could be due to higher boiling point of glycerol (290°C) compare to ethylene glycol (197.3°C). The effectiveness of solvent mixture as separating agent could decrease with reduction in the composition of high boiling point solvent in the mixture. It could also be observed from the table that the energy of the reboiler is decreasing while the energy of the condenser is approximately constant as the composition of ethylene glycol in the mixture is increasing. This could be due to lower heat capacity of ethylene glycol which is the dominant solvent in the ethylene glycol-glycerol mixture.

3.3.2 With PRSV fluid package

The results showing effects of solvent mixing ratio when 50 kmol/h of pure glycerol is used as basis for solvent mixing while the feed rate of ethanol-water mixture is kept constant at 100 kmol/h are presented in Table 3. As observed from the table, the trend of the results when PRSV fluid package is used is the same as for the NRTL fluid package since the same solvent mixture is used in both cases.



Fig. 5. Effect of solvent feed rate on composition of distillate for PRSV fluid package



Fig. 6. Effect of solvent feed rate on the reboiler and condenser energy for PRSV fluid package

3.4 Effects of Feed Temperature on Ethanol Composition in the Distillate, Reboiler and Condenser Energy When Pure Glycerol is Used as Solvent

3.4.1 With NRTL fluid package

The result of the effect of increasing the feed temperature on ethanol fraction in the distillate is presented in Fig. 7. The figure shows that the ethanol fraction in the distillate remains constant between 30°C to 70°C. However, beyond the boiling point of ethanol, the fraction of ethanol in the distillate is decreasing. This is because the boiling point of ethanol has been exceeded and solvent selectivity for ethanol or activity coefficient is also reducing as the temperature of the feed is raised. As shown in Table 4, an increase in temperature results to an increase in

the pressure and decrease in relative volatility. Hence, the fraction of ethanol in the distillate is decreasing with increasing temperature. As presented in Fig. 8, since the feed has been preheated, the energy required from the reboiler to vaporize the feed is decreasing with increasing temperature while the condenser energy remained constant.

3.4.2 With PRSV fluid package

The result of the effect of increasing feed temperature on the ethanol fraction in the

distillate, reboiler and condenser energy when PRSV equation of state is used is presented in Fig. 9. It could be observed from the figure that the fraction of ethanol in the distillate is increasing from 30°C till the boiling point of ethanol is reached. Beyond the boiling point of ethanol, the fraction of ethanol in the distillate begins to decrease possibly due to evaporation of water in the mixture. As shown in Fig. 10, the reboiler energy is decreasing with increasing temperature while feed the condenser temperature is constant.

Table 2. Effects of mixed solvent on separation ability and energy utilization for NRTL fluid
package at 80kmol/h of pure glycerol as basis

Ethylene glycol fraction:	Ethanol composition in	Reboiler energy		Conde	enser energy
Glycerol (Eg:G)	distillate	kW	kcal/hr	kW	kcal/hr
0.0:1.0	0.9993	2198	1.89E+06	1251	1.08E+06
0.1:0.9	0.9981	2148	1.85E+06	1251	1.08E+06
0.2:0.8	0.9957	2101	1.81E+06	1252	1.08E+06
0.3:0.7	0.9912	2055	1.77E+06	1253	1.08E+06
0.4:0.6	0.9839	2011	1.73E+06	1254	1.08E+06
0.5:0.5	0.9741	1969	1.70E+06	1255	1.08E+06
0.6:0.4	0.9626	1929	1.66E+06	1256	1.08E+06
0.7:0.3	0.9483	1889	1.66E+06	1256	1.08E+06
0.8:0.2	0.9345	1856	1.62E+06	1259	1.08E+06
0.9:0.1	0.9205	1827	1.59E+06	1261	1.09E+06
1.0:0.0	0.9097	1801	1.57E+06	1262	1.09E+06



Fig. 7. Effect of feed temperature on the ethanol fraction in distillate when pure glycerol used with NRTL fluid package

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Ethylene glycol (Eg)	Ethanol	Reboiler energy		Condenser energy	
fraction: Glycerol (G) (Eg:G)	composition in distillate	kW	kcal/hr	kW	kcal/hr
0.0: 1.0	0.9999	1922	1.65E+06	1317	1.13E+06
0.1:0.9	0.9998	1895	1.63E+06	1317	1.13E+06
0.2:0.8	0.9995	1871	1.61E+06	1317	1.13E+06
0.3:0.7	0.9987	1847	1.59E+06	1317	1.13E+06
0.4:0.6	0.9867	1821	1.57E+06	1317	1.13E+06
0.5:0.5	0.9923	1795	1.54E+06	1317	1.13E+06
0.6:0.4	0.9849	1768	1.52E+06	1317	1.13E+06
0.7:0.3	0.9737	1746	1.50E+06	1317	1.13E+06
0.8:0.2	0.9607	1730	1.49E+06	1317	1.13E+06
0.9:0.1	0.9474	1714	1.48E+06	1317	1.13E+06
1.0:0.0	0.9347	1696	1.46E+06	1317	1.13 E+06

Table 3. Effects of mixed solvent on separation	ability and Energy for PRSV fluid package at 50
kmol/hr of pure	glycerol as basis

Table 4. C	Calculated relative	volatility using	Antoine Equation	and Raoult's law
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Ethanol			Water				
T (°C)	InP ^{sat}	P ^{sat} (kPa)	K _i =P ^{sat} /P	InP ^{sat}	P ^{sat} (kPa)	K _J =P ^{sat} /P	α
20	1.770660	5.874727	0.057979	0.854962	2.351285	0.023205	2.498518
30	2.350349	10.48923	0.103521	1.451965	4.271501	0.042156	2.455631
40	2.887244	17.94379	0.177091	2.004774	7.424417	0.073273	2.416861
50	3.385915	29.54500	0.291586	2.518121	12.40526	0.12243	2.381651
60	3.850303	47.00729	0.463926	2.996084	20.00704	0.197454	2.349537
70	4.283826	72.51736	0.715691	3.442202	31.25571	0.30847	2.320131
80	4.689463	108.7947	1.073720	3.859554	47.44418	0.468238	2.293110
90	5.069820	159.1456	1.570645	4.250835	70.16397	0.692465	2.268196



Fig. 8. Effect of feed temperature on reboiler and condenser energy when pure glycerol is used with NRTL fluid package



Fig. 9. Effect of feed temperature on ethanol fraction in distillate when pure glycerol is used as solvent with PRSV fluid package



Fig. 10. Effect of feed temperature on energy of the reboiler and condenser when pure glycerol is used as solvent with PRSV fluid package

4. ALTERNATIVE DESIGN WHEN REGENERATED SOLVENT IS REUSED WITH FRESH SOLVENT

The spent solvent in the process is regenerated and heat recovered from the process is recycled and reused within the process. The alternative design evolved in which both the NRTL and PRSV are used as fluid packages for simulation is presented in Fig. 11. Economic analysis, energy requirements in both the reboiler and condenser and ethanol in the distillate are used as indices for performance evaluation of the designs. The results of comparative analyses of the base case and alternative designs are presented in Table 5. It was observed from the results that both the base case and the alternative designs achieved sufficiently high composition of ethanol in range of 99.46 – 99.96% in the distillate. The ethanol in the distillate for the base case and alternative designs when PRSV is used as fluid package achieved the highest composition of 99.96 and 99.67%, respectively. For the NRTL fluid package, the ethanol in the distillate for the base case and alternative designs are 99.54 and



Fig. 11. Alternative design when regenerated solvent and recovered heat is reused within the process

Parameters	Base case designs		Alternativ	ve designs
Extractive column	PRSV	NRTL	PRSV	NRTL
Equipment cost (USD)	156,900	177,200	142,600	149,400
Installed cost (USD)	509,600	531,800	466,700	494,200
Operating cost (USD/hr)	62	72	57	68
Reboiler energy (kW)	1869	2183	1691	2058
Ethanol in the distillate (%)	99.96	99.54	99.67	99.46
Recovery column	Base case d	lesigns	Alternative designs	
Equipment cost (USD)	85,500	87,300	71,100	73,300
Installed cost (USD)	371,600	375,300	331,900	339,200
Operating cost (USD/hr)	31	32	23	30
Reboiler energy (kW)	586.8	601	566.4	556.3

Table 5. Com	parative ana	yses of the base	e case designs	and alternative	design
		3			

99.46%, respectively. Observations from Table 5 show that:

- the equipment costs and their respective installation costs are lower when PRSV fluid package is used than for NRTL fluid package for the base case design and alternative design;
- i. the weight of equipment and their respective installed weight are lower when PRSV fluid package is used than for NRTL fluid package for the base case design than the for the alternative design;
- ii. the operating cost are lower when PRSV fluid package is used than for NRTL fluid package for the base case design. The same trend was observed in the alternative case design

The higher cost of equipment for NRTL fluid package over the PRSV for both the base case and alternative case could be due to higher weight of equipment required as a result of higher reboiler duty. As observed earlier, the NRTL fluid package required more solvent than the PRSV fluid package for effective separation of ethanol-water mixture, and this implies more reboiler energy requirements. The same trend was observed in the recovery column for both the base case and the alternative designs for the cost analysis and reboiler energy demand.

5. CONCLUSION

The study compared effect of fluid packages, separating agents and utilization of recovered heat on the extractive distillation of a mixture of ethanol-water system. The two fluid packages, considered, PRSV and NRTL, accurately modeled the process. However, it was concluded from the study that the PRSV performed better as it could save the cost of operation because of its lower solvent requirement and energy consumption in the reboiler for both the base case and alternative designs. Also, the mixing of additive solvent (ethylene glycol) to the basic

solvent (glycerol) reduced the reboiler energy requirement during the separation process.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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