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Energy-efficient heterogeneous extractive distillation system for the separation of close-boiling cyclohexane/cyclohexene mixture

Chun-Cheng Yi, Wen-Chi Huang, I-Lung Chien*

Department of Chemical Engineering, National Taiwan University, Taipei 10617, Taiwan

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ABSTRACT

Cyclohexane and cyclohexene are close-boiling compounds with relative volatility extremely close to unity, thus, separation via conventional distillation is impractical. In this paper, an energy-efficient heterogeneous extractive distillation system using ethylene glycol as heavy entrainer is proposed. By adding ethylene glycol into the system, large liquid-liquid envelope will form. However, the principle of separation is not exclusively the formation and split of heterogeneous azeotrope like that of a typical heterogeneous azeotropic distillation process, but also because of ethylene glycol greatly enhances the relative volatility of cyclohexane over cyclohexene when treated as a heavy entrainer. The proposed heterogeneous extractive distillation column includes a top decanter and no rectifying section. After establishing the base design of the proposed system, energy-saving schemes are also suggested to further reduce the total annual cost and the annual operating cost. The results show that the proposed heterogeneous extractive distillation system with a process-to-process heat exchanger has the predominant benefit of the lowest total annual cost. To compare the optimal result of this design flowsheet with a recently published separation method utilizing two reactive-distillation columns, our proposed design is much more economical with 61.4% savings in total annual cost and 64.6% savings in annual operating cost.

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1. Introduction

Cyclohexene (ENE) can be used to synthesize many kinds of chemicals of high commercial value, such as adipic acid, an important reagent in the manufacture of nylon. Also, various organic compounds can be prepared by reactions in which ENE is involved. Therefore, it appears that a process that makes ENE obtainable from inexpensive raw material is quite attractive. One of such technologies is to convert cyclohexane (ANE) to ENE by dehydrogenation or by dehydrohalogenation after ANE is halogenated [1]. Another process for the production of ENE is the partial hydrogenation of benzene under the existence of heterogeneous catalyst [2]. However, the above operations essentially let a certain amount of ANE present in the ENE product stream, either as the un-reacted material or as the byproduct. This situation seriously limits the application of this impure ENE product stream as an important raw material; therefore, the separation of ANE and ENE becomes a major concern in the relevant industrial process.

ANE and ENE are close boiling compounds with relative volatility very close to unity (1.074 at 1 atm). It is, consequently, a tough task to separate ANE from ENE, according to the difficulty given

by thermodynamics. Few studies have been reported on the separation of ANE and ENE nowadays, hence an economical process for the separation of these two compounds is desirable. Steyer et al. [3] proposed a conceptual design to reactively separate ANE and ENE using water as reactive entrainer. This process comprises two reactive distillation columns, wherein hydration and dehydration are taking place respectively. In that separation process, the principle is to hydrate ENE into cyclohexanol and separate ANE from all of the other components. After that, cyclohexanol is dehydrated into ENE and water, ENE is obtained from the top of the dehydration column while water is recycled to the hydration column. In 2016, Yu et al. [4] proposed a modified flowsheet wherein the two reactive distillation columns are thermally coupled to eliminate the remixing effect originally occurred in the bottom of the hydration column. Although the total annual cost (TAC) of this design is reduced compared to the conventional process, it can be observed that the whole process is still energy intensive and the number of stages required in this complex design structure is quite large.

In this study, an energy-efficient heterogeneous extractive distillation system will be proposed for this separation task. To assess the economic advantage of our proposed separation method, comparisons of TAC and energy consumption will be made with the thermally coupled reactive distillation system just mentioned in Yu et al. [4].

* Corresponding author.

E-mail address: ilungchien@ntu.edu.tw (I.-L. Chien).

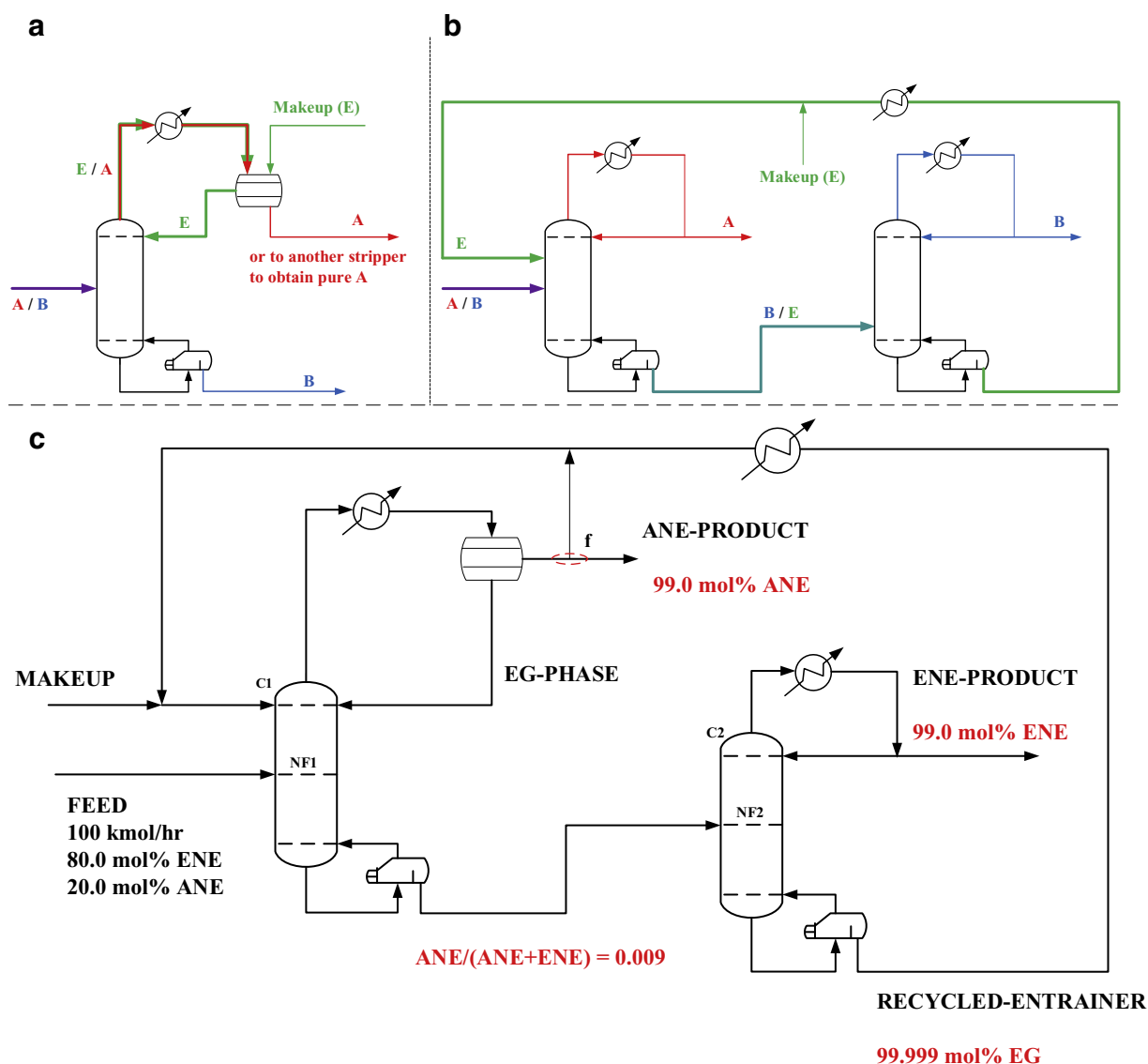


Fig. 1. Conceptual design for (a) heterogeneous azeotropic distillation (b) extractive distillation system (c) heterogeneous extractive distillation system.

2. Heterogeneous extractive distillation system

Extractive distillation and heterogeneous azeotropic distillation are two commonly used separation methods for the separation of azeotrope or close-boiling mixtures, both of which rely on the addition of entrainer. A conceptual design for the heterogeneous azeotropic distillation is illustrated in Fig. 1a. The principle of this separation process is to employ a light entrainer (E in Fig. 1a) to form a heterogeneous minimum-boiling azeotrope and carries one of the original component to the top of column, while another component exits the column from the bottom. The minimum heterogeneous azeotrope is condensed and sent to a decanter for liquid-liquid separation. Phase composed of light entrainer is recycled while another phase is obtained as top product or further purified in another stripping column to obtain another product. Typical applications of heterogeneous azeotropic distillation include acetic acid dehydration [5] and ethanol dehydration [6], which have been studied for decades. However, several recent studies have found that heterogeneous azeotropic distillation is more energy intensive to extractive distillation for some separation tasks [7–9].

A scheme of typical extractive distillation process can be seen in Fig. 1b. Differ from heterogeneous azeotropic distillation, this process employs a heavy entrainer (E in Fig. 1b) to enhance the relative volatility of the two original components. Light component is obtained as the distillate of the first column, while an entrainer recovery column is installed to purify another component and recycle the heavy entrainer. The recycled heavy entrainer is fed at a stage which is a few stages below from the top of the first column, that is to say, a rectifying section is needed to guarantee the separation of light component from heavy entrainer. Studies that focus on extractive distillation process [10,11] are common nowadays mainly because it is pervasively regarded as a promising separation process.

In this paper, a new design flowsheet is proposed as in Fig. 1c. For the first column, it is resemble heterogeneous azeotropic column in Fig. 1a with a top decanter. However in Fig. 1a, one product is obtained from the bottom stream. On the contrary in the proposed design, one product is obtained in one phase of the top decanter while the bottom stream containing a mixture of entrainer and the other component. The overall two-column sequence is similar to the design of a normal extractive distillation

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