

MODELING AND SIMULATION OF A BENZENE RECOVERY PROCESS BY EXTRACTIVE DISTILLATION

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Abstract - Extractive distillation processes with N-formylmorpholine (NFM) are used industrially to separate benzene from six carbon non-aromatics. In the process studied in this work, the stream of interest consists of nearly 20 different hydrocarbons. A new set of NRTL parameters was correlated based on literature experimental data. Both vapor-liquid equilibrium as well as infinite dilution activity coefficient data were taken into account; missing parameters were estimated with the UNIFAC group contribution model. The extractive distillation process was simulated using ASPEN Plus[®]. Very good agreement with plant data was obtained. The influences of the main operational parameters, solvent to feed ratio and solvent temperature, were studied. Theoretical optimum operating values were obtained and can be implemented to improve the industrial process. Extreme static sensitivity with respect to reboiler heat was observed, indicating that this can be the source of instabilities.

Keywords: Extractive distillation; N-formylmorpholine; NFM; Process simulation; Benzene.

INTRODUCTION

Benzene has being widely used in the chemical industry as a raw material for innumerable compounds. Due to its high toxicity, it is being replaced by other hydrocarbons with similar characteristics. This movement is also driven by stricter regulations. For instance, in Brazil, the commercialization of finished products containing more than 0.1% benzene by volume in their composition is not permitted. However, benzene is still widely employed in, for example, styrene polymers; phenol for resins and adhesives (via cumene); and cyclohexane, used in nylon manufacture.

This motivates the petrochemical industry to better understand and control high purity separation processes. Due to the similar boiling point of aromatics

and non-aromatics with the same carbon number, conventional distillation processes are impractical and extractive distillation processes become economically interesting.

According to Emmrich *et al.* (2001), Krupp Uhde originally developed the Morphylane process (extractive distillation process) for the recovery of high-purity benzene from hydro-refined coke oven benzole, the reason being that the existing liquid-liquid methods of extraction were unable to process coke oven benzole due to its high aromatic content. The liquid-liquid extraction techniques exploit the different solubilities of aromatics and non-aromatics in a polar solvent.

Different solvents can be used for the separation of aromatics from non-aromatics (such as sulfolane, N-methylpyrrolidone, diethylene glycol, etc.). In the

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process studied in this paper, NFM (N-formylmorpholine, shown in Figure 1) is used to accomplish the separation of benzene from a stream containing other hydrocarbons, mainly with six carbons.

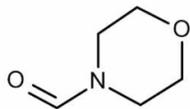


Figure 1: NFM molecule (N-formylmorpholine).

According to ZHIGANG *et al.* (2005), the addition of polar solvents to hydrocarbon mixtures results in increased volatilities of paraffins relative to naphthenes, olefins, diolefins and alkynes, and in increased volatilities of naphthenes relative to aromatics. Therefore, the addition of a polar solvent reduces the effort in the separation by distillation of certain mixtures which otherwise would be impractical. Thus, among other desired characteristics, its polarity and its high boiling point make NFM a good solvent for the required separation.

Figure 2 shows a schematic drawing of the unit studied.

The C6C8 stream from the pyrolysis gasoline hydrogenation process is first fractionated into C6 and C7+ cuts in the distillation column (T01). The C6 cut is sent to the extractive distillation column (T02). In this column, NFM is added as the entrainer to extract benzene, exiting at the bottom of the column. The overhead stream contains C6 non-aromatics (overhead product). The bottom stream is fed into a strip-

per column (T03) where the separation of benzene, at the top, from the solvent, at the bottom, occurs.

The column studied, T02, frequently does not specify the desired products with the desired stability. It should simultaneously specify both products: the overhead product (non-aromatic C6) and the bottom product (benzene). Therefore, the main goal of the work was to develop a model which agrees with plant data. A new set of NRTL parameters was estimated based on experimental data available in the literature. The insight obtained from this model will hopefully provide the directions for a more stable operation.

THERMODYNAMIC MODEL

Since extractive distillation processes are strongly affected by the non-ideal interactions between the entrainer and the stream of interest, one of the main difficulties when simulating these processes is the correct thermodynamic representation of the mixture behavior in contact with the solvent. Thus, most of the work was devoted to the thermodynamic modeling. First, a literature survey for phase equilibrium and infinite dilution activity coefficient (IDAC) data with NFM and the substances involved was accomplished. Information for mixtures with NFM and the following compounds was found in the literature: cyclopentane (CP), n-hexane (n-HX), methylcyclopentane (MCP), benzene (BZ), cyclohexane (CH), n-heptane (n-HP) and methylcyclohexane (MCH). Table 1 shows a summary of the collected information.

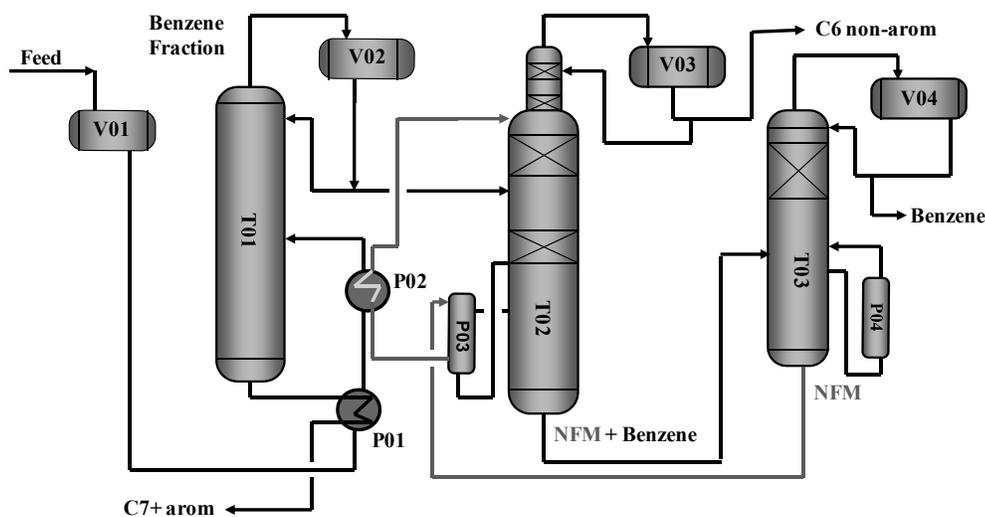


Figure 2: Benzene extractive distillation with a NFM recovery unit, schematic drawing.

Table 1: Available binary mixture data of the components of interest with NFM,

Component / NFM	Liquid-liquid equilibrium	Vapor-liquid equilibrium	Inf. Dil. Activity Coef.
CP	Ko <i>et al.</i> , 2003		DDBST
n-HX	Cincotti <i>et al.</i> , 1999; Ko <i>et al.</i> , 2002 (b)		DDBST; Krummen & Gmehling, 2004
MCP	Ko <i>et al.</i> , 2003		Knoop <i>et al.</i> , 1989 and Weidlich <i>et al.</i> , 1987
BZ		DDBST and Yin, 2010	DDBST
CH	Ko <i>et al.</i> , 2003		DDBST
n-HP	Cincotti <i>et al.</i> , 1999; Qattan <i>et al.</i> , 1995;		DDBST; Krummen & Gmehling, 2004
MCH	Ko <i>et al.</i> , 2003	DDBST	DDBST

From this information the parameters for the NRTL (Non-random two-liquid) model were estimated and the resulting correlation compared with experimental data for different temperatures. The choice of this model was based on its ability to represent deviations from ideality at low pressure and because it was already used successfully in similar works, like Ko *et al.*, 2002 (a), Zhu *et al.*, 2007 e Dongchu *et al.*, 2007.

The NRTL model variant used in this paper is given by:

$$\ln(y_i) = \frac{\sum_j x_j \tau_{ji} G_{ji}}{\sum_k x_k G_{ki}} + \sum_j \frac{x_j G_{ij}}{\sum_k x_k G_{kj}} \left(\frac{\sum_m x_m \tau_{mj} G_{mj}}{\sum_k x_k G_{kj}} \right) \quad (1)$$

with the other quantities given by:

$$G_{ij} = \exp(-\alpha_{ij} \tau_{ij}) \quad (2)$$

$$\tau_{ij} = a_{ij} + \frac{b_{ij}}{T} + e_{ij} \ln T + f_{ij} T \quad (3)$$

$$\alpha_{ij} = c_{ij} + d_{ij}(T - 273.15K) \quad (4)$$

For components with vapor-liquid equilibrium (VLE) and/or liquid-liquid equilibrium (LLE) data available in the literature (compounds showed in Table 1), the NRTL parameter regression was accomplished using this equilibrium information, performed by the built-in data regression functionality of Aspen Plus[®]. For those same components Aspen Plus[®] was used in order to obtain estimated NRTL parameters using IDAC data from the literature. Thus, two different groups of NRTL binary interaction parameters for each component with NFM were estimated, one based on phase equilibrium data (referred as VLE and/or LLE regression) and the other based on IDAC (referred as IDAC regression). Further, the NRTL parametrizations from KO *et al.*, 2002 (a) and ZHU *et al.*, 2008 were also compared with the collected experimental data.

Ko *et al.* (2002 a) was the only reference found

that used existing vapor-liquid equilibrium data to build the thermodynamic model with the NRTL equation and simulate the system with Aspen Plus[®]. The author used the NRTL equation and set binary parameters for NFM for the following pairs: pentane, hexane, heptane, octane, cyclopentane, cyclohexane, methylcyclopentane, methylcyclohexane, dimethylcyclohexane, benzene, toluene and ethylbenzene. In the study it was concluded that the NRTL equation was efficient to simulate the extractive distillation with NFM.

In their work, Ghaee *et al.* (2008) used the model proposed by Ko *et al.* (2002 a) to build a dynamic simulation of an extractive distillation column for benzene. On the other hand, Zhu *et al.* (2008) predicted vapor-liquid equilibrium data of NFM with hydrocarbons with an improved UNIFAC group contribution method modified by Dortmund. They also found their model to be in agreement with real industrial operation data.

Table 2 shows the availability of binary mixture parameters for each component with NFM for the different models.

Table 2: Binary mixture parameter availability for the different models.

Component/ NFM	VLE and/ or LLE regression	IDAC regression	Ko <i>et al.</i> , 2002 (a)	Zhu <i>et al.</i> , 2008
CP	X	X	X	X
n-HX	X	X	X	X
MCP	X	X		X
BZ	X	X	X	X
CH	X	X	X	X
n-HP	X	X	X	X
MCH	X	X		X

Since the non-aromatic compounds are present in low concentration in the NFM phase, the agreement with experimental data for the activity coefficients at infinite dilution is preferred. Thus, the IDAC regression parameters were used to assemble the model proposed in this paper for NFM/non-aromatics. For benzene, which is found in higher concentration in

the NFM-rich liquid phase, the selected model was the one estimated from experimental vapor-liquid equilibrium data.

As expected, in all cases, the model that best fit the experimental IDAC values was the one estimated with the same IDAC data. Figures 3, 4 and 5 show the model verification with IDAC data for the interaction of n-heptane, methylcyclohexane and benzene with NFM, respectively. They were built by comparing the experimental IDAC values for different temperatures with the ones calculated by the NRTL equation using the available binary parameters of each model. Thus, the closer the points are to the line, the better that model represents the experimental data.

Although, naturally, the model estimated from IDAC experimental values is superior when compared with the same IDAC data, for benzene the parameters obtained from vapor-liquid equilibrium data were selected for the simulation. As mentioned before, this is mainly because benzene is far from infinite dilution in the process studied. In this situation, the model built from the vapor-liquid equilibrium experimental data was assumed to be more appropriate for the final application. The VLE results for this mixture can be seen in Figure 6.

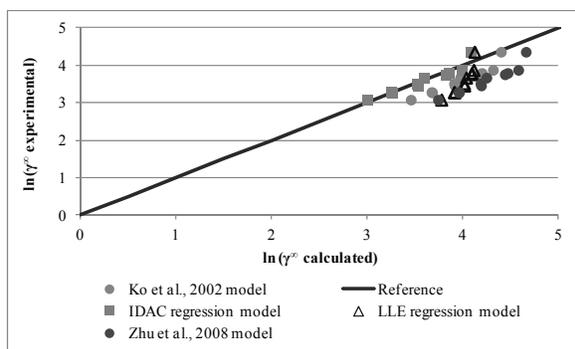


Figure 3: Model verification with IDAC experimental data for the interaction of n-heptane with NFM.

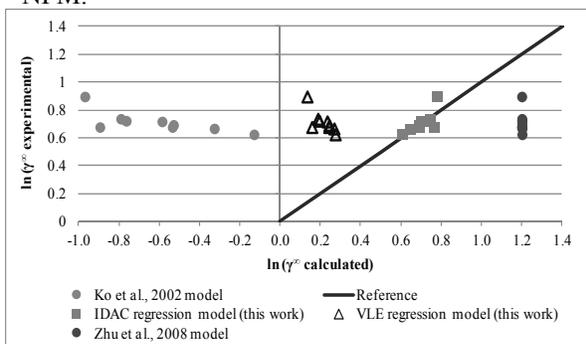


Figure 5: Model verification with IDAC experimental data for the interaction of benzene with NFM.

The overall performance of the model developed in this work, compared with experimental IDAC data, is shown in Figure 7, while the resulting binary parameter values are given in Table 3.

Table 3 presents the results of chosen the model for the pairs of components with experimental data. For benzene the following parameters were also considered: $d_{ij} = -0.11$, $e_{ij} = -2.42$ e $e_{ji} = 1.666$.

The UNIFAC model was used to estimate NRTL binary parameters for the components without equilibrium data available with the solvent NFM. These compounds are: 2,3-dimethylbutane (2,3DBM), 2-methylpentane (2MP), 3-methylpentane (3MP), 2,4-dimethylpentane (2,4DMP), 2-methylhexane (2MH), 2,3-dimethylpentane (2,3DMP), 1,1-dimethylcyclopentane (1,1DMCP), 3-methylhexane (3MH), cis-1,3-dimethylcyclopentane (c1,3DMCP), trans-1,3-dimethylcyclopentane (t1,3DMCP) and trans-1,2-dimethylcyclopentane (t1,2DMCP). According to the laboratory analyses, they represent about 6wt% of a typical feed for the process under study. In order to increase the reliability of the estimated parameters, the new UNIFAC model interaction parameters for $-CH_2$ and ACH groups with NFM, proposed by Zhao *et al.* (2001) were used. The results obtained from UNIFAC are shown in Table 4.

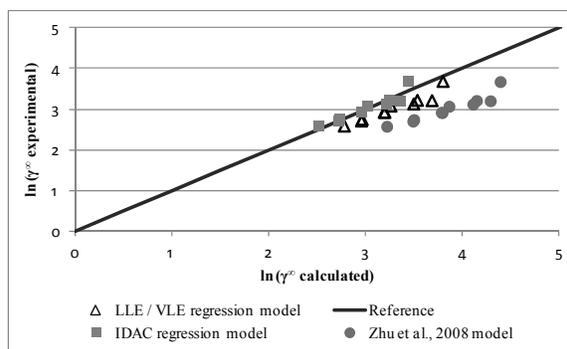


Figure 4: Model verification with IDAC experimental data for the interaction of methylcyclohexane with NFM.

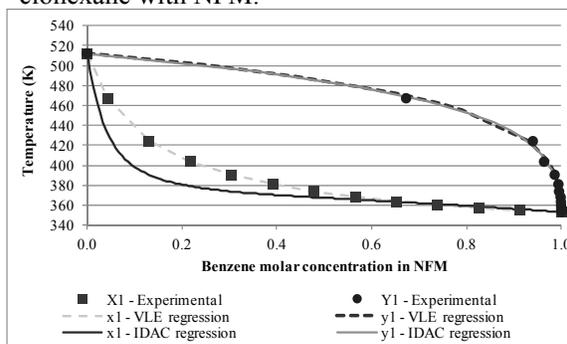


Figure 6: Comparison of the model generated from VLE data and from IDAC data for the benzene/NFM mixture.

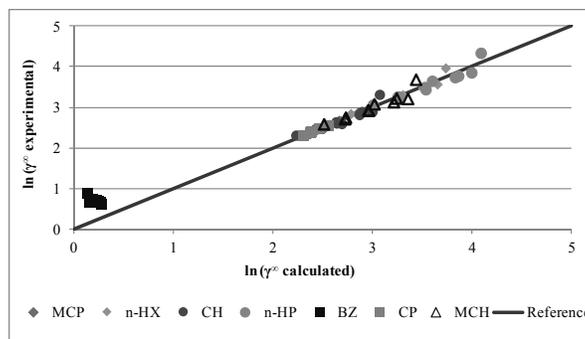


Figure 7: Comparison between gamma infinity data calculated by the model with experimental data.

Table 3: NRTL binary interaction parameters adjusted in this work.

Comp. i Comp. j	CP NFM	n-HX NFM	MCP NFM	BZ NFM	CH NFM	n-HP NFM	MCH NFM
a_{ij}	0.791	0.16	0.543	17.09	0.827	0.069	0.501
a_{ji}	-1.51	-2.13	-1.35	-12.3	-2.29	-2.39	-2.32
b_{ij}	891.5	1006	937.0	-1000	847.9	1036	922.8
b_{ji}	863.3	1380	932.1	952.7	1235	1563	1350
c_{ij}	0.300	0.300	0.300	0.300	0.300	0.300	0.300

Table 4: NRTL binary parameters estimated by UNIFAC.

Comp. i Comp. j	2,3DMB NFM	2MP NFM	3MP NFM	2,4DMP NFM	2MH NFM	2,3DMP NFM	1,1DMCP NFM	3MH NFM	c1,3DMCP NFM	t1,3DMCP NFM	t1,2DMCP NFM
b_{ij}	1101	1101	1101	1070	1070	1070	1101	1070	1109	1109	1109
b_{ji}	520	521	521	639	640	639	522	640	507	507	507
c_{ij}	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3

PROCESS MODELING AND SIMULATION

With the thermodynamic model parameters set, a simulation in the commercial simulator Aspen Plus[®] was assembled for the extractive distillation column, as schematically shown in Figure 8.

The extractive distillation column simulation was carried out using the Radfrac model. This is a rigorous model for simulating all types of multistage vapor-liquid fractionation operations. These operations include: ordinary distillation, absorption, reboiled absorption, stripping, reboiled stripping and extractive and azeotropic distillation. The model assumes thermodynamic equilibrium and rigorous mass and energy balances in all stages. It was considered that the extractive distillation column consisted of 71 stages. Of these, 33 refer to the trays section, 12 are the equivalent stages of the random packing section above the solvent feed, 24 are the equivalent stage of the structured packing section between the solvent and hydrocarbon feeds, one is the condenser and the other is the reboiler. The equivalent stages were calculated from the HETP (Height Equivalent to a

Theoretical Plate) given by the packing vendor. Table 5 shows the Radfrac simulation specifications.

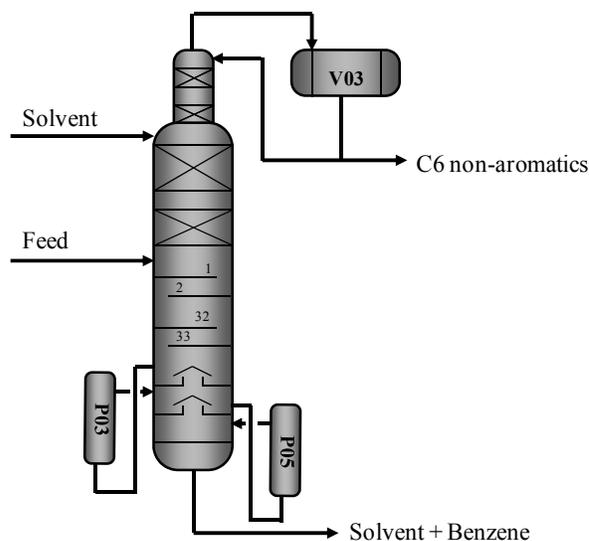


Figure 8: Schematic drawing to the extractive distillation column.

Table 5: Main specifications for the extractive distillation column simulated with the Radfrac model.

Configuration	Number of stages	71
	Condenser Reboiler Valid phases Convergence	Total Thermosiphon VLL Strongly non-ideal liquid
Streams	Solvent Feed	Above-stage 14 Above-stage 38
Pressure (bar _g)	Top stage pressure 2-13 section ΔP 14-37 section ΔP 38-70 section ΔP Bottom stage pressure	1.05 0.02 0.04 0.13 1.24
Condenser (°C)	Both reflux and liquid distillate are sub cooled	51.5
Reboiler (°C)	Temperature change	32

Another typical assumption in this work is adiabatic column operation, i.e., the metal thermal capacitance is negligible and heat exchange occurs only in the condenser and reboiler.

RESULTS

The proposed plant model was adjusted to match actual process conditions by varying the Murphree efficiencies. The efficiency was considered to be constant at each packing bed and tray section. Three different operating conditions were considered to determine the average efficiencies in the different zones. This step was accomplished manually by comparing the plant temperature profile and the simulated values. The result was satisfactory; however, some discrepancy was observed in the bottom temperature.

This difference is probably due to a small lack of fit of the benzene and NFM mixture vapor pressure. Since the pressure is specified in the model, discrepancies in the temperature prediction will directly impact the model response. Another possible reason for the temperature difference found in the bottom of the column is the feed composition used. Laboratory analyses are not reliable for the heaviest components in low concentration. Thus, the model included only components with up to seven carbons. However, this discrepancy affects only the bottom temperature profile and should not be considered relevant for the benzene/non-aromatics separation, which is the focus of this work.

In the absorption section (between stages 14 and 25), a small efficiency was found (aprox. 35%), indicating a poor liquid and/or vapor internal distribu-

tion. The simulation results were almost insensitive with respect to the efficiency in the other packing sections and trays. Further, this small sensitivity could not be verified with the currently measured data, due to the small number of temperature sensors available (see Figure 9). Thus, the following efficiency values were assumed: random packing from stages 2 to 13, 100%; structured packing from stages 26 to 37, 35%; and tray section, 80%.

Table 6 shows one of the unit feedstock compositions studied (feed composition used in the simulation was the same as that analyzed in the laboratory), the top composition sampled during operation, the composition obtained with the simulation and the absolute error (value measured – calculated value).

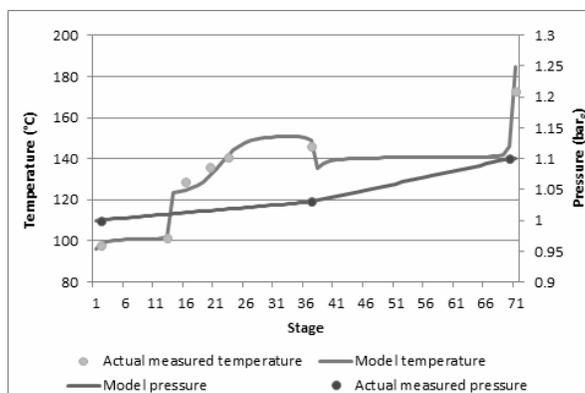
As can be seen in Table 6, the simulation results agree very well with plant data. The bigger composition errors were in the n-hexane and methylcyclopentane values. These components represent 15 and 45% of the stream, respectively, and an error of approximately 0.2 percent cannot be considered significant. Nevertheless, the actual plant mass balance also contains errors, probably due to measurement inaccuracies and inadequate sampling or laboratory analysis. No additional treatment, like data reconciliation, was applied to the measured plant data.

In Figure 9 it is possible to see the temperature and pressure profiles obtained and compare with the measured operating temperatures, the first stage being the condenser and the 71st stage the reboiler. The solvent NFM is added in the 14th stage and the feed is above the 38th stage.

As NFM is not fed into the top of the column, stages 2 to 13 only have the purpose of not letting the overhead product be contaminated with NFM, and therefore this region has practically constant temperature. The main influence of NFM on benzene occurs just below stage 14, where it is possible to notice a large increase in the temperature, even with the assumption of adiabatic operation. Note that this temperature profile is quite different from a conventional distillation process, with it being possible to observe higher temperatures in intermediate regions of the column. This is due to the phase changes promoted by the entrainer, making almost all of the benzene coming from the bottom vapor be transferred preferably to the liquid between stages 14 and 37. In the 38th stage a slight temperature decrease can be seen, due to a colder feed inlet. Between stages 39 and 70, we have a flatter profile because, in this region, we have a low benzene concentration in the vapor phase and not a lot of components are changing phases. Because the bottom mixture is mainly composed of solvent with benzene, the bottom section of the column has its boiling temperature.

Table 6: Feed composition, top composition and error found between simulation and sampled plant data (laboratory analyses).

Components	Feed mass composition in % (plant = model)	Top mass composition (%) - plant	Top mass composition (%) - model	Top composition absolute error (%)	Bottom mass fraction - model
Cyclopentene	0.11	0.41	0.37	0.03	0.00
2,3-dimethylbutane	0.14	0.44	0.49	-0.05	0.00
2-methylpentane	1.84	6.31	6.33	0.02	0.00
3-methylpentane	1.36	4.66	4.66	0.00	0.00
n-hexane	4.41	15.28	15.13	0.16	0.00
Methylcyclopentane	13.26	45.34	45.51	-0.17	0.00
2,4-dimethylpentane	0.10	0.33	0.33	0.01	0.00
Benzene	71.87	3.50	3.49	0.01	10.54
Cyclohexane	3.12	10.62	10.71	-0.08	0.00
2-methylhexane	0.58	2.05	1.99	0.06	0.00
2,3-dimethylpentane	0.05	0.18	0.18	0.00	0.00
1,1-dimethylcyclopentane	0.24	0.83	0.82	0.01	0.00
3-methylhexane	0.47	1.66	1.63	0.04	0.00
cis-1,3-dimethylcyclopentane	0.66	2.29	2.27	0.02	0.00
trans-1,3-dimethylcyclopentane	0.47	1.63	1.61	0.01	0.00
trans-1,2-dimethylcyclopentane	0.65	2.24	2.23	0.01	0.00
n-heptane	0.54	1.91	1.85	0.06	0.00
methylcyclohexane	0.12	0.40	0.41	-0.01	0.00
NFM	0.00	0.00	0.00	0.00	89.46

**Figure 9:** Extractive distillation column temperature and pressure profiles.

With the plant model adjusted, it was possible to study the column behavior by performing sensitivity analysis. The temperature profile of this column is very sensitive to the heat supplied. Along with other manipulated variables it determines the region where benzene absorption will occur. Figure 10 shows the static temperature profile change when the reboiler heat is varied. In these simulations the feed rates and reflux ratio were kept constant. It should be noted that the simulation results of Figure 10 differ by only

0.21 GJ/h of reboiling heat. Considering only the exchange of latent heat from a steam of 18bar_g (1859 kJ/kg), this corresponds to a variation of only 112 kg/h of steam. Thus, it is clear that small variations in the reboiler heat are enough to bring the products out of specification. It can also be noted that the major temperature variations for each reboiling heat are between stages 14 and 70. This happens because above stage 14, no solvent is present and the temperature is controlled by the non-aromatic

mixture boiling point. On the other hand, the bottom temperature is related to the boiling temperature of the solvent/benzene mixture. Hence, with reboiling heating variation, we are actually dislocating the stage where the benzene will change phase and, consequently, modifying the temperature profile mainly in the rectifying section.

In Figures 11 and 12 simulation results for benzene recovery (defined as benzene at the bottom / benzene at the feed) with respect to the solvent-to-feed ratio and solvent temperature are shown, respectively.

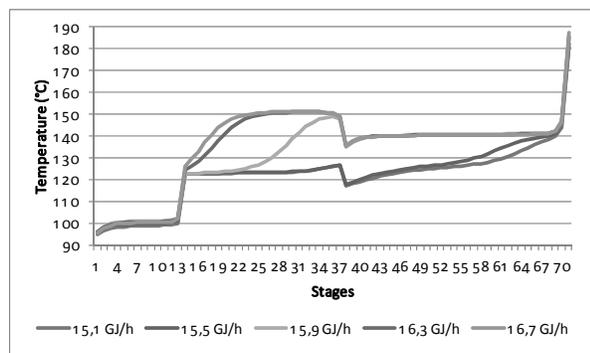


Figure 10: Effect of reboiler heat on the temperature profile.

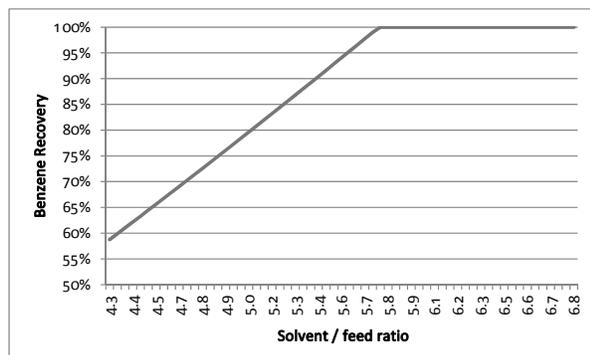


Figure 11: Effect of the solvent-to-feed ratio on the column benzene recovery.

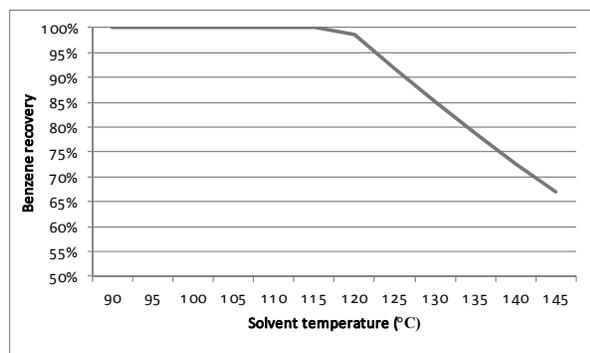


Figure 12: Effect of the solvent temperature on the benzene recovery.

From this analysis it can be seen that there is an optimal solvent-to-feed ratio for proper column operation. Above this value the benzene recovery does not improve substantially, only operational costs are increased. The same applies to solvent temperature. Temperatures below 115 °C, under the same reflux ratio and reboiler heat, are recommended to obtain a better benzene recovery.

CONCLUSIONS

In the present work an industrial system to separate benzene from a non-aromatic stream through extractive distillation with NFM as the solvent was studied. First, literature data were collected and different sets of parameters for the NRTL model were compared. For many pairs of substances a need to re-estimate the NRTL model parameters was identified. For the substance pairs with no experimental data available in the literature, the UNIFAC group contribution model was used to estimate the missing parameters. As a result of this step a new set of NRTL parameters was obtained for processes involving NFM, benzene and other non-aromatics.

Using the proposed NRTL parameter set, a plant model was assembled within *ASPEN Plus*[®]. Murphree efficiencies were then adjusted for better agreement with plant data using three different operating points. The model proposed represented satisfactorily the operation of the extractive distillation column and can be used to study the behavior of most components present in the column feed. From the model analysis it is possible to demonstrate that a great difficulty of the extractive distillation column studied is its extreme static sensitivity to variations of the supplied heat. The operational challenge is to control the contact point of benzene with NFM within a region that allows the simultaneous specification of the overhead product (non-aromatic C6) and bottom product (benzene). This behavior should be further investigated so that a new control strategy can be proposed, it perhaps being necessary to add additional thermocouples along the column.

Based on the sensitivity analysis of the column and the optimal operation point, it is possible to implement improvements in the operation site. However, in order to have a complete mapping of the possible operating problems, additional hydraulic analysis and internals verification are desirable.

NOMENCLATURE

a_{ij}, b_{ij}, c_{ij}	non-symmetrical constants	[-]
and f_{ij}		
x_i	component i mole fraction in the mixture	[-]
T	absolute temperature	[K]
ΔP	pressure drop	[kgf/cm ² _g]
y_i	activity coefficient of a component	[-]

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