



# Separating an azeotropic mixture of toluene and ethanol via heat integration pressure swing distillation

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## ARTICLE INFO

### Article history:

Received 17 November 2014  
Received in revised form 25 January 2015  
Accepted 28 February 2015  
Available online 10 March 2015

### Keywords:

Toluene  
Ethanol  
Pressure-swing distillation  
Heat integration  
Dynamic simulation

## ABSTRACT

A procedure is suggested to separating a minimum-boiling azeotrope of toluene and ethanol via heat integration pressure swing distillation (PSD), and an optimized separation configuration is obtained via taking the minimization of the total annual cost (TAC) as an objective function. The result demonstrates that PSD with heat integration is more economical than conventional PSD without heat integration. Based on steady-state simulation results, several control structures were explored using Aspen Dynamics. The results indicate that the composition/temperature cascade control structure and the pressure-compensated temperature control of a PSD process with partial heat integration with stage 21 selected as the control stage in the low pressure column can handle disturbances well. As for the PSD with full heat integration, stage 20 of the low pressure column can act as the control stage because of its more efficient controllability under feed flow rate and feed composition disturbances.

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## 1. Introduction

Toluene and ethanol are both commonly used solvents in the chemical and the pharmaceutical industries due to their excellent dissolution ability. In the production of steroid drugs, there is often a problem of separating mixed solvents. It remains a great challenge to effectively separate toluene and ethanol because their highly nonideal vapor–liquid equilibrium produces a minimum-boiling azeotrope (Luyben, 2013). Several methods have been used to overcome the natural distillation boundaries imposed by the presence of azeotropes. The addition of a third component is required for some separations to shift the vapor–liquid equilibrium, which tends to increase the operating costs. The books written by Gil and Botía introduce extractive distillation to achieve the separation of an acetone/methanol mixture (Gil et al., 2009). A comparison between extractive distillation and pressure swing distillation (PSD) to separate the azeotropic mixture of acetone and methanol was made by Luyben (2008a). It is clear that selecting a reasonable method is important to achieving the effective separation of mixed solvents.

In extractive distillation, two columns are used to achieve the separation of azeotropes, one for extractive separation and the other for solvent recuperation. A higher boiling solvent (or entrainer) needs to be added into the binary mixture to increase

the relative volatility of the original components (Langston et al., 2005; Munoz et al., 2006; Orchillés et al., 2009). In general, the azeotropic mixture and the solvent are fed to the first extraction column together, with the light key component obtained at the top of the column and the less volatile components including the extractant and heavy key components collected at the bottom. Subsequently, the less volatile components are fed to the second column, from which a high-purity key-component distillate product can be obtained, and the solvent at the bottom is recycled to the first extractive distillation column. The key issue is how to select the most proper solvent to reduce the energy consumption and total capital investment (Xu and Wang, 2006a,b).

Different from extractive distillation, azeotropic heterogeneous distillation often uses a lower boiling solvent to drive one of the key components overhead in the first column, where a low boiling azeotrope is formed between the entrainer and solvents. One key component leaves at the bottom with a high purity, while the low boiling-point azeotrope goes overhead as the distillate stream, which is then fed to a second column (the solvent recovery column) to generate the second key component at the bottom with a high purity. The distillate of the second column, which contains some key components and the entrainer, is recycled back into the first column. The use of azeotropic heterogeneous distillation can alleviate the problem of high energy consumption and investment (Luyben, 2012).

Another method widely used in the separation of azeotropes is pressure-swing azeotrope distillation (PSAD), which was proposed in detail by Lewis in 1982. Two operating columns including

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**Notation**

$A$	heat transfer area of heat exchanger [m <sup>2</sup> ]
$B$	bottom stream flow rate [kg/h]
$D$	distillate flow rate [kg/h]
$ID$	diameter of the column [m]
$K$	heat transfer coefficient
$H$	length of the vessel [m]
$N_T$	number of stages
$P$	pressure of column
$Q$	heat duty [kW]
HPC	high pressure column
$K_c$	gain of controller
LPC	low pressure column
NRTL	non-random two liquid
PSD	pressure swing distillation
HIPSD	heat integration pressure swing distillation
PC1, PC2	pressure controller of LPC, HPC
$Q_R$	reboiler duty
$Q_R/F$	reboiler duty/mol flow rate of $F$
RR	reflux ratio
TC1, TC2	temperature controller of LPC, HPC
TAC	total annual cost [\$/y]
M&S	Marshall & Swift index
$\tau_I$	integral time of controller
$x_D$	composition of the distillate stream
$x_B$	composition of the bottom stream

a high pressure column (HPC) and a low pressure column (LPC) are determined by the composition variation of the azeotropic mixtures at different pressures. Importantly, additional solvents are not involved in PSAD (Knapp and Doherty, 1992; Li et al., 2013). PSD can be employed to separate both minimum-boiling and maximum-boiling azeotropic mixtures. To date, many efforts have been devoted to study PSD, and it has been proven that PSD technology exhibits superiority over other separation methods for azeotropic mixtures.

There has been much interest in investigating the heat integration used for PSD processes in recent years. Heat integration PSD (HIPSD) can occur in one of two ways, either by integrating the condenser in the high pressure column (HPC) with the reboiler in the low pressure column (LPC) (condenser/reboiler type) or by integrating the stripping section in the LPC with the rectifying section in the HPC (rectifying/stripping type) (Sorensen, 2001; Suphanit, 2011; Wang et al., 2014). Both schemes have significant advantages in energy saving. Luyben presented a multiple-loop control system for HIPSD that can control this process very well (Luyben and Chien, 2011). Until now, there have been few reports on the rectifying/stripping type of heat integration. Nakaiwa studied the heat integration of the rectifying and stripping sections, proposed a program for its conceptually design, and ultimately developed the most optimal economic program by comparing it with heat integration of the condenser/reboiler type (Nakaiwa et al., 2003). The HIPSD process is an effective way to save energy. In this article, HIPSD processes for the separation of mixed toluene and ethanol with partial and full heat integration is optimized based on economic considerations using Aspen Plus and Aspen Dynamic. Both steady-state design and dynamic controllability are discussed.

## 2. Design

In this work, an HIPSD process was designed for separating an azeotropic mixture of toluene and ethanol using Aspen Plus. A simulation model based on the RADFRAC block using the NRTL property

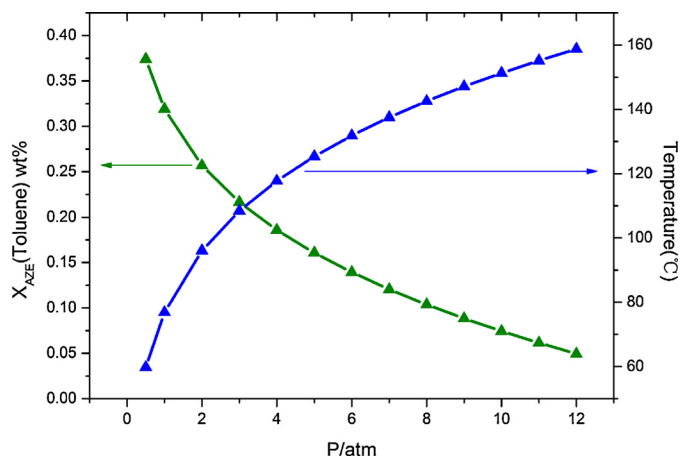


Fig. 1. Effect of pressure on azeotropic composition and temperature.

method was established based on the vapor–liquid equilibrium data of the binary system. The boiling points of toluene and ethanol at atmospheric pressure are 383.15 K and 349.85 K, respectively. A minimum-boiling azeotrope is formed when the mass content of ethanol in the binary system is 68.08% at 350.01 K and atmospheric pressure.

Fig. 1 shows the azeotropic compositions and azeotropic points for the binary system at different pressures. It is observed that the mass content of toluene in the azeotropic mixture decreases significantly upon increasing the pressure, while the azeotropic points increase quickly, both of which indicate that the pressure has an important effect on the separation of the azeotropic mixtures.

### 2.1. Optimization method

The HIPSD process is optimized by minimizing the total annual cost (TAC), which is one of the indexes most frequently used to measure the profit capability of a production process in its entirety. The capital cost and operating cost were also calculated over a five-year payback period. The operating time of the distillation system is set at 8000 h per year. The detailed calculations are shown as follows:

$$\text{TAC} = \frac{\text{total capital cost}}{\text{payback period}} + \text{annual energy cost} \quad (1)$$

$$\begin{aligned} \text{Capital cost} = & \text{column vessel cost} (C_1) + \text{plate cost} (C_2) \\ & + \text{heat exchangers cost} (C_3) \end{aligned} \quad (2)$$

$$\begin{aligned} \text{Operating cost} = & \text{annual steam cost} (C_4) \\ & + \text{annual cooling water cost} (C_5) \end{aligned} \quad (3)$$

The capital cost for the HIPSD process includes the column vessel, plate and two heat exchangers (i.e., reboiler and condenser). The column and sieve plate parameters were determined by the “tray sizing” function in Aspen Plus. The number of trays was determined using Aspen Plus, with the first stage being the reflux drum and the last stage the reboiler. The total heat transfer coefficients of the reboiler and condenser are 0.852 kW/(K m<sup>2</sup>) and 0.568 kW/(K m<sup>2</sup>), respectively. Additional costs including pumps, reflux drums, valves and pipes can be ignored because their costs are much lower than those of the column vessels and heat exchangers. The basis of the economics, the sizing relationships and parameters are listed in Table 1, and the relevant approximation methods were provided by Douglas (1988).

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