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Heat-Integrated Pressure-Swing Distillation Process for Separation of a Maximum-Boiling Azeotrope Ethylenediamine/Water

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ABSTRACT

A partially heat-integrated pressure-swing distillation process is designed for the separation of a maximum-boiling azeotrope of ethylenediamine/water. Aspen Plus and Aspen Dynamics are used to study rigorous steady state and dynamic simulations for this neat operation. An optimized configuration of this process is developed based on the proposed partial optimization and global economical optimization. From the results, it is found that the process with partial heat integration is more competitive than the non-heat-integrated one from the economic point view. The partially heat-integrated process helps save energy consumption of 19.79% and TAC of 15.30%, respectively. Basic and improved control structures are explored in this system, so that the better control solutions are discussed from the comparison. Purity fluctuations of products can be decreased by adding a ratio control block in the process. And the dynamic control is kept within acceptable limits.

1. Introduction

Ethylenediamine (EDA) is used for production of many industrial chemicals. It can form derivatives on proper conditions. Especially, EDA can form heterocycles because of its two amines. The EDA/water mixture can form a maximum-boiling homogeneous azeotrope at atmospheric pressure. Conventional distillation methods cannot reach efficient separation. Therefore, special distillation must be investigated to improve the separation efficiency. Lang et al. (2000a, 2000b) studied the separation of azeotropes by using extractive distillation and gave a separation method with continuous entrainer feeding. Salt-addition distillation was also explored by Lei et al. (2002). However, an extra component, entrainer, must be added in these processes. And it is believed that the entrainer would raise the possibility of products contamination. Pressure-swing distillation (PSD) is another method for azeotropes separation. Phimister and Seider (2000) studied the semicontinuous PSD. Modla and Lang (2008) suggested two new configurations for the batch PSD. One of the configurations is the double column batch rectifier for the separation of maximum azeotropes. And another is the double column batch stripper for the minimum azeotropes. It is considered as a special method of distillation based on the shift of the relative volatilities and azeotropic compositions by changing the system's pressure (Luyben and Chien, 2011). Because no other

Abbreviations: EDA, ethylenediamine; PSD, pressure-swing distillation; HPC, high-pressure column; LPC, low-pressure column; TAC, total annual cost; CS, control structure; VLE, vapor–liquid equilibrium; RR, reflux ratio; NT, number of total trays; SR, recycled flow rate; TC, condenser temperature; TR, reboiler temperature; Di, Diameter of column; D, distillate stream; NF, fresh feed tray; NR, recycled feed tray; HX, heat exchangers; P_I, proportional and integral; K_U, ultimate gains; P_U, ultimate periods; K_C, gain; τ_I, integral time; QR, , reboiler duty; Q_{aux}, auxiliary condenser duty; F, the fresh feed; TC, temperature controller; QRHP/F, reboiler duty of HPC/mole flow rate of F; BLP/F, bottom mass flow of LPC/mole flow rate of F.

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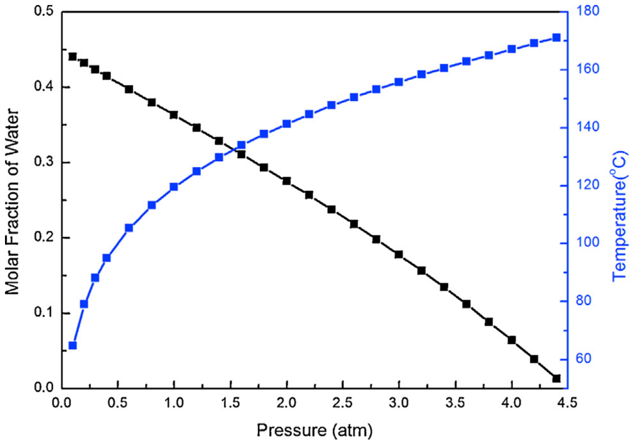

Fig. 1 – Effect of pressure on azeotropic composition and temperature.

component needs to be added, PSD process received much attention in azeotrope separation. PSD process is characterized by two columns operating at two different pressures. And it is possible for the condenser of the high-pressure column (HPC) to provide heat for the reboiler of low-pressure column (LPC) (Löwe, 2001). From the economic viewpoint, it is much more attractive for PSD process because the process can be generally heat integrated. In the past, heat integration was widely studied to improve the heat efficiency of the distillation process. Huang et al. (2008) investigated a PSD process of rectifying/stripping section type heat integration. Wang et al. (2015) studied a PSD process of condenser/reboiler type heat integration. Both of these systems can reach the requirement of energy saving (Jana, 2010).

The control of PSD systems for minimum-boiling azeotropes had been discussed by many papers in the past years. Repke et al. (2007) investigated a pressure-swing batch distillation process based on pilot-plant experiments. The article also gave a dynamic control example of separating a minimum-boiling acetonitrile/water mixture. Yu et al. (2012) separated methylal/methanol and Wang et al. (2014) dealt with tetrahydrofuran/methanol mixture by using PSD process. They all investigated the optimized configuration about separating minimum-boiling azeotropes and suggested several dynamic control solutions. Nowadays, the study of maximum-boiling azeotropes has received more and more attention. As is rarely discussed in the literature, the dynamic control appears to be challenging in these systems, which separate maximum-boiling azeotropes with heat-integrated PSD process. Luyben (2012, 2014a, 2014b) studied the separation of maximum-boiling azeotropic mixtures in detail from steady-stage design to dynamic control. A control structure (CS) had been developed for a heat-integrated pressure-swing distillation process. The dynamic control was much complex because of the variable pressure of HPC. Adding a flow controller in the bottom of HPC can prevent it from losing control.

The purpose of this article is to provide details of the EDA/water separation system and to develop proper dynamic control methods. However, the investigation of the design and control of PSD systems for maximum-boiling azeotropes is very limited. Thus, great efforts must be made to acquire data in this work. By ascertaining proper parameters such as reflux ratio, number of trays, and feeding locations, TAC is minimized to meet an optimum one of the system. Partial heat integration is used to reach the potential of large energy savings. Moreover, the control structures are also explored for the separation system of partially heat-integrated PSD.

2. Steady state design

The separation of the maximum-boiling azeotrope of EDA/water appears to be challenging. The PSD process is considered as one of the effective methods. In the PSD process, the feed rate is set up as 100 mol/h. And the feed mixture, made up of 40 mol% EDA and 60 mol% water, is separated into EDA product (with a purity of 99.5 mol%) and water product (the EDA impurity is not more than 0.5 mol%).

2.1. *Vapor–liquid equilibrium*

The simulation result is mainly based on Aspen Plus v7.1. Under the UNIQUAC physical properties, a maximum-boiling azeotrope, with composition of 63.70 mol% EDA, is formed at 119.54 °C at atmospheric pressure.

PSD process is based on the pressure sensitivity of the azeotrope. The effect of pressure on the azeotropic composition and temperature for the EDA/water binary azeotrope is shown in Fig. 1. The illustration clearly shows that the molar concentration of water changes significantly with pressure, which makes it feasible to separate the azeotrope by using PSD. In the EDA/water system, azeotropic composition and boiling point under different pressure are calculated. As is shown in Fig. 2 of T-xy curves, the maximum-boiling point appears at

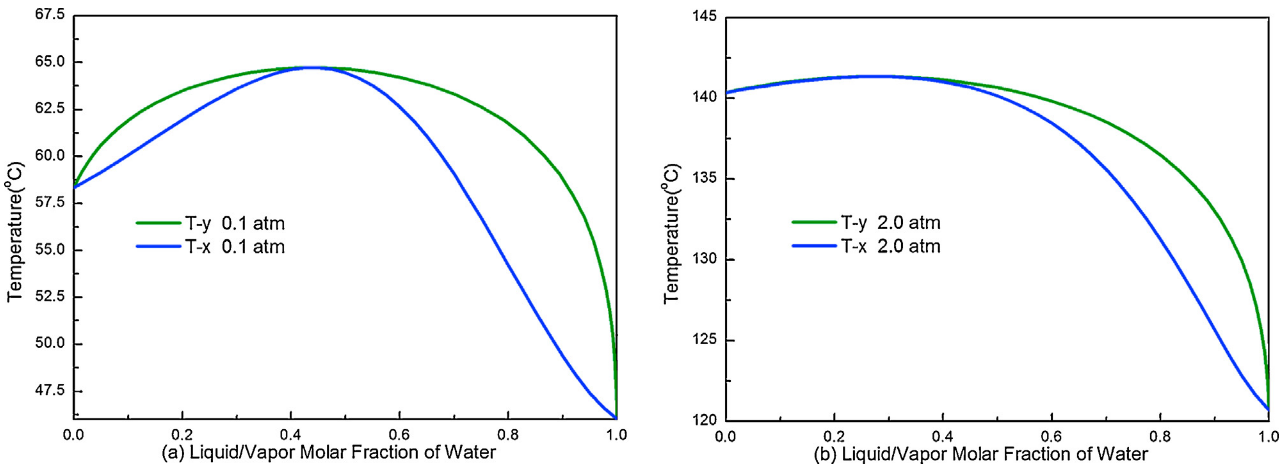

Fig. 2 – T-xy for EDA/water system at (a) 0.1 atm and (b) 2.0 atm.