Separation and Purification Technology 207 (2018) 489–497

Contents lists available at ScienceDirect

Separation and Purification Technology

journal homepage: www.elsevier.com/locate/seppur

Design and control of an energy-efficient alternative process for the separation of methanol/toluene/water ternary azeotropic mixture

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ARTICLE INFO

Keywords:
Solvent extraction
Multiple azeotropes
Separation system
Process design
Process control

ABSTRACT

Separation of a ternary mixture containing methanol/toluene/water is a tough task because toluene forms azeotropes with both methanol and water. A recent paper proposed using *N*-methyl-2-pyrrolidone as heavy entrainer and included a two-column extractive distillation system plus a decanter for the separation of this ternary mixture. In this paper, an energy-efficient simpler process composed of an extraction column and a regular distillation column is investigated in order to achieve the same separation task. Water is used as the solvent to extract methanol from toluene in the extraction column. Toluene is obtained as raffinate while extract phase containing water and methanol is sent to a distillation column for the purification of these two components. Because water itself is a component in the separation mixture, there is no need to introduce any foreign component into the system and hence avoid the possibility of contaminating the products. To compare the optimal result of this design flowsheet with the recently published separation method, our proposed design provides 56.3% savings in total annual cost and 57.1% savings in annual operating cost. Furthermore, a control structure with adjustable solvent flowrate is devised for our proposed process based on the results of open-loop and closed-loop sensitivity tests. Closed-loop dynamic responses show that all products can be maintained at high purity despite large throughput and feed composition disturbances.

1. Introduction

Mixture of methanol, toluene and water is commonly produced in pharmaceutical [1] and methanol-to-aromatics [2,3] processes. Usually, it is necessary to separate organic chemicals from water for the purpose of reuse in production section. However, separation of this methanol/toluene/water mixture is a tough task because toluene forms azeotropes with both methanol and water. Methods aiming to deal with only the separation of methanol/toluene mixture were studied in the past few years. Garg et al. [4] indicated the possibility of using pervaporation to separate methanol/toluene azeotrope. Nevertheless, this kind of process is still not suitable for industrial application due to the concern of high operating costs [5]. In 2013, Tang et al. [5] proposed an extractive distillation process using *o*-xylene as heavy entrainer and the simulation results were confirmed via experiments. Luyben [6] suggested an extractive distillation system, which adopted triethylamine as intermediate entrainer, for the separation of mixture containing equal amount of methanol and toluene. In 2016, Ma et al. [7] investigated two kinds of extractive distillation processes in which heavy and intermediate entrainers were adopted respectively, the results showed that process with triethylamine (intermediate entrainer) performs better from the standpoint of economics and controllability. Subsequently, a corresponding extractive dividing-wall column using triethylamine as entrainer was studied for the purpose of saving energy and total annual cost (TAC) [8]. Besides extractive distillation process, method based on the principle of azeotropic distillation combining pressure-swing is also feasible for separating methanol/toluene azeotrope [9]. Although the above studies gave remarkable insights about the separation of methanol and toluene, they failed to consider a more practical situation that ternary methanol/toluene/water mixture is encountered.

In 2018, Wang et al. [10] proposed a two column plus decanter extractive distillation system for the separation of this ternary azeotropic mixture. By using *N*-methyl-2-pyrrolidone (NMP) as heavy entrainer, methanol is obtained from the top of the first extractive distillation column while bottom stream containing toluene, water and NMP is sent to another column. Toluene and water are obtained as the distillate of the second column and a decanter is installed to split this mixture into toluene phase and water phase. Heavy entrainer from the bottom of the second column is cooled and recycled to extractive

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https://doi.org/10.1016/j.seppur.2018.07.003
Received 21 April 2018; Received in revised form 18 June 2018; Accepted 3 July 2018
Available online 04 July 2018
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distillation column. The most noticeable drawback of this process is that expensive high pressure steam must be used as the heating source at the bottom of distillation column from which heavy entrainer is recovered. Furthermore, large energy requirement is observed for the second distillation column and the TAC associated with this second column accounts for about 52.4% in the overall TAC. Recently, Yang et al. [11] proposed a corresponding extractive dividing-wall column with heat integration based on the design mentioned in Wang et al. [10]. The results show that this thermally-coupled counterpart provides further reduction in TAC for the separation of methanol/toluene/water mixture. In this paper, an energy-efficient alternative separation process based on totally different design concept is proposed for the separation of this ternary mixture having same feed condition as the one mentioned in Wang et al. [10].

Solvent extraction remains to be one of the most preferable separation methods because no reboiler duty is required and hence low operating cost is expectable. Garcia-Chavez et al. [12] adopted ionic liquid to perform solvent extraction for the recovery of glycols from aqueous stream, and the results showed that noticeable energy saving is obtained compared to a conventional multi-effect distillation system. For more common solvent extraction processes which adopt water as solvent, Perederic et al. [13] mentioned an alternative process that gasoline is obtained as raffinate from an extraction column, while methanol goes with water as extract stream. In a pilot testing, methanol is extracted from an organic stream under a relatively low water flowrate [14]. In most of the cases, solvent extraction is combined with other technologies for the further purification of products and the recovery of solvent. Martínez et al. [15] designed a bioethanol dehydration process which uses n-dodecane as solvent to remove some of the water in the dilute feed stream, while further purification of ethanol is completed in the following extractive distillation sequence. Recently, hybrid extraction-distillation processes which incorporate solvent extraction with distillation show potential on TAC saving in various separation issues [16–21]. Owing to the fact that solvent extraction is a separation method based on the principle of liquid-liquid equilibrium, it is possible for an extraction column to cross distillation boundary formed by azeotropes. Solvent can be recovered by a regular distillation column and sent back to the extraction column after cooling, this kind of design can be viewed as a simplest hybrid extraction-distillation process.

In the following section, water is selected as the solvent to extract methanol from the ternary methanol/toluene/water mixture. Because water itself is the component being separated, this process will not introduce any new component into the system and hence avoid the possibility of contaminating the products. A regular distillation column is installed after the extraction column to complete the purification of methanol product and the recovery of water. To assess the economic advantage of our proposed separation method, comparisons of TAC and energy consumption will be made with the extractive distillation system recently published by Wang et al. [10] and also with the dividing-wall counterpart proposed by Yang et al. [11]. Furthermore, a feasible control strategy will be devised for our proposed process and tested for the rejection of large throughput and feed composition disturbances.

2. Steady-state simulation of this process

2.1. Thermodynamic model and conceptual design

Wang et al. [22] measured a series of liquid-liquid equilibrium data for ternary methanol/toluene/water mixture at atmospheric pressure. In their work, experimental data were correlated using nonrandom two-liquid (NRTL) and universal quasichemical (UNIQUAC) models, and the results showed that UNIQUAC model predicts the equilibrium behavior better than NRTL model for this system. In Wang et al. [10], azeotropes predicted by UNIQUAC model were also compared with the experimental data, and the consistency of azeotropic composition confirmed the precision of UNIQUAC model. Hence, UNIQUAC model will be adopted in this paper to describe the vapor-liquid and liquid-liquid equilibrium behavior of ternary methanol/toluene/water (MeOH/Tol/H_2O) system in the following process design. UNIQUAC model parameters adopted in this study are listed in Table 1.

Table 1
UNIQUAC model parameters of the studied system.

Comp. *i* Comp. *j*	MeOH Tol	MeOH H_2O	Tol H_2O
a_{ij}	0	−1.066	0
a_{ji}	0	0.644	0
b_{ij} (K)	27.831	432.879	−950.6
b_{ji} (K)	−563.013	−322.131	−350.21

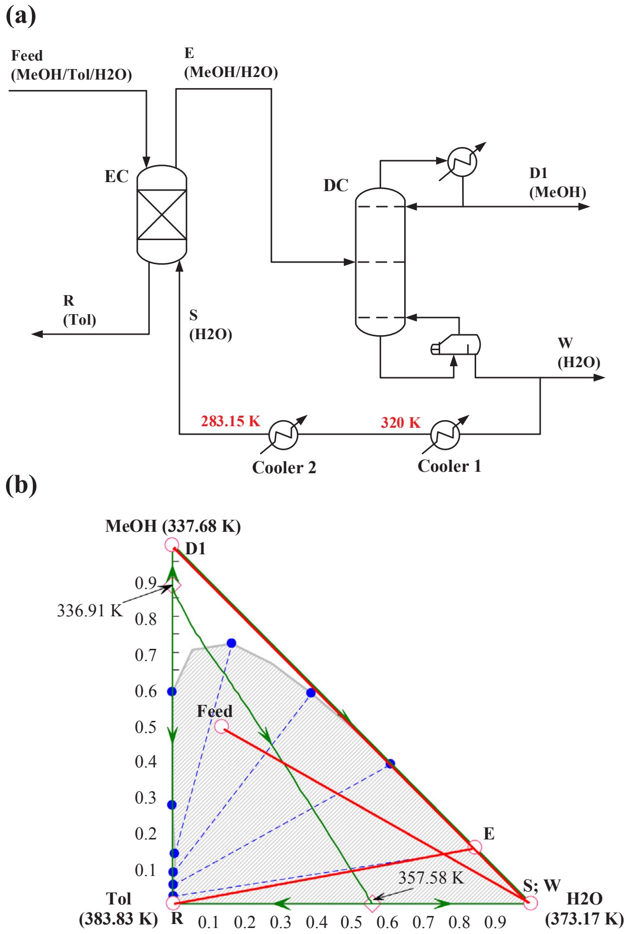

Fig. 1. (a) Conceptual design for the proposed system (b) Material balance lines and liquid-liquid envelope for the studied system.

Conceptual design of the proposed separation process is shown in Fig. 1a, in this design flowsheet, fresh feed is sent into an extraction column (EC) while solvent (S) enters the column from the opposite direction. After a multi-stage liquid-liquid separation, Tol product is obtained as raffinate stream (R). Extract stream (E) containing MeOH and H_2O is fed into a regular distillation column (DC), MeOH product is collected from the top while pure H_2O leaves the column from the bottom. A large portion of bottom stream is recycled and cooled before acting as solvent, while stream W leaves the process for the overall material balance of H_2O component. Fig. 1b illustrates these design concepts by locating the composition of every main stream in a ternary diagram at 1 atm. As shown in this figure, Tol forms a homogeneous azeotrope with MeOH (336.91 K) and a heterogeneous azeotrope with

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