

# Controllability Analysis of Thermally Coupled Distillation Systems: Five-Component Mixtures

Juan Gabriel Segovia-Hernández,\* Minerva Ledezma-Martínez, Marcelino Carrera-Rodríguez, and Salvador Hernández

Universidad de Guanajuato, Facultad de Química, Noria Alta S/N, Guanajuato, Gto., 36050, México

The theoretical control properties and dynamic responses under closed-loop operation of thermally coupled distillation sequences for the separation of five-component mixtures of hydrocarbons were compared to those of conventional distillation sequences. Seven thermally coupled arrangements were investigated: five thermally coupled distillation sequences with three recycles and two thermally coupled distillation schemes with two recycles. The preliminary steady-state design of complex schemes was obtained by starting from a conventional distillation sequences and then optimizing for minimum energy consumption [Calzon-McConville, C. J.; Rosales-Zamora, M. B.; Segovia-Hernández, J. G.; Hernández, S.; Rico-Ramírez, V. Design and Optimization of Thermally Coupled Distillation Schemes for the Separation of Multicomponent Mixtures. *Ind. Eng. Chem. Res.* 2006, 45, 724]. The control properties of the sequences considered were obtained by using the singular value decomposition technique at zero frequency. It was found that, in general, the coupled schemes present theoretical control properties similar to or better than those of conventional distillation sequences. This result was corroborated by using rigorous closed-loop dynamic simulations. As a result, one can conclude that the energy savings predicted for thermally coupled distillation sequences are achieved without introducing additional control problems if we assume constant pressure drops in columns.

## 1. Introduction

Effective screening of separation systems constitutes a critical stage, as engineers need to review and understand tradeoffs ahead of detailed modeling and simulation. In separations, it is often desired to explore the use of complex rather than simple columns, because the complex units reduce mixing losses, use available vapor and liquid more effectively, and improve the separation efficiency.<sup>1,2</sup> Among all possible complex schemes for a multicomponent distillation process, thermally coupled distillation sequences are very promising for both energy and capital cost savings. Much detailed work has contributed to some specific ternary configurations aiming at performance analysis.<sup>3–9</sup> In these studies, it has reported that the thermally coupled configurations are capable of achieving energy savings of up to 30% in contrast to the conventional direct and indirect distillation sequences for the separation of feeds with low or high content of the intermediate component, and that the energy savings depends on the amount of the intermediate component. Also, the thermally coupled distillation sequences for the separation of ternary mixtures, over a wide range of relative volatilities and feed compositions, have been reported to provide a better thermodynamic efficiency than the conventional distillation configurations.<sup>10</sup> For mixtures with four or more components, the combinatorial problem makes it even more difficult to construct all possible schemes. There are a few works on extensions toward the design of integrated systems for mixtures of more than three components.<sup>11–13</sup> Recently, Rong et al.<sup>14–16</sup> have parametrically studied some thermally coupled distillation sequences for five-component mixtures from the viewpoint of economic evaluation and optimal synthesis of the multicomponent thermally coupled distillation flow sheets. In general, the economic potential of thermally coupled sequences has already been recognized, but their control properties have not

been studied to the same degree. Recent efforts have contributed to the understanding of the dynamic properties of integrated schemes for the separation of ternary and quaternary mixtures.<sup>17–23</sup>

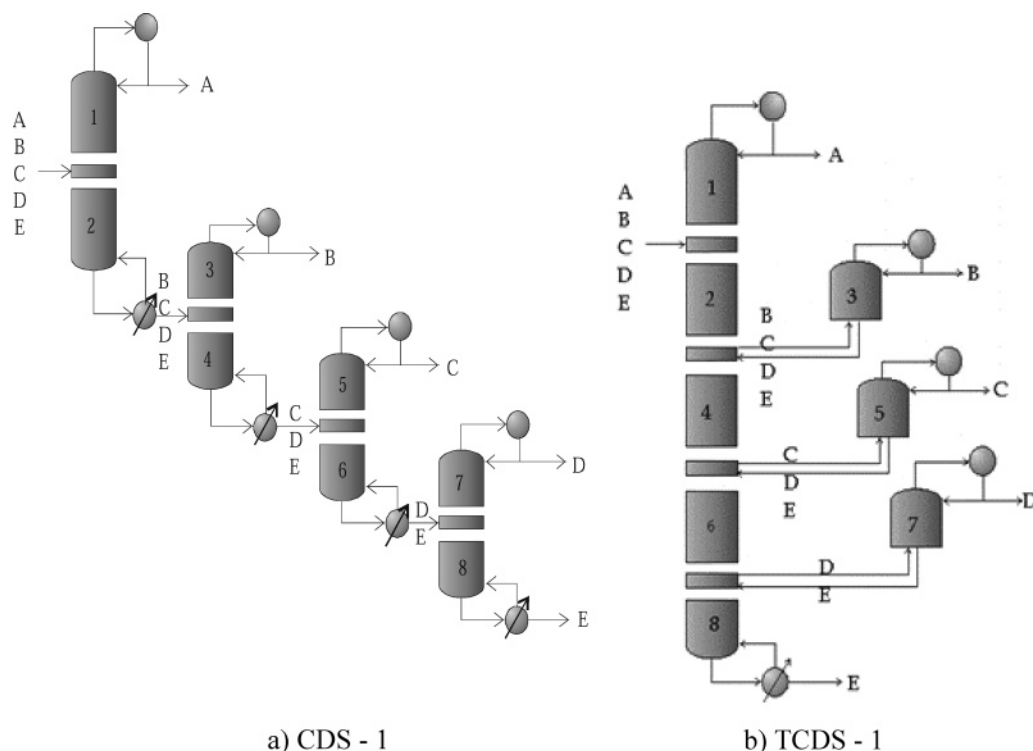
Alatqi and Luyben<sup>24</sup> compared quantitatively the dynamic responses of the conventional direct distillation sequence and the thermally coupled distillation with a side stripper, and they found the unexpected result that the integrated distillation sequence presented better dynamic responses than those of the uncoupled distillation sequence. They reported that the coupling nature and recycle stream present in the thermally coupled distillation with a side stripper contributed positively to disturbance attenuation. Kaibel<sup>25</sup> reported that the control of the fully thermally coupled distillation sequence corresponds to that of a conventional distillation column with a side product. This result has gained importance, since thermally coupled distillation columns have successfully been implemented and operated in industrial practice.<sup>26,27</sup>

Jiménez et al.<sup>28</sup> presented a controllability analysis of seven distillation schemes, and they found that the thermally coupled distillation sequences presented better theoretical control properties than those of the conventional distillation sequences. The result was corroborated through the use of dynamic simulations under closed-loop operation.

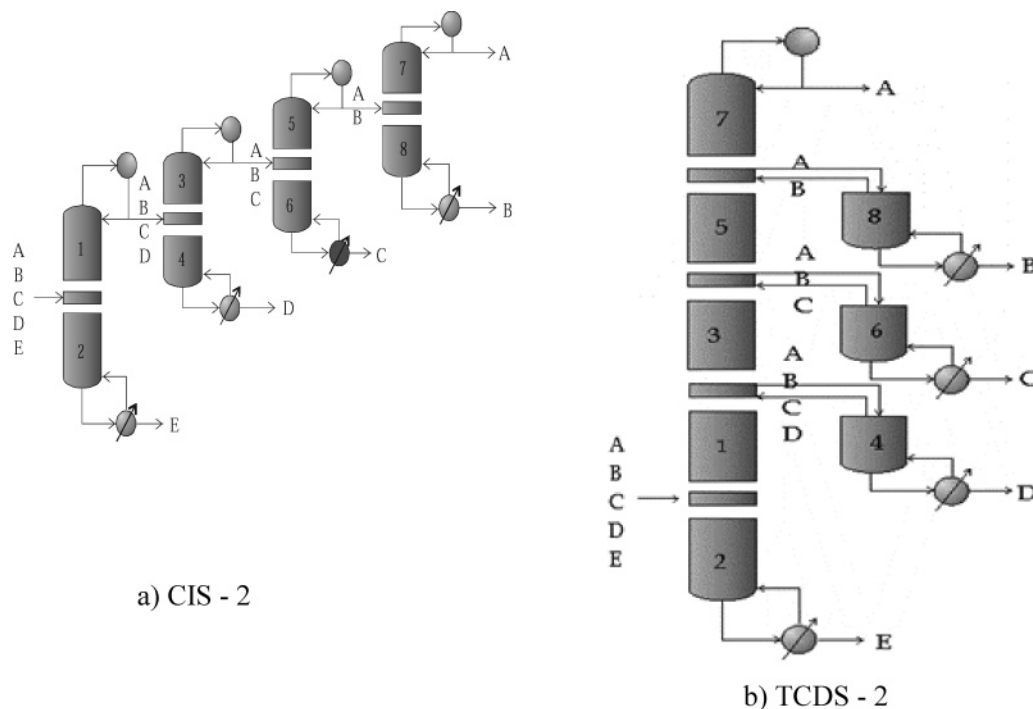
In the case of quaternary separations some results have been reported; for example, Cárdenas et al.<sup>23</sup> found that the theoretical control properties of a fully thermally coupled distillation sequence and a thermally coupled distillation sequence with two side columns were better than those of an uncoupled distillation sequence. This result was corroborated using dynamic simulations for different pairings in the control loops.<sup>29</sup>

The expectation that the dynamic properties of those coupled systems may cause more operational problems than the conventional sequences is one of the factors that has contributed to their lack of industrial implementation. This conflict is commonly observed in cases where the optimization of some energy-efficient systems leads to tight designs, which in turn are more difficult to control.

\* To whom correspondence should be addressed. Tel.: (52) 473-73-20006, ext 8142. Fax: (52) 473-73-20006, ext 8108. E-mail: gsegovia@quijote.ugto.mx.



**Figure 1.** Thermally coupled sequence (TCDS-1) and the conventional arrangement where it is obtained.



**Figure 2.** Thermally coupled sequence (TCDS-2) and the conventional arrangement where it is obtained.

In this work we developed a comparative study of the control properties of seven thermally coupled distillation sequences (previously proposed by Rong et al.<sup>14,15</sup>) for the separations of five-component mixtures and those of conventional sequences (Figures 1–7). It is important to mention that constant pressure drops in columns were assumed.

## 2. Energy-Efficient Design

Strictly, the design of the thermally coupled distillation sequences could be modeled through superstructures suitable

for optimization procedures with mathematical programming techniques. However, the task is complicated and is likely to fail to achieve convergence. In this case, to overcome the complexity of the simultaneous solution of the tray arrangement and energy consumption within a formal optimization algorithm, we decoupled the design problem in two stages: (1) tray configuration; (2) energy-efficient design (optimal energy consumption).

The first stage of our approach begins with the development of preliminary designs for the complex systems from the design aspects of conventional distillation columns (see Figures 1–7).

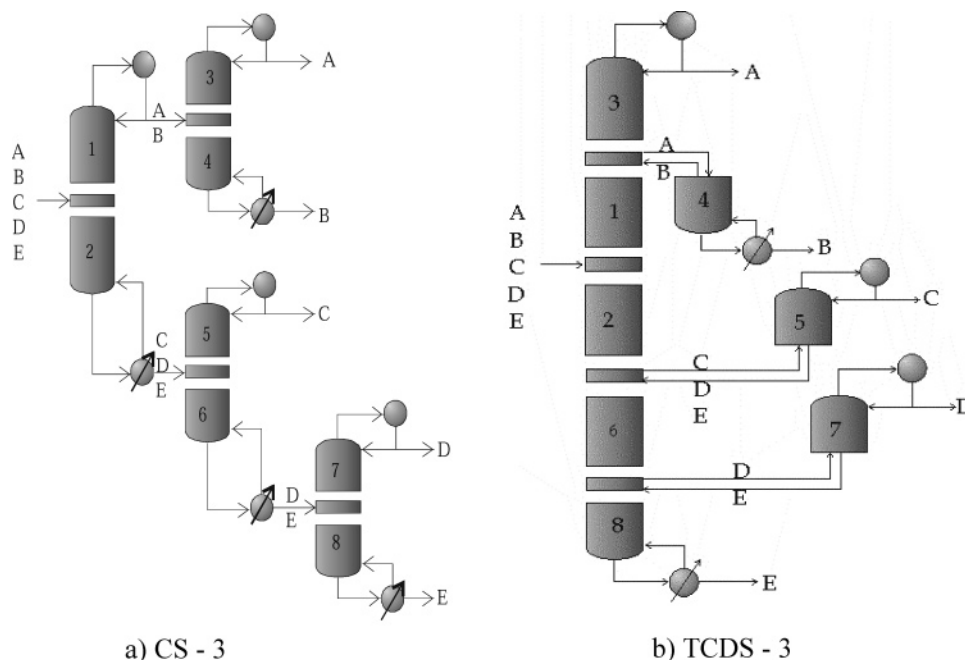


Figure 3. Thermally coupled sequence (TCDS-3) and the conventional arrangement where it is obtained.

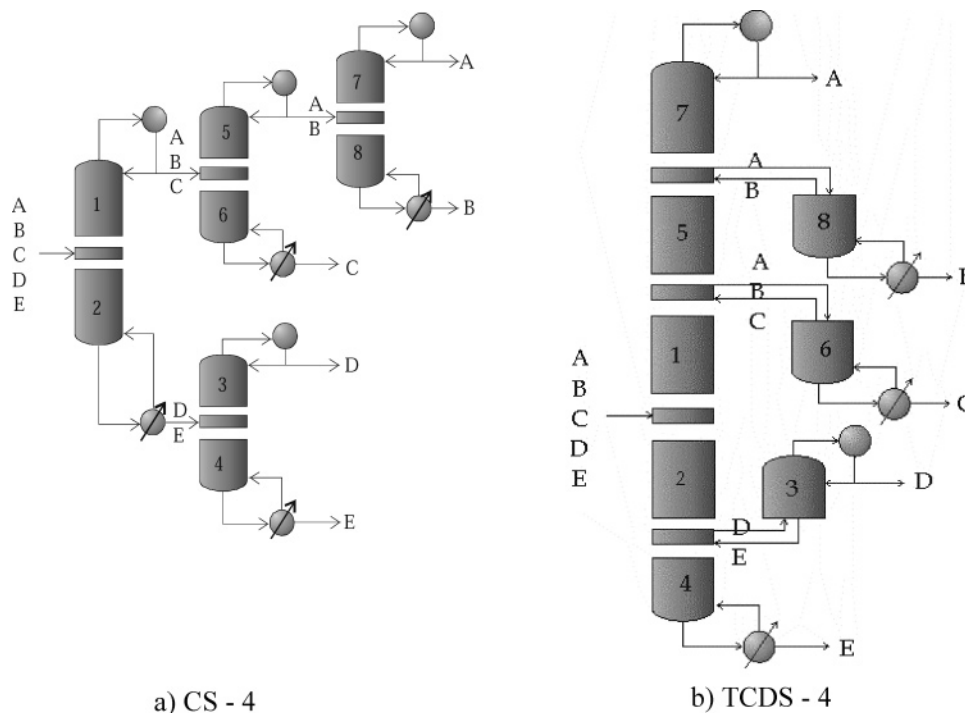


Figure 4. Thermally coupled sequence (TCDS-4) and the conventional arrangement where it is obtained.

In the conventional sequences, each column performs its respective split (i.e., the separation of the light and heavy key components) with a recovery of 98%. Using the well-known shortcut methods of Fenske-Underwood and Gilliland, the tray structure of conventional distillations schemes was obtained. The number of the trays was obtained using a reflux ratio of 1.33 times the minimum value for each separation.

The conventional distillation sequences (Figures 1–7) show eight different tray sections. These sections are used as a basis for the arrangement of the tray structure of the coupled schemes through a section analogy procedure. For instance, in the main column of the integrated sequence of Figure 1, the total number of trays is obtained by conceptually moving stripper sections from the second to fourth columns of the conventional sequence

to the bottom of the first column. The reboiler of the first column is replaced by a vapor–liquid interconnection with a side rectifier. The number of trays in the side rectifiers of the complex scheme is equal to the number of trays in the rectifier zone in the second, third, and fourth columns (in the conventional arrangement), respectively. Two more vapor–liquid interconnections are used to get the coupling scheme. A similar procedure is applied to obtain the other thermally coupled schemes. The complete design and optimization procedures are reported in detail in the work by Calzon-McConville et al.<sup>30</sup>

After the tray arrangements for the integrated designs have been obtained, an optimization procedure is used to minimize the heat duty supplied to the reboilers of each coupled scheme, taking into account constraints imposed by the required purity

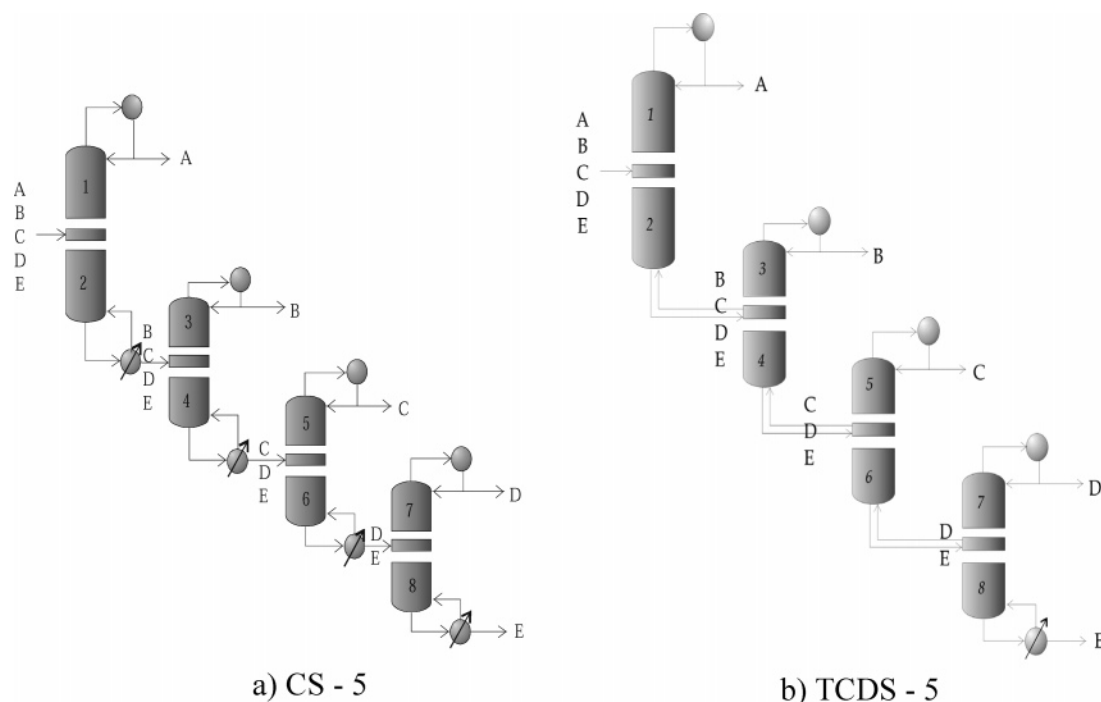


Figure 5. Thermally coupled sequence (TCDS-5) and the conventional arrangement where it is obtained.

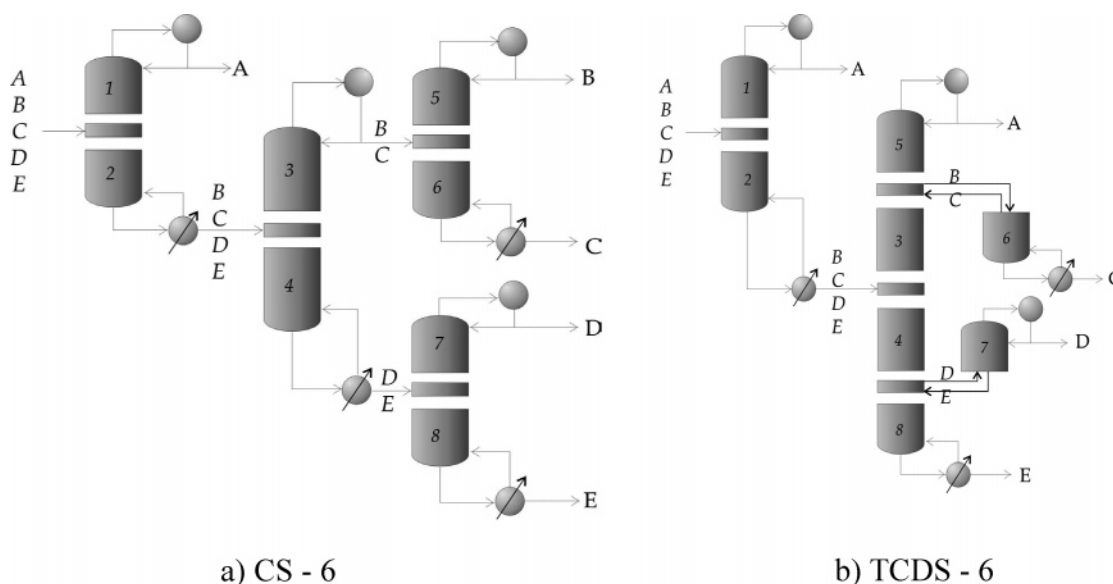


Figure 6. Thermally coupled sequence (TCDS-6) and the conventional arrangement where it is obtained.

of the five product streams. Although the number of trays is not formally optimized, a parametric analysis can be carried out to test different tray arrangements by changing the recoveries of the key components. Depending on the recovery of the key components, the number of trays changes. Because the resulting sections serve as a basis for the tray arrangements of the complex scheme, such a procedure allows for the comparison of different designs to detect the design with superior performance in terms of energy consumption. In practice, the procedure is limited by the number of tray arrangements that the designer decides to consider.

Then, the degrees of freedom that remain, after design specifications and tray arrangement are used to obtain the operating conditions, are used to get the integrated designs that provide minimum energy consumption. Three or two degrees of freedom (see Figures 1–7) remain for each integrated sequence. They are the interconnecting flows (vapor or liquid,

depending on the scheme). The search procedure provides the optimal values of the interconnecting flow to minimize the energy consumption for the separation. The design is successful if it meets the product specifications.

### 3. Theoretical Control Properties

Open-loop dynamic responses to step changes around the assumed operating point (which corresponds to that with minimum energy consumption for each configuration) were obtained. The responses were obtained through the use of Aspen Dynamics 11.1. Transfer function matrices (**G**) were then collected for each case, and they were subjected to singular value decomposition (SVD):

$$\mathbf{G} = \mathbf{V}\mathbf{\Sigma}\mathbf{W}^H \quad (1)$$

where  $\mathbf{\Sigma} = \text{diag}(\sigma_1, \dots, \sigma_n)$  and  $\sigma_i$  = singular value of **G** =

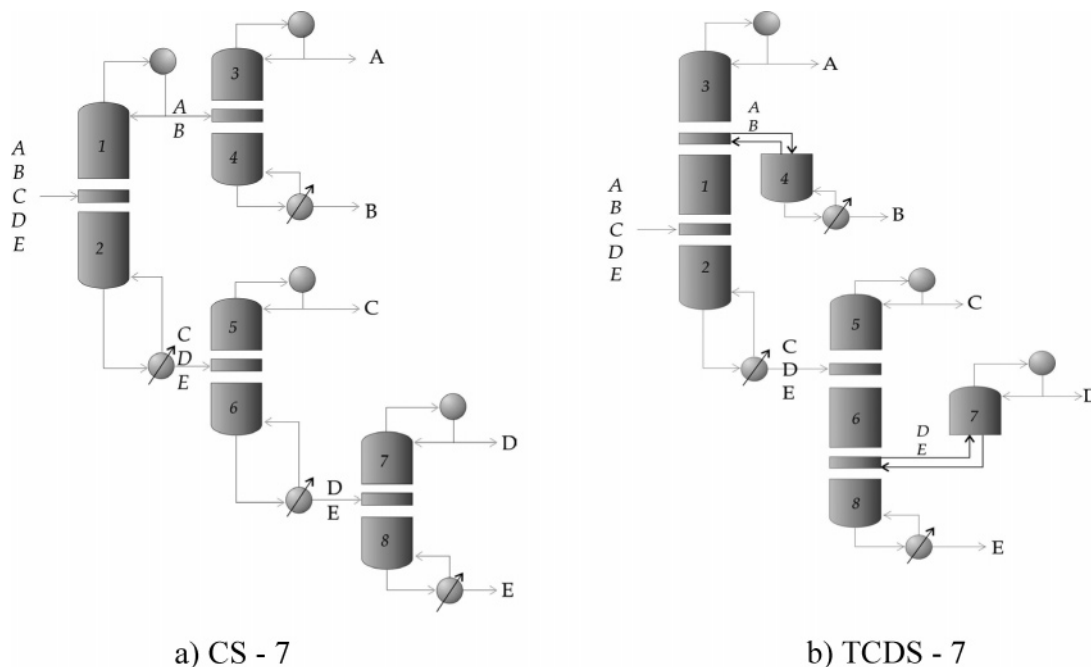


Figure 7. Thermally coupled sequence (TCDS-7) and the conventional arrangement where it is obtained.

$\lambda_i^{1/2}(\mathbf{G}\mathbf{G}^H)$ ,  $\mathbf{V} = (v_1, v_2, \dots)$  matrix of left singular vectors, and  $\mathbf{W} = (w_1, w_2, \dots)$  matrix of right singular vectors. Two parameters of interest are the minimum singular value,  $\sigma_*$ , and the ratio of maximum to minimum singular values, or condition number:

$$\gamma^* = \sigma^*/\sigma_* \quad (2)$$

The minimum singular value is a measure of the invertibility of the system and represents a measure of the potential problems of the system under feedback control. The condition number reflects the sensitivity of the system under uncertainties in process parameters and modeling errors. These parameters provide a qualitative assessment of the theoretical control properties of the alternative designs. The systems with higher minimum singular values and lower condition numbers are expected to show the best dynamic performance under feedback control. A full SVD analysis should cover a sufficiently complete range of frequencies. For this initial analysis of the coupled schemes to the conventional configurations, we simply estimated the SVD properties for each separation system at zero frequency. Such analysis should give some preliminary indication of the control properties of each system around the nominal operating point. To support the theoretical control properties, dynamic simulations under closed-loop fashion were conducted.

#### 4. Case Study

The analysis presented in this work is based on the separation problem of two different five-component mixtures with molar compositions (A, B, C, D, E) equal to 0.35, 0.10, 0.10, 0.10, and 0.35 for F1 and 0.125, 0.25, 0.25, 0.25, and 0.125 for F2, to examine the effect of the content of the intermediate components, and product purities of 98, 94, 94, 94, and 97%, respectively. The two mixtures considered were *n*-butane, *n*-pentane, *n*-hexane, *n*-heptane, and *n*-octane (mixture 1), and *n*-butane, isopentane, *n*-pentane, *n*-hexane, and *n*-heptane (mixture 2). A feed flow rate of 45.5 kmol/h as saturated liquid was taken. The design pressure for each sequence was chosen such that all condensers could be operated with cooling water. To ensure a proper driving force for vapor flow in the columns,

Table 1. Sequence Design for the TCDS-1, Mixture 1 (F1)

column	parameters
main column	stages = 41 feed stage = 9 reflux ratio = 1.72 FV1 = 16.83 kmol/h FV2 = 20 kmol/h FV3 = 22.75 kmol/h pressure = 4.5 atm
side rectifier 1 (where component B is purified)	stages = 10 distillate flow rate = 4.6 kmol/h
side rectifier 2 (where component C is purified)	stages = 11 distillate flow rate = 4.45 kmol/h
side rectifier 3 (where component D is purified)	stages = 11 distillate flow rate = 4.54 kmol/h

a total pressure drop of 10 psi was considered in each distillation column. The pressure drop for a single tray is given based on the heuristics of Kister.<sup>31</sup> Since the feed involves a hydrocarbon mixture, the Chao-Seader correlation was used for the prediction of thermodynamic properties. The tray arrangements and some parameters for TCDS-1 after an optimization task for mixture 1 (F1) is given in Table 1. It is important to establish that studying a five-component mixture of hydrocarbons is a suitable example, given the applications of hydrocarbon mixtures in the petrochemical industry.<sup>32</sup> As far as energy consumption is concerned, the optimized steady-state design provides energy savings of ~35% with respect to the best energy-efficient sequence based on conventional distillation columns. During the search for the optimum energy consumption, five design specifications for product purities (A, B, C, D, E) were set in order to avoid deviations in the required purities.

#### 5. Results

The theoretical control properties of conventional and thermally coupled distillation sequences were obtained. The SVD technique requires transfer function matrices, which are generated by implementing step changes in the manipulated variables of the optimum design of the distillation sequences (base designs) and registering the dynamic responses of the five products. In this stage, the interconnecting flows were assumed



**Table 2. Minimum Singular Values and Condition Numbers for Sequences with Three Recycles in Mixture 1**

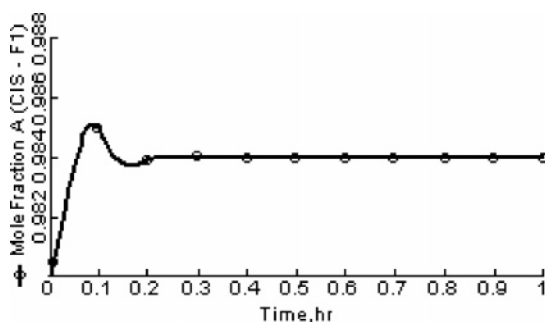
feed	sequence	$\sigma_*$	$\gamma^*$
F1	CDS-1	0.0219	562.85
	TCDS-1	0.0218	564.29
	CIS-2	0.0012	14106
	TCDS-2	0.2026	33.37
	CS-3	0.0005	33318
	TCDS-3	0.0418	576.8
	CS-4	0.0064	59934
	TCDS-4	0.0071	56650
	CS-5	0.0350	562.85
	TCDS-5	0.0001	14000
F2	CDS-1	0.0401	490.01
	TCDS-1	0.0405	488.7
	CIS-2	0.0176	109.43
	TCDS-2	0.0560	13.99
	CS-3	0.0008	10007
	TCDS-3	0.0988	689.9
	CS-4	0.0111	40098
	TCDS-4	0.0998	4765.6
	CS-5	0.0500	129.05
	TCDS-5	0.0009	9000.8

**Table 3. Minimum Singular Values and Condition Numbers for Sequences with Three Recycles in Mixture 2**

feed	sequence	$\sigma_*$	$\gamma^*$
F1	CDS-1	0.0906	600.9
	TCDS-1	0.0900	605.8
	CIS-2	0.0456	308.9
	TCDS-2	0.0908	35.05
	CS-3	0.0007	9005
	TCDS-3	0.0605	977.2
	CS-4	0.0028	43982
	TCDS-4	0.0109	1008.6
	CS-5	0.0621	908.5
	TCDS-5	0.0003	7406.21
F2	CDS-1	0.0821	375.9
	TCDS-1	0.0829	369.4
	CIS-2	0.0198	469.3
	TCDS-2	0.0576	90.5
	CS-3	0.0004	7005
	TCDS-3	0.0129	884.9
	CS-4	0.0060	28065
	TCDS-4	0.0281	2974.6
	CS-5	0.0807	1004.9
	TCDS-5	0.0004	12754.9

to be constant. In fact, during the operation manipulation of the flows is difficult, but it is important to say that, in industrial practice, the thermally coupled distillation schemes are implemented by using a single shell and dividing walls. Also, some new designs are emerging, for instance, designs that consider moving dividing walls. Open-loop and closed-loop dynamic simulations were carried out in Aspen Dynamic 11.1 in order to obtain the transfer function matrix.

**5.1. Sequences with Three Thermal Couplings.** Tables 2 and 3 give the results for the SVD test for each sequence.

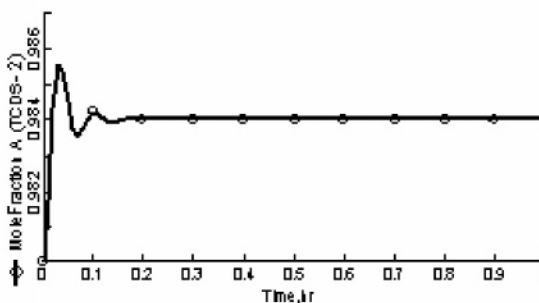
**Table 4. Minimum Singular Values and Condition Numbers for Sequences with Two Recycles in Mixture 1**

feed	sequence	$\sigma_*$	$\gamma^*$
F1	CS-6	0.00220	13588
	TCDS-6	0.00500	2630.14
	CS-7	0.0005	33318
	TCDS-7	0.00009	1973.8
F2	CS-6	0.00010	21349
	TCDS-6	0.00024	9264.46
	CS-7	0.0008	10007
	TCDS-7	0.00079	3884.01

TCDS-1 has values of minimum singular value and condition number similar to those of a conventional sequence (CS-1), which implies that the coupled arrangement has similar control properties (lower control efforts under feedback operation and better conditioned to the effect of disturbances) compared to the conventional design. TCDS-2, TCDS-3, and TCDS-4 present higher values of the minimum singular value (Table 2 and 3); therefore, it can be expected that these coupled systems exhibit better control properties than the conventional sequences under feedback control. The results for the condition number show that the complex sequences offer the best values (Tables 2 and 3). As a result, it can be expected that thermally coupled distillation systems are better conditioned to the effect of disturbances than the conventional arrangements.

One detail is worth highlighting: TCDS-1 and TCDS-5 are thermodynamically equivalent schemes. A simple column has two sections—the rectifying and stripping sections. Based on the function of each of the column sections in a complex distillation schemes (i.e., either rectifying sections or stripping sections), a complex scheme can be converted into a sequence in which each unit has only one rectifying column section and one stripping column section. Then the connections of the units are determined according to the interconnections of their streams. Thus, for the TCDS-1 scheme, TCDS-5 could be obtained. That converted sequence is a thermodynamically equivalent scheme of the corresponding TCDS-1.<sup>5</sup> The results (Tables 2 and 3) show that TCDS-5 has the worst control properties in comparison with TCDS-1 to TCDS-4. In this case the conventional scheme (CS-5) has better values in the minimum singular value and condition number. This result is important because two thermodynamically equivalent schemes have different dynamic properties. In other words, the structure has a different effect on dynamic performance for a specific separation.

**5.2. Sequences with Two Thermal Couplings.** For the case of structures with reduction in the number of thermal couplings, the results are presented in Table 4. In both cases analyzed the complex schemes show higher values of the minimum singular value and offer the best values in the condition number. Therefore, it can be expected that these coupled systems exhibit better control properties than the conventional sequences under

**Figure 8.** Dynamic responses of component A for a positive set point change (feed composition F1 for the distillation sequences of Figure 2).

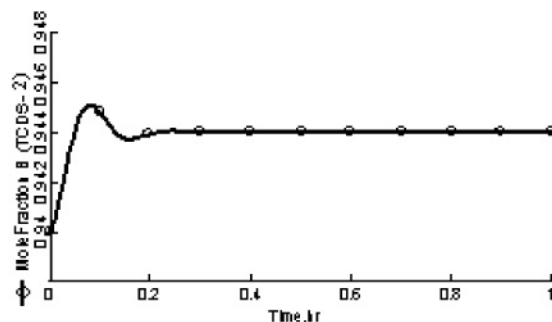
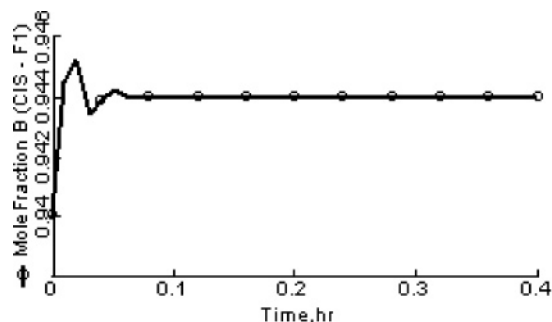


Figure 9. Dynamic responses of component B for a positive set point change (feed composition F1 for the distillation sequences of Figure 2).

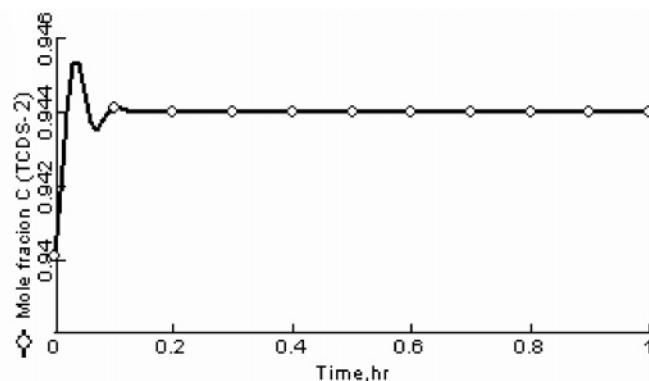
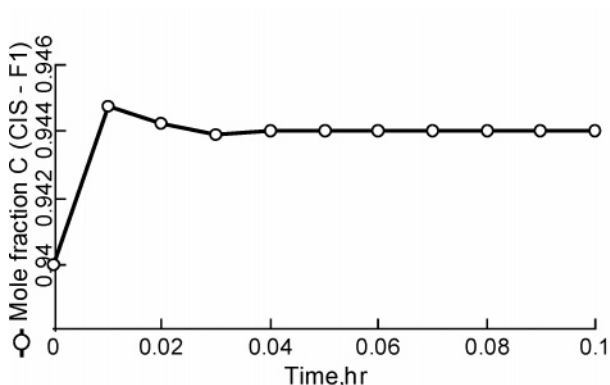


Figure 10. Dynamic responses of component C for a positive set point change (feed composition F1 for the distillation sequences of Figure 2).

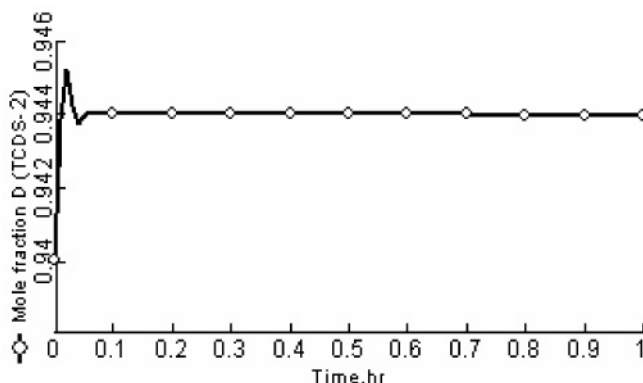
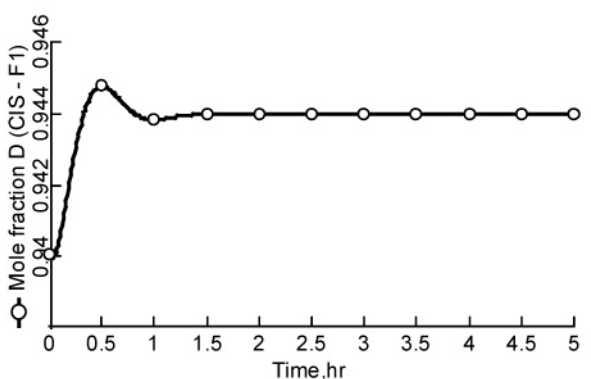


Figure 11. Dynamic responses of component D for a positive set point change (feed composition F1 for the distillation sequences of Figure 2).

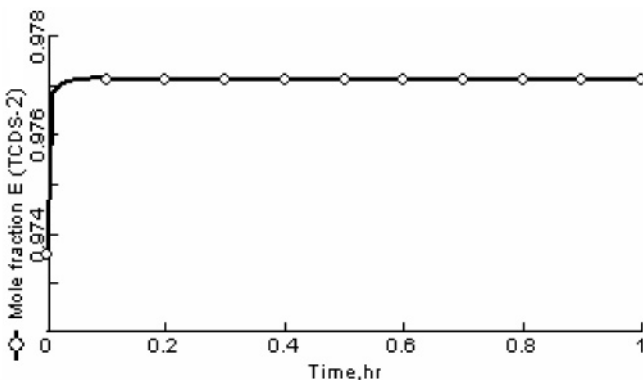
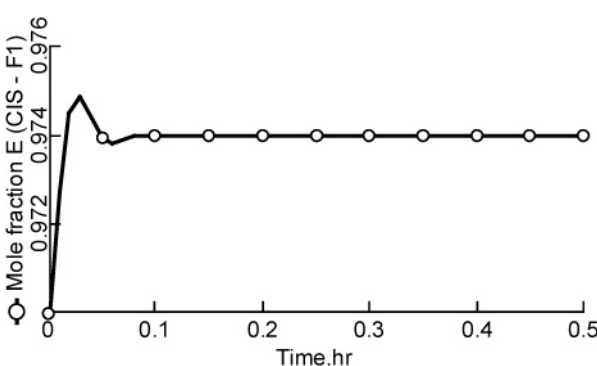


Figure 12. Dynamic responses of component E for a positive set point change (feed composition F1 for the distillation sequences of Figure 2).

feedback control, and it can be expected that these systems are better conditioned to the effect of disturbances than the conventional arrangements.

Some remarks on thermally coupled structures with two recycles can be made. Some authors<sup>11,33</sup> have claimed that the reduction of interconnection flows in complex arrangements might provide better operating properties. For example, TCDS-7

and TCDS-3 are produced from the same conventional sequence, but one structure has two recycles and the other has three recycles. The scheme with more recycles shows better dynamic properties. In other words, the topology of the scheme has a different effect on the control properties for a specific separation. When we do a general comparison of control properties of the seven coupled schemes (Tables 2–4), the thermally coupled

schemes with two recycles (TCDS-6 and TCDS-7) show worse values (minimum singular value and condition number) than the thermally coupled arrangements with three recycles (TCDS-1 to TCDS-4). This observation is interesting because the number of the thermal links affects the control properties of coupled systems. In this case, the control properties have been improved with the presence of three recycles.

### 5.3. Dynamic Responses under Closed-Loop Operation.

In the second part of the study, dynamic responses under the action of proportional integral (PI) controllers were obtained for changes in the set points. According to previous studies in thermally coupled distillation sequences,<sup>23,29</sup> TCDS options can have good dynamic responses in comparison to those obtained in conventional distillation sequences considering heuristic pairings in the control loops, i.e., distillation composition–reflux rate, bottoms composition–reboiler heat duty, and side stream purity–side product flow rate. Also, they reported that some control-loop pairings obtained by using the relative gain array provided some minor improvements in the dynamic responses.

The relative gain arrays were obtained for the distillation sequences under study, and many control-loop pairings were obtained. For instance, for the distillation sequence shown in Figure 2b, the relative gain array for the feed composition F2 is indicated in matrix 3.

$$\Lambda_{F2} = \begin{bmatrix} 0.7978 & -0.0031 & 0.0143 & 0.0434 & 0.0415 \\ -0.0011 & 0.7259 & -0.0725 & -0.0044 & 0.0007 \\ 0.3273 & -0.0387 & 0.1827 & -0.1944 & -0.0652 \\ 1.7198 & 0.0078 & -0.6594 & 3.6476 & -0.4217 \\ -2.1376 & -0.0246 & 1.1562 & 0.9552 & 1.2863 \end{bmatrix} \quad (3)$$

There are five controlled variables (compositions A, B, C, D, and E) and five manipulated variables (one reflux rate and four heat duties supplied to the reboilers). In the first row, we can see that composition A should be controlled by manipulating the reflux rate of the main column; the second row of the relative gain array suggests that a good pairing is composition B and the heat duty supplied to the reboiler of the first side stripper; in the third row the pairing obtained is not allowed since the reflux ratio of the first columns has been tied to the distillate composition, as a result, the control loop for composition C should be made by manipulating the heat duty required in the second side stripper; the fourth row indicates a pairing between the composition D and the heat duty of the third side stripper; finally, the last row indicates that the bottoms composition of the main column should be controlled by manipulating the heat duty of the reboiler of the main column. It is important to note that the relative gain array of matrix 3 shows diagonal control-loop pairings.

In the case of feed composition F1 for the same distillation sequence, the relative gain array of matrix 4 indicates control-loop pairings very different from those obtained through the use of the heuristic rules used in the control of distillation columns.

$$\Lambda_{F1} = \begin{bmatrix} -29.438 & -0.0072 & -0.7463 & 2.0432 & 29.1466 \\ 12.6630 & 0.3539 & 0.3205 & -0.8611 & -11.4763 \\ 18.9472 & 0.7092 & 0.4991 & -1.3127 & -17.8430 \\ -1.8230 & -0.0689 & 1.0680 & 0.3271 & 1.4966 \\ 0.6513 & 0.0131 & -0.1433 & 0.8032 & -0.3241 \end{bmatrix} \quad (4)$$

Also, large relative gains are presented that indicate strong interactions. For example, in matrix 4, the first row establishes a pairing between the composition of the distillate A and the heat duty supplied to the reboiler of the main column. If we study the second row of matrix 4, the control of the composition of the second side stripper should be by manipulating the reflux rate of the main column. This of course indicates nonconventional pairings in the control loops. As a result, we tested the conventional pairings and others suggested by the relative gain array. This, of course, is a very tedious study that can be avoided by using the singular value decomposition. As a result, the dynamic responses of the distillation sequences are representative and can be used to support the theoretical control properties obtained by using the singular value decomposition technique.

Many studies can be obtained for the distillation sequences under the action of PI controllers; as a result, an illustrative example is described for a positive set point change in the product compositions. The PI controllers were tuned by minimizing the integral absolute of error (IAE). For the distillation sequences of Figure 2, the dynamic responses are depicted in Figures 8–12. For the case of component A, Figure 8 shows good dynamic responses for both distillation sequences: they reach the new steady state at the same time, but the oscillations in the integrated distillation sequence are slightly bigger than those in the conventional distillation sequence. The integrated distillation sequence gives an IAE value of  $9.5736 \times 10^{-5}$ , which is lower than that of the conventional distillation sequence ( $IAE = 1.00042 \times 10^{-4}$ ). As a result, the integrated distillation sequence presents a better dynamic response.

When a positive set point change in component B is implemented in both distillation sequences, the dynamic response of the conventional distillation sequence reaches the new steady state faster, but it presents more oscillations. Both distillation sequences present similar IAE values ( $TCDS-2 = 1.72 \times 10^{-4}$  and  $CIS-2 = 1.69 \times 10^{-4}$ ).

For component C, the dynamic response of the integrated distillation sequence presents more oscillations; however, both compositions are met at a similar time. Now, the integrated distillation sequence presents a higher IAE value ( $TCDS-2 = 8.7138 \times 10^{-5}$  and  $CIS-2 = 6.9842 \times 10^{-5}$ ).

Figures 11 and 12 present similar behavior for compositions D and E of the heat integrated distillation sequences: the new compositions are obtained in a very short time in contrast to that exhibited in the conventional distillation sequence. Also, the IAE values are very similar.

In general, the dynamic responses of TCDS options can be as good as those obtained in the conventional distillation sequences.

## 6. Conclusions

Controllability properties of seven thermally coupled distillation sequences for the separation of five-component mixtures (five thermally coupled schemes with three recycles and two thermally coupled schemes with two recycles), an issue of major importance, have been shown to be similar to those of the conventional sequences (the integrated sequences present similar or better minimum singular values and condition numbers in comparison to those obtained in the conventional distillation sequences for both mixtures analyzed). Also, in some cases the thermally coupled distillation sequences outperformed the dynamic responses of the conventional distillation sequences in closed-loop fashion. The results show that the structure has a different effect on dynamic performance for a specific separation in the case of thermodynamically equivalent schemes.



These results are significant because they let us establish that coupled schemes not only require energy demands lower than the conventional distillation sequences but also present theoretical control properties similar to those of the conventional distillation sequences used in the preliminary design of the thermally coupled distillation sequences. It is apparent that the presence of recycle streams, instead of deteriorating the dynamic behavior of separation sequences, may contribute positively to their dynamic properties.

## Acknowledgment

The authors acknowledge financial support received from PROMEP and Universidad de Guanajuato, México.

## Literature Cited

- (1) Triantafyllou, C.; Smith, R. The Design and Optimization of Fully Thermally Coupled Distillation Columns. *Trans. Inst. Chem. Eng.* **1992**, 70, 118.
- (2) Hernández, S.; Pereira-Pech, S.; Jiménez, A.; Rico-Ramírez, V. Energy Efficiency of an Indirect Thermally Coupled Distillation Sequence. *Can. J. Chem. Eng.* **2003**, 81 (5), 1087.
- (3) Tedder, D.; Rudd, D. Parametric Studies in Industrial Distillation: Part I. Design Comparisons. *AIChE J.* **1978**, 24, 303.
- (4) Glinos, K.; Malone, M. F. Optimality Regions for Complex Column Alternatives in Distillation Systems. *Chem. Eng. Res. Des.* **1988**, 66, 229.
- (5) Carlberg, N.; Westerberg, W. Temperature-Heat Diagrams for Complex Columns. 2. Underwood's Method Side Strippers and Enrichers. *Ind. Eng. Chem. Res.* **1989**, 28, 1379.
- (6) Hernández, S.; Jiménez, A. Design of Optimal Thermally-Coupled Distillation Systems Using a Dynamic Model. *Trans. Inst. Chem. Eng.* **1996**, 74, 357.
- (7) Hernández, S.; Jiménez, A. Design of Energy-Efficient Petlyuk Systems. *Comput. Chem. Eng.* **1999**, 23 (8), 1005.
- (8) Yeomans, H.; Grossmann, I. Optimal Design of Complex Distillation Columns Using Rigorous Tray-by-Tray Disjunctive Programming Models. *Ind. Eng. Chem. Res.* **2000**, 39 (11), 4326.
- (9) Rév, E.; Emtir, M.; Sztik, Z.; Mizsey, P.; Fonyó, Z. Energy Savings of Integrated and Coupled Distillation Systems. *Comput. Chem. Eng.* **2001**, 25, 119.
- (10) Flores, O. A.; Cárdenas, J. C.; Hernández, S.; Rico-Ramírez, V. Thermodynamic Analysis of Thermally Coupled Distillation Sequences. *Ind. Eng. Chem. Res.* **2003**, 42, 5940.
- (11) Agrawal, R. More Operable Fully Thermally Coupled Distillation Column Configurations for Multicomponent Distillation. *Trans. Inst. Chem. Eng.* **1999**, 77, 543.
- (12) Christiansen, A.; Skogestad, S.; Lien, K. Complex Distillation Arrangements: Extending the Petlyuk Ideas. *Comput. Chem. Eng.* **1997**, 21, S237.
- (13) Blacarte-Palacios, J. L.; Bautista-Valdés, M. N.; Hernández, S.; Rico-Ramírez, V.; Jiménez, A. Energy-Efficient Designs of Thermally Coupled Distillation Sequences for Four-Component Mixture. *Ind. Eng. Chem. Res.* **2003**, 42, 5157.
- (14) Rong, B. G.; Kraslawski, A.; Nystrom, L. The Synthesis of Thermally Coupled Distillation Flowsheets for Separations of Five-Component Mixtures. *Comput. Chem. Eng.* **2000**, 24, 247.
- (15) Rong, B. G.; Kraslawski, A.; Nystrom, L. Design and Synthesis of Multicomponent Thermally Coupled Distillation Flowsheets. *Comput. Chem. Eng.* **2001**, 25, 807.
- (16) Rong, B. G.; Kraslawski, A. Partially Thermally Coupled Distillation Systems for Multicomponent Separations. *AIChE J.* **2003**, 49, 1340.
- (17) Abdul Mutalib, M. I.; Smith, R. Operation and Control of Dividing Wall Distillation Columns. Part I: Degrees of Freedom and Dynamic Simulation. *Trans. Inst. Chem. Eng.* **1998**, 76, 308.
- (18) Hernández, S.; Jiménez, A. Controllability Analysis of Thermally Coupled Systems. *Ind. Eng. Chem. Res.* **1999**, 38 (10), 3957.
- (19) Jiménez, A.; Hernández, S.; Montoy, F. A.; Zavala-García, M. Analysis of Control Properties of Conventional and Nonconventional Distillation Sequences. *Ind. Eng. Chem. Res.* **2001**, 40 (17), 3757.
- (20) Segovia-Hernández, J. G.; Hernández, S.; Jiménez, A. Control Behaviour of Thermally Coupled Distillation Sequences. *Trans. Inst. Chem. Eng.* **2002**, 80, 783.
- (21) Segovia-Hernández, J. G.; Hernández, S.; Rico-Ramírez, V.; Jiménez, A. A Comparison of the Feedback Control Behavior between Thermally Coupled and Conventional Distillation Schemes. *Comput. Chem. Eng.* **2004**, 28, 811.
- (22) Segovia-Hernández, J. G.; Hernández, S.; Jiménez, A. Analysis of Dynamic Properties of Alternative Sequences to the Petlyuk Column. *Comput. Chem. Eng.* **2005**, 29, 1389.
- (23) Cardenas, J. C.; Hernández, S.; Gudiño-Mares, I. R.; Esparza-Hernández, F.; Irianda-Araujo, C. Y.; Domínguez-Lira, L. M. Analysis of Control Properties of Thermally Coupled Distillation Sequences for Four-Component Mixtures. *Ind. Eng. Chem. Res.* **2005**, 44, 391.
- (24) Alatiqi, I.; Luyben, W. L. Control of a Complex Sidestream Column/Stripper Distillation Configuration. *Ind. Eng. Chem. Process Des. Dev.* **1986**, 25, 762.
- (25) Kaibel, G. Distillation Columns with Vertical Partitions. *Chem. Eng. Technol.* **1987**, 10, 92.
- (26) Kaibel, G.; Schoenmakers, H. Process Synthesis and Design in Industrial Practice. *Proceedings ESCAPE-12*; Grievink, J., Schijndel, J. V., Eds.; Computer Aided Process Engineering 10; Elsevier: Amsterdam, 2002; p 9.
- (27) Schultz, M. A.; O'Brien, D. E.; Hoehn, R. K.; Luebke, C. P.; Stewart, D. G. Innovative Flowschemes using Dividing Wall Columns. *Proceedings ESCAPE-16 and PSE 2006*; Marquardt, W., Pantelides, C., Eds.; Computer Aided Process Engineering 15; Elsevier: Frankfurt, 2006; p 695.
- (28) Jiménez, A.; Hernández, S.; Montoy, F. A.; Zavala-García, M. Analysis of Control Properties of Conventional and Nonconventional Distillation Sequences. *Ind. Eng. Chem. Res.* **2001**, 40 (17), 3757.
- (29) Hernández, S.; Gudiño-Mares, I. R.; Cárdenas, J. C.; Segovia-Hernández, J. G.; Rico-Ramírez, V. A Short Note on Control Structures for Thermally Coupled Distillation Sequences for Four-Component Mixtures. *Ind. Eng. Chem. Res.* **2005**, 44 (15), 5857.
- (30) Calzon-McConville, C. J.; Rosales-Zamora, M. B.; Segovia-Hernández, J. G.; Hernández, S.; Rico-Ramírez, V. Design and Optimization of Thermally Coupled Distillation Schemes for the Separation of Multicomponent Mixtures. *Ind. Eng. Chem. Res.* **2006**, 45, 724.
- (31) Kister, H. Z. *Distillation Design*; McGraw-Hill: New York.
- (32) Harmsen, G. J. Industrial Best Practices of Conceptual Process Design. *Chem. Eng. Process.* **2004**, 43, 671.
- (33) Agrawal, R.; Fidkowski, Z. New Thermally Coupled Schemes for Ternary Distillation. *AIChE J.* **1999**, 45, 485.

Received for review May 22, 2006

Revised manuscript received November 1, 2006

Accepted November 2, 2006

IE060635S