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# Energy-saving dividing-wall column design and control for benzene extraction distillation via mixed entrainer



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#### ABSTRACT

Benzene and cyclohexane form an azeotrope (Benzene, 49.7 w.t%, 1 atm), which can be separated by extractive distillation. In this study, o-xylene is added into the sulfolane entrainer for mixed entrainer to make the operation conditions more convenient and controllable. The TAC (total annual cost, generally utilized in global economic optimization) of the process with o-xylene as mixed entrainer is less than that of the process with SULF as entrainer. In addition, as the most energy saving process, the process of extractive dividing wall column (EDWC) is proposed, for it can save 44.0% of energy and 35.8% of TAC, compared with the conventional extractive distillation arrangement with pure entrainer. Besides, two control structures are established for EDWC, and their dynamic performances are evaluated by feed flow rate disturbances and feed composition disturbances. Finally, it is found that the disturbances are well handled with the second control structure.

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#### 1. Introduction

Benzene (BEN) is an important basic chemical materials, which can be used to synthesize a variety of fine organic chemical intermediates. The market has great demand in high purity BEN, so it has great social effects to produce BEN products to suit market demand. In the production of catalytic reforming or pyrolysis hydrogenation, there is usually existing a mixture of BEN and cyclohexane (CYH). BEN and CYH can form azeotrope, which makes their separation via traditional distillation column unrealized. Therefore, an effective alternative separation technique is highly required in industry.

There are two common methods to separate this system in industry. One is liquid–liquid extraction. Liquid–liquid extraction is the unit operation which uses different solubility of components in solvents to separate the mixture. It is widely used in chemical, metallurgy, food industry and petroleum refining industry. In the aromatics extraction process, the ideal solvent is sulfolane (SULF). Because it can obtain higher aromatic hydrocarbon recovery rate and can be dissolved into two phase separation and dissolution agent. Schuur et al. studied different entrainer in liquid–liquid extraction, they considered crown ether based extractants, metal complexes, metalloids and so on [1]. Billard et al. discussed actinides and lanthanides by using ionic liquids in liquid-liquid extraction [2].

The other method is aromatics (BEN) extraction distillation which is an extractive distillation process. Extractive distillation is a special distillation method, which continuous adding entrainer into the distillation column to change the relative volatility of the separation components. Many different entrainers can be used in extractive distillation. SUFL can also be used in extractive distillation. Oin et al. discussed the use of extractive distillation for the separation of BEN-CYH via pure entrainer [3]. SUFL was used as entrainer and two control structures were proposed. The SUFL would be thermally decomposed when the base temperature was over 200°C. The pressure of the condenser in the entrainer recovery column (ERC) was 0.08 atm and the top temperature of ERC was 15.7 °C. Because the temperature was too low to use the convenient water as condensing intermedium, the operating cost would increase. Sun et al. studied separation of BEN-CYH system by extractive dividing wall column with furfural as entrainer [4]. However, because the boiling point of furfural was too low, there were some problems after treatment.

Due to the continuous improvement of limited energy resources, the process has been strengthened, which has caused wide attention in the world [5–7]. For further energy savings, the new configuration with integrated two columns into one shell, which is identified as a dividing wall distillation column (DWC) [8]. Gómez-Castro et al. found that 33% reduction of energy had been saved in DWC when they studied six cases of different feed

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Nomenclature		
EDWC	Extractive dividing well column	
	Dividing wall distillation column	
BEN	Benzene	
CVH	Cyclohevane	
	Sulfolane	
F	The fresh feed of the extractive distillation column:	
R/F	The mass ratio of reflux flow rate/flow rate	
FF	The flow rate of entrainer	
FDC	The extractive distillation column	
FRC	The entrainer recovery column	
EDC-C	The extractive distillation column of CYH	
EDC-B	The BEN recovery column	
RC	The entrainer recovery column	
F2	The feed of ERC	
RR	The reflux ratio	
EF/F	The molar ratio of mixture entrainer to feed	
N <sub>F</sub>	The stage of fresh feed	
N <sub>EF</sub>	The mixture entrainer feed stage	
NT <sub>EDC-C</sub>	The number of total stages of EDC-C	
NT <sub>EDC-B</sub>	The number of total stages of EDC-B	
NT <sub>RC</sub>	The number of total stages of RC	
XD1	The purity of top product in EDC-C	
XD2	The purity of top product in EDC-B	
HX	Heat exchangers	
K <sub>U</sub>	Ultimate gains	
$P_{\rm U}$	Ultimate periods	
Кс	Gain	
$ au_{\mathrm{I}}$	Integral time	
QR	Reboiler duty	
TC	Temperature controller	
TP	The top pressure of column	
QR/F	Reboiler duty/mole flow rate of F	

components of DWC [9]. Emtir et al. found that there was 30% reduction of the total annual cost of DWC when they studied a ternary mixture system with three different feed compositions [10]. Compared with the conventional structures, DWCs can reduce the cost of capital and energy [11–14]. Kiss and Suszwalak [11] studied an extractive dividing wall column (EDWC) for the purpose of bioethanol dehydration, and the result showed that 9.4% reboiler duty can be saved as compared with conventional extractive distillation process. Zhang et al. studied EDWC simulated as three columns model. They found that EDWC can save more energy than conventional extractive distillation arrangement [14].

Another important aspect of EDWC should be considered is dynamic control. EDWC have interactions and inner structures among control loops which make it much more difficult to control than that of conventional extractive distillation arrangement [13]. Sun et al. used the composition control cascaded with the molar ratio of mixture entrainer to feed and the composition control cascaded with temperature control in the EDWC [4]. Zhang et al. studied the separation of ethyl acetate-isopropyl alcohol system by extractive dividing wall column and the temperature control was cooperated with the composition control [14]. Xia et al. studied separation of methylal-methanol system by extractive dividing wall column and two control schemes were all evaluated [15].

In this study, BEN and CYH azeotrope is separated through extractive distillation. The entrainer is the mixture of SULF and oxylene (OX). Therefore, the temperature of bottom is lower than the decomposition temperature of SULF, and water can be utilized as the cooling medium at the top of the column. In order to save energy, EDWC simulated as a three-column model is studied in this paper. In terms of simulation studies, three-column model is more close to EDWC concept compared with the models simulated as two columns. Total annual cost (TAC) is calculated to obtain the optimal conventional extractive distillation process and EDWC with mixed entrainer. The separation of BEN-CYH using mixed entrainer, which is SULF mixed with OX via EDWC is studied for the first time. Subsequently, two control structures are proposed. There has been no published literature about the detailed control of EDWC with mixed entrainer simulated as a three-column model.

#### 2. Optimization of extractive distillation process

2.1. Thermodynamic model and mixed entrainer screening and selection

In this work, NRTL model is used in the Aspen simulations. BEN and CYH have very similar boiling points (80.13 °C and 80.78 °C), and the BEN/CYH mixture has an azeotrope with composition of 55.01 mol% BEN at atmospheric pressure. In the earlier work [3], BEN–CYH system was separated by extractive distillation using SULF as entrainer. To avoid thermally decomposition, the entrainer recovery column (ERC) was operated at 0.08 atm. However, the top temperature of ERC was 15.7 °C, which was too low to use water as cooling medium. To solve this problem, another lower boiling point component is added into the entrainer so that the temperature in bottom of column can be lower than that of thermally decomposition (200 °C) and meanwhile the top temperature in column is high enough to use water as cooling medium.

O-xylene (OX) has no selectivity for the separation of BEN–CYH system and its boiling point (144.4 °C) is between BEN (80.13 °C) and SULF (287.3 °C). OX had no selectivity for separation of aromatics but can help increase mutual solubility of SUFL and BEN. So, it is used as a cosolvent of SULF in the process and the compatibility is commendable. The concentration of OX in mixed entrainer is important to the effect of separation. Usually the top pressure of the EDWC is designed to be able to use water as the cooling medium. Comparison of the relative volatilities in the presence of different entrainers is a criterion for the entrainer selection [16]. The relative volatility is more higher, the separation process is more easier. Fig. 1 shows the pseudo-binary VLE of BEN/CYH mixture with SULF and OX to feed mole ratio 1.1 by NRTL



**Fig. 1.** Pseudo-binary *x*-*y* plot for the BEN–CYH system with mixed entrainer to feed molar ratio 1.1.

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