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# Comparison of different extractive distillation processes for 2-methoxyethanol/toluene separation: Design and control



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#### ABSTRACT

In this work, the extractive distillation process is proposed for the first time to separate the 2-methoxyethanol/toluene mixture. Three configurations, including the conventional extractive distillation (CED), extractive distillation under reduced pressure (EDRP) and extractive dividing-wall column (EDWC) processes are investigated in order to obtain the optimal separation configurations. Their economic feasibilities are estimated by calculating the total annual cost (TAC). The results show that the EDRP process operating at a relatively low pressure is more economic than that operating at the lowest pressure. The TAC of the EDRP and EDWC processes can be saved by 4.88% and 11.04% respectively compared to the CED process, thus the EDWC sequence is superior to the other two processes in economic benefits. Three control strategies are proposed to investigate the controllability of the EDWC process. The results show that the improved dynamic structure can overcome large feed disturbances in flowrate and composition effectively.

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#### 1. Introduction

2-Methoxyethanol has received a great deal of attention in chemical industry since it is an ether alcohol which has unique physicochemical characteristics (Singh et al., 2013). It is not only used as raw material in organic synthesis but also adopted as solvents (Pla-Franco et al., 2013). In addition, 2-methoxyethanol can be used as anti-microbial additive and diluent as well. Toluene is an important raw material widely applied in the fine-chemical and synthetic material industries, and it is also used as solvents in the chemical industry (Modla, 2013; Wang and Huang, 2011). The derivatives of toluene such as xylene, phenylcarbinol and benzaldehyde are extensively applied to the fuel, pharmaceutical, pesticide production. However, a large volume of toluene and 2methoxyethanol mixed solution may be produced in the dyestuffs, varnishes, printing inks and electrochemical industrial processes, which would form a minimum azeotrope of the 2-Methoxyethanol and toluene (Zhao et al., 2014). Hence it is difficult to effectively separate 2-Methoxyethanol and toluene mixture by ordinary distillation process.

Some special distillation technologies such as pressure-swing distillation (PSD), azeotropic distillation and extractive distillation

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can be used to separate the azeotropic mixtures or close boiling point mixtures (Tabari and Ahmad, 2015; Luyben, 2012; Modla, 2013). Though the PSD process can accomplish without using a third component, it is inefficient for the mixtures whose azeotropic composition is moderately sensitive to pressure. Luyben (2008) compared the extractive distillation and the PSD processes for the separation of the acetone-methanol mixture, and the results show that the PSD system consumes more energy and has a 15% higher total annual cost (TAC). In azeotropic distillation process, the entrainer can generate a new azeotrope with the original one or more components, which can be separated as the distillate, thus the feed mixture can be separated. However, the azeotropic distillation can bring some challenges like multiple steady states and large energy demand (Yu et al., 2015; Zhu et al., 2015). As for extractive distillation process, the introduced entrainer can alter the relative volatility of the azeotropic components, through which the mixture can be separated. It is worth noting that both in the azeotropic distillation and extractive distillation, the use of entrainer has some disadvantages, for example, the third component entrainer may exist in the products since it cannot be completely removed. Moreover, the entrainer recovery is an energy-intensive process that will cause additional cost.

Although it is characterized by a large amount of energy consumption, extractive distillation is still a widely used method in chemical industry. In recent years lots of energy-saving technologies have been proposed and applied to the extractive distillation

#### Nomenclature

Nomenciature					
$A_i$	Heat exchanger areas of i				
$B_2$	Bottom stream of the recovery column				
C1	Extractive column				
C2	Recovery column				
C3	Stripping column				
CED	Conventional extractive distillation				
CS1	Basic control structure				
CS2	Control structure with $Q_R/F$ and $\alpha_V$				
CS3	Improved control structure				
$D_1$	Distillate of the extractive column				
DMF	N,N-dimethyl formamide				
DMSO	Dimethyl sulfoxide				
EDRP	Extractive distillation under reduced pressure				
<b>EDWC</b>	Extractive dividing-wall column				
$N_{\rm F1}$	Feed location of feed mixture				
$N_{\rm F2}$	Feed location of recovery column				
$N_{\rm FS}$	Feed location of entrainer				
NMP	N-methyl-2-pyrrolidone				
$N_{\mathrm{T}i}$	the number of stages in column <i>i</i>				
NRTL	Nonrandom two liquid				
$P_{C1}$	Operating pressure of extractive column				
PSD	Pressure-swing distillation				
$Q_{\rm R}$	Reboiler duty of the EDWC				
$Q_{R1}$	Reboiler duty of the extractive column				
$Q_{R2}$	Reboiler duty of the recovery column				
$Q_R/F$	Ratio of reboiler duty to feed flowrate				
RCM	Residue curve map				
$RR_i$	Reflux ratio of column i				
S	Entrainer flowrate				
$T_4$	Temperature on stage 4 of stripping column				
$T_7$	Temperature on stage 7 of recovery column				
$T_{34}$	Temperature on stage 34 of extractive column				
TAC	Total annual cost				
$T_{\rm RC1}$	Reboiler temperature in extractive column				
$\alpha_{v}$	Vapor split ratio				

(An et al., 2015; Anokhina and Timoshenko, 2015; Chen et al., 2015). When a heavy entrainer with high boiling point is adopted in the extractive distillation system, it usually requires high quality energy as the heat source. When operating the extractive distillation under vacuum pressure, the boiling points of the components will decrease, thus the low-grade steam can be adopted instead of the high-grade steam. Qin et al. (2013) studied the extractive distillation process for the separation of benzene-cyclohexane mixture, and they operated the entrainer recovery column at 0.08 atm to reduce the base temperature. You et al. (2015a, 2015b, 2016) investigated several extractive distillation configurations for the separation of acetone-methanol mixture, and the extractive distillation columns were all operated at 0.6 atm to reduce the operating cost. In their work, lower pressure means less entrainer required in the extractive distillation system. However, an unusual phenomenon occurs in the separation case of 2-methoxyethanol and toluene mixture using Dimethyl sulfoxide (DMSO) as entrainer, and thus it is valuable to study the extractive distillation process at low pressure.

Nowadays extractive dividing-wall column (EDWC) has drawn much attention in the azeotropic mixture separation processes (Dai et al., 2016; Sun et al., 2014; Zhang et al., 2014). However, according to a recent report by Wu et al. (2013), EDWC sequence may have energy savings but with adversely increasing the total steam cost compared to the conventional extractive distillation (CED) process.

**Table 1**Correlation parameters for the NRTL model<sup>a</sup> in toluene (1) + 2-methoxyethanol (2) + DMSO (3) ternary system.

System i+j	Interaction parameters			
	a <sub>ij</sub>	a <sub>ji</sub>	b <sub>ij</sub>	b <sub>ji</sub>
1+2	-8.9660	1.6576	3850.0675	-495.9146
2+3	-0.8504	-0.4775	126.5416	214.3344
1+3	7.5944	2.8612	-2331.0058	-1300.7055

<sup>&</sup>lt;sup>a</sup> NRTL model:  $\tau_{ii} = a_{ii} + b_{ii}/T$ , the value of  $c_{ii}$  was fixed at 0.3 for the binary systems.

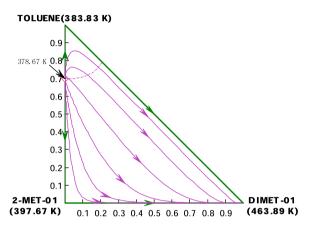


Fig. 1. Residue curve map for 2-methoxyethanol/toluene/DMSO system at 1 atm.

Therefore, the EDWC cannot guarantee a simultaneous reduction on both energy requirement and TAC. The economic benefits should be carefully checked when the EDWC scheme is applied to different system.

To the best of our knowledge, the simulation studies on the extractive distillation of 2-methoxyethanol and toluene mixture are not reported in the open literature. The purpose of this work is to investigate the design and control of extractive distillation for the separation of 2-Methoxyethanol and toluene mixture. In present work, two energy-efficient extractive distillation processes, including the extractive distillation under reduced pressure (EDRP) and EDWC configurations, are developed based on the conventional process. The optimization of sequential iterative search is carried out to achieve the minimum TAC. The EDRP process shows an interesting phenomenon that there exists an economic design with the column operating at a relatively low pressure rather than the lowest pressure. The EDWC configuration is superior to the EDRP one in economic benefits. In addition, the dynamic controllability of the EDWC process is examined on the feed disturbances. Finally, some conclusions are drawn for the proposed processes.

#### 2. Steady-state design of extractive distillation process

#### 2.1. Flowsheet of the CED

DMSO is a kind of promising solvent in chemical industrial applications, which is a less harmful solvent than the aprotic highly polar solvent *N*-methyl-2-pyrrolidone (NMP) and *N*,*N*-dimethyl formamide (DMF) (Chen et al., 2005). Zhao et al. (2014) studied the vapor-liquid equilibrium for the ternary system of 2-methoxyethanol/toluene/DMSO at 1 atm, which provides the fundamental data for the mixture separation. Table 1 presents the correlation parameters reported by Zhao et al. for the nonrandom two liquid (NRTL) thermodynamic model, which fits the ternary system well with minor deviations (Zhao et al., 2014). Therefore, in this work the NRTL model is used to calculate the physical properties of this ternary system. Fig. 1 displays the corresponding

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