Optimal Retrofit Design of Extractive Distillation to Energy Efficient Thermally Coupled Distillation Scheme

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Design and optimization procedures employing the response surface methodology (RSM) for retrofitting the conventional extractive distillation sequence to a thermally coupled extractive distillation scheme (TCEDS—SR) is presented. The optimum TCEDS—SR structure can be found in a practical manner with minimal simulation runs. Furthermore, the RSM allows the interactions between variables to be identified and quantified. The separation of close boiling point mixtures and azeotropic mixtures was examined to test the proposed method. The predictions agreed well with the results of a rigorous simulation. The results showed that a retrofit of the extractive distillation sequence to TCED—SR can achieve significant energy savings compared to the conventional extractive distillation sequence. © 2012 American Institute of Chemical Engineers *AIChE J,* 59: 1175–1182, 2013
Keywords: thermally coupled extractive distillation, thermally coupled azeotrope distillation, response surface methodology, distillation process retrofit, process integration

Introduction

Azeotropic and close boiling components are commonly encountered in fine-chemical and specialty industries. The separation of these mixtures is a challenging task in many chemical processes, which is impossible using a single conventional distillation column, and many nonconventional distillation techniques have been proposed to solve this problem.[1–4] The most common alternatives involve changing the operating pressure or adding a separation solvent. The pressure option is economically feasible only for mixtures sufficiently sensitive to pressure. The second alternative is the typical nonconventional distillation process encountered in industry and involves what is known as extractive distillation. This method uses a solvent that is generally nonvolatile, has a high-boiling point, and is miscible with the mixture. This solvent interacts differently with the components of the mixture, thereby causing a change in their relative volatility.[5] This enables new three-part mixtures to be separated by normal distillation. Moreover, the process makes it possible to obtain a pure compound at the top of the column with the other, together with the solvent, collected at the bottom, which can be separated easily in a secondary distillation or solvent recovery column due to the high-boiling point of the solvent.[3]

Extractive distillation (Figure 1a) is used widely in a number of processes,[3,6–11] but its high-energy consumption is a major main disadvantage, which requires dispatching. Process integration is an interesting option for solving this problem and it has been successful in reducing the energy requirements compared to processes where all the units are configured with little or no integration.[5] In particular, the use of columns with thermal coupling has attracted considerable attention in recent years.[12–24] Thermally coupled distillation systems (TCDS), which are shown schematically in Figure 1b, were obtained through the implementation of interconnecting streams (one in the vapor phase and the other one in the liquid phase) between two columns. Each interconnection replaces one condenser or one reboiler from one of the columns, thereby providing potential cost savings. Thermally coupled extractive distillation sequences (TCEDS—SR) can have lower energy consumption than conventional extractive distillation sequences.[2,3,25]

At the end of the 1980s, 70–80% of capital investment projects in the processing industry were retrofit projects.[26] Several studies examined the application of dividing wall columns in a retrofit.[27–31] They stressed that the dividing wall column can be used to save energy and expense. On the other hand, this can have practical difficulties.[27] Apart from existing column constraints, such as a fixed column diameter, most of these concerns were focused on the need to modify column shells to accommodate the dividing wall, and have another nozzle fitted to withdraw the middle product. Such modifications to install a dividing wall, which may involve the removal of tray support rings and replacement of the existing internals, can be a major undertaking. In addition, the mechanical design aspects of the column, such as mechanical stress, are affected by changing the feed or side-draw locations. Therefore, although the dividing wall column may initially be the best option for retrofit projects, these concerns must be addressed fully during the retrofit design. These modifications require plant downtime, which leads to a loss of production and an interruption of the product supply to the customers.

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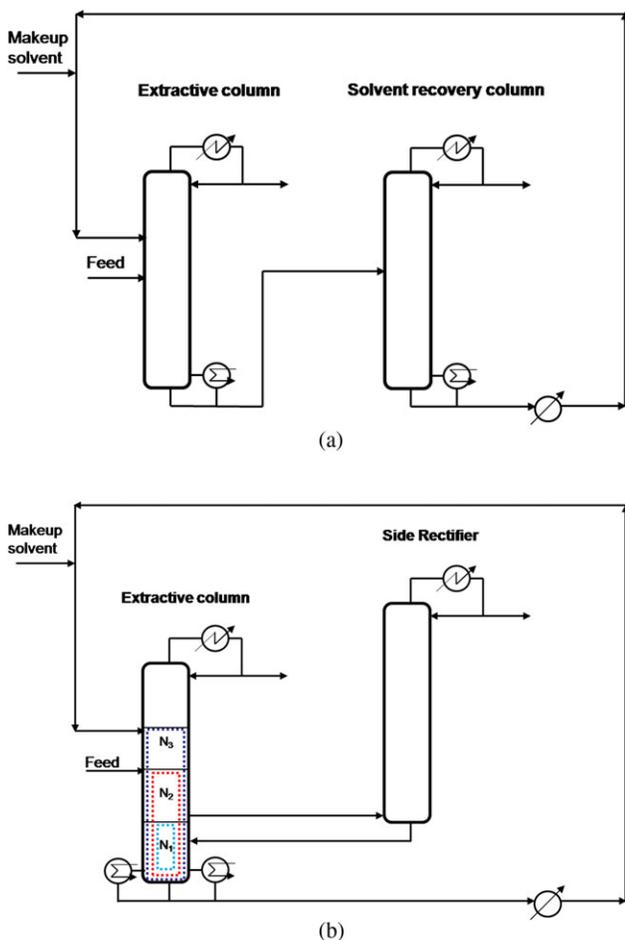
Figure 1. Schematic diagram of (a) conventional extractive distillation sequence, and (b) thermally coupled extractive distillation sequence.

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The response surface methodology (RSM) is a general technique for an empirical study of the relationships between the measured responses and independent input variables.[32] A response surface is normally a polynomial, whose coefficients are extracted by a simple least-square fit to the experimental data. The RSM is quite powerful because, in addition to modeling, it can also optimize the conditions of the process under investigation.[33–36] Normally, a low-order polynomial is used in some regions of the independent variables.[37] A simple first-order model can be used as an approximating function if the response is modeled well by a linear function of the independent variables. On the other hand, a polynomial of higher degree, such as a second-order model, must be used if there is curvature in the system. According to the sparsity-of effects principle in the response surface methodology, main (single factor) and quadratic effects and two-factor interactions are likely to be the most significant effects, and the higher-order interactions are negligible.[38–39] In other words, higher-order interactions, such as three-factor interactions, are quite rare and are considered to be residual, which are dispersed randomly. Almost all RSM problems use one or both of these models. A polynomial model is unlikely to be a reasonable approximation of the true functional relationship over the entire space of independent variables, but usually works well for a relatively small region.[37]

Table 1. Mixture Analyzed in this Study

Mixture	Feed components	Extractive solvent	Feed flow (kmole/hr)	Feed composition (mole fraction)
M1	Acetone/methanol	water	540.00	0.5/0.5
M2	Heptane/toluene	aniline	181.44	0.5/0.5

On the other hand, the RSM encounters some difficulties in approximating the sufficient quality on the entire design space when the behavior of the function is highly nonlinear with respect to the variables.[40] The optimum may not be defined if the range of factors is too narrow or too wide. Furthermore, critical factors may not be defined or specified correctly. In the case of many factors, the problem becomes complex and the RSM cannot be predicted well.

This article reports the design and optimization procedures employing the RSM for retrofitting conventional extractive distillation sequences to TCEDS—SRs with particular emphasis on utilization of the existing hardware with minimal investment costs and construction effort. Several mixtures were used to show that the TCEDS—SR requires less energy consumption than the conventional extractive distillation sequences.

Design

Distillation column retrofitting is performed more often than the installation of new equipment because distillation is an energy-intensive process requiring large capital investment.[27,41] In general, a retrofit project in the distillation process covers a broad range of modifications and uses existing process ranging from a simple modification or replacement of column internals only to large modifications of the column configuration, partial enlargement of the shell diameter and height, and the modification of auxiliary equipment. In any case, the key to a successful retrofit lies in exploiting the existing hardware by maximizing the use of existing
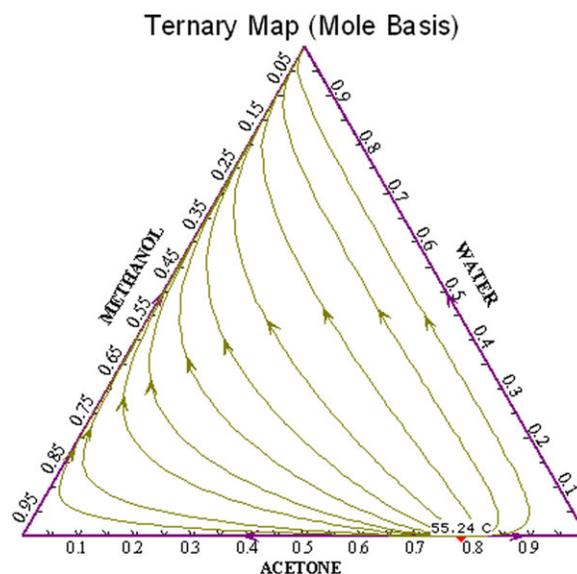

Figure 2. The residue curve map of mixture acetone-methanol with water as an extractive solvent.

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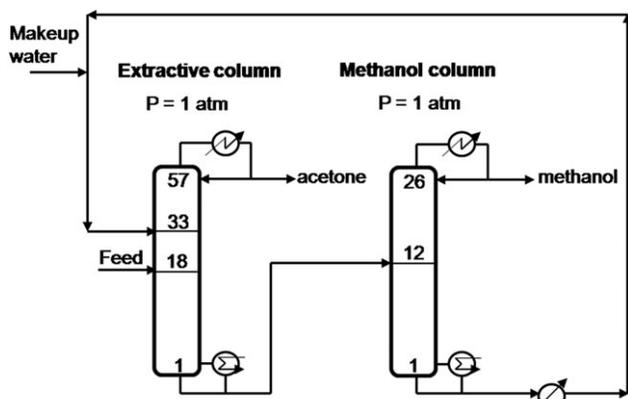
Figure 3. Simplified flow sheet illustrating the existing separation train of two conventional columns for mixture M1.

Table 2. Coded Levels of Factors for Mixture M1

	Levels		
Factor	−1	0	1
Vapor stream section (N1)	5	7	9
Feed section (N2)	13	15	17
Solvent section (N3)	28	31	34

in the conventional direct sequence, which eliminates the reboiler in the second column of the conventional scheme.[3] Modification for the retrofit involves a change in the draw trays for the vapor and liquid stream. More pipe work is also needed for the thermal link and reassignment of the recovery column reboiler to the main column.

Optimization

To minimize the modifications in a retrofit, the number of trays of all columns was fixed to the same number in conventional extractive distillation columns. Furthermore, another variable, the solvent/feed ratio, was also kept constant as the optimal value in the conventional extractive distillation sequence. Therefore, after a preliminary design for the TCEDS─SR column, the main design variables, such as the internal vapor flow (FV) to the side rectifier, vapor (N1), feed (N2) and solvent (N3) stream locations, were then optimized. The response surface methodology was used to examine the effects on the reboiler. After determining the preliminary ranges of the variables through single-factor testing, a Box-Behnken design was used to determine how the variables interact and to optimize the system in terms of the reboiler duty.

The simulation data was fitted to a second-order polynomial model and the regression coefficients were obtained.
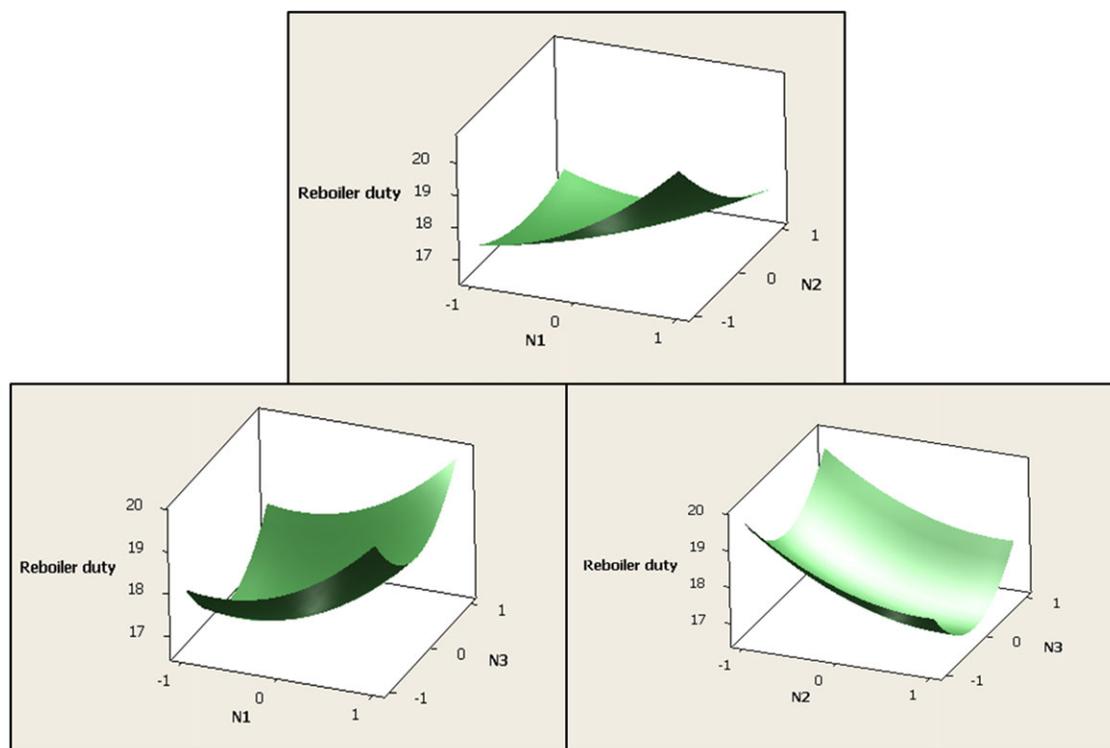
equipment while, at the same time, minimizing the need for new hardware to minimize the capital cost.

Figure 1b shows the possibility of rearranging two existing columns into a thermally coupled extractive distillation scheme, where the extractive and side rectifier columns of the retrofit scheme use extractive and solvent recovery columns of the conventional scheme, respectively. To highlight the optimal use of the existing columns, the study assumes no change in the diameter or the total number of stages of each column. Retrofitting the extractive distillation sequence begins with the development of preliminary designs for complex systems and minimizes the heat duty supplied to reboilers through optimization procedures. The TCEDS─SR column was designed using a thermal link in the vapor phase

Figure 4. Three-dimensional (3-D) response surface plots of interactions between main design variables N1, N2, and N3 for mixture M1.

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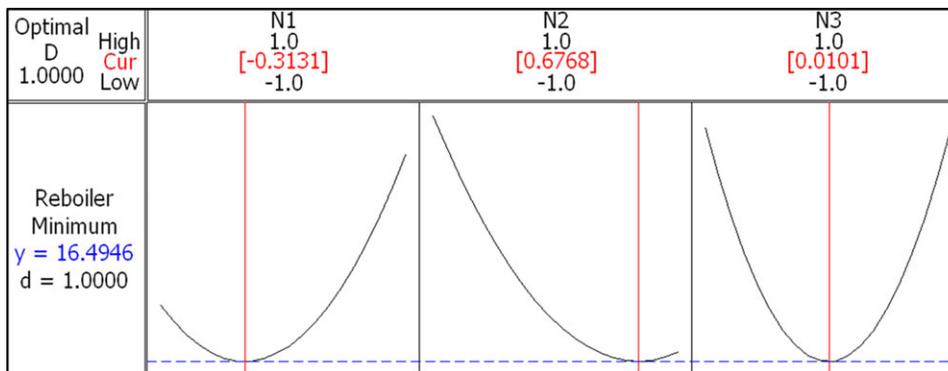
Figure 5. Optimization plot by the RSM for mixture M1.

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The generalized second-order polynomial model used in the response surface analysis can be expressed as follows

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum \sum_{i<j} \beta_{ij} X_i X_j + \varepsilon \quad (1)$$

where Y is the predicted response (reboiler duty), X_i are the uncoded or coded values of the variables, β_0 is a constant, β_i, β_{ii} and β_{ij} are the coefficients of the linear, quadratic and interactive terms, respectively, and ϵ is the error term. MINITAB software was used for response surface fitting and optimizing the reboiler duty.

Case Study

Optimal designs of the retrofitted TCEDS—SRs were obtained for two binary mixtures with different extractive solvents using the RSM, which is shown in Table 1. The conventional extractive sequences and solvent were taken from previous extractive distillation papers.[3,42] The simulation was performed using Aspen HYSYS V7.1. The UNIQUAC model was used to describe the thermodynamic properties.

To retrofit the extractive distillation sequences to the TCEDS—SRs, the main design parameters of the existing columns were checked. The maximum flooding, which is the ratio of the actual vapor velocity to the maximum/flooding vapor velocity, was in the acceptable range. This is one of the main determining factors for retrofitting extractive distillation sequences to the TCEDS—SRs.[25] Furthermore, existing reboilers and condensers should be checked for reusability with minimal modifications.

To determine the maximum flooding of a particular column, the rating mode was simulated using the internal
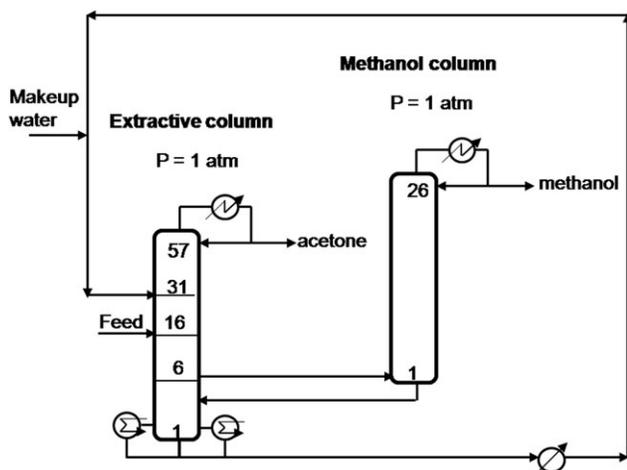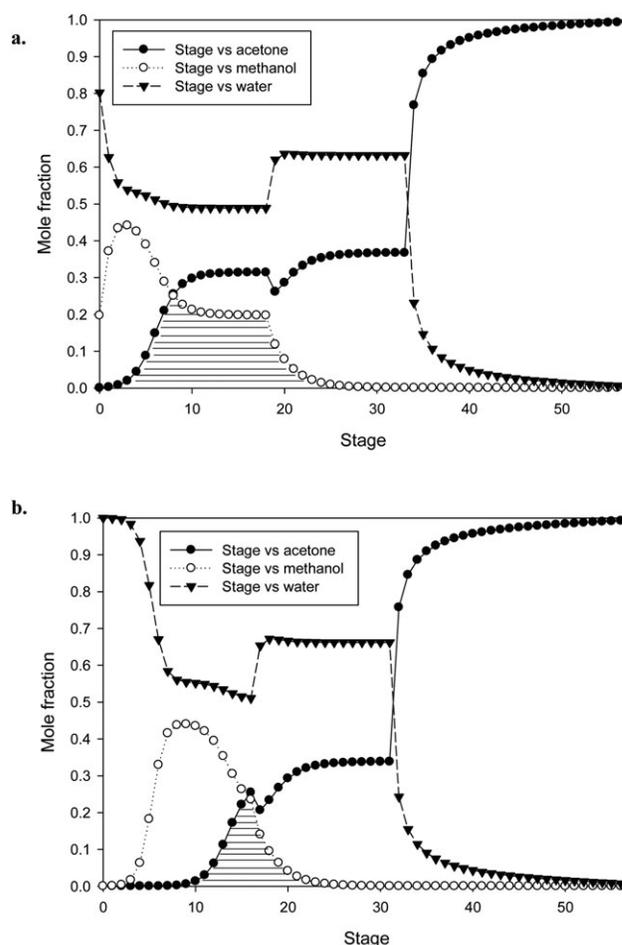

Figure 7. Composition profiles for the extractive column of (a) conventional extractive distillation sequence, and (b) thermally coupled extractive distillation sequence for mixture M1.

Figure 6. Simplified flow sheet illustrating the retrofitted TCEDS-SR for mixture M1.
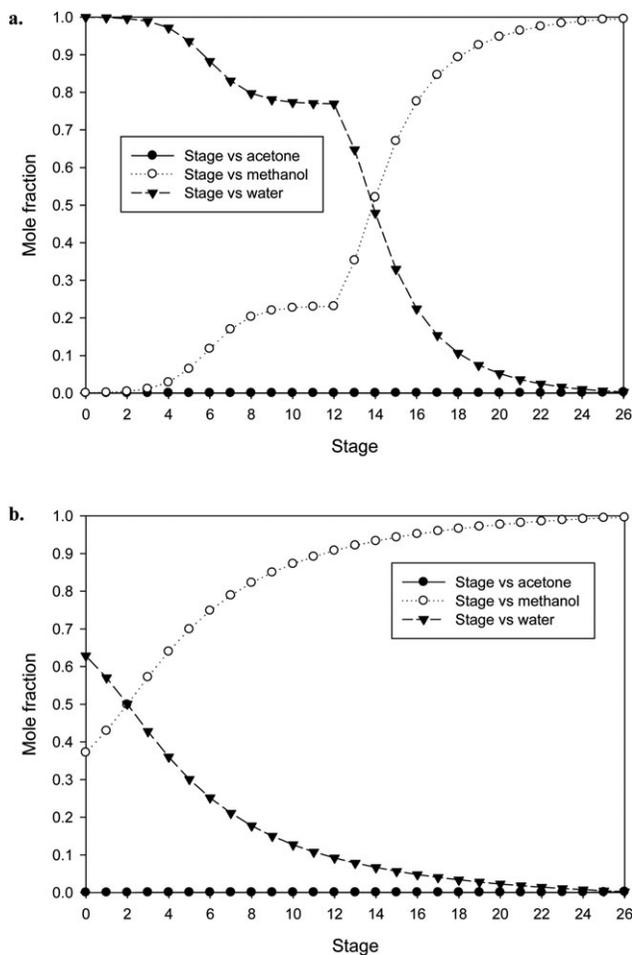
Figure 8. Composition profiles for the (a) solvent recovery column of conventional extractive distillation sequence, and (b) side rectifier of thermally coupled extractive distillation sequence for mixture M1.
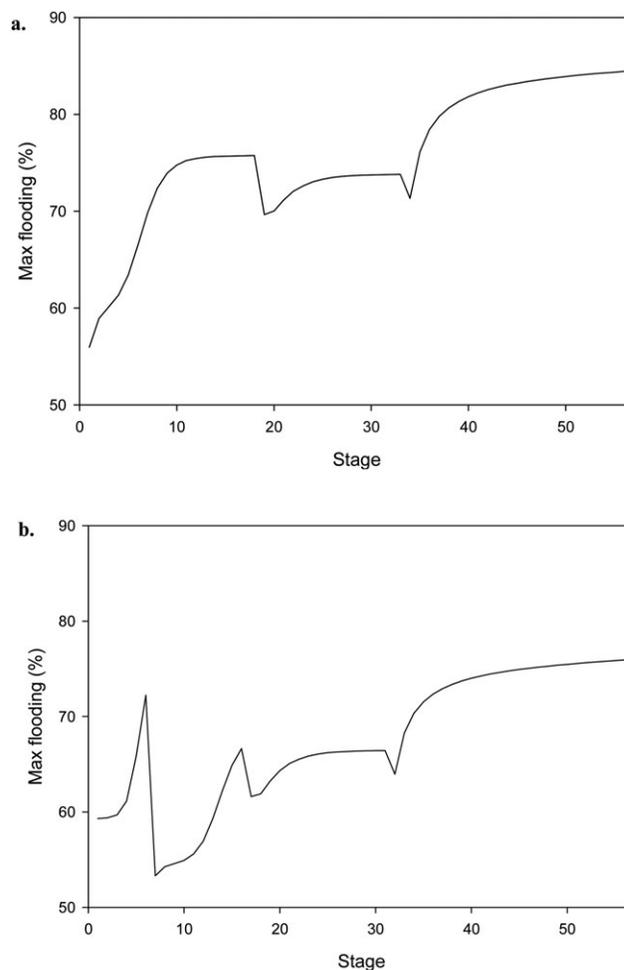
Figure 9. Max flooding profiles for the extractive column of (a) conventional extractive distillation sequence, and (b) thermally coupled extractive distillation sequence for mixture M1.

specifications of the column, such as the type of trays, column diameter, tray spacing and number of passes. All columns were designed with a load near 85% of the flooding point load to prevent flooding in the columns.

Mixture M1

As shown in Figure 2, there was no distillation boundary that divides the composition profile into different regions, and separation was feasible. Figure 3 shows a simplified flow sheet illustrating the existing separation train of two conventional columns for the mixture M1.[42] The process has two columns, one for extractive separation and the other for solvent recuperation. The optimum design had a 57-stage extractive column and a 26-stage methanol column, both operating at 1 atm. The solvent was fed into the extractive column on a tray above the feed tray and below the top of the column so high-purity acetone could be produced in the distillate. The solvent flow rate must be 1,100 kmol/h to achieve the desired 99.5 mol % acetone purity.

Table 2 lists the factors and levels used in this case study. Fifteen simulations were run to optimize 3 parameters of the TCEDS—SR structure. For each run, internal vapor flows to the side rectifier were varied to minimize the reboiler duty while still achieving the required product purity. Figure 4
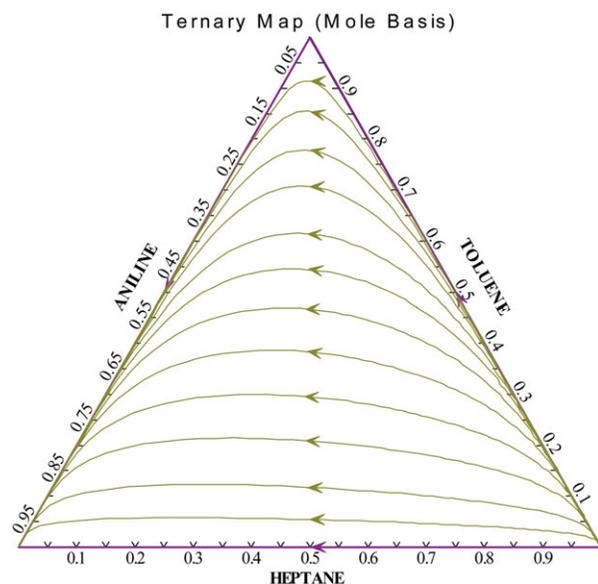

Figure 10. The residue curve map of mixture heptane-toluene with aniline as an extractive solvent.

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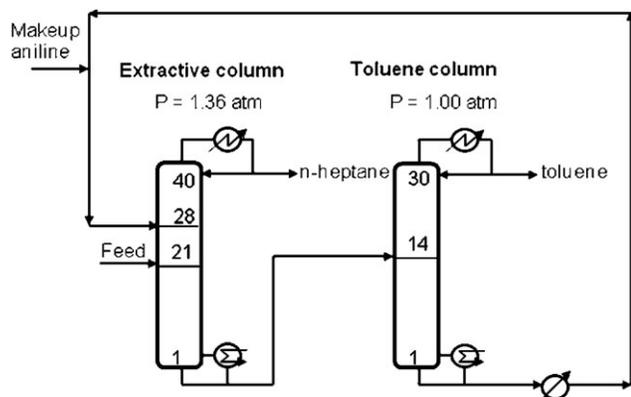
Figure 11. Simplified flow sheet illustrating the existing separation train of two conventional columns for mixture M2.

Table 3. Coded Levels of Factors for Mixture M2

Factor	Levels −1	0	1
Vapor stream section (N1)	6	9	12
Feed section (N2)	18	21	24
Solvent section (N3)	26	29	32

shows the three-dimensional (3-D) response surface plots of the interactions between the main design variables N1, N2 and N3, for the mixture M1. Two parameters of each model were plotted on each set of X- and Y-axes. The reboiler duty was plotted on the Z-axis. The remaining parameters were set automatically at their center point values by the software while constructing the plots. The smallest reboiler duty was observed at the coded levels of the number of trays in the vapor stream, feed and solvent sections of −0.3131, 0.6768
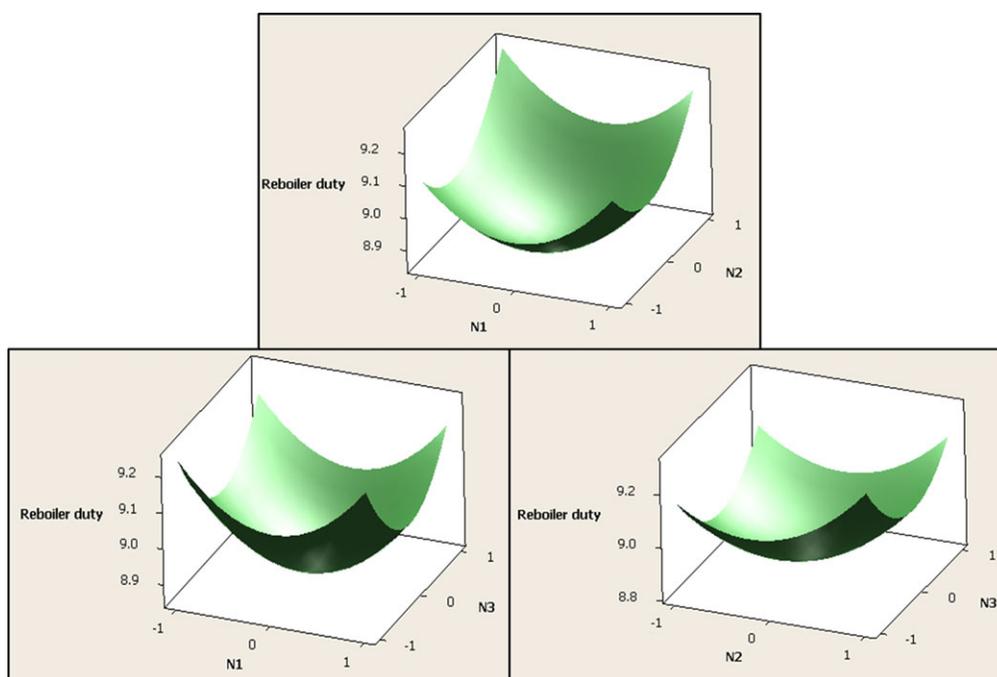

Figure 12. Three-dimensional response surface plots of interactions between main design variables N1, N2, and N3 for mixture M2.

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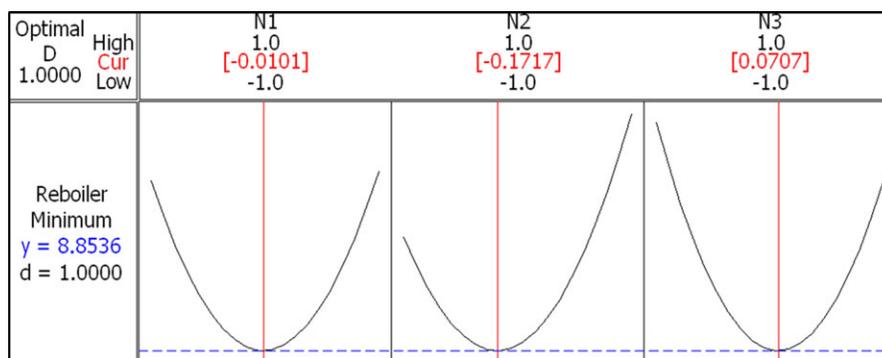

Figure 13. Optimization plot by the RSM for mixture M2.

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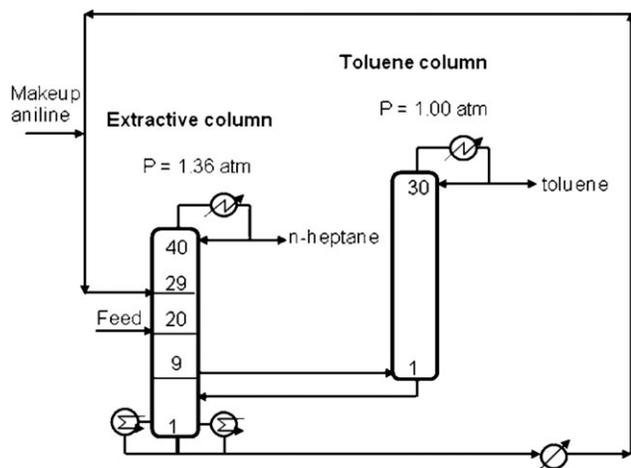
Figure 14. Simplified flow sheet illustrating the retrofitted TCEDS─SR for mixture M2.

and 0.0101, respectively (Figure 5). Under these conditions, the minimum reboiler duty by the retrofitted TCEDS─SR was predicted to be 16.49 MW. The natural values of the variables can be derived from the coded levels. The internal vapor flows to the side rectifier were then optimized to minimize the reboiler duty, while maintaining sufficient product purity. Figure 6 shows a simplified flow sheet illustrating the retrofitted TCEDS─SR for the mixture M1. The simulation also showed that the TCEDS─SR consumes 16.58 MW, which is in good agreement with the result predicted by the RSM. Considerable savings (13.36%) in reboiler energy could be achieved. The reboiler in the methanol column can be used together with the existing reboiler in the extractive column. Figure 7 shows the composition profiles of the extractive column for the conventional extractive distillation sequence and the TCEDS─SR sequence. The composition profile of methanol was quite smooth with no pinch zones in the retrofitted TCEDS─SR. The separation zone of acetone and methanol was lower than that in the conventional extractive distillation sequence. Figure 8 presents the composition profiles for the solvent recovery column of the conventional extractive distillation sequence and side rectifier of the thermally coupled extractive distillation sequence. Pinch zones also appear from the 9th stage to the 12th stage for both the acetone and methanol composition profiles, which were eliminated in the retrofitted TCEDS─SR. Furthermore, maximum flooding in the retrofitted TCEDS─SR was reduced to 76% compared to the conventional extractive distillation

sequence (85%), as shown in Figure 9, which offers 10% additional capacity.

Mixture M2

Figure 10 depicts the residue curve map of the heptane-toluene mixture with the extractive solvent, aniline. A separation split is feasible if the composition of the products and feed need to be located in the same region of the composition space.[5] Mixture M2 does not have an azeotropic point, which makes separation feasible. Figure 11 shows the simplified flow sheet illustrating the existing separation train of two conventional columns for mixture M2.[3] For the conventional case, the feed flow rate and optimal solvent/feed ratio was 184.44 kmol/h and 2.5, respectively. Table 3 presents the factors and levels. Figure 12 shows the 3-D response surface plots of the interactions between the main design variables, N1, N2 and N3, for the mixture M2. Figure 13 shows the RSM optimization plot for M2. Figure 14 shows the structure of the retrofitted TCEDS─SR. The energy saving generated from this arrangement was 32.03% (Table 4), which is comparable to the performance of the existing column arrangement. The TCEDS─SR offers greater capacity to the separation train while exploiting the existing columns. On the other hand, the column operating pressure may be changed. Therefore, the mechanical calculations for the column might require checking. Fortunately, these columns are normally operated at the same or similar operating pressures.

These modifications require plant downtime.[27] In many retrofit projects, downtime is the largest economic factor, and a significant amount can lead to a loss of production and an interruption in product supply. In an integrated facility, where plants depend on each other, such downtime can disrupt other downstream processes. Here, the feed and aniline stream nozzle moves from the 21st and 28th trays to the 20th and 29th trays, respectively, which require some modifications. Energy savings of up to 31.65% are still possible when the feed locations remain in the conventional case. Even the optimum point provides better operating cost savings, and keeping the same location of the feeds is preferred due to fewer requirements for modification. The operating cost and investment are a trade-off to be considered for a retrofit design.[28]

Gas is normally transported when a compressor applies a pressure difference between the inlet and outlet, which results in more investment and operating cost. Fortunately, the pressure of the vapor stream tray location is 1.70 atm, which is higher than that of the bottom section of the side rectifier. Therefore, a compressor is not required in this retrofit design.

Table 4. Results for Different Mixtures

	M1				M2			
	Conventional sequence		Retrofitted TCEDS─SR		Conventional sequence		Retrofitted TCEDS─SR	
	Extractive column	Solvent recovery column	Extractive column	Side rectifier column	Extractive column	Solvent recovery column	Extractive column	Side rectifier column
Tray number	57	26	57	26	40	30	40	30
Column diameter (m)	2.7	1.7	2.7	1.7	2.4	2.1	2.4	2.1
Pressure (atm)	1.00	1.00	1.00	1.00	1.36	1.00	1.36	1.00
Reflux ratio	3.9	1.6	3.4	1.0	3.4	5.8	3.3	0.5
Reboiler duty (MW)	12.01	7.13	16.58		7.38	5.70	8.89	
FV (kmol/h)			514.5				115.9	
Reboiler saving (%)	0		13.36		0		32.03	

Conclusions

This article reports the design and optimization of retrofitting an extractive distillation sequence to a thermally coupled extractive distillation sequence with a side rectifier. This can be adapted easily to include the hydraulic capacity and the total number of stages of the existing columns involved in the retrofit. The response surface methodology was used to obtain the optimal designs of the TCEDS−SR. Retrofitting of the extractive distillation sequence can achieve significant energy savings with few modifications. The predicted RSM results showed good agreement with a rigorous simulation. Furthermore, retrofitting to a TCEDS−SR could not only use existing columns, but could also increase the process capacity.

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