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# Chemical Engineering Research and Design

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## Control comparison of conventional and thermally coupled ternary extractive distillation processes

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### ARTICLE INFO

#### Article history:

Received 13 October 2015

Received in revised form 24

November 2015

Accepted 27 November 2015

Available online 11 December 2015

#### Keywords:

Extractive distillation

Ternary systems

Controllability

### ABSTRACT

Extractive distillation is widely used to separate binary mixtures with azeotropes that prevent separation in a single distillation column. The conventional binary extractive distillation configuration uses two columns with one key component going overhead in the extractive column and the other key component going either overhead or out the bottom of the solvent recovery column, depending on the effect of the solvent on the volatilities of the key components.

Extractive distillation can also be applied to separate ternary mixtures with azeotropes. The conventional ternary extractive process requires three columns. Unconventional processes can also be used, such as thermally coupled sidestream/rectifier columns. Economic advantages of the thermally coupled process have been reported for some separations.

The controllability of extractive distillation system for binary mixtures has been explored in many papers. The novel contribution of this paper is the exploration of the dynamic controllability of extractive distillation systems for a ternary mixture. The dynamics of a conventional three-column process are compared with a thermally coupled column/rectifier process.

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## 1. Introduction

There is a rich literature covering the use of extractive distillation to separate binary mixtures with azeotropes. Many papers and books (Wankat, 1988; Stichlmair and Fair, 1998; Doherty and Malone, 2001; Luyben and Chien, 2010) have appeared that discuss the various types of configurations that can be used. The conventional configuration uses two distillation columns with a solvent fed near the top of the first column, and one of the key components taken overhead. The essentially binary mixture leaving in the bottoms is separated in the second column with the solvent recycled from either the bottom (high-boiling solvent) or the top (intermediate-boiling solvent) of the column.

Nonconventional configurations for binary mixtures have also been explored (Kiss and Suszwalak, 2012; Wu et al., 2013). The E-DWC features a single divided-wall column with the

wall extending from the top of the stripping section all the way to the top of the column. The bottoms stream is the high-boiling solvent, which is recycled to the near the top of the feed side of the wall. Each side of the wall has its independent condenser and reflux. The two distillate streams are the high-purity key components of the binary feed mixture. An important application is the dehydration of ethanol using ethylene glycol solvent.

However, the single-column E-DWC cannot be applied when the initial feed is a ternary mixture since three product streams must be produced. In a very comprehensive and insightful paper, Timoshenko et al. (2015) discuss the many alternative extractive distillation configurations for ternary mixtures, both conventional three-column flowsheets and partially thermally coupled flowsheets, for a variety of phase equilibrium relationships. A case study is presented for the ternary mixture of benzene (boiling point

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<http://dx.doi.org/10.1016/j.cherd.2015.11.021>

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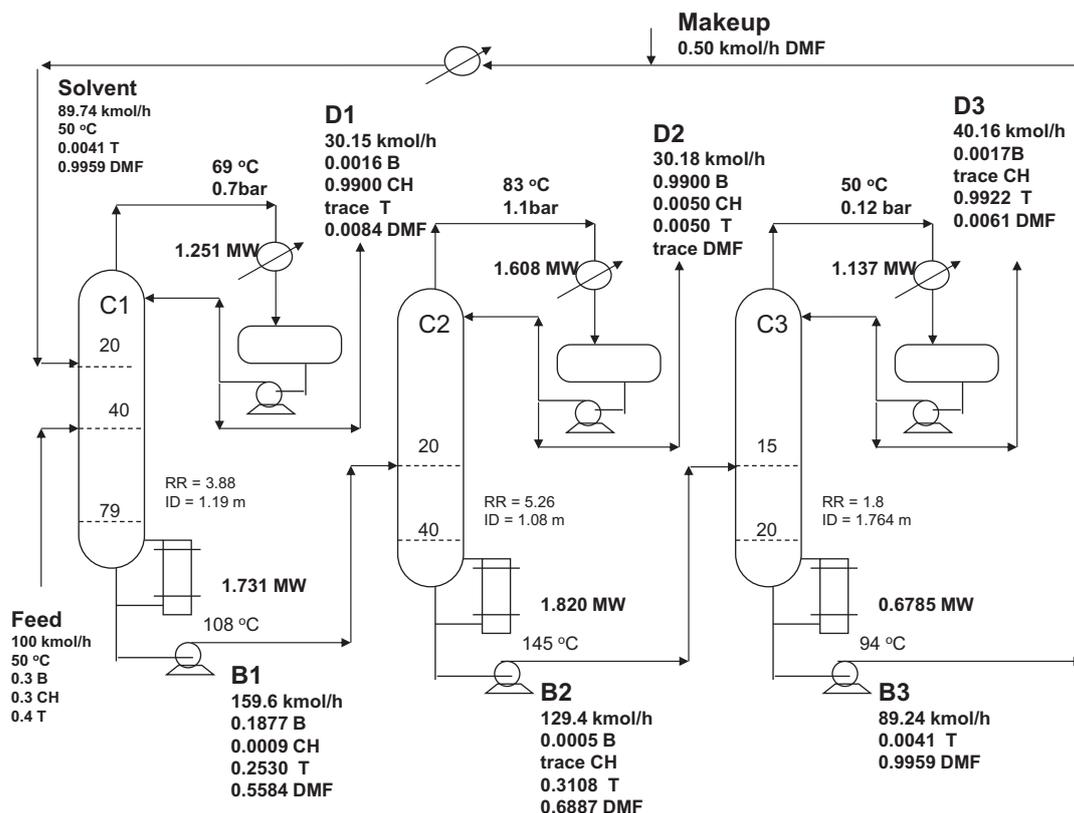


Fig. 1 – Conventional ternary extractive distillation.

78.11 °C), cyclohexane (80.72 °C) and toluene (110.6 °C) using *N*-methylpyrrolidone as a heavy solvent (204.3 °C). The conventional three-column flowsheet was shown to have 13% higher total reboiler duty than a non-conventional two-column flowsheet with a side rectifier on the extractive column. No discussion of the dynamic controllability of the alternative flowsheets was presented. That is the purpose of this paper.

## 2. Process studied

A conventional three-column configuration (Fig. 1) is compared with a two-column configuration with a side rectifier (Fig. 2). We study the same ternary mixture of key components considered by Timoshenko et al. (2015) (benzene, cyclohexane and toluene). The minimum-boiling azeotrope in the benzene/cyclohexane system is shown in Fig. 3.

Timoshenko used the high-boiling solvent *N*-methylpyrrolidone (NMP). In our Aspen simulations using NRTL physical properties, we were unable to duplicate the Timoshenko designs since the separation in the extractive column between cyclohexane and benzene using NMP was inadequate to achieve the desired product purities even with very large solvent-to-feed ratios. Instead we switched to dimethyl formamide (DMF).

As shown in Fig. 4, DMF has a stronger effect on the volatility between benzene and cyclohexane. The ordinate and abscissa are the normalized mole fractions in the vapor and liquid phases excluding the solvent. The plots are generated by using a *Flash2* Aspen model. The flash pressure and the feed composition are fixed. For example, with a feed of 50 mol% DMF, 25 mol% benzene and 25 mol% cyclohexane, the normalized composition is 50 mol% benzene. The vapor fraction in the

*Flash2* is specified to be very small ( $10^{-5}$ ), so the liquid composition is the same as the feed. The resulting calculated vapor composition in equilibrium with the known liquid composition is then used to calculate the normalized vapor benzene composition.

Since our objective is to make a comparative study of dynamic controllability between the two processes, we feel the using DMF instead of NMP should have little effect on the comparison.

Notice in Fig. 4 that the normalized *xy* curve is below the 45-degree line. This means that cyclohexane goes out the top of the extractive column even though it is higher boiling than benzene. Fig. 5 gives a ternary diagram for the benzene/cyclohexane/DMF system with the distillate (D1), fresh feed (F), solvent (S), mix point (M) and bottoms (B1) points are indicated (neglecting the third key component toluene). In the region below the isovolatility line as we go towards more solvent, the relative volatility of benzene to cyclohexane is less than unity. So the cyclohexane goes overhead in the extractive column.

### 2.1. Conventional process

Fig. 1 gives the flowsheet of the conventional process studied. The fresh feed is 100 kmol/h with composition 30 mol% benzene (B), 30 mol% cyclohexane (CH) and 40 mol% toluene (T). Fresh feed is fed on Stage 40 of an 80-stage column. The DMF solvent is fed on Stage 20 at 89.74 kmol/h with composition 0.41 mol% toluene and 99.59 mol% DMF.

The operating pressure of the extractive column (C1) is set at 0.7 bar because there is an azeotrope between toluene and DMF at pressures higher than 0.7 bar that adversely affects the separation. At 1 atm this azeotrope is 1.54 mol% DMF at 110 °C. At 0.7 bar, the azeotrope disappears.

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