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# Extractive Dividing Wall Column: Design and Optimization

**Cristofer Bravo-Bravo,[†] Juan Gabriel Segovia-Hernández,*[,†] Claudia Gutiérrez-Antonio,[‡] Ana Luisa Durán,[§] Adrián Bonilla-Petriciolet,[§] and Abel Briones-Ramírez[||]**

*Departamento de Ingeniería Química, División de Ciencias Naturales y Exactas, Universidad de Guanajuato, Campus Guanajuato, Noria Alta s/n, 36050, Guanajuato, Gto., México, CIATEQ, A.C., Av. del Retablo 150, Col. Fovissste, 76150, Querétaro, Querétaro, México., Departamento de Ingeniería Química, Instituto Tecnológico de Aguascalientes, Av. Adolfo López Mateos #1801 Ote. Fracc. Bonagens, 20256, Aguascalientes, Aguascalientes, México, and Exxerpro Solutions, Av. del Sol 1B Local 4B Plaza Comercial El Sol, Col. El Sol, 76113, Querétaro, Querétaro, México*

This paper proposes a novel extractive dividing wall distillation column, which has been designed using a constrained stochastic multiobjective optimization technique. The approach is based on the use of genetic algorithms to determine the design that minimizes energy consumption and total annualized cost. Several case studies are used to show the feasibility of performing extractive separations in dividing wall distillation columns. The simulation results show the effect of the main variables on the complex extractive distillation process.

## 1. Introduction

The separation of fluid mixtures can be performed using several different methods; nevertheless, distillation is still the best and most used. The separation task requires a considerable effort when mixtures consisting of several components are to be separated; the separation of a mixture of $n$ components requires $(n - 1)$ distillation columns. In spite of the high number of units required, distillation handles about 3% of total US energy consumption, over 90% of all product recovery and purification separations in the US, and over 95% of chemical industry consumption worldwide. Data from the United States Department of Energy indicate that distillation columns in the U.S. consume 5.07 million TJ per year; this is 43% of the total net installed capacity of the 439 nuclear power plants in operation worldwide.[1] It is clear that the main disadvantage of distillation columns is their high energy consumption.

Process integration is an interesting option for solving this problem, and it has been successful in reducing energy requirements, when compared to a process where all units are configured with minimal or no integration. With process integration, for example, the heat duty of a reboiler of one distillation column can be fully satisfied with the condenser heat output of another distillation column, thereby reducing the overall utility requirement. However, it may sometimes not be desirable to integrate a process because of operational problems such as the occurrence of excessive fouling in the reboiler of one of the distillation columns. Moreover, it may not be feasible to perform this distillation column integration due to heat flow limitations in the background process. In such cases, the distillation column may have to be operated in a nonintegrating mode, and opportunities to reduce energy consumption must be sought through column design and configuration. It is in this context that one explores the potential of complex column arrangements. Motivated by the large energy requirements of distillation, researchers have developed several column arrangements that can bring savings in both energy and capital cost. Any reduction in energy consumption will not only bring economic benefits but also environmental benefits in terms of reduction in fossil fuel usage and their associated emissions.[2] Reported studies reveal that the fully thermally coupled distillation system (also called the Petlyuk column) provides the maximum energy reduction in distillation columns.[3−9] In most cases, this separation scheme is implemented in the form of a dividing wall column (DWC), in which both columns are installed in a single shell. This reduces investment cost by 25%, operating cost by 35%, and space requirements by 40%, as compared to the conventional column system.[10] The savings in space requirements are due to reduction in the number of reboilers, condensers and associated equipment such as pumps, their supports, etc.[7,10,11]

A number of design and optimization methods for the Petlyuk or the DWC have been proposed by several researchers.[5,6,12−14] Despite the energy and capital advantages of DWC, its industrial application began two decades ago; the world's first DWC was established by BASF in 1985. In addition, understanding of control and operability issues has improved greatly.[15−17] Since then, many DWCs have been established worldwide, such as in Europe, South Africa, and the US.[18] Amminudin et al.[19] noted

**Table 1. Process Stream Flow of the Extractive Distillation Dividing Wall Column**

| stream | description |
|---|---|
| FEED | feed streamflow of the binary mixture to the main column (B1), lb-mol/h |
| FEED 2 | feed streamflow of the extractive agent to the main column (B1), lb-mol/h |
| DIST | top product streamflow from the main column (B1), lb-mol/h |
| SIDE | side product streamflow from postfractionator (B2), lb-mol/h |
| BOTT | bottom product streamflow from the main column (B1), lb-mol/h |
| FV1 | vapor interconnection streamflow from postfractionator (B2) to the main column (B1), lb-mol/h |
| FL1 | liquid interconnection streamflow from main column (B1) to the postfractionator (B2), lb-mol/h |
| FV2 | vapor interconnection streamflow from main column (B1) to the postfractionator (B2), lb-mol/h |
| FL2 | liquid interconnection streamflow from postfractionator (B2) to the main column (B1), lb-mol/h |

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* To whom all correspondence should be addressed. E-mail: gsegovia@quijote.ugto.mx. Phone: +52 (473) 732-0006 ext 8142.

[†] Universidad de Guanajuato.

[‡] CIATEQ.

[§] Instituto Tecnológico de Aguascalientes.

[||] Exxerpro Solutions.

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**Table 2. Mixtures Analyzed in This Study**

| mixture | feed components | extracting agent | feed flow (lb-mol/h) | feed composition (mol fraction) |
|---|---|---|---|---|
| M1 | *n*-heptane/ toluene | aniline | 400 | 0.5/0.5 |
| M2 | tetrahydrofuran/ water | 1,2-propanediol | 100 | 0.9/0.1 |
| M3 | isopropyl alcohol/ water | dimethyl sulfoxide | 100 | 0.5/0.5 |
| M4 | acetone/ water | octanoic acid | 100 | 0.5/0.5 |

the industrial acceptance and commercialization of DWCs by organizations such as BASF AG, M.W. Kellogg (together with BP, later known as BP Amoco), and Sumitomo Heavy Industries Co. together with Kyowa Yuka. Linde AG constructed the world's largest DWC for Sasol, an estimated 107 m tall and 5 m in diameter. Hence, there are better prospects for DWCs in the near future, and it might become a standard distillation configuration in chemical process industries in the next 50 years.[10] The increasing energy cost, and the concern about global warming in recent times have made the DWC an attractive alternative for reducing energy usage in chemical process industries.

On the other hand, azeotropic and low-relative volatility mixtures are commonly encountered in the fine-chemical and specialty industries, and many chemical processes depend on efficient and economical methods for their separation. These mixtures can be separated in a distillation column by altering relative volatilities or shifting the azeotropic point to a more

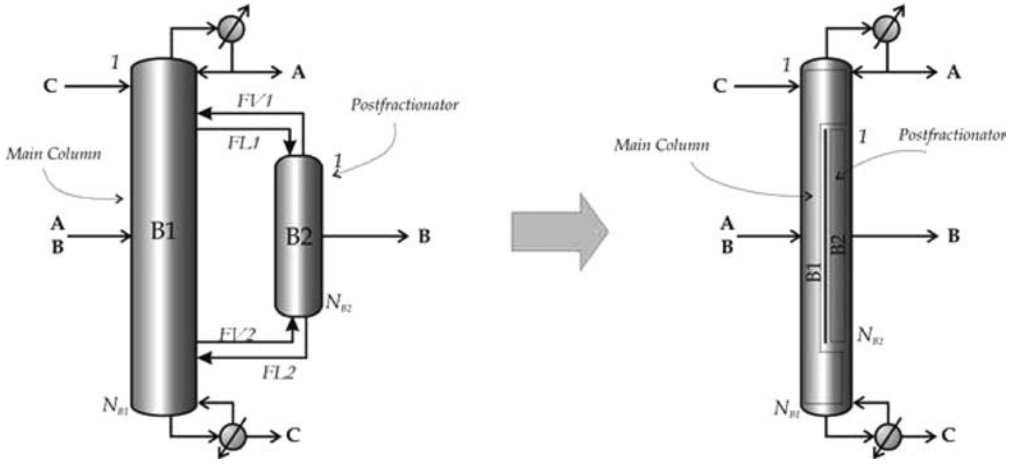


**Figure 1.** Extractive dividing wall distillation column (EDWC).

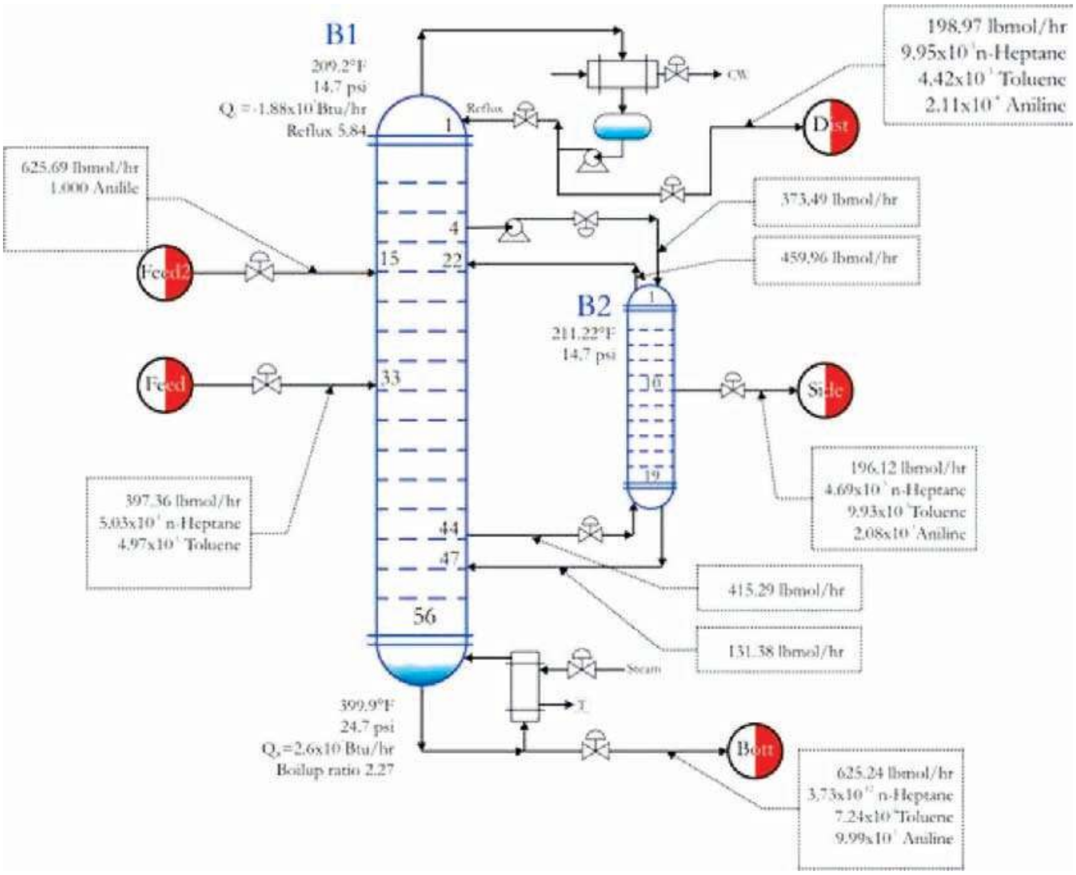


**Figure 2.** Implementation of the extractive distillation dividing wall column design in the simulator (case M1).

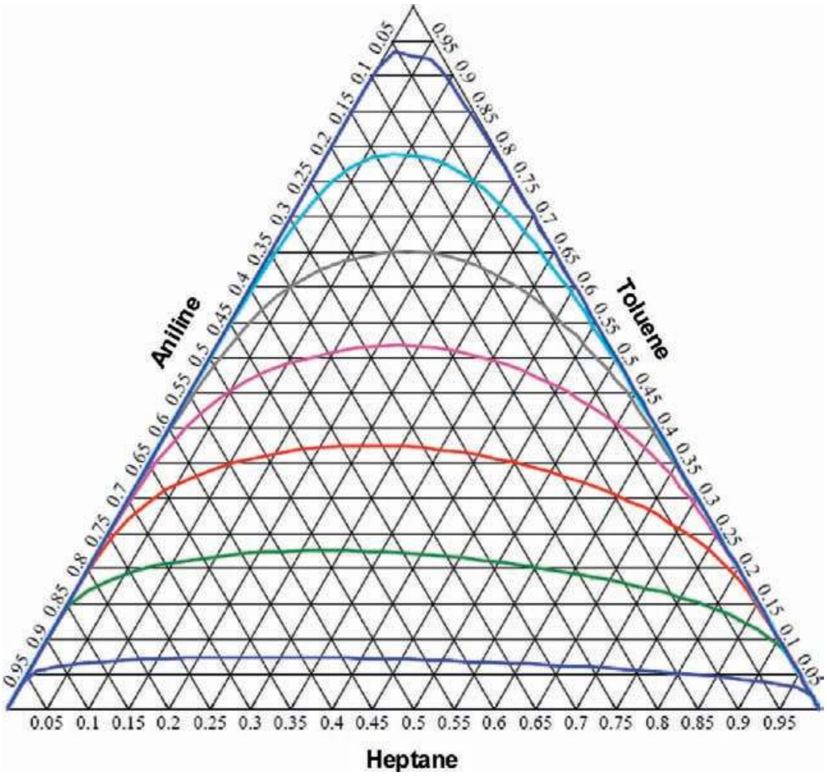


**Figure 3.** Residue curve map of mixture *n*-heptane−toluene with the extracting agent aniline.

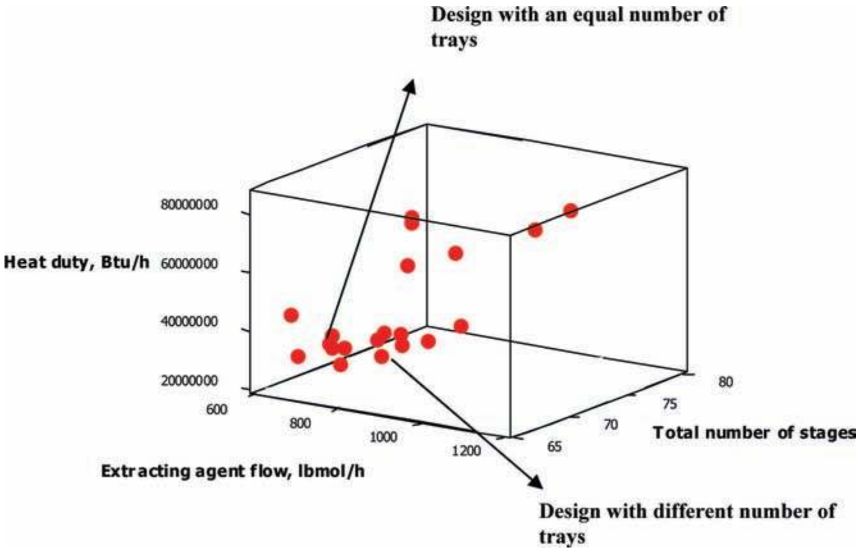


**Figure 4.** Pareto front of extractive dividing wall distillation columns for mixture M1.

favorable position. Extractive distillation is defined as distillation in the presence of a miscible, high-boiling, and relatively nonvolatile component, the solvent, which forms no azeotrope with the other components in the mixture. The method is used for mixtures having a low relative volatility value, nearing unity. Such mixtures cannot be separated by simple distillation, because the volatility of the two components in the mixture is nearly the same, causing them to evaporate at nearly the same temperature at a similar rate, making normal distillation impractical.

The method of extractive distillation uses a separation solvent, which is generally nonvolatile, has a high boiling point, and is miscible with the mixture, but does not form an azeotropic mixture. The solvent interacts differently with the components of the mixture, thereby causing their relative volatilities to change. This enables the new three-part mixture to be separated by normal distillation. The original component with the greatest volatility separates out as the top product. The bottom product consists of a mixture of the solvent and the other component, which can again be separated easily because the solvent does not form an azeotrope with it. The bottom product can be separated by any of the methods available.[20,21] It is important to select a suitable separation solvent for this type of distillation. The solvent must alter the relative volatility by a wide enough margin for a successful result. The quantity, cost, and availability of the solvent should be considered. The solvent should be easily separable from the bottom product and should not react chemically with the components or the mixture or cause corrosion in the equipment. Extractive distillation is a widely used technology in a number of different processes, such as

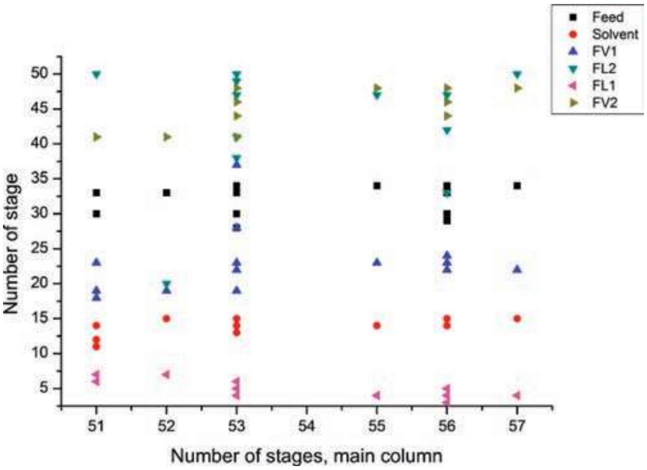


**Figure 5.** Structures of main column for all optimal designs of Pareto front, mixture M1.

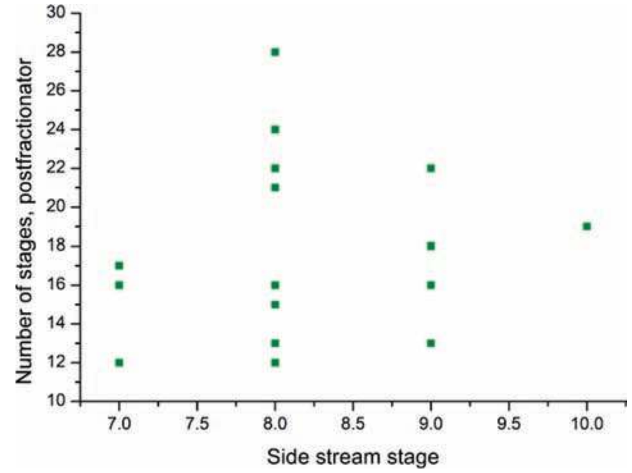


**Figure 6.** Structures of postfractionator for all optimal designs of Pareto front, mixture M1.

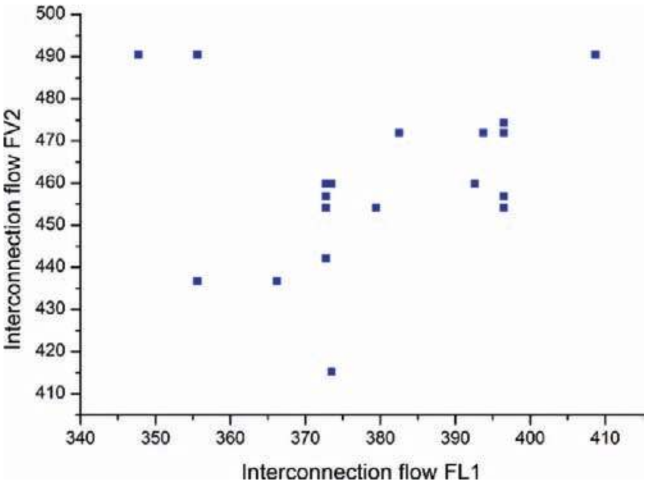


**Figure 7.** Relationship between interconnection flows of liquid and vapor, mixture M1.

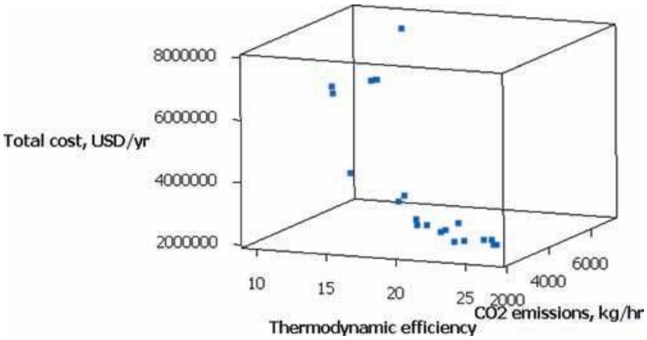


**Figure 8.** Total annual cost, $CO_2$ emissions, and thermodynamic efficiency for mixture M1.

recovery of aromas or fragrances,[22] separation of aqueous alcohol solutions,[23] mixtures that exhibit an azeotrope,[24] and separation of hydrocarbons with close boiling points.[25] Recently, the use of ionic liquids as entrainers has also been introduced in the extractive distillation of certain mixtures.[26,27]

The optimization of a complex distillation system or an extractive distillation is usually characterized as being of large problem size, since the significant number of strongly nonlinear equations results in serious difficulty in solving the model. Moreover, good initial values are needed for solving the nonlinear programming (NLP) subproblems. The search for optimal extractive distillation design has been addressed by several authors. In a recent work by Farkas et al.,[28] the original outer approximation algorithm is modified in order to provide good initial values in each iteration. Abdulfatah et al.[29] have studied the extractive distillation with mathematical programming. Application of the method is demonstrated on two different extractive processes. The optimal structures are found widely independent of the weights of different cost parts.

In 2008, Hernández[30] studied the separation of a typical mixture of ethanol and water from a fermentation process. The results show that the extractive dividing wall column can produce energy savings of ca. 30% in comparison to a conventional extractive distillation column. Also, Gutiérrez-Guerra et al.[31] have shown energy savings in an extractive coupled system with a side rectifier for separation of several azeotropic mixtures. Further studies must be done on the complex extractive distillation systems relating to design and optimization.

Despite the prior work of Hernández[30] and Gutiérrez-Guerra et al.[31] where they have shown the energy savings of thermally coupled extractive distillation configurations as compared to a conventional extractive distillation sequence, there is a lack of rigorous design methodology for this kind of structures. In this study we analyze the feasibility of separating different mixtures using an extractive dividing wall column, EDWC; see Figure 1. The design and optimization was carried out using, as a design tool, a multiobjective genetic algorithm with restrictions coupled with the process simulator Aspen Plus,[32] for the evaluation of the objective function, ensuring that all results obtained are rigorous. A main advantage of this procedure is that instead of obtaining just one optimal design, a set of optimal designs, called Pareto front, is obtained. In this context, stochastic optimization methods are playing an important role because they are generally robust numerical tools that present a reasonable computational effort in the optimization of multivariable functions; they are also applicable to unknown structure problems, requiring only calculations of the objective function, and can be used with all models without problem reformulation.[33] Moreover, a major advantage of genetic algorithms over other stochastic techniques is the availability of several multiobjective techniques such as VEGA,[34] MOGA,[35] NSGA,[36] Niched Pareto GA,[37] and NSGA-II.[38] These stochastic methods are very useful for the reliable design and optimization of chemical processes, where several decision variables are involved. To the best of our knowledge, multiobjective stochastic methods have not been reported for

**Table 3. Optimal Design of EDWC with the Lowest Total Annual Cost and Lowest Greenhouse Gas Emissions, M1**

| design parameters | lowest greenhouse gas emissions | lowest total annual cost |
|---|---|---|
| reflux ratio column B1 | 5.84 | 5.84 |
| number of stages of column B1 | 56 | 56 |
| number of stages of column B2 | 19 | 19 |
| total number of stages | 75 | 75 |
| stage of the binary mixture | 33 | 33 |
| stage of the extractive agent | 15 | 15 |
| feed streamflow of *n*-heptane (lb-mol/h) | 199.68 | 199.89 |
| feed streamflow of toluene (lb-mol/h) | 197.68 | 197.46 |
| total feed streamflow (lb-mol/h) | 397.36 | 397.36 |
| extractive agent flow aniline (lb-mol/h) | 625.69 | 625.69 |
| heat duty (Btu/h) | 26033316.30 | 26033316.30 |
| *n*-heptane recovered (lb-mol/h) | 198.97 | 198.97 |
| toluene recovered (lb-mol/h) | 196.12 | 196.12 |
| aniline recovered (lb-mol/h) | 625.24 | 625.24 |
| stage of the interconnection flow FV1 | 22 | 22 |
| stage of the interconnection flow FV2 | 44 | 44 |
| stage of the interconnection flow FL1 | 4 | 4 |
| stage of the interconnection flow FL2 | 47 | 47 |
| interconnection liquid flow FL1 (lb-mol/h) | 373.49 | 373.49 |
| interconnection vapor flow FV2 (lb-mol/h) | 415.29 | 415.29 |
| stage of the side stream | 10 | 10 |
| operating pressure of column B1 (psi) | 14.7 | 14.7 |
| operating pressure of column B2 (psi) | 14.7 | 14.7 |
| column B1 diameter (ft) | 8.48 | 8.48 |
| column B2 diameter (ft) | 4.48 | 4.48 |
| purity of the *n*-heptane recovered (%) | 99.54 | 99.54 |
| purity of the toluene recovered (%) | 99.32 | 99.32 |
| purity of the aniline recovered (%) | 99.93 | 99.93 |
| thermodynamic efficiency (%) | 23.70 | 23.70 |
| $CO_2$ emissions (lb/h) | 4692.36 | 4692.36 |
| total annual cost ($/y) | 2477452.78 | 2477452.78 |

process design of extractive dividing wall columns. The results show that this complex configuration is a feasible option in terms of energy savings (consequently, reductions in greenhouse gas emissions) and capital investment.

## 2. Optimization Strategy

For the extractive dividing wall distillation columns, the objectives of the optimization problem include minimization of total number of stages on both sides of the shell (main column and postfractionator according to Figure 1), the extracting agent flow, and the heat duty of the sequence, but constrained by the desired purities and recoveries:

$$\begin{aligned} &\min(Q_i, N_i, F_{\mathrm{EA}},) = f(R_i, N_i, N_{\mathrm{F},i}, F_{\mathrm{EA}}, F_k, N_k, F_{\mathrm{ps,o}}) \\ &\text{subject to} \qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad (1) \\ &\vec{y}_m \geq \vec{x}_m \end{aligned}$$

where $R_i$ is the reflux ratio, $N_{\mathrm{F},i}$ is the number of the feed stage, $N_i$ is the number of stages of column $i$ of the sequence, $F_{\mathrm{EA}}$ is the extracting agent flow, and $F_k$ and $N_k$ are the value and location of the interconnection flow $k$. Also, the product stream flows, $F_{\mathrm{ps,o}}$, are manipulated due to this also being required to manage the recoveries of the components along with their

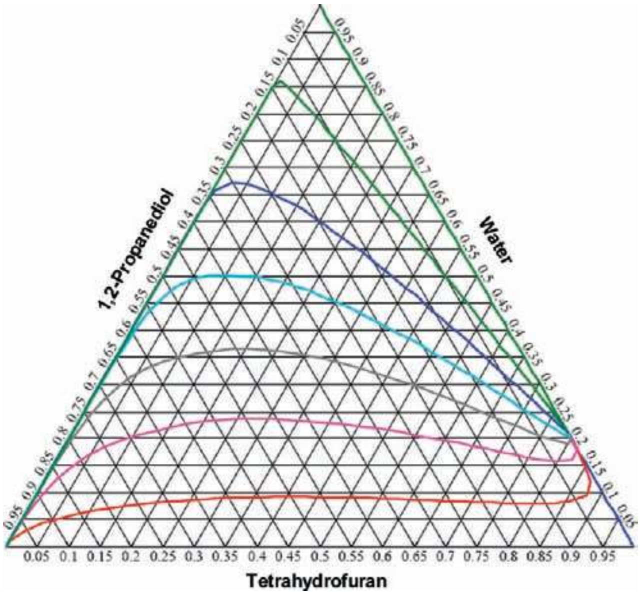


**Figure 9.** Residue curve map of mixture tetrahydrofuran−water with the extracting agent 1,2-propanediol.

**Table 4. Optimal Design of EDWC with the Lowest Total Annual Cost and Lowest Greenhouse Gas Emissions, M2**

| design parameters | lowest greenhouse gas emissions | lowest total annual cost |
|---|---|---|
| reflux ratio column B1 | 1.48 | 1.52 |
| number of stages of column B1 | 64 | 47 |
| number of stages of column B2 | 6 | 6 |
| total number of stages | 70 | 53 |
| stage of the binary mixture | 29 | 29 |
| stage of the extractive agent | 5 | 5 |
| feed streamflow of tetrahydrofuran (lb-mol/h) | 90 | 90 |
| feed streamflow of water (lb-mol/h) | 10 | 10 |
| total feed streamflow (lb-mol/h) | 100 | 100 |
| extractive agent flow 1,2-propanediol (lb-mol/h) | 153.38 | 164.53 |
| heat duty (Btu/h) | 5103626.23 | 5318971.77 |
| tetrahydrofuran recovered (lb-mol/h) | 90 | 89.99 |
| water recovered (lb-mol/h) | 8.29 | 8.75 |
| 1,2-propanediol recovered (lb-mol/h) | 153.35 | 164.47 |
| stage of the interconnection flow FV1 | 50 | 39 |
| stage of the interconnection flow FV2 | 57 | 41 |
| stage of the interconnection flow FL1 | 2 | 2 |
| stage of the interconnection flow FL2 | 31 | 46 |
| interconnection liquid flow FL1 (lb-mol/h) | 17.63 | 17.63 |
| interconnection vapor flow FV2 (lb-mol/h) | 18.54 | 31.28 |
| stage of the side stream | 4 | 3 |
| operating pressure of column B1 (psi) | 16.7 | 16.7 |
| operating pressure of column B2 (psi) | 16.7 | 16.7 |
| column B1 diameter (ft) | 2.8615 | 2.89 |
| column B2 diameter (ft) | 0.5510 | 0.72 |
| purity of the tetrahydrofuran recovered (%) | 99.00 | 99.04 |
| purity of the water recovered (%) | 99.70 | 99.35 |
| purity of the 1,2-propanodiol recovered (%) | 99.48 | 99.77 |
| thermodynamic efficiency (%) | 27.33 | 28.38 |
| $CO_2$ emissions (lb/h) | 920.56 | 962.51 |
| total annual cost ($/y) | 657839.63 | 627458.07 |

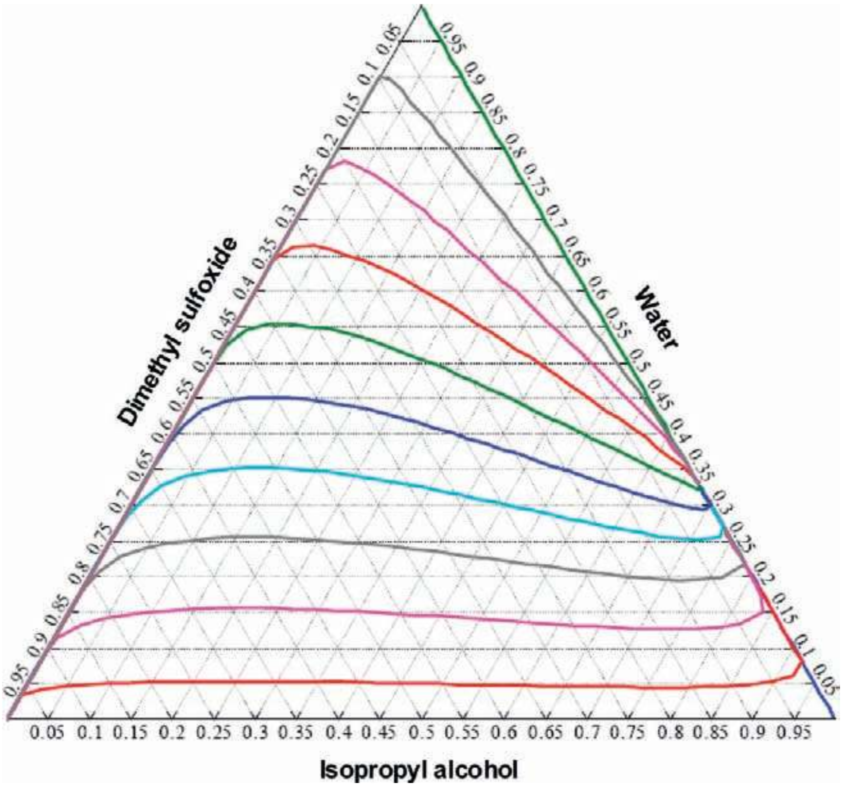


**Figure 10.** Residue curve map of mixture isopropyl alcohol−water with the extracting agent dimethyl sulfoxide.

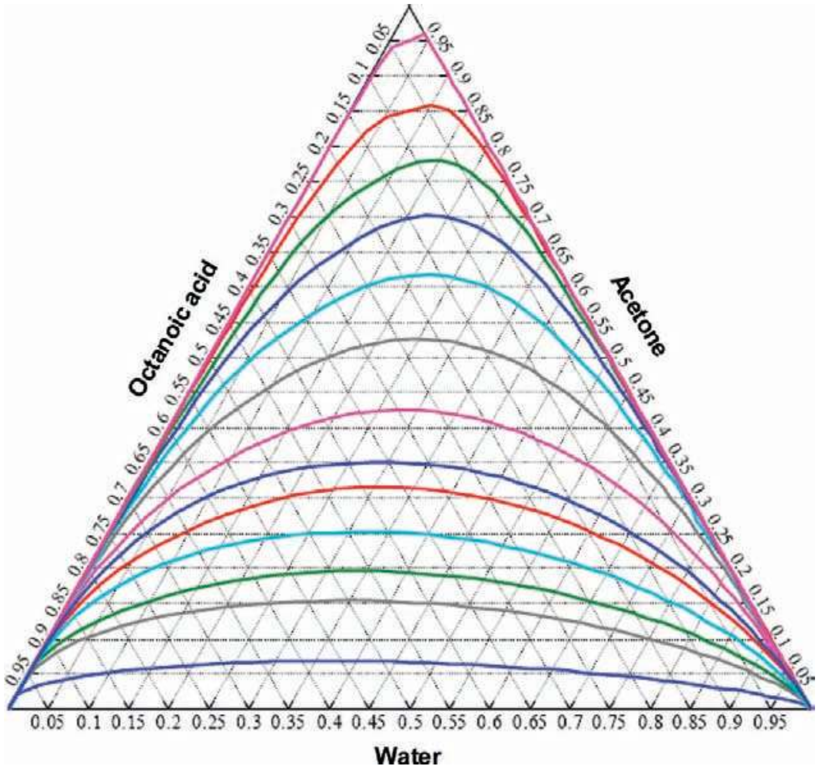


**Figure 11.** Residue curve map of mixture acetone−water with the extracting agent octanoic acid.

purities; $y_m$ and $x_m$ are the vectors of obtained and required purities and recoveries for the $m$ components, respectively.

At this point, it is important to mention that the extractive divided wall column is simulated using a main column (with a condenser and a reboiler) and a postfractionator (absorber column); see Figure 1. This is the most common way to simulate a divided wall column as seen in the works of Sotudeh and Shahrak[39] and Ling and Luyben,[40,41] among others.

In the extractive dividing wall distillation column there are four objectives to minimize: the number of stages in each side of the shell, the extracting agent flow, and the heat duty of the sequence. For these sequences the objectives are in competition,

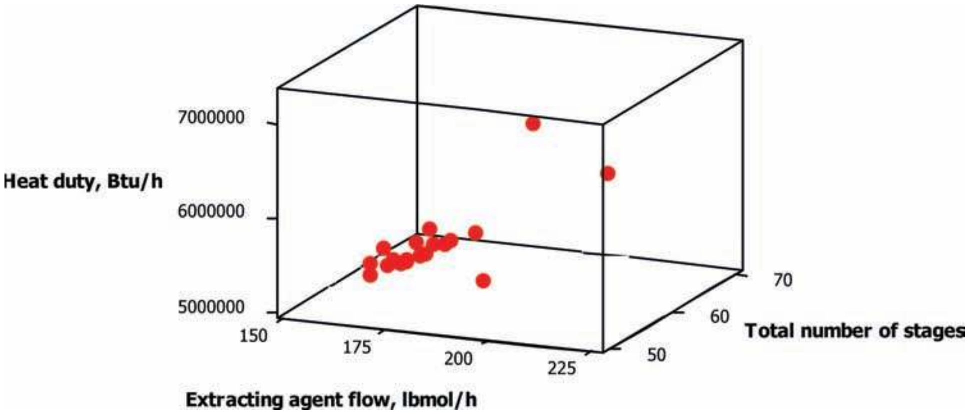


**Figure 12.** Pareto front of extractive dividing wall distillation columns for mixture M2.

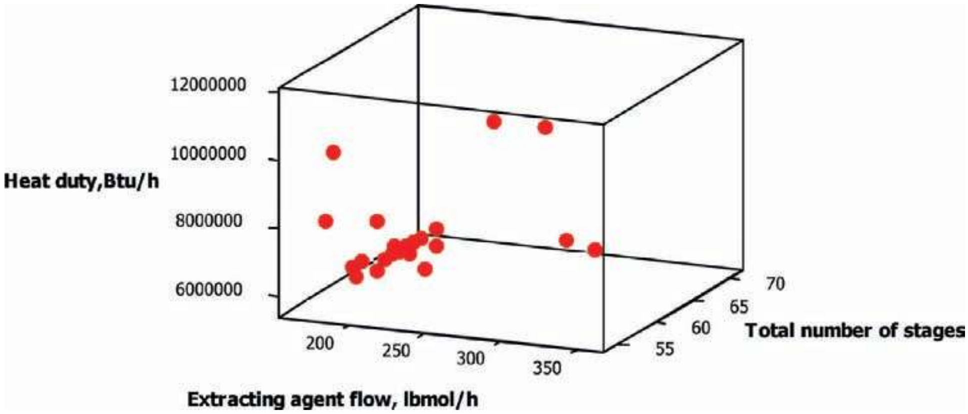


**Figure 13.** Pareto front of extractive dividing wall distillation columns for mixture M3.

so they have to be optimized simultaneously. The manipulated variables include reflux ratio, total number of stages, the stage number and value of liquid and vapor interconnection flows, product streams flows, and extracting agent flow.

The use of the multiobjective genetic algorithm with constraints allows obtaining the rigorous Pareto front of the extractive thermally coupled distillation systems: a set of nondominated, optimal, and rigorous designs that satisfied the purities required.[32] The term "nondominated" means that there is no other design that can improve one objective without worsening another one. The term "rigorous" means that all designs presented were obtained considering the complete set of MESH equations along with the phase equilibrium calculations, using the Radfrac module of Aspen Plus; in this module the dividing wall distillation column is considered a main column followed by a postfractionator—see Figure 2—with their corresponding process stream flows reported in Table 1.

The multiobjective genetic algorithm works as follows: For each run, a feasible initial design of the extractive DWC is given as initial solution to the algorithm. From this initial solution, the algorithm generates *N* individuals (i.e., new designs) to make up the initial population. The manipulated variables of each of the *N* individuals are sent to Aspen Plus to perform the simulation; then, the algorithm retrieves, from Aspen Plus, the values of objective functions and constraints for each individual. With the retrieved information, the population is divided in subpopulations according to the number of satisfied constraints; at this time, the best individuals are those that satisfy the $c$ constraints, followed by those individuals that reach $c - 1$ constraints, and so on. Inside each subpopulation, the individuals

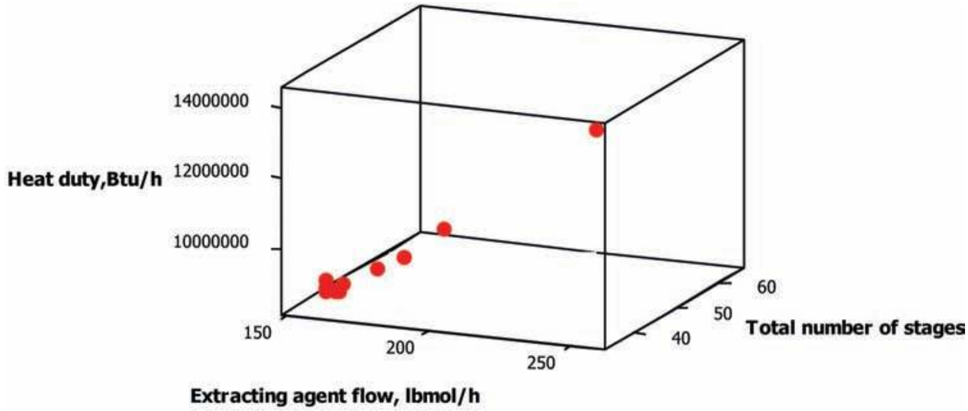


**Figure 14.** Pareto front of extractive dividing wall distillation columns for mixture M4.

**Table 5. Optimal Design of EDWC with the Lowest Total Annual Cost and Lowest Greenhouse Gas Emissions, M3**

| design parameters | lowest greenhouse gas emissions | lowest total annual cost |
|---|---|---|
| reflux ratio column B1 | 3.49 | 3.21 |
| number of stages of column B1 | 55 | 48 |
| number of stages of column B2 | 14 | 12 |
| total number of stages | 69 | 60 |
| stage of the binary mixture | 28 | 28 |
| stage of the extractive agent | 7 | 5 |
| feed streamflow of isopropyl-alcohol (lb-mol/h) | 50 | 50 |
| feed streamflow of water (lb-mol/h) | 50 | 50 |
| total feed streamflow (lb-mol/h) | 100 | 100 |
| extractive agent flow dimethyl-sulfoxide (lb-mol/h) | 169.89 | 169.89 |
| heat duty (Btu/h) | 5733134.64 | 5790483.17 |
| isopropyl-alcohol recovered (lb-mol/h) | 49.77 | 49.99 |
| water recovered (lb-mol/h) | 49.38 | 47.13 |
| dimethyl-sulfoxide recovered (lb-mol/h) | 169.88 | 167.34 |
| stage of the interconnection flow FV1 | 29 | 29 |
| stage of the interconnection flow FV2 | 37 | 38 |
| stage of the interconnection flow FL1 | 4 | 4 |
| stage of the interconnection flow FL2 | 51 | 39 |
| interconnection liquid flow FL1 (lb-mol/h) | 77.93 | 72.69 |
| interconnection vapor flow FV2 (lb-mol/h) | 144.68 | 116.43 |
| stage of the side stream | 8 | 7 |
| operating pressure of column B1 (psi) | 14.7 | 14.7 |
| operating pressure of column B2 (psi) | 14.7 | 14.7 |
| column B1 diameter (ft) | 3.44 | 3.03 |
| column B2 diameter (ft) | 1.97 | 1.63 |
| purity of the isopropyl-alcohol recovered (%) | 99.98 | 99.10 |
| purity of the water recovered (%) | 99.53 | 94.86 |
| purity of the dimethyl-sulfoxide recovered (%) | 99.65 | 98.58 |
| thermodynamic efficiency (%) | 23 | 24.44 |
| $CO_2$ emissions (lb/h) | 992.78 | 1050.12 |
| total annual cost ($/y) | 704707.98 | 682794.13 |

**Table 6. Optimal Design of EDWC with the Lowest Total Annual Cost and Lowest Greenhouse Gas Emissions, M4**

| design parameters | lowest greenhouse gas emissions | lowest total annual cost |
|---|---|---|
| reflux ratio column B1 | 1.96 | 2.07 |
| number of stages of column B1 | 42 | 32 |
| number of stages of column B2 | 11 | 7 |
| total number of stages | 53 | 39 |
| stage of the binary mixture | 13 | 9 |
| stage of the extractive agent | 7 | 6 |
| feed streamflow of water (lb-mol/h) | 50 | 50 |
| feed streamflow of acetic acid (lb-mol/h) | 50 | 50 |
| total feed streamflow (lb-mol/h) | 100 | 100 |
| extractive agent flow *n*-octanoic acid (lb-mol/h) | 161.08 | 159.12 |
| heat duty (Btu/h) | 8487165.58 | 8555275.81 |
| water recovered (lb-mol/h) | 49.56 | 49.73 |
| acetic acid recovered (lb-mol/h) | 48.41 | 48.52 |
| *n*-octanoic acid recovered (lb-mol/h) | 160.84 | 158.87 |
| stage of the interconnection flow FV1 | 21 | 18 |
| stage of the interconnection flow FV2 | 38 | 27 |
| stage of the interconnection flow FL1 | 6 | 5 |
| stage of the interconnection flow FL2 | 21 | 24 |
| interconnection liquid flow FL1 (lb-mol/h) | 46.36 | 41.72 |
| interconnection vapor flow FV2 (lb-mol/h) | 158.26 | 116.57 |
| stage of the side stream | 6 | 6 |
| operating pressure of column B1 (psi) | 14.7 | 14.7 |
| operating pressure of column B2 (psi) | 14.7 | 14.7 |
| column B1 diameter (ft) | 4.11 | 4.18 |
| column B2 diameter (ft) | 2.18 | 1.81 |
| purity of the water recovered (%) | 99.93 | 99.57 |
| purity of the acetic acid recovered (%) | 98.64 | 98.93 |
| purity of the *n*-octanoic acid recovered (%) | 99.04 | 99.21 |
| thermodynamic efficiency (%) | 50.35 | 49.70 |
| $CO_2$ emissions (lb/h) | 1798.11 | 1817.49 |
| total annual cost ($/y) | 887888.12 | 859771.52 |

are ranked based on the value of the fitness function. The classification of the population makes it possible to optimize the original objective functions, but also minimizes the difference between the required and obtained constraints (recoveries and purities). At the end, a set of nondominated optimal designs of the extractive dividing wall distillation columns are obtained. It is worthy of mention that an infinite heat duty is assigned by the algorithm to the individual where the simulation converges with errors; if the simulation does not converge, the algorithm also settles as zero the values of purities and recoveries. For more detailed information about this algorithm and its link to Aspen Plus, the reader is referred to the original work.[32]

## 3. Cases of Study

Optimal designs of the extractive dividing wall distillation columns were obtained for four binary mixtures with different extracting agents, see Table 2, using the multiobjective genetic algorithm with the aforementioned constraints. The extractive agents selected were taken from a review of previous works of extractive distillation[23,24] those papers indicate the best entrainer option for each case analyzed in this study. For the extractive dividing wall distillation sequences, we used 2500 individuals and 40 generations as parameters of the genetic algorithm, with 0.80 and 0.05 of crossover and mutation fraction. These parameters were obtained through a tuning process, where several runs of the algorithm were performed with different numbers of individuals and generations. Phase equilibrium of all mixtures is calculated with the solution model UNIQUAC. For all cases of study, the purities and recoveries were fixed at 99% for the compositions of the products and the solvent recovered. The optimum economic design of the divided wall column was based on minimizing total annual cost, which includes both energy and capital costs. The sizing relationships and economic factors are taken from the method of Guthrie.[42] The best design can present the same or different numbers of trays in the column sections on both sides of the wall. In this study, the restriction of equal number of stages in both sides of the column is relaxed in order to find the optimal design, regardless of the tray spacing on both sides of the wall. At this point, it is important to note that the dividing wall column with symmetry on both sides of the wall may not correspond to the scheme best suited to achieve separation at a lower cost of operation. In this study, the sizing on both sides of the wall is also considered as optimization variable, i.e. it is a degree of freedom. It is also convenient to remark that some previous works have considered the option of modifying the design of

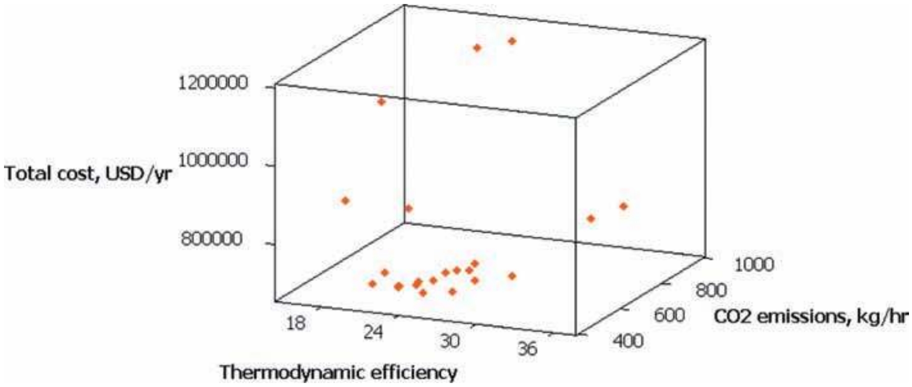


**Figure 15.** Total annual cost, $CO_2$ emissions, and thermodynamic efficiency for mixture M2.

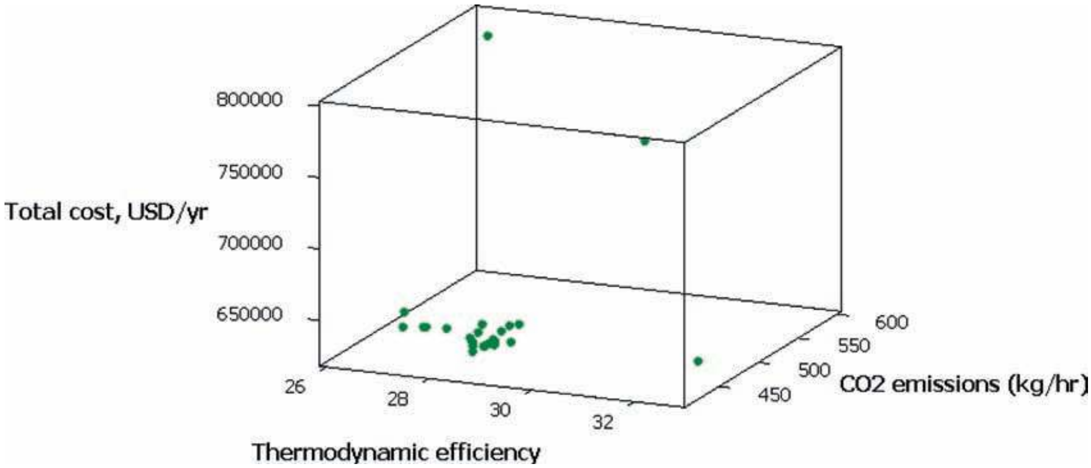


**Figure 16.** Total annual cost, $CO_2$ emissions, and thermodynamic efficiency for mixture M3.

the wall of the DWC in order to cope with some inadequacies of the conventional dividing wall columns. Specifically, these studies suggest novel wall geometrical complex structures such as the triangular wall column[43] or with special self-adjusting packing to avoid assembling problems.[44] At this point, it is worth noting that in this paper unlike the approach generally used, that of equal numbers of trays in both sides of the wall aiming to give the same pressure drop on both sides of the wall, the number of trays on either part of the DWC is not the same. From a constructive point of view, an equal number of trays would be preferable. In our analysis, we consider that the number of stages in both sides of the wall can be different; thereby, the physical implementation of the extractive dividing wall has to ensure the same cross-section in both the prefractionator part and in the side-draw region (This kind of configuration has already been realized in industrial practice. The first applications of this rather simple configuration in industrial practice occurred in 2004 with good results. Off-center positions of the dividing wall can be used in special process requirements as, e.g., high vacuum.[45]). The liquid split, defined as the ratio between the reflux ratio flow rate in the prefractionator and total reflux flow rate in the column, was adjusted in order to provide the product purities. The vapor split is free to be adjusted by imposing the same pressure drop on both sides of the extractive dividing wall column, which implied a different number of trays. In practical applications, the issue of having a nonequal number of trays can be overcome by using packing elements with different heights.[44] Nevertheless, the most common situation in the industrial implementation of the dividing wall column is assuming identical tray spacing on both sides of the wall.

Finally, the cross-sectional area of the middle section of the EDWC is calculated as a function of the area of the postfractionator and the main column (values taken as function of the diameters of extractive complex sequence simulated as Petlyuk column). Nevertheless, the EDWC should be sized for the maximum vapor load in each of the top, middle, and bottoms sections, available from rigorous optimization. Thus, in this paper, to calculate the cost, the diameter of each of the three sections of the extractive DWC shall be calculated based on vapor rate. For the middle and bottom sections, it will be usually the same as or bigger than the top section. Depending upon the change in diameter, one can choose either to use a single diameter or step diameter (top section with a smaller diameter). In the former case, the larger diameter of all the three sections should be used. Premkumar and Rangaiah[2] have presented a similar assumption for the design and costing of a DWC.

## 4. Analysis of Results

In this section, we analyze the resulting Pareto fronts of the extractive dividing wall distillation columns for the different mixtures studied. We begin with a detailed analysis of mixture 1, based on which we study the rest of the cases.

**Mixture M1.** The first mixture is made up of *n*-heptane and toluene, while aniline is used as the extracting agent. Figure 3 shows the residue curve map of this mixture at the selected operating pressure. According to the residue curve map, we observe that the separation is feasible, since there are no distillation boundaries that divide the composition space into different regions. Therefore, we calculate the Pareto front using, as design tool, the multiobjective aforementioned genetic

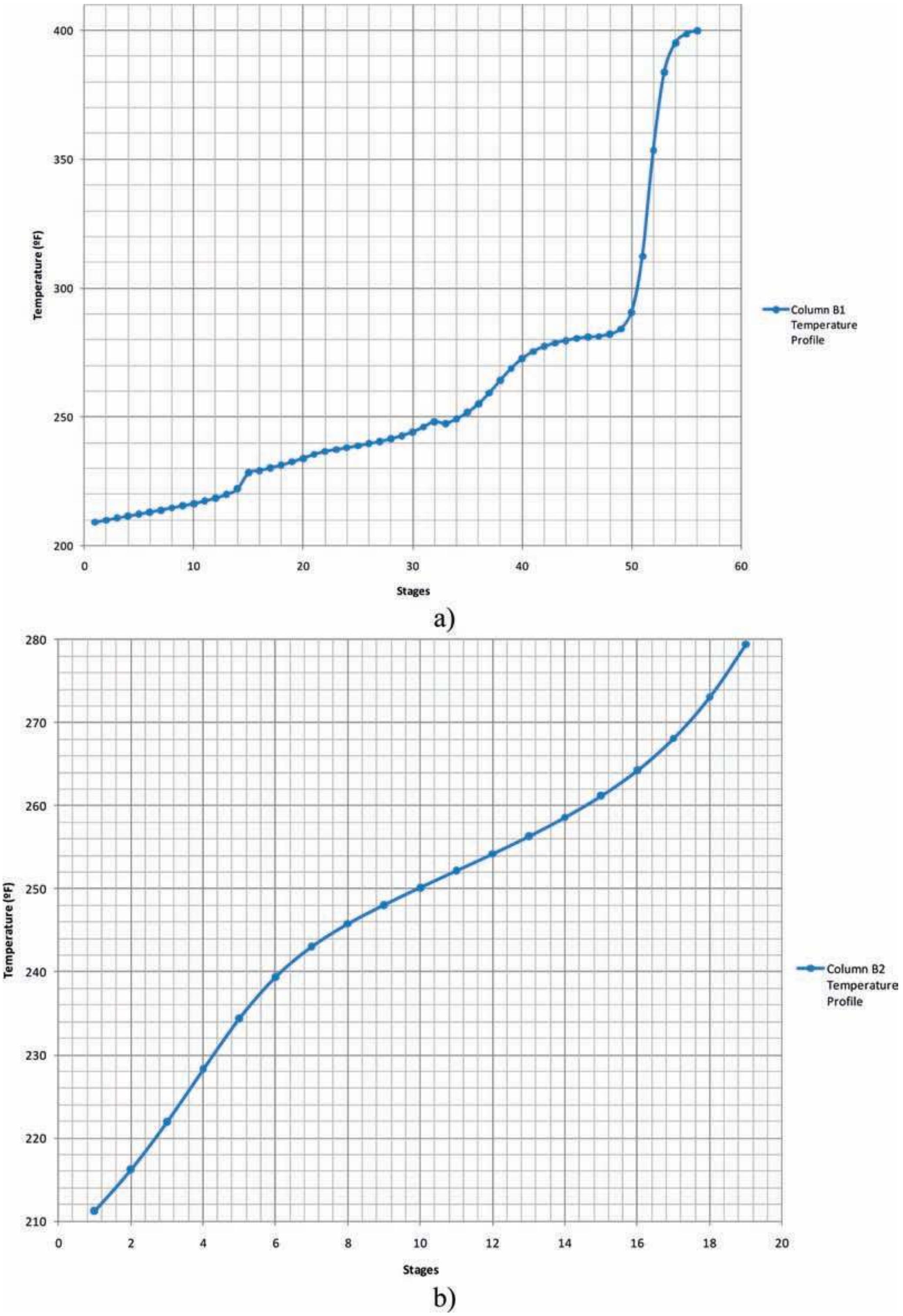


**Figure 17.** Temperature profiles for the extractive distillation dividing wall column design with the minor total annual cost, energy consumption, and carbon dioxide emissions (mixture *n*-heptane/toluene): (a) main column (B1), (b) postfractionator (B2).

algorithm. Figure 4 shows the Pareto front for mixture 1, which includes the objectives to minimize: heat duty of the sequence, extracting agent flow, and the number of stages on both sides of the shell. The first observation is that a dividing wall distillation column can perform an extractive separation; as proof of this affirmation, we can observe the 25 optimal designs that made up the Pareto front. These optimal designs satisfy the specified purities and recoveries with different structures and solvent flows, but always with the lowest energy possible. Thus, the engineer can choose the best design for his particular needs. Each design in the Pareto front is an optimal design, and this set includes designs from minimum number of stages to minimum reflux ratio, along with all designs between these extremes. Also, from this figure we can observe a good diversity in the designs that made up the Pareto front; solvent flows, number of stages, and heat duties cover a wide range of values. With illustrative purposes, Figure 4 shows the comparison of heat duties between an optimal design with an equal number of trays (i.e., the common industrial implementation of DWC) and an optimal design with different number of trays. Our results

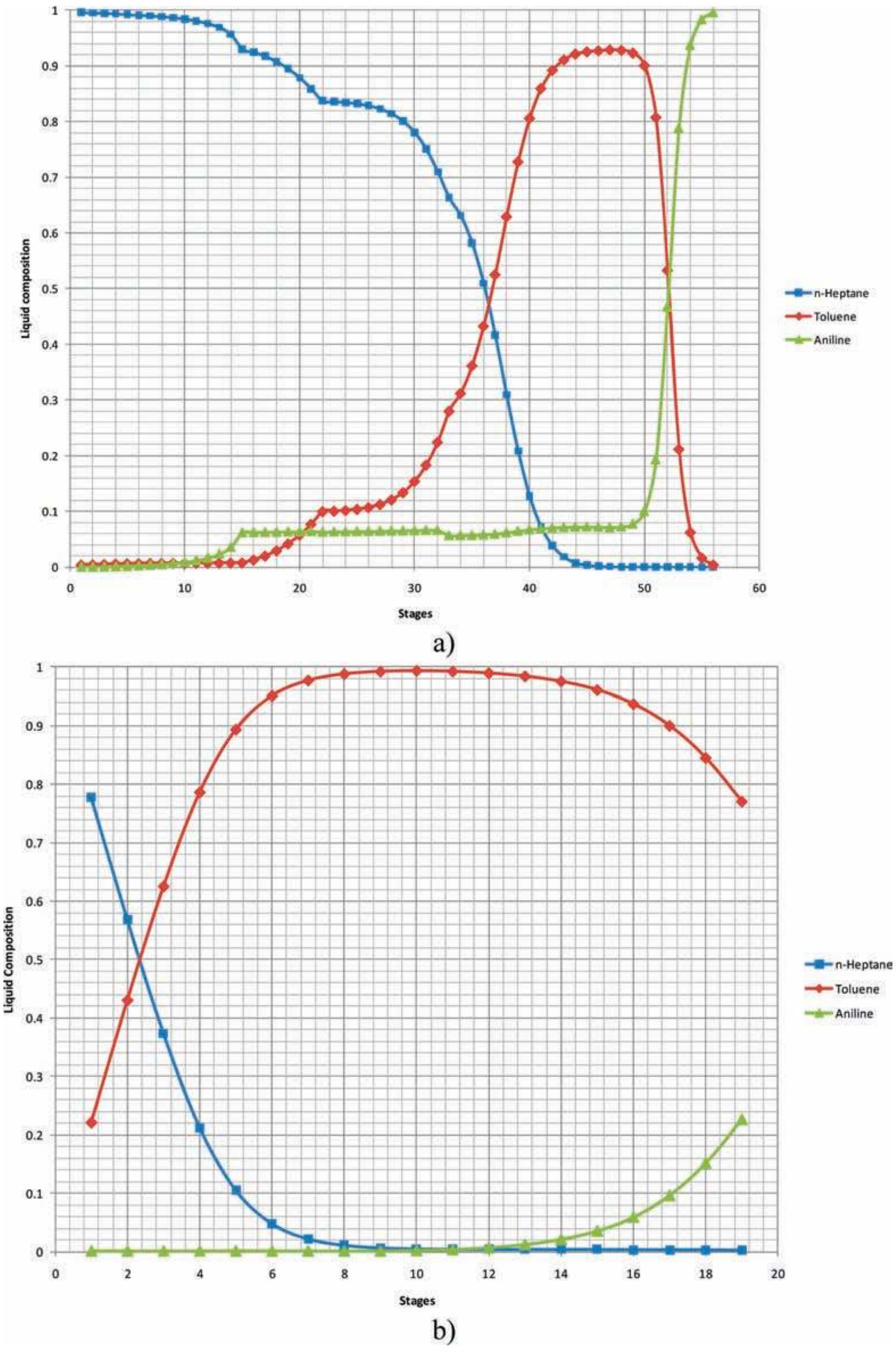


**Figure 18.** Composition profiles for the extractive distillation dividing wall column design with the minor total annual cost, energy consumption, and carbon dioxide emissions (mixture *n*-heptane/toluene): (a) main column (B1), (b) postfractionator (B2).

indicate that the optimum energy consumption design is obtained with different sizing on both sides of the wall. Similar results have been obtained for remaining cases of study considered in this paper. As stated, the classical dividing wall column with symmetry on both sides of the wall may not correspond to the best scheme with lower energy consumption. These results justify the use of sizing on both sides of the wall as the optimization variable for the optimal design of DWC.

Now, with respect to the structure of the main column of all optimal designs of the Pareto front, we can easily observe in Figure 5 that the proportions between the different stages are kept when the number of stages of the main column is increased. From this figure, we can easily observe linear relationships between the different feed and product flows; this finding concurs with that previously found in the rigorous and stochastic optimization of the Petlyuk sequence.[32] On the other hand, Figure 6 shows the structure of the postfractionator of all designs that integrated the Pareto front. We observe that, in spite of the size of the postfractionator varying considerably, the location of the side stream stage is kept nearly constant. Thus, the size

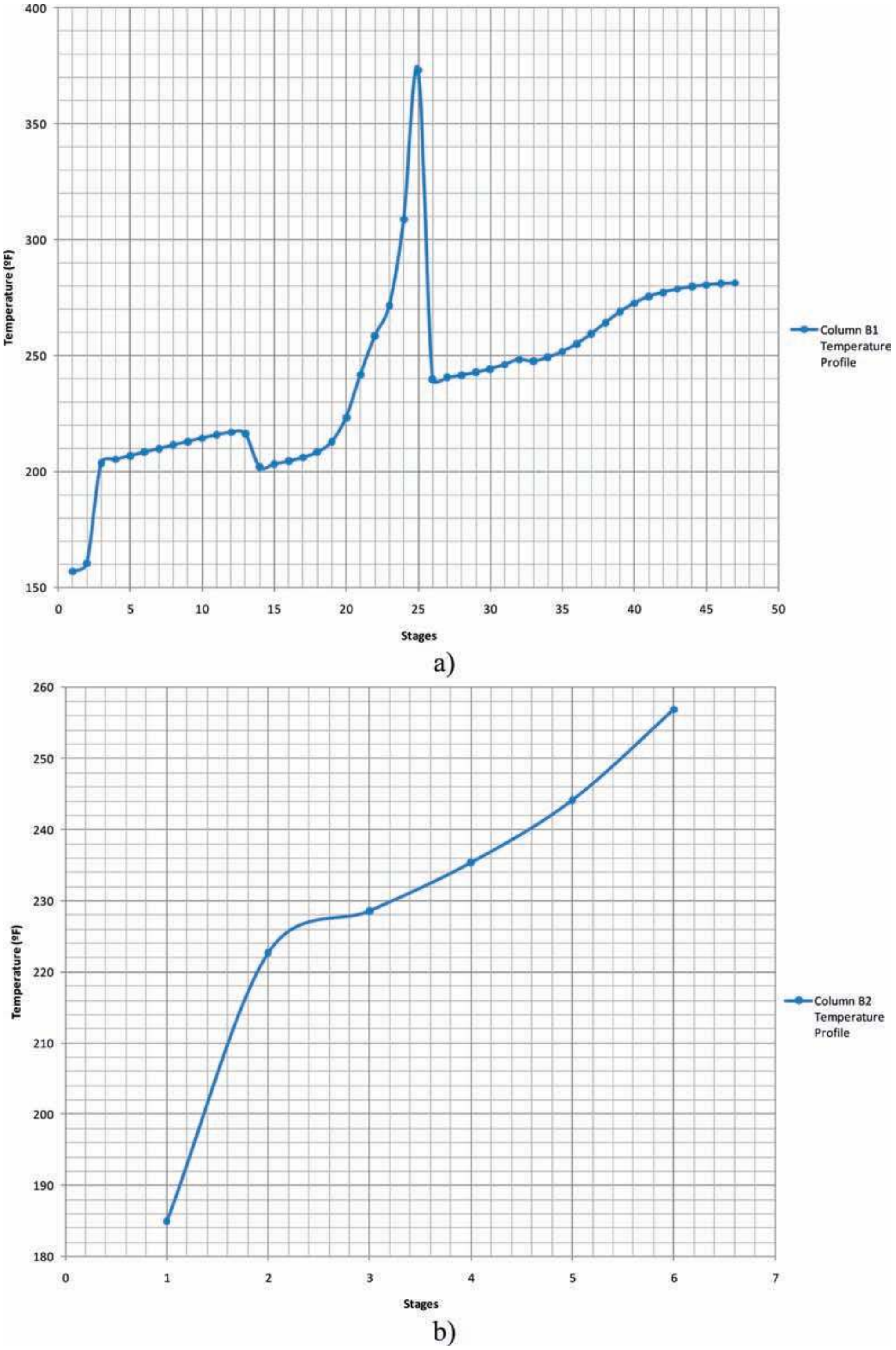


**Figure 19.** Temperature profiles for the extractive distillation dividing wall column design with the minor total annual cost, mixture tetrahydrofuran/water: (a) main column (B1), (b) postfractionator (B2).

of the postfractionator varies, but the separation performed is the same for all cases. From both figures, we observe that, in the main column (first side of the shell), the total number of stages varies around 53, with 51 and 57 as the highest and lowest values, respectively. On the other hand, in the postfractionator (second side of the shell), the number of stages varies considerably from 12 to 28; in other words, this means that the main column remains almost constant, while the Pareto front is integrated with the variations in the structure of the postfractionator. Moreover, the ratio of flows extracting agent/feed varies from 1.55 to 2.95, with 1.88 being the average ratio, which means that, in spite of all the interconnection flows, the ratio value does not increase considerably; therefore, competitive operating costs can be expected. Also, it appears that the interconnection flows of optimal designs present a linear relationship between one another, as can be appreciated in Figure 7. Finally, it was found that the optimum energy consumption design can be related to the minimum total annual operating

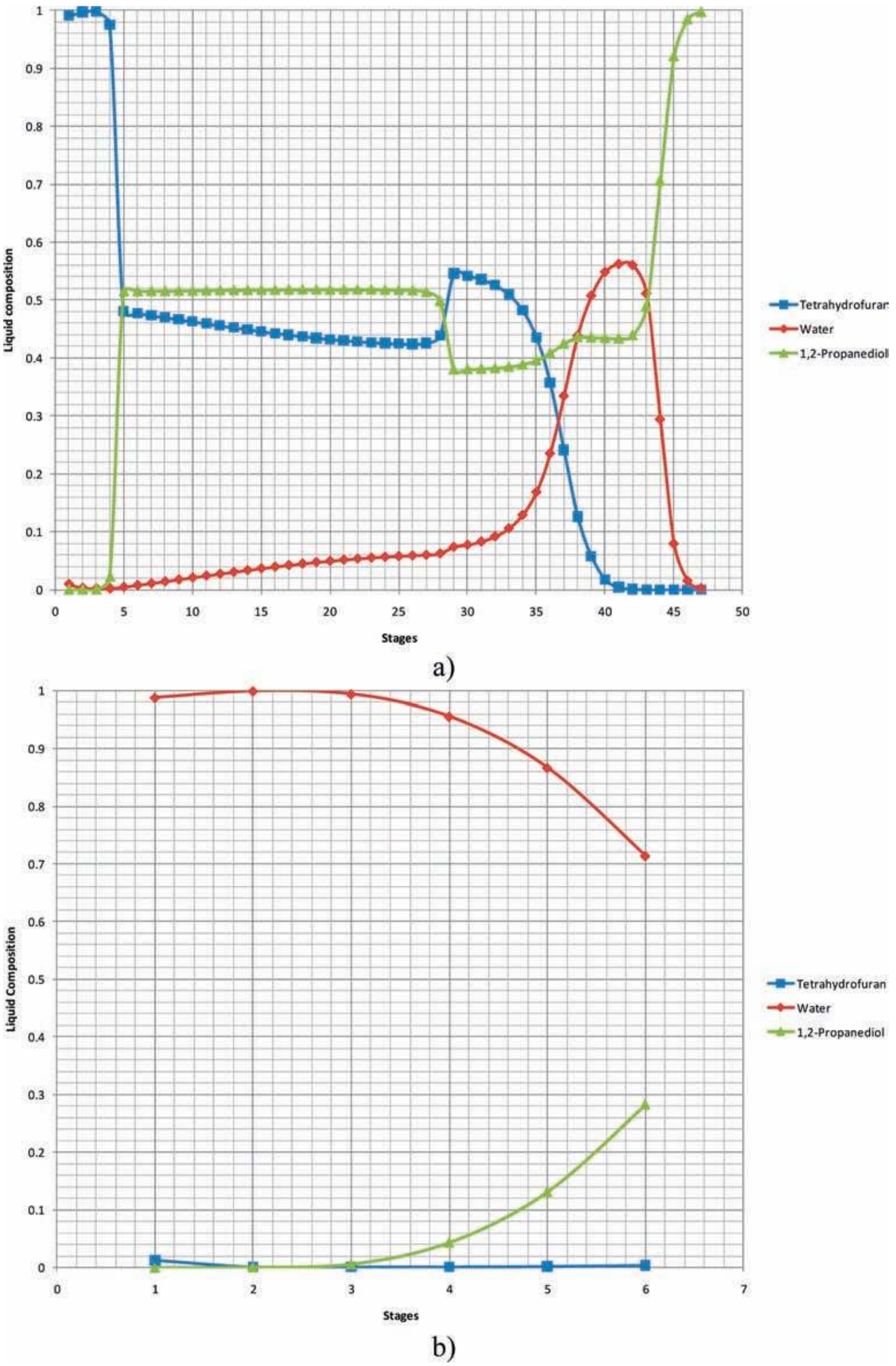


**Figure 20.** Composition profiles for the extractive distillation dividing wall column design with the minor total annual cost (mixture tetrahydrofuran/water): (a) main column (B1), (b) postfractionator (B2).

cost (calculated using the method of Guthrie[42]), minimum greenhouse gas emissions[46] and higher thermodynamic efficiencies[47] (see Figure 8). We clearly observe from this figure that, with the increase in solvent flow and total number of stages, the total annual cost also increases.

From all designs of the Pareto front, we have selected the optimal design of lowest total annual cost and the one of lowest $CO_2$ emissions. In Table 3, we can observe that, for this mixture, the optimal design represents the lowest annual cost and the lowest greenhouse gas emissions. This observation is quite important, since in the original formulation of the problem the minimization of total annual cost and greenhouse gas emissions was not considered. The thermodynamic efficiency of this sequence is 23.70%, which is slightly higher than the efficiency of a conventional extractive sequence, 21.42%; this value was obtained from an optimization of a conventional sequence only for comparative purposes. Therefore, it is important to note that the thermodynamic efficiency of the extractive coupled system is slightly higher than the conventional one, in spite of the presence of the interconnection flows.

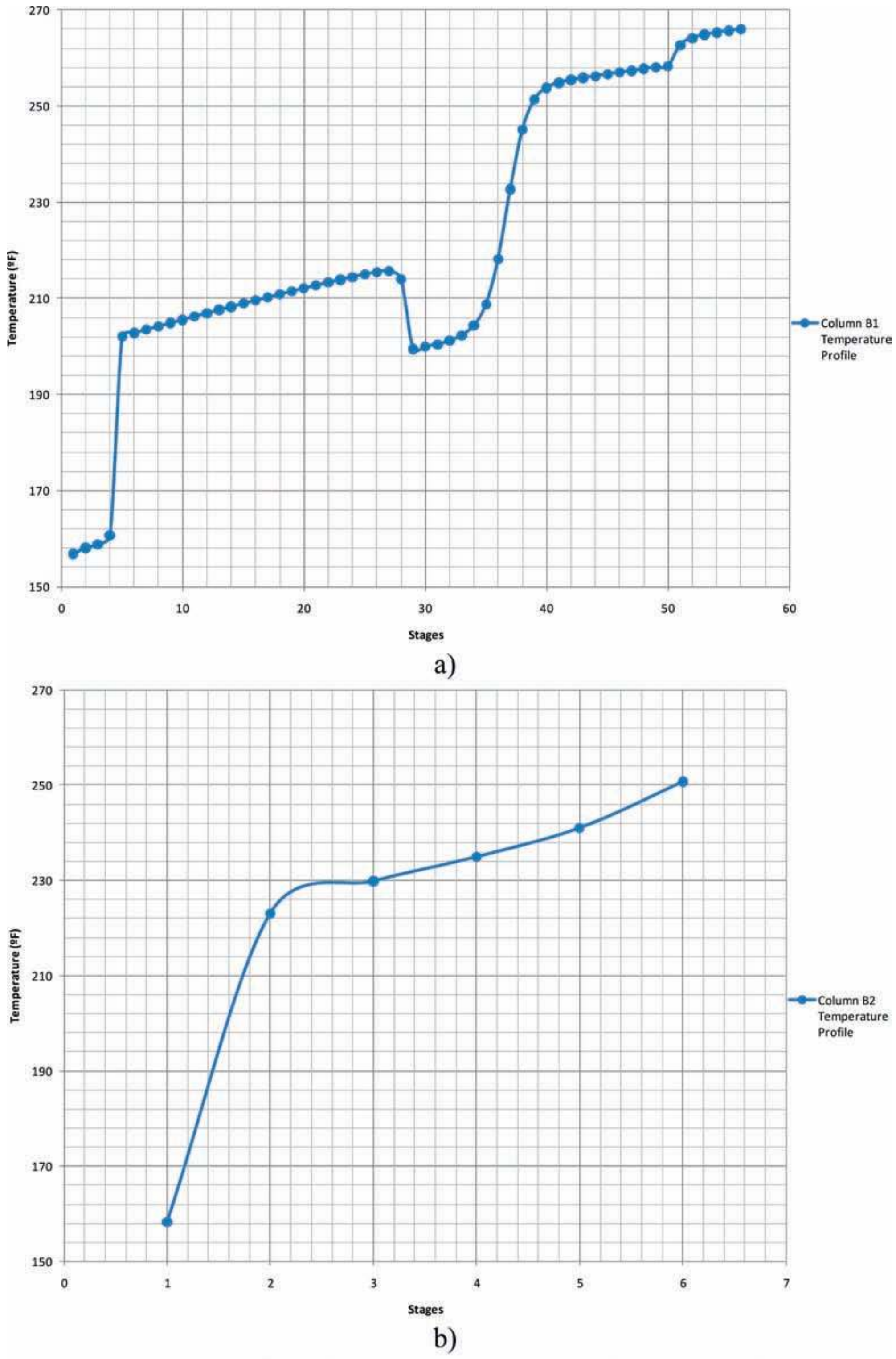
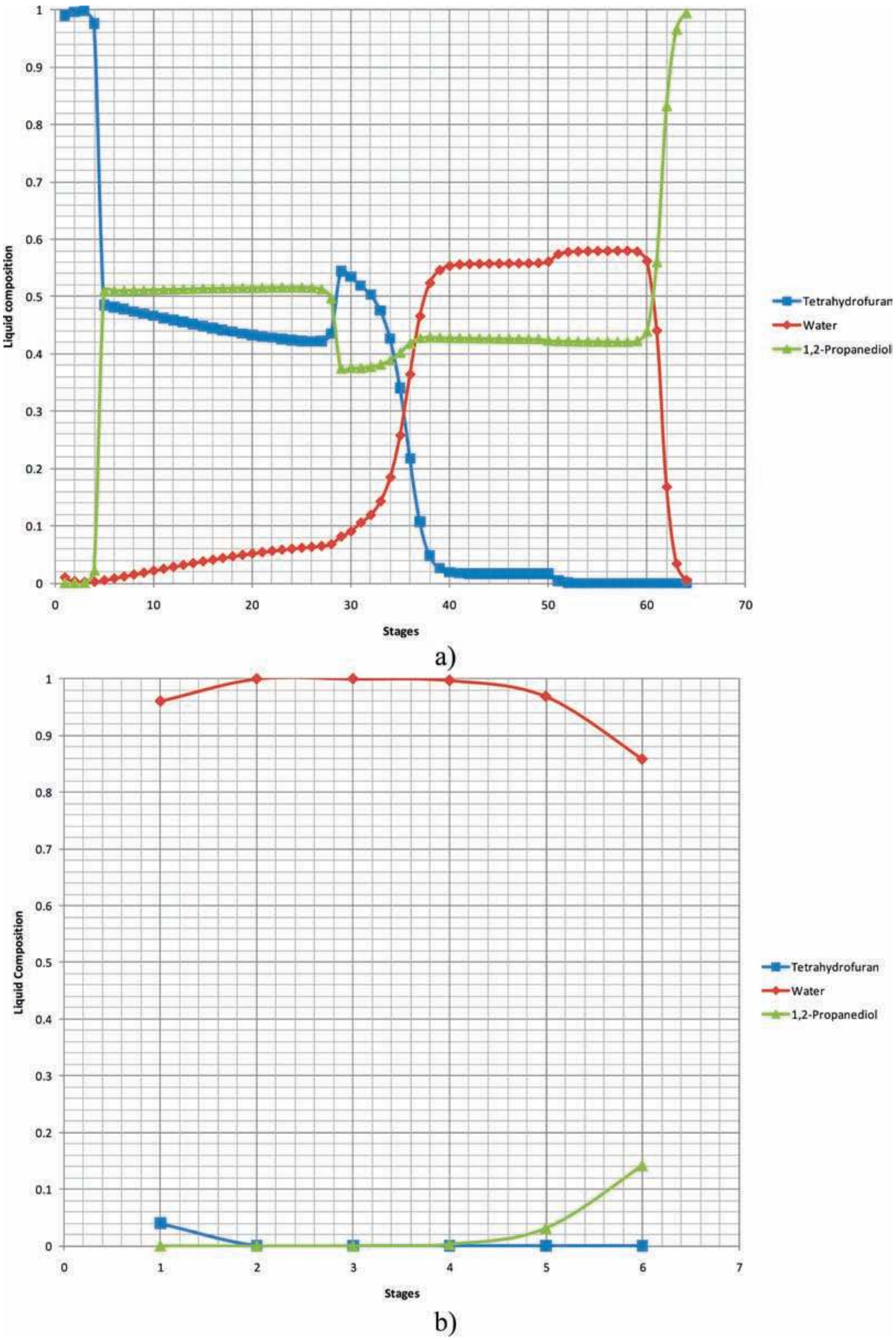


**Figure 21.** Temperature profiles for the extractive distillation dividing wall column design with the minor energy consumption and carbon dioxide emissions (mixture tetrahydrofuran/water): (a) main column (B1), (b) postfractionator (B2).

**Mixtures M2, M3, and M4.** Figures 9−11 present the residue curve maps for mixtures M2, M3, and M4, respectively. It is worthy of mentioning that a separation split is feasible if the compositions of products and feed have to be located in the same region of the composition space. From Figure 9, we observe that there is one homogeneous azeotropic point composed by tetrahydrofuran and water. However, the interaction with 1,2-propanediol allows having just one region in the composition space, making possible the separation of three components with high purity. Mixture M3 also presents an azeotropic point composed of isopropyl alcohol and water, Figure 10; however, the separation is feasible since just one region is observed in the composition space. On the other hand, mixture M4 does not have an azeotropic point, which makes the separation feasible; see Figure 11. According to the residue curve maps, we observe that separation splits are feasible for mixtures M2, M3, and M4, since there are no distillation boundaries dividing the composition space into different regions. Therefore, we calculate the Pareto front using, as design tool, the aforementioned multiobjective genetic algorithm.



a)

b)

**Figure 22.** Composition profiles for the extractive distillation dividing wall column design with the minor energy consumption and carbon dioxide emissions (mixture tetrahydrofuran/water): (a) main column (B1), (b) postfractionator (B2).

Figures 12−14 show the Pareto front for mixtures M2, M3, and M4; these graphs present the objectives in competition that were optimized: heat duty of the sequence, extracting agent flow, and the number of stages on both sides of the shell. From these figures, we observe that the tendency is the same for all mixtures: with the increase in the number of stages and solvent flow, the heat duty also increases. As reported for mixture M1, obtaining the Pareto front for the rest of study mixtures allows checking that dividing wall distillation columns can perform an extractive separation.

With respect to the structure of the dividing wall columns, we found tendencies in the location of interconnection and feed streams. In the main column, FL1 and FV2 leave the column, while FL2, FV1, solvent, and feed flows enter it. For all mixtures, we found that the exiting streams are always located at the ends the distribution stage, with all the feeds between them. After the FL1 interconnection flow, the first stream leaving the column, the solvent flow is present. Locations of FL2, FV1, and feed are always between the solvent flow stage and FV2 interconnection flow. The distribution of the interconnection and feed flows obeys the basic principle of increasing the interaction between the mixture and the solvent as long as possible; this is the reason why the exit flows are located at the ends of the column. In the postfractionator, the location of the side stream with respect to the number of stages is the same for all optimal Pareto front designs; the size of the postfractionator varies, but since the specifications of the separation are the same, the ratio of number of stages remains unchanged. This can be observed in Tables 4−6 where optimal designs that represent the lowest annual cost and the lowest greenhouse gas emissions of mixtures M2, M3, and M4 are presented.

The range for the minimum−maximum ratio of the solvent/feed flows oscillates around [1.5−2.2], [1.6−3.5], and [1.5−2.5] for mixtures M2, M3, and M4. These ratios show that the presence of four interconnection flows does not necessarily increase the solvent flow; therefore, competitive operating costs can be expected. Moreover, for all mixtures, we found linear relationships between the interconnection vapor and liquid flows. The ratio between FV2 and FV1 oscillates around 1.5 for mixtures M1, M2, and M3, but for mixture M4 this value is around 3.2. The value of this ratio depends on the modified nature of the mixture after the addition of the extracting agent.

The optimal designs of the extractive dividing wall distillation columns were obtained considering the minimization of heat duty, extracting agent flow, and the total number of each shell of the sequence. However, these optimal designs can be transferred in terms of minimum total annual cost and $CO_2$ emissions, as well as maximum energy efficiency, in spite of these variables not being considered explicitly in the optimization process. This can be seen in Figures 15 and 16.

As additional information, Figures 17−22 show the temperature and composition profiles of the designs reported in Tables 3 and 4. For mixtures M3 and M4, Tables 5 and 6, a similar behavior to that observed in mixture M2 was found. Figures 17 and 18 show temperature and composition optimal profiles for mixture M1 in both main column and postfractionator. We can observe that the location of feed and product stages allows having smooth profiles in both sides of the column; in spite of the third component added (solvent). For mixture M1, the same design satisfies the implicit (energy consumption) and nonimplicit (total annual cost and $CO_2$ emissions) optimization criteria. On the other hand, for mixture M2 there were slight differences between designs with minor total annual costs and minor as energy consumption as $CO_2$ emissions; however, it is important to mention that these differences were 3% at its maximum value. Nevertheless, Figures 19−22 show temperature and composition profiles for mixture M2. It is worth mentioning that all designs presented here are optimal designs, since the composition profiles are very smooth and they do not present pinch zones. The smoothness of the composition profiles means that the location of the inlet and oulet streams are properly located in the structure of the column; also, this structure is correctly dimensioned since pinch zones do not occur (oversizing) and the recoveries and purities are reached (undersized is not present). Also, in Figure 18, we observe that the composition profile in the postfractionator is practically a replica of the section of the composition profile in main column included between stages 31 and 51. This finding shows that there is, also, a smooth transition between the two sides of the shell, which allows reducing the entropy generation and, as consequence, the energy is properly used to perform the separation. Hereby, as in the structure as in the energy utilization, the designs that integrate the Pareto front are optimal.

## 5. Concluding Remarks

In this study, a multiobjective stochastic procedure is presented to obtain optimal designs of extractive dividing wall distillation columns. The stochastic procedure allows manipulation of 15 variables simultaneously; six being continuous and the rest being integer. All resulting optimal designs are rigorous, since the optimization procedure is coupled to Aspen Plus. The results show that dividing wall distillation columns are a feasible option to separate extractive mixtures, despite their highly nonideal nature. The Pareto fronts obtained for extractive dividing wall distillation columns present good diversity, in terms of the different structures of the columns, and also with respect to energy consumption. Moreover, it was found that the optimum energy consumption design can be related to the minimum total annual operating cost, minimum greenhouse gas emissions, and higher thermodynamic efficiencies. The Pareto front is obtained from keeping constant the structure of the main column, and varying the size of the postfractionator; this behavior is because the hard separation is preferably performed in the main column. The design of the main column remains almost constant; however, the postfractionator structure varies considerably. In general, the ratio of solvent flows with respect to feed is around 1.6, inside the range recommended by the heuristic rule for conventional extractive sequences. For all cases, there are linear relationships between the interconnection flows of the dividing wall distillation columns.

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