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Combining the preconcentration column and recovery column for the extractive distillation of ethanol dehydration with low transition temperature mixtures as entrainers

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ABSTRACT

Extractive distillation using low transition temperature mixtures (LTTMs) is a promising technology to separate ethanol and water azeotrope. In this work, the separation of water and ethanol by extractive distillation using LTTMs (choline chloride/urea 1:2) as entrainer was designed and simulated in Aspen Plus. Firstly, database of ChCl/urea = 1:2 (Reline) was established in Aspen Plus. Secondly, the simulation and optimization of the conventional extractive distillation process for ethanol dehydration with Reline or ethylene glycol as entrainer were worked out. The results showed that the TAC (total annual cost) of the process with Reline as entrainer was reduce by 14.61% than that of the process with EG as entrainer. In addition, in order to reduce energy consumption in distillation, an energy-efficient extractive distillation process which combining the preconcentration section and entrainer recovery section in one column was developed. The results showed that the proposed process with Reline as entrainer could save 29.48% and 17.42% of TAC compared with the conventional extractive distillation with EG and Reline as entrainer, respectively.

1. Introduction

Ethanol is widely used as fuel, solvent, raw material in the chemical industry. Separation of the aqueous mixture of ethanol is often demanded in its production and recovery. Ethanol forms an azeotrope with water at 95.5 wt%, therefore, the ethanol dehydration by ordinary distillation is impossible. Several special separation techniques are needed, such as extraction, extractive distillation, azeotropic distillation, pervaporation, adsorption and so on [1,2]. Extractive distillation, in which a new heavy chemical compound (entrainer) is added at the top of the column, has been widely used for this separation [3]. The entrainer increases the volatility of ethanol, so that the separation of the ethanol and water components can be achieved in the column. The research areas in extractive distillation are primarily focused on a potent entrainer, process intensified configurations and process optimization strategies [4].

The effectiveness of the extractive distillation process relies on the choice of entrainer [5]. Recent studies on extractive distillation have focused on the use of low transition temperature mixtures (LTTMs) as entrainer [1,2,6–9]. LTTMs which have much lower melting point than the initial compounds are composed of hydrogen bond donors and hydrogen bond acceptors. The mixtures are sometimes referred to as deep eutectic solvents (DESs). LTTMs as a new type of entrainer have attracted much attention due to its low vapour pressure, wide liquid range, water compatibility, biodegradability, non-flammability, easy and cheap preparation [2,10]. And the use of LTTMs as azeotropic breakers in liquid-liquid extraction and extractive distillation has been presented [9,11,12]. However, most of these studies on LTTMs as potential entrainer have been focus on vapor-liquid equilibrium (VLE) experiments and liquid-liquid equilibrium (LLE) experiments. It is necessary to make a technical and economic evaluation via process simulation for the design of the industrial-scale applications [5]. Recently, Ma [9] et al introduced the methodology of an extractive distillation column using LTTMs (glycolic acid-choline 3:1) in ethanol dehydration system. In the simulation, an extractive distillation column and a flash tank for LTTMs entrainer recovery were included. The pressure in the flashing tank was only 0.004 kPa, however it was difficult to reach such a high vacuum in industry [13,14]. Therefore the flashing tank was replaced by an entrainer recovery column to recycle the LTTMs entrainer in this paper.

Although extractive distillation was widely used in industry, its energy consumption was very large. Different methods for reducing energy cost have been reported. Heat integration is a useful option to reduce energy cost compared to the conventional process with no

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integration. Gu et al [15] studied the separation of tetrahydrofuran and water through extractive distillation with and without heat integration, the results showed that the optimal heat integration process could achieve a 26.7% saving in energy cost compared with the conventional extractive distillation process. But the heat integrated extractive distillation might lead to high investment cost [16]. Nowadays extractive dividing wall column (EDWC) has drawn much attention. EDWC is a typical example of process intensification which can result in reductions in both capital investment and energy costs [17–19]. However, it was reported that EDWC system might provide energy saving but with increasing the total steam cost [20]. Also the design and control of the EDWC system were complicated. Recently, it was reported that when separating minimum boiling azeotrope with a heavy entrainer by extractive distillation, a lower pressure in the extractive distillation column could reduce the total annual cost (TAC) [19,21–24]. But this method is not widely used, so each case needs to be considered in detail. Liang and An et al [25,26] explored an innovative energy-saving extractive distillation by combining the preconcentration column and entrainer recovery column in one column, and the new process could provide potential energy savings and capital investment reduction. But it should be noted that the preconcentration column was already included in the extractive distillation system and the fresh feed was diluted.

In this paper, database of LTTMs (ChCl/urea = 1:2) was firstly established in Aspen Plus by the user-defined method, and then simulation and optimization of the extractive distillation process for ethanol dehydration with choline chloride/urea 1:2 (Reline) or ethylene glycol (EG) as entrainers were worked out. In addition, an energy-efficient extractive distillation process which combining the preconcentration section and entrainer recovery section in one column was developed. The ethanol-water-Reline system was studied to verify the energy and economic advantages of the new process. In the studies, an optimum design for the conventional and new process was investigated based on global economic optimization.

2. Thermodynamic models

In this work, Aspen Plus 7.2 was used to simulate all the processes. The thermodynamic model NRTL was used to describe the non-ideality of liquid and ideal vapor phase behavior.

There is no data of LTTMs (Reline) in Aspen databank, therefore it is necessary to create a user-defined LTTMs database in order to allow LTTMs to be selected as components in a process simulation. The necessary specifications required are: molecular weight, normal boiling temperature, the critical properties (T_c, P_c and V_c), the acentric factor (ω), ideal gas heat capacity and liquid vapor pressure. All these parameters came from literatures [27,28]. The thermodynamic properties of Reline were listed in Table 1. The NRTL binary parameters for Reline-water-ethanol system were taken from the literature [1] (Table 2). For ethanol-water-EG system, the binary interaction parameters were retrieved from Aspen Plus database.

Table 1
Thermodynamic properties of the LTTMs Reline.

	M_w	T_b, K	T_c, K	P_c, bar	V_c, cm^3/mol	ω	Freezing Point, K
Reline	86.58	445.6	644.4	49.35	0.661	0.952	285.15

CPIG ($C_p = C_1 + C_2{*}T + C_3{*}T^2$), C_p, J. mol^{-1}. K^{-1}

	C_1	C_2	C_3
Reline	247.4	−0.5633	1.141×10^{-3}

Wanger ($\text{Ln}P_r = 1/T_r[A(1\text{-}T_r) + B(1\text{-}T_r)^{1.5} + C(1\text{-}T_r)^3 + D(1\text{-}T_r)^6]$)

	A	B	C	D
Reline	109.29	−308.4	484.22	−1009.34

Table 2
Parameters of NRTL binary parameters used in Ethanol/Water/Reline system.

Component i Component j	ethanol Water	water Reline	ethanol Reline
A_{ij}	0	0	0
A_{ji}	0	0	0
B_{ij}	−29.228	145.898	7698.22
B_{ji}	613.423	−672.841	502.646
C_{ij}	0.3	0.3	0.3

In order to confirm the feasibility of the extractive distillation process using Reline or ethylene glycol as entrainer, the residue curve maps for both ethanol-water-EG and ethanol-water-Reline systems at 1 atm were analyzed (Fig. 1). It could be seen from the Fig. 1 that pure ethanol and water were saddle points while Reline and ethylene glycol were stable nodes. There was no distillation region in the residual curves for both the two systems. As shown in Fig. 1, the volatility between ethanol and water was unity in the binary system at the azeotrope. The isovolatility line moved towards entrainer-ethanol side of the triangle by gradually adding Reline or ethylene glycol, at the same time the concentration of water went to zero. It could concluded that by adding ethylene glycol or Reline into the system, the relative volatility between ethanol and water became greater than unity and resulted in ethanol going up the extractive distillation column as the products. In addition, the location where the isovolatility curve intercepted the ethanol-entrainer edge of the triangle reflected the minimal entrainer flowrate for the separation. The closer the intersection point was to the ethanol corner, the less entrainer was required, which meant lower operating and capital costs [29]. Fig. 1 showed that the location was closer to the ethanol corner when using Reline as entrainer. It indicated that Reline was a more effective entrainer than ethylene glycol to some extent.

3. Steady state design and economic analysis

3.1. Design flowsheet via conventional extractive distillation

The simulation flowsheet for ethanol dehydration extractive distillation system consisted of extractive distillation column (EDC) and entrainer recovery column (ERC). The fresh feed containing ethanol and water (60 wt% of ethanol and 40 wt% of water) with the flowrate of 3000 kg/h was fed together with entrainer to the extractive distillation column. With the presence of entrainer, the relative volatility of ethanol to water was enhanced, thus causing high purity ethanol to be obtained at the top of EDC, then the bottom stream was sent to the entrainer recovery column to produce high purity water and high purity entrainer. The entrainer was then recycled back to the EDC. For EDC, the distillate composition was set at 99.9 wt% ethanol. For ERC, the distillate and bottom compositions were set at 99.5 wt% water and 99.9 wt % entrainer, respectively.

To find the optimal design of the extractive distillation system, total annual cost (TAC) was used as the objective function to be minimized. The equations of TAC [18,30] were listed in Table 3. As illustrated in Table 3, TAC included annual total operating cost and total capital cost divided by 3 years (payback period). Total capital cost included the costs of column, cooler, condenser and reboiler. Operating cost included the cost of steams.

There were quite a number of parameters in the process to be determined for optimum performance, including the total number stages of EDC (N_{T1}) and ERC (N_{T2}), molar reflux ratio of EDC (RR_1) and ERC (RR_2), the feed stage locations (N_{F1}, N_{F2} and N_{FS}), recycle entrainer flowrate (S), the pressure of EDC (P) and the temperature of the entrainer (T_{ent}). During the optimization, the pressure in ERC kept consistent with that in EDC. Because if the operating pressure of ERC

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