



## Separation of ethylbenzene and *p*-xylene using extractive distillation with *p*-dinitrobenzene



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### ABSTRACT

Though *p*-xylene derived from naphtha reformat is in high demand as a raw material in the production of polyesters and polyethylene terephthalates, its separation from the FCC reformat is not easy due to close boiling points among the components in the reformat. When a proper solvent for the *p*-xylene separation is available, an extractive distillation can be applied in the separation. Currently the separation utilizes an adsorption process requiring a large amount of costly adsorbent and a desorption process followed by distillation. In the proposed extractive distillation here, *p*-dinitrobenzene has been utilized to improve the separation of *p*-xylene from its ethylbenzene mixture. The VLE data of the ternary mixture estimated with the UNIFAC were compared to those computed from molecular simulation to show their reliability. An extractive distillation and solvent recovery were applied to obtain 99.6% purity *p*-xylene with 99.1% recovery. The economics of the proposed process demonstrates its comparability to the existing Parex process.

### 1. Introduction

Xylene isomers and ethylbenzene are coexisting components in naphtha reformat, and they are separated by distillation as a group. Before the separation, the C8 compounds are in the aromatic compounds mixture, including benzene and toluene, contained in the extracts of extractive distillation using sulfolane [1]. Further distillation splits the C8 components of close boiling points from others. Among the mixture *p*-xylene has high demand to produce terephthalic acid, a raw material in the production of polyesters and polyethylene terephthalates (PETs). The separation processes of *p*-xylene based on solvent extraction [2] or crystallization [3,4] were not efficient with only 60–65% recovery rate. The extractive distillation of ethylbenzene and *p*-xylene has been studied for many years [2,5,6–9], but no commercial process was developed due to no effective extractant found. Recently searching effective extractants extended to ionic liquid solvents in many extractive distillation processes [10–12].

In 1971, the Universal Oil Products (UOP) commercialized a new separation method of *p*-xylene, an adsorption process, called the Parex process with a 95% recovery rate [13,14]. Though the adsorption process is largely effective and efficient in the separation of a specific component from a mixture with high recovery and high purity, it requires a large volume of high-cost adsorbents. Moreover the next stage of the adsorption includes a desorption process to recover the adsorbate

from the adsorbent using a desorbent [15]. The recovery and recycling of the desorbent accommodate a distillation column separating the targeted *p*-xylene. A continuous processing has been desired for a large throughput of chemical production, and distillation and extraction are suitable for the processes. On the other hand, because the adsorption was a batch process, a simulated moving bed (SMB) operation [16], a quasi-continuous process, requiring multiple beds of adsorbents was introduced to the Parex process [13,17]. While one of the beds releases the adsorbate, the product, other three beds are preparing for the next production in sequence. Consequently, the SMB operation necessitates a large volume of high-cost adsorbents [18].

In this study, an efficient solvent, *p*-dinitrobenzene, for the separation of *p*-xylene from the mixture of ethylbenzene and *p*-xylene, two main components in the naphtha reformat, is suggested, and the separation process of *p*-xylene is designed for 99.1% recovery and 99.6% purity of *p*-xylene using an extractive distillation. The design procedure is explained to present the performance of the proposed separation process. The estimated VLE data of ternary mixture of ethylbenzene, *p*-xylene and *p*-dinitrobenzene used in the process design are probed by comparing to the computed ones with molecular simulation. The separation performance and economics of the proposed process are measured to the Parex process.

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Nomenclature		$U$	potential ( $\text{J mol}^{-1}$ )
$A$	probability (–)	$V$	volume ( $\text{m}^3$ )
$N$	number of particles (–)	Greek	
$p$	pressure (Pa)	$\beta$	scaling factor ( $\text{mol J}^{-1}$ )
$Q$	canonical partition function (–)	$\Lambda$	de Broglie wavelength (m)
$\mathbf{r}$	position vector (–)		
$T$	temperature (K)		

## 2. Process overview

Ethylbenzene and *p*-xylene are isomers having close boiling points, difficult to be separated by common distillation. To assist the separation of the mixture, *p*-dinitrobenzene was added to the distillation as an extractant separated from the product and recycled in sequence as shown in Fig. 1. The extractive distillation used here is the same as applied in the conventional processes. The homogeneous solvent prevents from applying an extraction in the process rather than the distillation. The solvent introduced in the extractive distillation was recovered at the solvent recovery column after separating the product. A small amount of ethylbenzene accompanying solvent recycles to the extractive distillation column to be endlessly accumulated. Therefore, a purge and a make-up stream were attached in the process streams.

The process proposed here was designed with the commercial software HYSYS using the thermodynamic model NRTL for vapor–liquid equilibrium (VLE). The program was developed for chemical process simulation using the rigorous models of material and energy balances, thermodynamic equilibrium equations, kinetic equations of chemical reactions, and various constituent equations of physical and thermodynamic properties [27]. The simulation results of chemical process equipment show the product flow rate and specification for a given set of feeds. Therefore, providing various sets of equipment size, feed information, and operating condition yields the information of products. When the specification and flow rates of products are as desired, the used equipment size and operating condition in the computation are the variables of equipment in the process design.

Because no experimental binary VLE data with *p*-dinitrobenzene were available, the NRTL parameters were calculated with the group contribution technique UNIFAC. However, the technique considers the type and number of the comprising groups in a molecule for the

parameter estimation. In other words, the UNIFAC does not distinguish the difference among the location of nitro groups at the dinitrobenzene molecules, such as ortho, meta and para locations. The difference is counted, when the fugacity applied to the computation of vapor pressure is computed. For the examination of the parameters used in the HYSYS design, a molecular simulation was applied to the computation of equilibrium VLE compositions and the results were compared with the HYSYS compositions. The vapor–liquid equilibrium computed by the HYSYS has been widely used in the chemical process design in field applications. The computed VLE data by molecular simulation were not accurate enough to be applied in the process design, but the comparison indicated that the selection of *p*-dinitrobenzene for the solvent of *p*-xylene separation considering the principle of molecular simulation was acceptable.

## 3. Molecular simulation

By positioning molecules in a box at their optimized coordinates, the total potential energy of the molecules becomes the minimum in equilibrium. The Monte Carlo simulation places the molecules toward the minimum energy in statistical way. In the canonical ensemble, the partition function is given by [19]

$$Q(N, V, T) = \frac{1}{\Lambda^{2NN!}} \int e^{-\beta U(\mathbf{r})} d\mathbf{r} \quad (1)$$

where  $U$  is the potential of molecules at a position vector  $\mathbf{r}$ , and the integration ranges at the number of molecules  $N$ . The potential includes bonded and intermolecular unbonded forces such as the Lennard-Jones potential. The de Broglie wavelength  $\Lambda$  is the length at which intermolecular interactions are neglected [19]. When the integrand in Eq. (1) is proportional to the probability of the desired configuration  $\mathbf{r}$  of all the molecules, the average probability is found as [19]

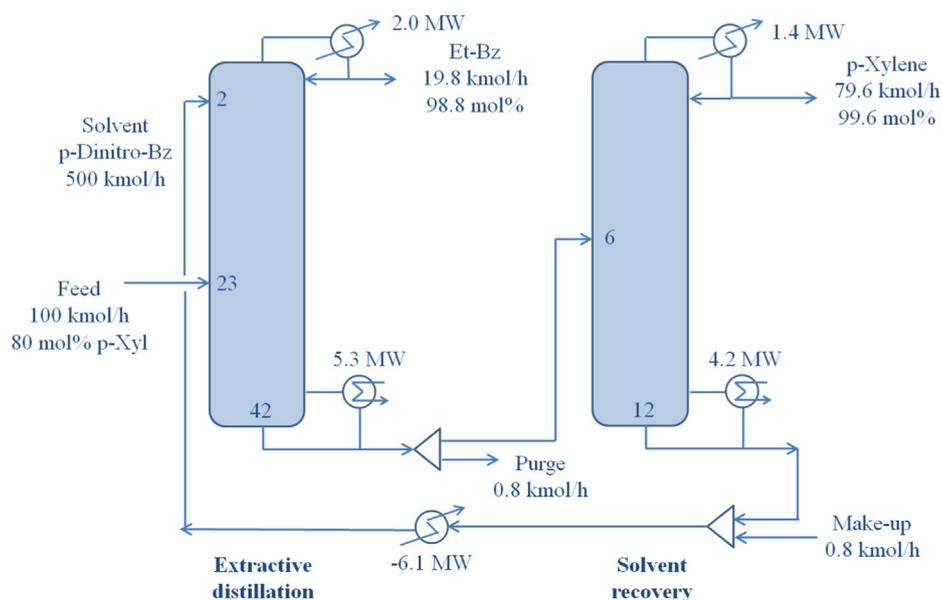


Fig. 1. Schematic diagram of the proposed extractive distillation process.

$$\langle A(\mathbf{r}) \rangle = \frac{\int A(\mathbf{r}) e^{-\beta U(\mathbf{r})} d\mathbf{r}}{\int e^{-\beta U(\mathbf{r})} d\mathbf{r}} \quad (2)$$

In the Monte Carlo simulation, the positions of molecules are not deterministically calculated but stochastically sought, where a random number is generated to find the new position of each molecule. The newly computed potential at the position gives the average probability in Eq. (2). Here the Metropolis criterion [20] is applied to determine if the newly suggested positions of molecules are accepted.

In this study the Monte Carlo simulation in the NVT ensemble was conducted using the open program RASPA [21,22] that includes the procedures explained above. The molecular structure and bond and bend stretches were from the transferable potentials for phase equilibria (TraPPE) potentials [23]. The box size in liquid phase was 40 Å cubic, and that of vapor was 150 Å. The simulation parameters are listed in Table 1. The input data in the simulation were referenced to the result of the HYSYS VLE simulation for fast settlement at equilibrium. The numbers of molecules were about 250 and 50 for liquid and vapor, respectively. The numbers were adjusted for the density calculated with the HYSYS. The VLE compositions computed from the molecular simulation were compared to those of the HYSYS calculation.

#### 4. Process design

The designs of extraction and distillation utilize the equilibrium-stage design procedures, and the processes in this study do not accommodate any energy-saving feature, such as divided wall column or internal heat integration. Therefore, the columns design has followed a common procedure using the commercial program HYSYS. The thermodynamic equilibrium model was the NRTL model, because no experimental VLE data were available to estimate the model parameters for other thermodynamic model. The parameters of the NRTL model can be computed using the UNIFAC estimation.

A well-known extraction process, a sulfolane process separating aromatic compounds from naphtha reformat provides the solvent at a rate of 3 times feed flow rate [1]. An experimental study of extractive distillation of ethylbenzene and *p*-xylene suggested 6 times the flow rate of solvents gives the best performance [7]. In this study the solvent was supplied at a rate of 5 times the feed rate in the design of extractive distillation column. The number of trays in the extractive distillation is inversely proportional to reflux flow rate. While the former increases the investment cost, the latter raises the operating cost. For the optimum of the total annual cost (TAC), an optimization procedure, differential evolution method [24], was used to determine the number of trays at the extractive distillation. The number of trays and reflux ratio are inversely proportional variables determining the cost in the design of distillation columns. While the former is related to investment, the latter is to operating cost. Minimizing the TAC gives the optimal design of a distillation column. The TAC comprises the annual investment cost, total investment divided by the payback time of the investment, and annual operating cost, mostly steam, cooling water and electricity. The cost equations are listed in [25,26], and the cost index, Chemical Engineering Plant Cost Index (CEPCI) of 2016, 541.7, was used here. Payback time of the investment was 5 years, and annual operation day was 330. The parameters used in the differential evolution are listed in Table 2. The same procedure was applied to the design of solvent recovery column. After the column tray number and reflux flow were calculated, the number of feed location was iteratively adjusted for the minimum reflux. The design results are listed in Table 3.

#### 5. Results and discussion

For the separation of ethylbenzene and *p*-xylene consisted of close boiling compounds, an effective solvent was proposed and its separation performance and economics were examined below.

##### 5.1. Solvent selection and thermodynamic parameters

There were many studies of extractive distillation, but none of the proposed solvent worked as efficient as *p*-dinitrobenzene. Berg [6] introduced methyl formate, *n*-butanol and *c*-pentanol as solvents in the extractive distillation. He has published the experimental data of ethylbenzene separation using polychloro-organic compounds as solvents [2]. A variety of solvents were experimentally examined for possible utilization as solvents in the extractive distillation [7]. These previously introduced solvents were not efficient as *p*-dinitrobenzene proposed here. Applying large amount of reflux flow in a tall distillation column separates the close boiling mixtures, which cannot be commercialized due to high operating cost with large investment. Various solvents introduced in the previous studies were evaluated by comparing their separation factors, but no satisfactory separation was found from the study.

Searching a new solvent using the HYSYS simulation with the assistance of the molecular simulation led to *p*-dinitrobenzene being the most efficient. Molecular simulation optimizes the placement of molecules in a cell by minimizing the sum of intermolecular potential among molecules. When a similar molecular structure to *p*-xylene is used as a solvent, their intermolecular potential becomes lower than with the solvent of non-similar molecules because the similarity of molecular structure makes the potential small. Note that the intermolecular potential is computed by adding the potentials with the constituent atoms in paired molecules. Therefore, molecules having two radicals in para position with benzene structure were selected in this study. After comparing boiling points of *p*-xylene and the potential solvents for the easy separation of solvent later on, *p*-dinitrobenzene was selected finally.

Because the experimental vapor–liquid equilibrium (VLE) data of ethylbenzene/*p*-xylene/*p*-dinitrobenzene system are not available, the data computed in the HYSYS thermodynamics were compared to the results of the molecular simulation explained in the above. Fig. 2 demonstrates their comparison. The compositions of vapor and liquid are connected with lines for 4 different sets of mixtures. The dotted lines are of HYSYS computation, while the dash-dot lines are of molecular simulation. The average error in absolute values between the computed values was 0.036. The average error was computed from the total difference in absolute values between the estimated compositions by the HYSYS and molecular simulation in vapor and liquid phases, respectively, divided by the number of difference counts. When the numbers of molecules, 250 and 50 in liquid and vapor phases, respectively, are considered, the error is not significant. Though no experimental VLE data of the ternary system of ethylbenzene/*p*-xylene/*p*-dinitrobenzene are compared to the HYSYS VLE calculation using the NRTL parameters derived from the UNIFAC estimation, the comparison demonstrated in Fig. 2 indicates the estimated NRTL parameters are relatively reliable for the process design of the proposed *p*-xylene separation system.

##### 5.2. Design results

The proposed extractive distillation process was designed using a

**Table 1**  
Parameters in molecular simulations.

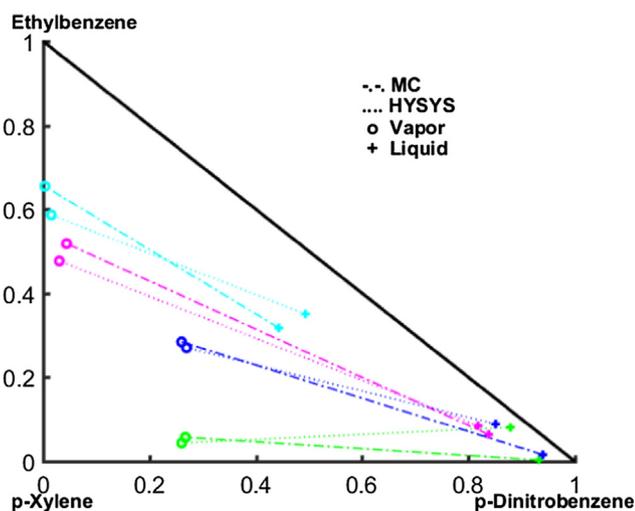
Parameter	Value
Simulation method	Monte Carlo
Initialization cycles	3000
Computation cycles	10,000
Charge method	Coulomb smoothed
Box size liquid (angstrom)	40
Box size vapor (angstrom)	150
Gibbs volume change probability	0
Gibbs swap probability	0.001

**Table 2**  
Parameters in the differential evolution optimization.

Parameter	Value
Mutation factor	0.001
Crossover ratio	0.5
Population size	30
Maximum iteration	30
Bounds in tray number, extractive	32–60
Bounds in tray number, recovery	6–16

**Table 3**  
Structural information and operating conditions in the proposed process. Tray numbers are counted from the top.

Variable	Extractive distillation	Recovery
<i>Structural</i>		
Tray No.	42	12
Feed	23	6
Solvent	2	
<i>Operating</i>		
Pressure (MPa)- top	0.12	0.12
<i>Temperature (°C)</i>		
Overhead	143	150
Bottom	188	310
Feed (kmol/h)	100	580
Solvent (kmol/h)	500	
Make-up/Purge (kmol/h)	0.8	0.8
<i>Product (kmol/h)</i>		
Overhead	20	80
Bottom	580	499
Reflux (kmol/h)	184	61
Vap. boilup (kmol/h)	255	248
Cooling duty (MW)	2.0/6.1	1.4
Reboiler duty (MW)	5.3	4.2
<i>Comp. (mole frac.)</i>		
Feed		
p-Xylene	0.8	0.138
Product		
Ovhd.		Ovhd.
p-Xylene	0.012	0.996



**Fig. 2.** Comparison of vapor liquid compositions computed by molecular simulation and HYSYS. Different colors indicate difference in mixture compositions. Dotted lines connect the vapor and liquid compositions in equilibrium computed by HYSYS, and dashed dot lines do those by molecular simulation.

commercial design program HYSYS [27]. Because varying pressure does not improve the separation, the column pressure at the top trays was set at 120 kPa. The number of trays was determined using the

differential evolution method [24] for the minimum total annual cost consisted of investment and operating cost. The locations of feed and solvent were adjusted, after the number of trays and reflux flow rate were set. The amount of solvent was also adjusted for the product specification. Table 3 lists the design results of the structural and operating variables of the extractive distillation along with product specification. Because no phase separation in liquid was observed in the ternary system, the distillation simulation was not complex as in the design of an azeotropic distillation having 3-phase separation. The contamination of a small amount of ethylbenzene in the extract stream accumulates the ethylbenzene at the recycled solvent. A purge from the bottom product of the extractive distillation column removes the ethylbenzene, and the make-up of solvent replaces the solvent loss by the purge. The recovery of *p*-xylene was 99.1% at a purity of 99.6%. The performance of production is comparable to the Parex process that has 89.5% recovery at a purity of 99.7% [17]. The purity is assumed at the total removal of desorbent, *p*-Diethylbenzene, from the effluent product of *p*-xylene at the SMB adsorption column. The adsorption was applied in the separation of *p*-xylene, because an efficient solvent of extractive distillation was not available for the distillation. The performance of *p*-dinitrobenzene in this study indicates that a new distillation process can be used for the separation of *p*-xylene.

### 5.3. Economic evaluation

The investment cost of the proposed *p*-xylene separation process includes two columns cost composed of column shell, tray and heat exchangers. Table 4 summarizes the investment and utility costs for the extractive distillation column and solvent recovery column. The production of *p*-xylene is equivalent to 67,000 tons per year. The investment of 3.1 million US dollars of the proposed process is comparable to the Parex process. The investment cost of 135 million US dollars (adjusted at 2016 price) for the Parex process at a 99.7% *p*-xylene production of 700,000 tons per year [28], while the Parex process processes the mixtures of three xylene isomers and ethylbenzene mostly. The purity and recovery of the Parex process are 99.7% and 89.5%, respectively [17]. When the production is increased for the same capacity as in the Parex process, the investment is equivalent to 32 million US dollars for the purity and recovery of 99.6% and 99.1%, respectively, in this study. The Parex process consumes 20 ton/hr of medium pressure steam, while the proposed process does 4.6 ton/hr (deduced amount of the recovered heat at the cooler of recycled solvent) for about one tenth of *p*-xylene production. Because no adsorbent, expensive and large amount necessary, is utilized in the distillation, the utility cost does not discourage the application of distillation process in the separation of *p*-xylene.

## 6. Conclusions

An extractive distillation process for the separation of ethylbenzene

**Table 4**  
Economic evaluation of the proposed separation process. Units are in million U.S. dollars, and the utility cost is per annum.

Variable	Extractive distillation	Recovery
<i>Investment</i>		
Column	0.262	0.062
Tray	0.110	0.019
Condenser	0.357	0.193
Reboiler	1.353	0.788
Total		3.144
<i>Utility</i>		
Coolant	0.070	0.020
Steam	2.456	1.621
Total		4.167

and *p*-xylene was developed, and its design procedure was presented here. The solvent used in the process has similar structure of *p*-xylene with higher boiling point. The molecular simulation in the ternary system of ethylbenzene/*p*-xylene/*p*-dinitrobenzene indicates that the solvent improves the separation factor between ethylbenzene and *p*-xylene. The design results of the extractive distillation and solvent recovery columns are presented, and the economics of the proposed process was briefly compared to that of the Parex process. The process design demonstrated the feasibility of distillation separation of ethylbenzene and *p*-xylene with 99.1% recovery and 99.6% purity of *p*-xylene. The economic evaluation also exhibits the distillation process is comparable to the adsorption process, a current commercial process. The VLE computation in a ternary system using the molecular simulation was applied to the examination of the NRTL thermodynamic model for the ternary system.

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