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Integrated process design and control of divided-wall distillation columns using molecular tracking

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Abstract
This work explores the dynamic and control behavior of dividing wall distillation columns from two different steady-state design approaches (molecular tracking and optimization method) for three different mixtures. The controllability of the six design cases was evaluated using singular value decomposition and the closed-loop performance was evaluated using integral absolute error in Aspen Dynamics. The results demonstrate that the side-draw location obtained by molecular tracking (MT) provides optimal controllability. As a result, there is a slight advantage in control properties while obtaining designs by reducing the time to find the optimal solution through the MT method.

KEYWORDS
controllability, divided-wall distillation column, molecular tracking, optimization

1 | INTRODUCTION

Distillation is a widely used method to separate chemicals on a large scale. It is estimated that distillation accounts for up to 50% of both operating and capital costs in industrial processes and makes up about 50% of the energy demand in chemical and petroleum refining industries.[1] For a more sustainable product separation and product purification, optimal design and control of distillation columns, optimal distillation column sequences, and intensified distillation alternatives are essential.

Thermally coupled distillation column sequences were introduced in the late 1940s to reduce the energy demand of separation.[2] Subsequently, the Petlyuk column,[3] along with several modifications[4] was introduced. The dividing wall column (DWC) is an intensified thermally coupled distillation column that enables multicomponent separation in a single tower with a single reboiler and condenser.[2] The DWC has shown a reduction in energy demand, capital cost, and footprint at an industrial scale.[5] A drawback to this reduction, however, is the increased complexity in the design of thermally coupled distillation columns due to the reduced number of degrees of freedom compared to other conventional schemes.[6] This makes the optimal design and control of this unit operation challenging[7] due to the integration of tasks and phenomena.

Reported design approaches can be broadly grouped into three categories: (1) approximate methods, (2) rigorous simulation methods, and (3) optimization-based design methods. Approximate methods, also known as (Class 1), are useful in providing starting points for rigorous simulation methods, or (Class 2), as well as for optimization-based design methods also called (Class 3). These methods include but are not limited to, graphical methods, and short-cut methods.[8,9] Rigorous simulation methods solve Mesh equations[10] which are guided by a design algorithm, typically heuristically, to minimize a specified cost function.[11] Optimization-based design methods can be used to simultaneously minimize a cost function by varying the possible degrees of freedom at the expense of an elevated complexity and computational power.[12] The current advantage of Class 2 design methods, is the fact that they can be incorporated directly into the design engineers' explorative working process involving commercial process simulators. In this way, the challenges of ensuring convergence, global minima, and fast, are handled in a manual manner, at the price of requiring expert-guided decision-making.

In a (Class 2) design method, molecular tracking (MT) was applied to DWC configurations based on the thermodynamically equivalent Petlyuk arrangement.[11] At first, MT was developed for multicomponent mixtures with intermediate boiling components at an infinite dilution for side-draw distillation units[13] as one of the simplest intensified distillation configurations. This concept has been employed to identify the near-optimal side-draw location in terms of the minimal energy demand of separation. MT has proved to be intuitive and provides a good compromise between available process details (accounts for rigorous internal column flows from the simulator) while being computationally straightforward to implement (can be evaluated in spreadsheets), which are common tools for process design.

Controllability has been demonstrated to be an inherent function of the Morari[14] design. As Morari[14] describes "With increased process integration it has become important to evaluate and compare the dynamic operability characteristics (dynamic resilience) of alternate designs." Several examples of the integrated process design-control have been proposed in the literature, more specifically, on reactive distillation[15–18]; demonstrating the optimal design that can address conflicting trade-offs between design and control strategies. The intention of this work is to demonstrate and evaluate the application of MT as a method for integrated design and control for divided-wall distillation columns and to evaluate the controllability of DWC operating at design specifications using MT. Vega et al.[19] present successful applications of the integrated process design and control problem. However, they also suggest that the computational effort required for integrated design and control increases considerably. Techniques that perform design and control integration usually use stochastic formulations. In contrast, MT is focused as a methodology for reducing the number of random samples of perturbation needed in the analysis, allowing its application to large-scale processes. Yuan et al.[20] indicate that the integration of design and control represents the central and most critical component of a sustainable approach. They suggest that effective scheduling techniques, mature data analysis procedures, and high-performance computing environments are required for design and control integration.[21] Mansouri[22] argues that there are limitations (violations of dynamic constraints) in the use of the sequential approach in which the process is designed first and then the control. More recent studies have shown that good operational robustness can be achieved with complex column dynamics such as high-purity distillation columns[23] and for distillation columns operating in significantly different operating modes.[24]

Steady-state performance metrics resulting from the application of MT on side-draw distillation units have previously been compared with the driving force-based design method[25] on multicomponent systems.[13] Nazemzadeh et al.[13] developed the MT concept for the design of side-draw distillation and illustrated the application of the developed method on the separation of a few model systems: (1) benzene–toluene–*p*-xylene (BTX) and (2) *n*-pentane–*n*-hexane–*n*-heptane (PHH) and on the purification unit of a retired bioethanol production plant (Inbicon A/S). The configuration proposed by MT for the BTX system showed a 25% lower energy demand compared to the configuration obtained by applying the driving force-based method. Energy demand reductions were found to be 20% for the PHH system. However, the design configuration proposed by MT led to a 2% lower distillate recovery rate compared to the design proposed by the original driving force-based method. Minor energy demand reductions were reported for the application of MT on the bioethanol side-draw distillation configuration compared to the configuration proposed by Bisgaard et al.[26]

The primary findings of the MT concept application on side-draw distillation units prompted us to research the application of this concept on a rather more complex unit operation, that is, the divided-wall distillation column. By applying MT on such a unit, the molecular pathways inside the column are like those of a conventional distillation column. A schematic figure of these pathways, for a ternary mixture, is illustrated in Figure 1. Monotonati et al.[11] developed a modeling framework for the design of DWCs by using an MT concept that provided a near-optimal configuration for a unit operation. The results have been compared to the configurations obtained by optimization methods from Nazemzadeh et al.[13] in terms of total annualized cost (TAC). Taking into consideration the computational time and effort for an optimization algorithm, MT has shown an advantage over these algorithms in the design of such units. Although a chemical process may have been initially designed effectively with a given methodology, the study of the dynamic properties allows to identify opportunities for continuous improvement and optimization. By analyzing how the process behaves under operating conditions and how it responds to disturbances, possible adjustments in design and/or control strategies, in operating variables or even in the process design methodology can be identified to maximize its efficiency and performance. The study of the dynamic properties of a chemical process after its design can provide valuable information for future research and development projects. The insights gained about the behavior and dynamic characteristics of the process can help improve theoretical models, develop new control strategies, and generate innovative ideas for the optimization of future chemical processes. In other words, the study of the dynamic properties of a chemical process, after it has been designed, provides valuable information for continuous improvement and optimization of the process. By analyzing the dynamic behavior of the system, it is possible to identify possible improvements in the design methodology, control strategies, or adjustments in the operating variables. This allows us to continuously evolve and optimize the process

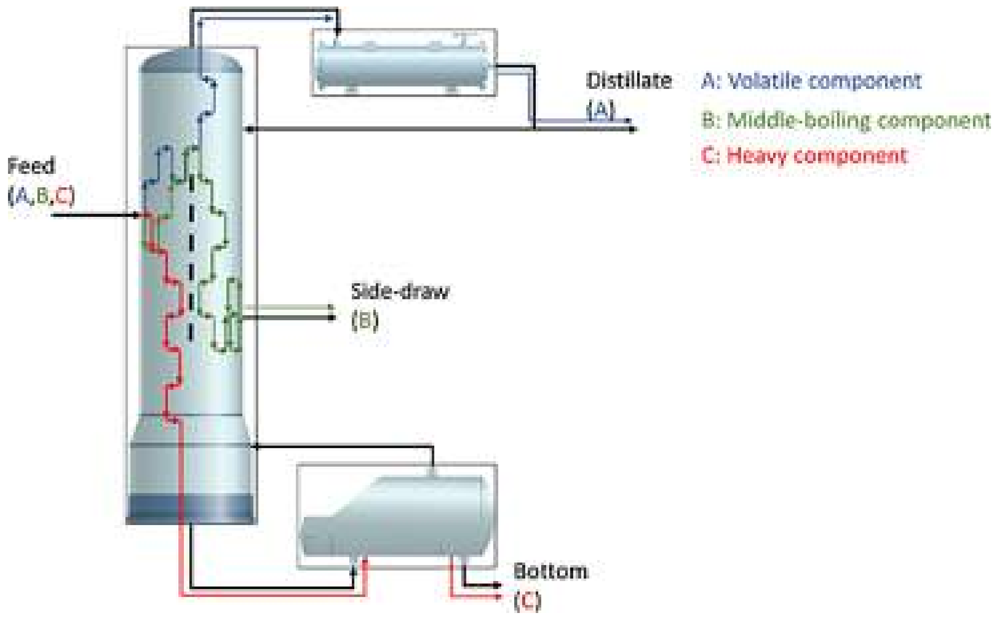

FIGURE 1 Schematic representation of molecular pathways for the separation of a ternary mixture in a divided-wall distillation column.

to obtain better results and stay competitive in the market. As a result, this work further explores the control properties of DWC steady-state designs by MT versus rigorous optimization in a qualitative manner. Details of the DWC design using MT are provided in the Supporting Information Material.

2 | METHODOLOGY

This methodology section will show the case study, as well as the methods to perform the open-loop control study and the closed-loop control study. It is important to note that in the proposed methodology the process design and control pairing/tuning were performed separately. An optimal design that can resolve conflicts between design and control strategies will be demonstrated. Thus, by evaluating the controllability and dynamic performance of various DWC designs obtained with the MT methodology it will be possible to integrate control and design.

2.1 | Design methods

2.1.1 | Molecular tracking

In this work, the case studies and the results from Montonati et al.[11] regarding the application of MT for the design of DWCs are adopted. However, as the MT method is relatively new, a simple illustration of the concept is provided below. Overall, the MT method proposes a near-optimal column stage from where a middle-boiling component can be removed in a side-stream, given a rigorous column simulation of main mixture constituents and suitable VLE models. This approach can be repeated for any middle-boiling components. For example, this can be used to select a side-stream location for middle-boiling trace components using infinite dilution activity coefficients, or more complex separations such as DWC.[13]

The name of the method refers to the fact that the molecular pathway of a targeted component inside the distillation column is tracked mathematically via a probability function based on the thermodynamic properties of a mixture (VLE data) and the internal flows (vapor and liquid flow rates). This probability function, as shown in Equation (1), defines the probability of a component that moves upwards (in the vapor phase) or downwards (in the liquid phase) at any given location. A more detailed description of this function can be found in the work of Nazemzadeh et al.[13]

$$\beta_i^n = \frac{V^n K_i^n}{V^n K_i^n + L^n}; K_i^n = \frac{y_i^n}{x_i^n} \quad (1)$$

where β represents the probability function; V denotes vapor internal flow rate while L is the liquid internal flow rate; K represents the VLE equilibrium constant; y is the vapor phase composition, and x is the liquid phase composition; i and n identify the component and the stage number, respectively.

To describe the probability function in a more detailed manner let us consider a multicomponent mixture to be separated by distillation. Based on the boiling temperature of the pure components at the operating pressure of the column (in the ideal mixture case), the components in the mixture can be classified as follows: most volatile, middle-boiling, and least volatile compounds. The most volatile compound effortlessly flows toward the distillate of the column as this corresponds to a high probability value based on Equation (1), while the opposite occurs with the least volatile component. However, Nazemzadeh et al.[13] discuss that the middle-boiling component does not follow the same pathway as either of the previous two components. Middle-boiling components move inside the column and accumulate somewhere between the top and bottom trays. The tray, at which this component has the highest tendency of accumulating, corresponds to a 0.5 (50%) value of the probability function. This is known as the optimum/near optimum location of the side-draw to withdraw the middle-boiling compound from the column.

2.1.2 | Optimization method

This work directly incorporates the optimization method and results from Ramirez-Corona et al.,[12] in which the TAC, is minimized (see Equation 2). Note the work presented by Ramírez-Corona et al. propose an optimization model for energy-saving distillation systems. The model uses a shortcut design procedure as a basis, which allows the system to be modeled as an NLP problem. The model was used to detect Petlyuk and DWC structures that minimize the total annual cost for a variety of case studies and validated with rigorous simulations. On the other hand, it was also evaluated the same case studies with the methodology provided by Nazemzadeh et al. As mentioned above, this method is a systematic method developed for the design of side-draw distillation units based on the concept of MT, integrated with the conventional methods of distillation design such as McCabe–Thiele, driving force-based method or other existing methods. This method guarantees optimal design configurations for side-draw distillations.

$$\mathrm{TAC} = \frac{\text{Capital costs}}{\text{Payback period}} + \text{Operating costs}. \tag{2}$$

2.2 | Singular value decomposition: Open-loop analysis

The singular value decomposition (SVD) technique provides a measure of the controllability properties of a dynamic system. The SVD is not a quantitative measure, but a qualitative one in the comparison of theoretical control properties between the considered sequences.

Minimum singular value (Morari Resilience Index [MRI]) and condition number are two concepts related to the SVD of an array. The SVD is a mathematical tool that decomposes a matrix into three components: a left singular vector matrix, a diagonal singular value matrix, and a right singular vector matrix.

The dynamic responses were obtained through the use of the Aspen Dynamics simulator. Once all responses were obtained, the transfer function matrices (G) were collected and subjected to SVD; the calculation of SVD was performed as follows:

$$G = V \Sigma W^H \tag{3}$$

where $\Sigma = \mathrm{diag}\,(\sigma_1, \sigma_2, \ldots, \sigma_n)$, $\sigma_1 =$ singular value of $G = \lambda^{\frac{1}{2}}\left(GG^H\right)$, $V = (v_1, v_2, \ldots, v_n)$ matrix of the left singular vector and $W = (w_1, w_2, \ldots, w_n)$ is the matrix of the right singular vectors. Inside the calculation of G, the two parameters of interest are the minimum singular value σ_* and the ratio of the maximum to minimum singular values, named the condition number, which is calculated as follows:

$$\gamma^* = \sigma^* / \sigma_* \tag{4}$$

The engaging aspect of the SVD study regarding process control is that when applied to a matrix that describes the steady-state characteristics of a multivariable process, the singular values have a strong physical interpretation. In practice, the minimum singular value (MRI) measures the invertibility of the evaluated scheme, and it also measures the potential problems of the system under feedback control. Small singular values could indicate that in spite of a good condition number, the system is simply not sensitive enough to control. On the other hand, large singular values indicate a practical control problem. Furthermore, the condition number could be interpreted as the sensitivity of the system under uncertainties and modeling errors. However, the condition number only provides a qualitative assessment of the theoretical control properties of the schemes under analysis. In general, schemes presenting lower values of the condition number were expected to show better dynamic performance under feedback control. In physical terms, the condition number represents the ratio of the maximum and minimum open-loop, decoupling gains of the system. A large condition number indicates that the relative sensitivity of the system in one multivariable direction is very weak. SVD analysis does not solve all the control problems which may be found in industrial multivariable control; however, it is relatively easy to understand and identify basic control difficulties. The SVD technique has been used by several authors to study the dynamic properties of complex designs.[27]

We analyzed the control properties of the designs obtained with the optimization method and with the MT strategy. For more details about SVD see Klema and Laub.[28] To begin with, open-loop dynamic responses to changes in the manipulated variables around the assumed operating point were obtained. An important point to note here is that the Aspen Plus models are also available in Aspen Dynamics which make it possible to rigorously solve the dynamic model controllability studies. To achieve the open-loop dynamic responses, a step change around the nominal operation point was applied. The extent of the step change was 1% of the manipulated variable (reflux ratio, reboiler duty, or side-stream flow). The responses were obtained using Aspen Dynamics and adjusted to proper transfer functions (i.e., first order, second order, etc., depending on the performance of the dynamic response). Transfer function matrices (K) were then collected for each case and subjected to SVD.

The SVD is appealing and interesting in terms of controllability for when it is applied to a matrix that reflects the steady-state component of a multivariable process, the singular vectors, and singular values have a very specific physical interpretation: the physical scaled steady-state sensitivity to the changes of each process sensor in each of the controlled variables is represented by K, the steady-state gain matrix.

The singular values of the open-loop frequency function matrix of a process at a given frequency are the gains of the process at this frequency in the directions of the corresponding input singular vectors (as the input of the singular vectors form a basis in the input space, the gain can be calculated in every direction). These gains play an important role when performing the controllability analysis of a process. For a complex analysis, the gains must be evaluated at various frequencies. The MRI is the smallest singular value (σ_*) of the open-loop frequency function matrix process. The larger the value is, the more controllable the process becomes. When the value is zero there is an input direction where the gain is zero and the matrix is not invertible. The condition number (γ) is the ratio of the largest and smallest singular values of the open-loop frequency function matrix process. When the value is great the matrix has strong directionality; this means that the gains will vary strongly depending on the input directions. Such a matrix is said to be ill-conditioned. A large γ means that the system is sensitive to input and model uncertainty and therefore the process is less controllable. Systems with higher σ_* value and a lower γ are expected to show the best dynamic performance under feedback control.[29]

2.3 | Closed-loop analysis

In the comparison of the control properties of study cases, a control analysis was conducted by means of a closed-loop control test. The choice of control outputs and the corresponding controlled variables was a crucial component in the analysis of each loop. Thereby, structures based on energy balancing considerations were utilized to regulate the distillate and bottom output compositions. This structure produces the LV control structure which employs the reflux flow rate *L* and the vapor boil-up rate *V* as the manipulated variables. In other words, we selected, as the manipulated variables, the corresponding reflux flow rate for the top of the column to control a distillate component concentration, the reboiler heat duty at the bottom of the column to control a bottom component concentration, and the side-stream flow rate to control the concentration of the intermediate component. The closed-loop control performance was evaluated under compositional perturbation scenarios and taking into consideration the feed composition perturbation (−5 wt% of medium boiling component). These types of analyses are very useful in the research of theoretical dynamic properties under feedback control, just as several authors have done before.[30,31] In addition, the control test will show which is the best scheme among all the ones analyzed from a dynamic point of view.

The steady-state results were exported to Aspen Plus Dynamic. Within this simulator the control test was performed as follows: a step change was induced in the set point for each product composition set points. Under single-input single-output feedback control at each output flow rate (a 1% perturbation was considered for the present work). For the closed-loop control policy, the analysis considered proportional–integral (PI) controllers. This kind of controller was chosen because it is greatly used in industrial procedures. In the closed-loop control test the tunning of the controller was the main issue to solve. In this study, a common strategy was considered to compare and optimize the controller parameters. Because we used PI controllers, the proportional gain (K_c) and the integral times (τ_i) were tuned up for each scheme studied. In addition, we compared the dynamic performance by using the integral of the absolute error (IAE) criterion.[29] Similarly, a key issue is the selection of control outputs and their respective manipulated variables. In this study, to control the distillate, the bottom and the side-stream output compositions, structures based on energy balance considerations were used. This structure yields the LV control structure, which uses the reflux flow rate *L*, vapor boil-up rate *V*, and side-stream flow *S* as the manipulated variables. This common strategy is founded on the principle of balancing energy, resulting in the LV control configuration. This involves manipulating the rates of reflux flow (*L*) and vapor boil-up (*V*), which are influenced by the amount of heat provided to the reboiler, to

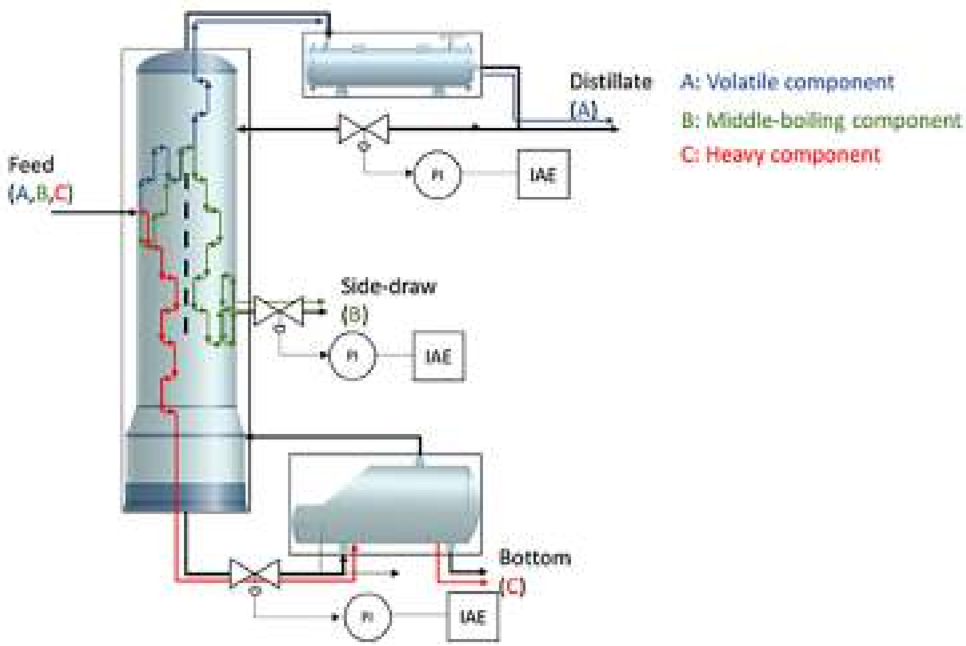

FIGURE 2 Implementation of PI controller.

regulate the compositions of the distillate and bottom outputs (as shown in Figure 2).[32] This kind of control loop has been applied with satisfying results in DWC schemes.[33,34] The IAE has the advantage of reducing the maximum overshoot, more than other evaluation criteria such as integral of square error (ISE) or integral of time multiplied by absolute error, but the latter has the advantage of reducing the stabilization time more than IAE and ISE. Considering the properties of the criteria, the present work makes use of the IAE to reduce the maximum overshoot of the error signal. Reducing the overshoot of the error signal as long as they are autonomously reduced by changing the set-point and perturbation of the compositions.[35]

To tune up each controller, an initial value of proportional gain was added, and a range of integral time was tested with this fixed value until a local optimum in the IAE value was obtained.[36]

$$\mathrm{IAE} = \int_0^{\infty} |\varepsilon(t)| dt \tag{5}$$

where $\varepsilon(t)$ is the function of integral time, which is given by:

$$\varepsilon(t) = y_{\mathrm{d}} - y(t) \tag{6}$$

The sequences were compared by evaluating the IAE obtained following the methodology suggested by Ramirez-Marquez et al.[37]

3 | BENCHMARK CASE STUDIES

The following sections will show and analyze the results obtained in the analysis of the dynamic properties, open-loop control, and closed-loop control, that were performed.

3.1 | Case study selection

In the present work, three mixtures were taken as case studies. Both design methodologies show the difference in the operating costs of the process, with the MT method resulting in economically favorable designs. However, economics do not guarantee that the designs will be industrially operable, therefore, a control study of the DWC designs presented in both works[11,12] was carried out in this investigation. The design features of each DWC can be found in the Supporting Information Material. Table 1 shows the components of each mixture, as well as their composition and purity. Three ternary mixtures with different values of the ease of separability index, as defined by Ramìrez-Corona et al.,[12] were considered to observe the effect of volatility on the design and control properties of the designs under the different methodologies. The Equation of State (EOS) was selected for two reasons; (1) because of the similarity between molecules, and (2) because it adequately represents the properties of the mixture in the Peng–Robinson EOS.[11]

3.2 | Open-loop results

The minimum singular value (σ_*) is the smallest value in the diagonal matrix of singular values obtained in the decomposition. It represents the minimum magnitude of changes that occur in the system. In the context of a chemical process, it can be related to the sensitivity of the system to disturbances or changes in operating conditions. A small minimum singular value indicates that even small disturbances can have a significant impact on the behavior of the system. The condition number (γ), on the other hand, is a measure of the numerical stability of the matrix and is calculated as the quotient between the maximum singular value and the minimum singular value. Provides information about the sensitivity of the numerical calculations used to solve systems of linear equations associated with the matrix. In the context of a chemical process, the condition number can indicate how sensitive the system is to numerical errors during the calculation of solutions. In summary, the minimum singular value and the condition number of a matrix obtained from a chemical process can provide information about the stability and sensitivity of the system to perturbations or numerical errors. These properties may be relevant to understand the dynamic characteristics of the chemical process and to assess the reliability of the associated calculations and predictions. Figure 3 shows the results of the condition numbers for the three studied mixtures, and Figure 4 shows the results for the minimum singular value.

Figure 3 shows the condition number throughout the frequency domain, and as mentioned in the methodology, a low condition number indicates that the system is well-positioned for possible perturbations. Although all the mixtures were designed using both methods (molecular tracking and optimization method), the designs obtained through MT, particularly those for Mixtures 1 and 2, exhibited the lowest condition number values. However, unlike the other mixtures, Mixture 2 is less straightforward because, at low frequencies, the designs obtained through the optimization method demonstrate superior dynamic behavior. Please note that, in an open-loop control study, the best-conditioned designs are those that have a smaller value of the condition number and a larger value of the minimum condition number. Likewise, it is possible to have values of condition number and minimum singular value in a relatively large range of frequency. Both values, when located in low-frequency ranges, refer to the

TABLE 1 Study mixtures.

	Components	Composition (% mol)	Purities
Mix 1	Isopentane–*n*-pentane–*n*-hexane	40–20–40	0.99–0.92–0.99
Mix 2	*n*-Butane–isopentane–*n*-pentane	40–20–40	0.99–0.92–0.99
Mix 3	*n*-Pentane–*n*-hexane–*n*-heptane	40–20–40	0.99–0.92–0.99

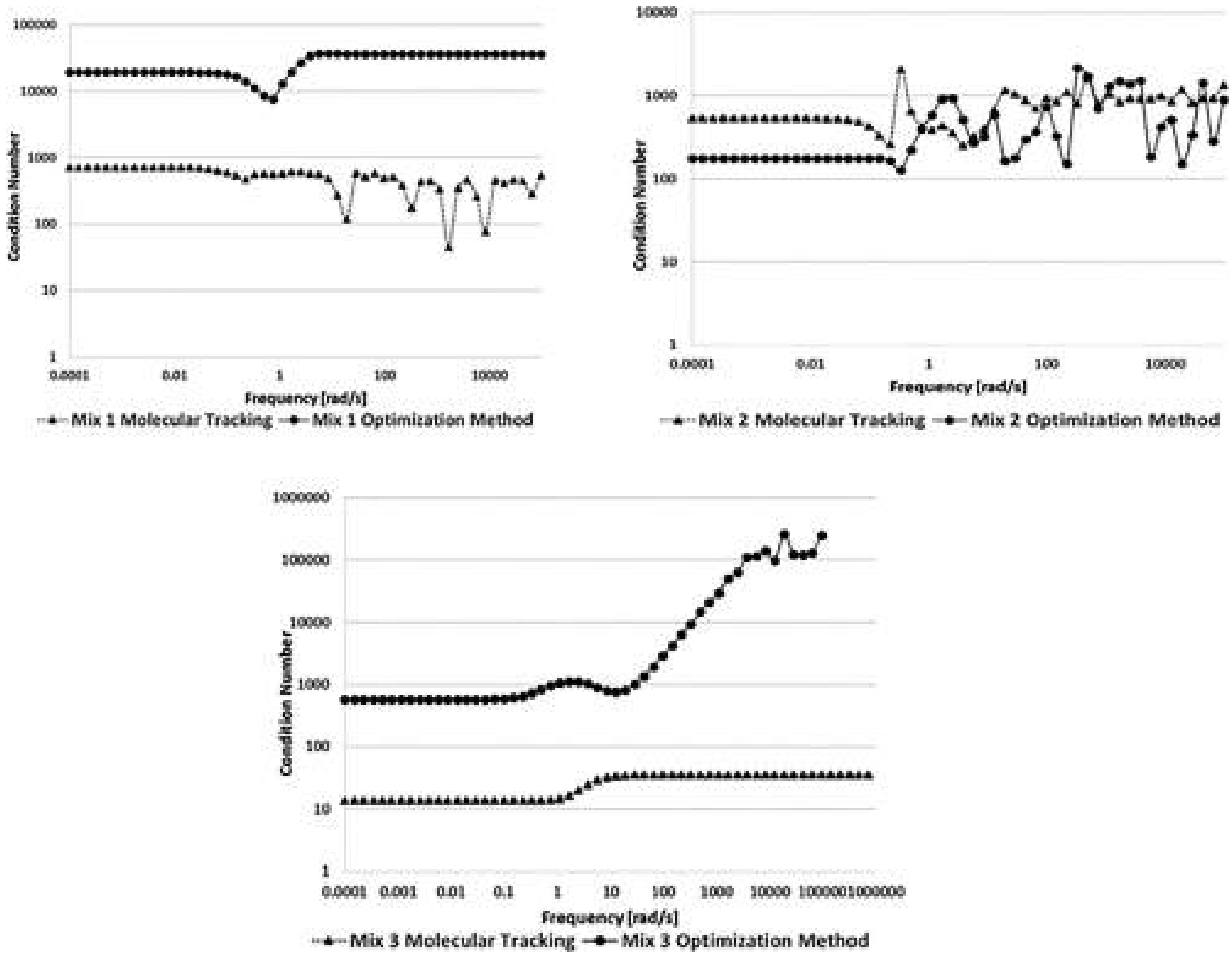

FIGURE 3 Condition number for Mixtures 1–3 both cases study.

controllability that processes can have in the presence of small disturbances, and vice versa. Hence, Mixture 2 indicates that in a scenario with small perturbations, the optimization method designs will respond better to such perturbations; on the other hand, with larger perturbations, the MT designs will exhibit better control.

Overall, schemes presenting lower values of the condition number were expected to show better dynamic performance under feedback control. In physical terms, the condition number signifies the ratio of the maximum and minimum open-loop, decoupling gains of the system. A large condition number implies that the relative sensitivity of the system in one multivariable direction is very weak. SVD analysis does not solve all the control problems which may be found in industrial multivariable control, however, it is relatively easy to understand and identify basic control difficulties.[27]

Figure 4 shows the results regarding the minimum singular value as it adequately complements what was observed in Figure 3, that is, in Mixtures 1 and 3, the MT method generated better designs in terms of control. On the other hand, a similar behavior was observed with the condition number in the frequency range of Mixture 2.

Overall, taking only into consideration the open-loop study, the process designs obtained by the MT method show better control properties. A closed-loop control study is also expected to yield similar results in terms of controllability. The observed values of the condition number and minimum singular value is an indication of the sensitivity of the associated sensor to its manipulated variable, in other words, good dynamic behavior is expected under a closed-loop control policy. In summary, designs that present lower condition number and higher values of the minimum singular value in the frequency range are expected to exhibit better control properties under feedback control and are better conditioned to the effect of disturbances. The minimum singular value and the condition number can indicate the stability of the system. A minimum singular value close to zero or a high condition number suggests that the system is unstable and can undergo significant changes in response to small disturbances. This can be relevant to assess the stability of a process. The minimum singular value and the condition number are measurements that can provide valuable information about the dynamic properties of a chemical process. These measurements can help to understand system stability,

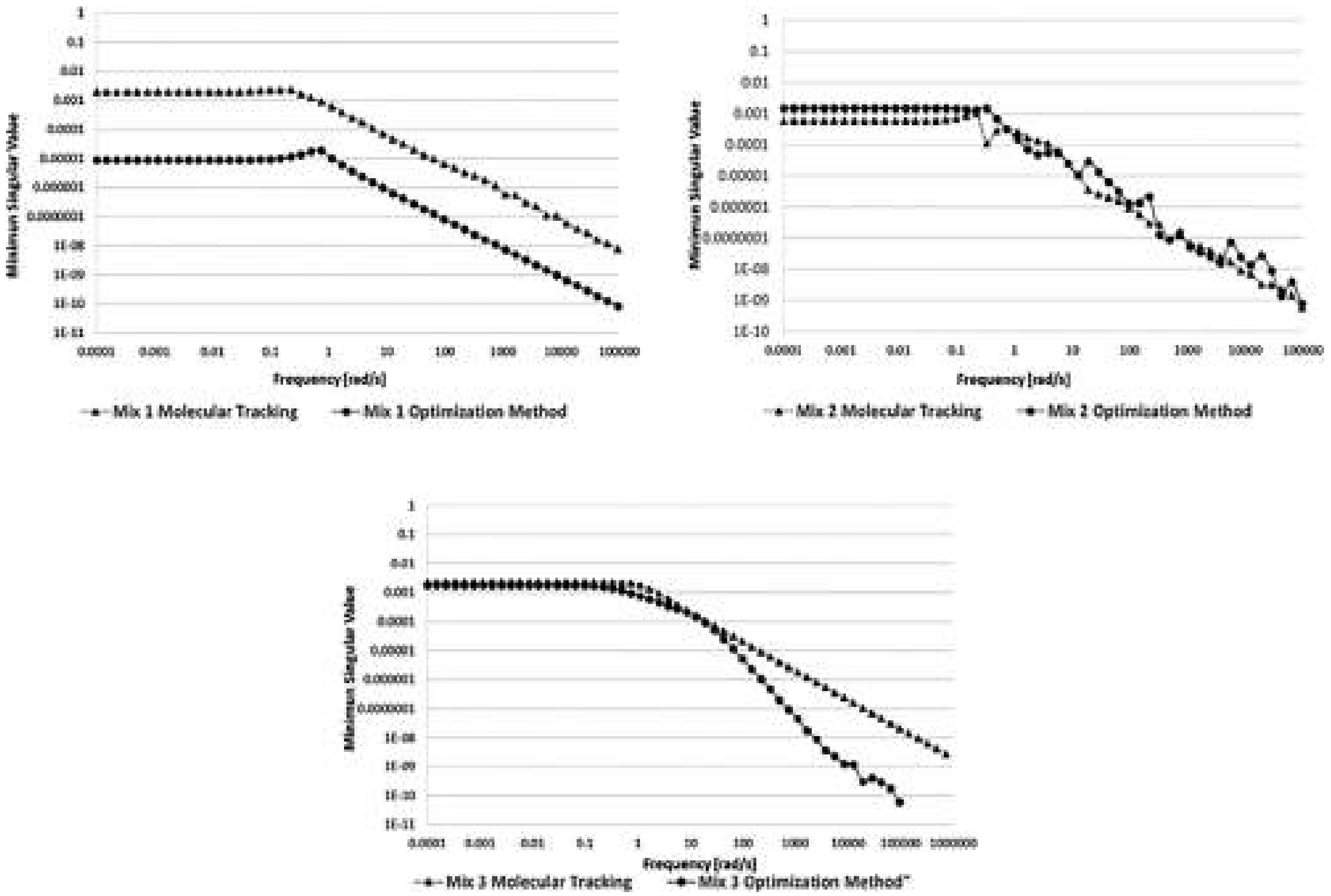

FIGURE 4 Minimum singular value for Mixture 1–3 both cases study.

TABLE 2 Matrix of transfer functions for M1 of molecular tracking.

$$
\begin{array}{c}
\begin{array}{ccc} \textbf{iC5} & \textbf{nC5} & \textbf{nC6} \end{array}\\
\begin{bmatrix}
\frac{(-0.0531)-((-0.0531)(2.8246\omega j)}{1+2.8240^2\omega^2} & \frac{(-0.0016)-((-0.0016)(-0.0016\omega j)}{1+-0.00165^2\omega^2} & \frac{(0.0095)-((0.0095)(1.7869\omega j)}{1+1.7869^2\omega^2} \\
\frac{(0.03)-((0.03)(0.2386\omega j)}{1+0.238^2\omega^2}+\frac{(-0.1)-((-0.1)(1.5051686\omega j)}{1+1.505168^2\omega^2} & \frac{(-0.0022)-((-0.0022)(4.55239\omega j)}{1+4.5523^2\omega^2} & \frac{(-0.14997)-((-0.14997)(7.3443\omega j)}{1+7.34439018750041^2\omega^2} \\
\frac{(0.0096)-((0.0096)(5.27493\omega j)}{1+5.2749^2\omega^2} & \frac{(-0.081678)-((-0.0816)(0.9787\omega j)}{1+0.9787^2\omega^2} & \frac{(-0.081678)-((-0.081678)(0.9787\omega j)}{1+0.9787^2\omega^2}
\end{bmatrix}
\end{array}
$$

reaction rate, and sensitivity to disturbances, which is essential for the design, control, and optimization of chemical processes.

Thus, it can be concluded from this open-loop analysis methodology that the schemes obtained by the MT method presented better dynamic properties. Note that in terms of condition number and minimum singular value, the process schemes derived from MT had the best values of both indicators for Mixtures 1 and 3. On the other hand, for Mixture 2, the schemes derived from the optimization method were only better when evaluated at low frequencies, that is, they presented better control properties against small disturbances, but not against larger disturbances as shown by the designs obtained with the MT method.

Tables 2–4 show the matrix of transfer functions for all MT cases to exemplify the kind of transfer functions obtained in the study. It has been observed that even for the complex system under study, all the responses are adjusted as first order or first order in competence, as seen in Figures 5–7. There are several reasons why a distillation column may exhibit first-order open-loop dynamic responses. For example, (a) linear behavior: in general, systems that present a linear and stable behavior tend to respond in a first-order open-loop dynamic way. In the case of a distillation column, the separation process is a physical process that can be modeled linearly; (b) response speed: the response speed of the distillation column can be quite fast compared to other chemical processes. This is because the column is in constant equilibrium, so you can quickly adjust the separation of the mixture components; (c) flow control: the operation of a distillation column is based on the control of the inlet and outlet flow of the mixture. This flow control can easily be modeled as a first-order

TABLE 3 Matrix of transfer functions for M2 of molecular tracking.

$$\begin{array}{c}
\begin{array}{ccc} \mathbf{nC4} & \mathbf{iC5} & \mathbf{nC5} \end{array} \\
\begin{bmatrix}
\dfrac{-0.0735) - ((-0.0735)(0.9946\omega j)}{1 + 0.9946^2\omega^2} & \dfrac{(0.0004) - ((0.0004)(4.69387\omega j)}{1 + 4.6938^2\omega^2} & \dfrac{(0.0098) - ((0.0098)(0.5150\omega j)}{1 + 0.5150^2\omega^2} \\
\dfrac{(-0.1342) - ((-0.1342)(3.4168\omega j)}{1 + 3.4168^2\omega^2} & \dfrac{(0.0064) - ((0.00641)(3.9501\omega j)}{1 + 3.950157^2\omega^2} & \dfrac{(0.02) - ((0.02)(2.82\omega j)}{1 + 2.82^2\omega^2} + \dfrac{(-0.09) - ((-0.09)(1.82\omega j)}{1 + 1.82^2\omega^2} \\
\dfrac{(0.0085) - ((0.00852)(2.1025\omega j)}{1 + 2.1021^2\omega^2} & \dfrac{(-0.00182) - ((-0.00184)(5.6506\omega j)}{1 + 5.6506^2\omega^2} & \dfrac{(-0.0343) - ((-0.0343)(4.9997\omega j)}{1 + 4.9997^2\omega^2}
\end{bmatrix}
\end{array}$$

TABLE 4 Matrix of transfer functions for M3 of molecular tracking.

$$\begin{array}{c}
\begin{array}{ccc} \mathbf{nC5} & \mathbf{nC6} & \mathbf{nC7} \end{array} \\
\begin{bmatrix}
\dfrac{(-0.0141) - ((-0.0141)(0.9934\omega j)}{1 + 0.9934^2\omega^2} & \dfrac{(0.0002) - ((0.0002)(1.9022\omega j)}{1 + 1.9022^2\omega^2} & \dfrac{(0.0026) - ((0.0026)(0.2887\omega j)}{1 + 0.2887^2\omega^2} \\
\dfrac{(-0.02078) - ((-0.0207)(0.3108\omega j)}{1 + 0.31084^2\omega^2} & \dfrac{(0.0067) - ((0.0067)(0.5290\omega j)}{1 + 0.529046^2\omega^2} & \dfrac{(0.083) - ((0.083)(2.8834\omega j)}{1 + 2.8834^2\omega^2} + \dfrac{(-0.03) - ((-0.03)(1.7108\omega j)}{1 + 1.7108^2\omega^2} \\
\dfrac{(0.0018) - ((0.0018)(0.29215\omega j)}{1 + 0.2921^2\omega^2} & \dfrac{(-0.0009) - ((-0.0009)(0.5087\omega j)}{1 + 0.5087^2\omega^2} & \dfrac{(-0.0081) - ((-0.0081)(0.9558\omega j)}{1 + 0.9558^2\omega^2}
\end{bmatrix}
\end{array}$$

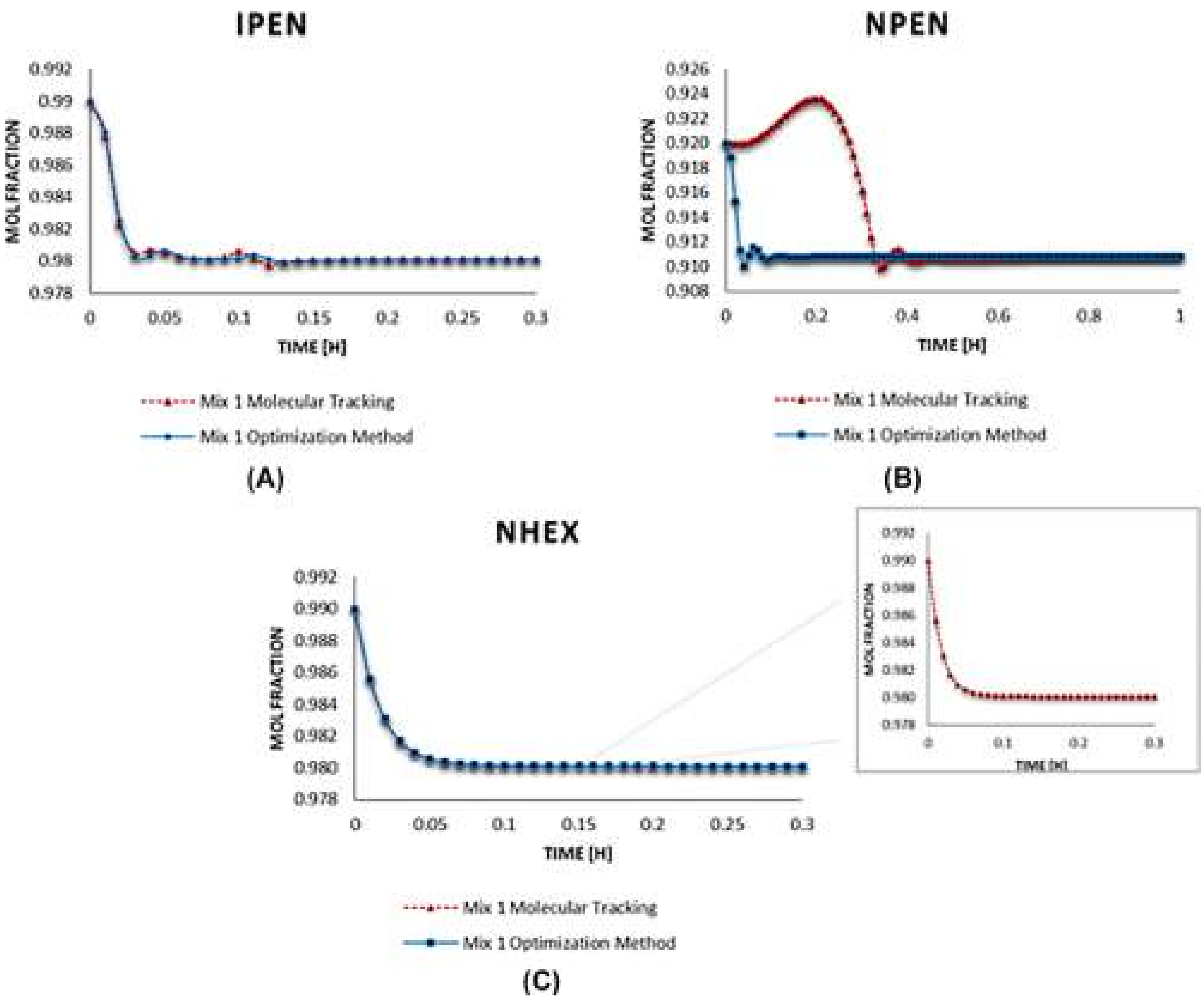

FIGURE 5 Closed-loop dynamic response for the Mix 1. (A) Isopentane loop, (B) *n*-pentane loop, and (C) *n*-hexane loop.

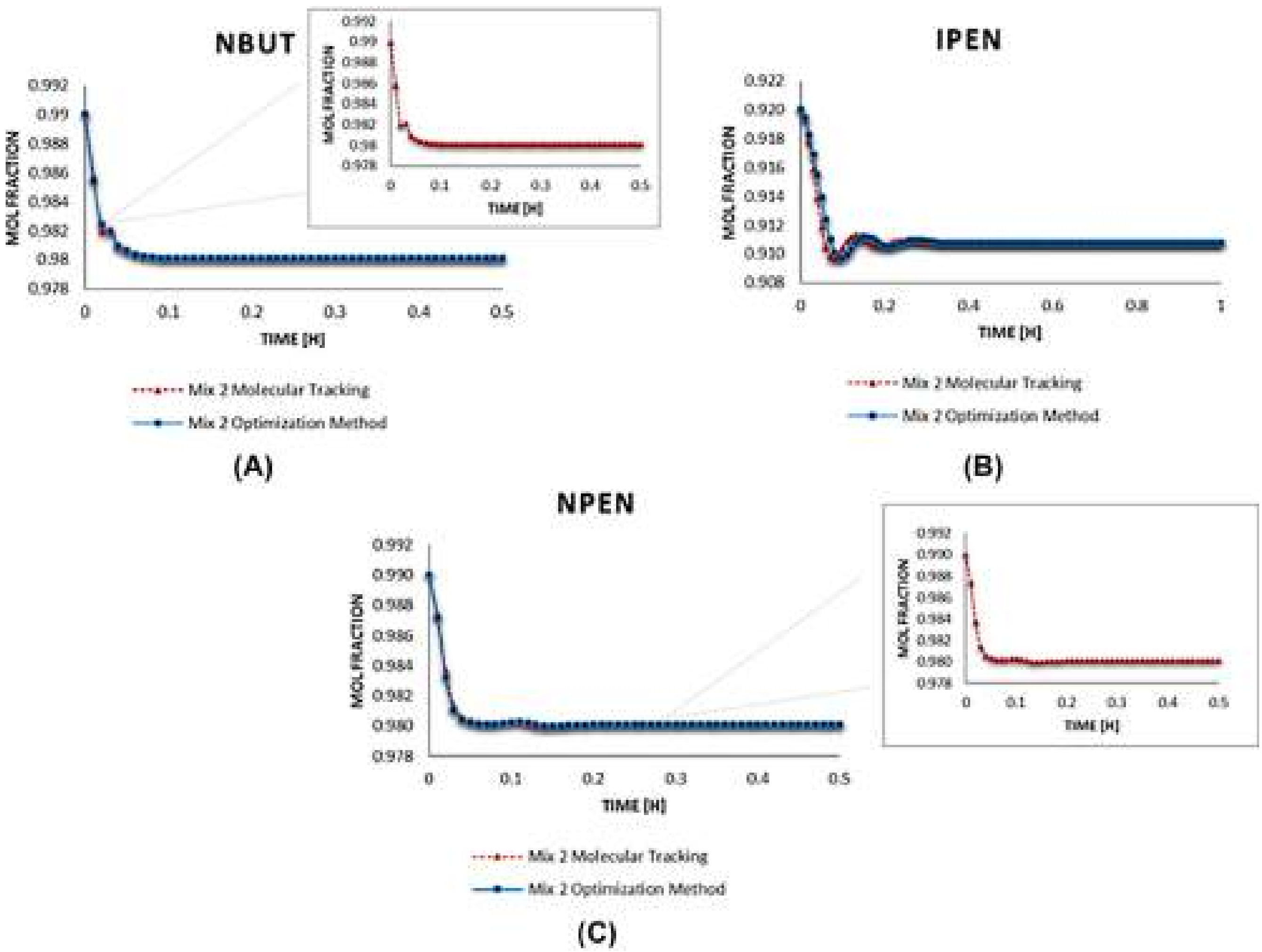

FIGURE 6 Closed-loop dynamic response for the Mix 2. (A) *n*-Butane loop, (B) isopentane loop, and (C) *n*-pentane loop.

system; (d) level control: another important variable in a distillation column is the liquid level in the reflux vessel. The level control can also be modeled as a first-order system; (e) the type of mixture, the percentage of the perturbation performed. That is, the thermodynamic properties and the value of the disturbance.[38] In this case, the type of mixture and the percentage of the disturbance to which the column is subjected primarily impacts. It has been observed that the ratio between the components that make up the feed directly affects the dynamics of the process when first-order dynamic responses are assumed. This behavior, in general, is relatively well documented in controllability studies. The fact that most of the responses are of the first order has various explanations. These reasons are, for example, the type of mixing, the percentage of the perturbation performed, and so forth. It has been observed that the ratio between the components that make up the feed directly affects the dynamic of the process when first-order dynamic responses are assumed.[39] This behavior, in general, is relatively well documented in several controllability studies and has allowed for the proposal of various control strategies derived from the use of SVD.[40]

3.3 | Closed-loop results

It is important to obtain a transient response of complex systems such as DWCs to understand the efficiency of the process operation. This work will help us evaluate which DWC design methodology is more appropriate in terms of control properties. It is important to remember that the objective of the closed-loop study methodology is to minimize the IAE. The results for Mixture 1, after the closed-loop study for both DWC design methodologies was completed, can be seen in Table 5. The steady-state design for Montonati et al.'s[11] mixture illustrates that the MT method ended up having a 4% decrease in reboiler duty, but a 10% increase in the TAC.

The results show that IAE values are obtained in the same order of magnitude for the isopentane and *n*-hexane loops. The optimization method, in the isopentane loop, has only a 4% difference with respect to MT. In the case of the optimization method, the *n*-hexane loop shows a 7% quantitative improvement in dynamic properties. Both percentages do not represent a significant advantage in terms of process dynamics. And finally, in the case of the intermediate loop or

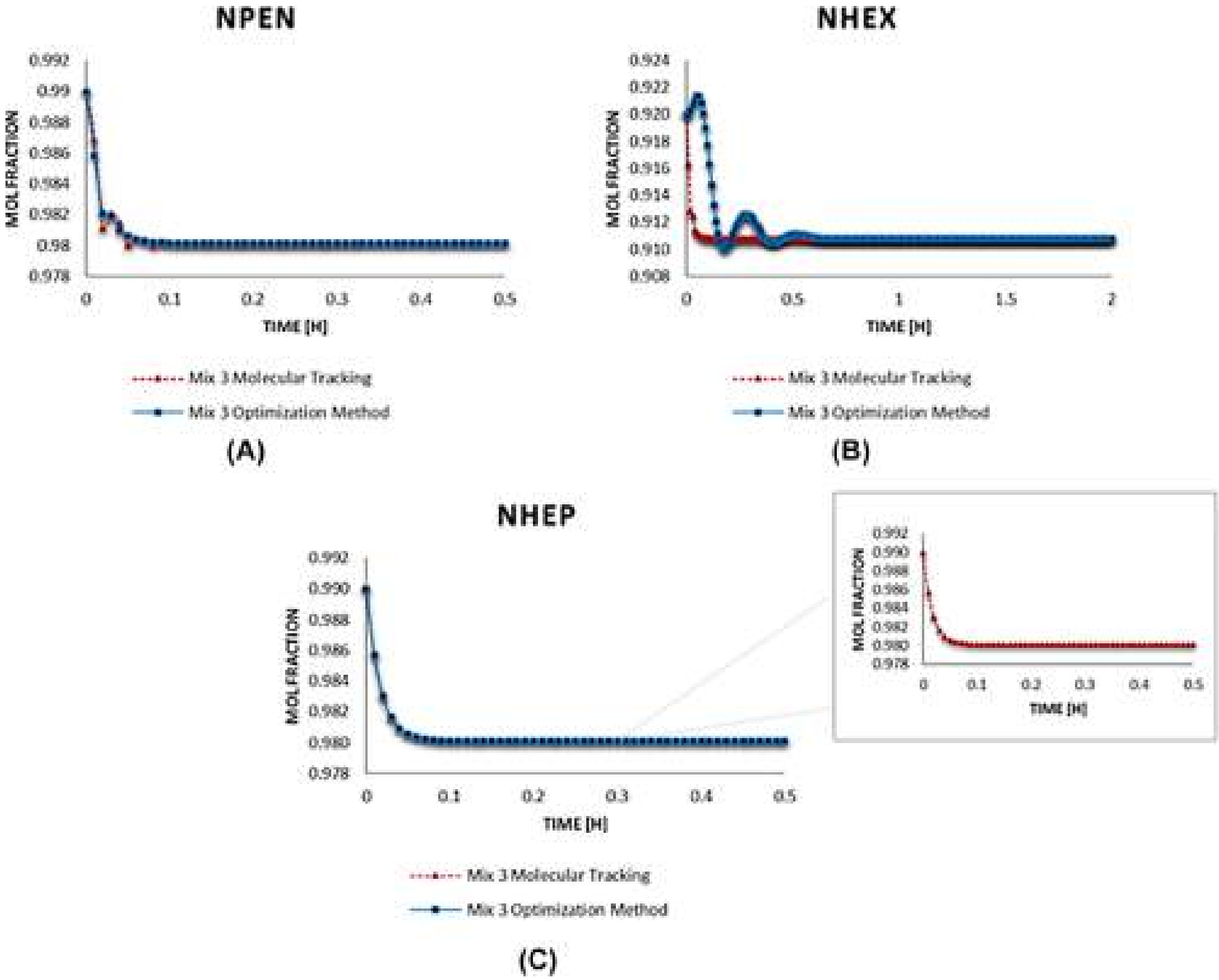

FIGURE 7 Closed-loop dynamic response for the Mix 3. (A) *n*-Pentane loop, (B) isohexane loop, and (C) *n*-heptane loop.

TABLE 5 IAE values for the set point change for Mix 1.

	Mix 1 (molecular tracking)			Mix 1 (optimization method)		
	K	Ti	IAE	K	Ti	IAE
Isopentane	250.00	1.00	1.7489E−04	250.00	1.00	1.6862E−04
n-Pentane	250.00	1.00	3.2994E−03	250.00	1.00	2.0110E−04
n-Hexane	250.00	1.00	1.7645E−04	250.00	1.00	1.6353E−04

Abbreviation: IAE, integral of the absolute error.

n-pentane, the order of magnitude is better. It is evident that the results do not match those obtained in the open-loop control study where qualitatively, the MT method has better control properties. This is basically due to the quantitative results of the intermediate loop. However, it should be noted that the quantitative results in the closed-loop control study are very similar to one another in design methodologies with a slight advantage in terms of control properties for the optimization method. This does not mean that the MT has a bad performance, on the contrary, it shows that both design methods, in this specific mixture, have excellent control properties. This is evident in Figure 5 where it can be seen that in both cases, the responses are almost identical. In Figure 5A,C there is a practicable overlap in the response. In all the loops the responses stabilize in less than half an hour after the perturbation. In the case of Figure 5B, the MT presents an initial growth and a small overshoot from 0 to 0.2 h, this occasionally happens in the control of side-streams which is linked to the high nonlinearity of the system and to the direct action of the controller. For Mixture 1, it is important to note that for both the MT and the optimization method, the responses stabilize in less than 30 min after the perturbation.

TABLE 6 IAE values for the set point change for Mix 2.

	Mix 2 (molecular tracking)			Mix 2 (optimization method)		
	K	Ti	IAE	K	Ti	IAE
n-Butane	250.00	1.00	1.5989E−04	250.00	1.00	1.5965E−04
Isopentane	250.00	1.00	3.3830E−04	250.00	1.00	4.15010E−04
n-Pentane	250.00	1.10	1.9972E−04	250.00	1.00	1.88740E−04

Abbreviation: IAE, integral of the absolute error.

TABLE 7 IAE values for the set point change for Mix 3.

	Mix 3 (molecular tracking)			Mix 3 (optimization method)		
	K	Ti	IAE	K	Ti	IAE
n-Pentane	250.00	1.00	1.63754E−04	250.00	1.00	1.5986E−04
n-Hexane	250.00	1.00	1.49302E−04	170.00	7.00	1.3582E−03
n-Heptane	250.00	1.00	1.61955E−04	250.00	1.00	1.61267E−04

Abbreviation: IAE, integral of the absolute error.

The results for the case of Mixture 2 are shown in Table 6. These results are interesting in as much as in the case of the external component loops, *n*-butane and *n*-pentane, the IAE values are practically the same; with percentages of less than 1% in the case of the *n*-butane loop, and 6% in the case of *n*-pentane loop. Both loops have a slight advantage over the optimization method. However, the intermediate loop, isopentane, is better in the case of MT with 22.67% in quantitative terms. It is worth noting that contrary to better controllability, in the case of the steady-state design of the divided-wall unit for this mixture, the MT design suggests a 9% increase in the reboiler duty and approximately a 20% increase in the TAC compared to the optimization method.[11]

In this sequence, the results do match the open-loop results as the MT design methodology presents a slight advantage in the control properties. This is made evident by Lucero-Robles et al.,[41] where they show that the intermediate loops are determinant in the control properties in complex columns as in the case of DWC. Figure 6 shows that both design methods respond the same with Mixture 2, with a slight advantage in the intermediate loop in the case of MT. And in all Mixture 2 cases the responses stabilize after or before 25 min of perturbation.

The results of the closed-loop control study for Mixture 3 are shown in Table 7. Here, similar results to Mixture 2 can be observed. The extreme loops, *n*-pentane and *n*-heptane, show practically the same IAE value, and in the same order of magnitude with only a slight advantage for the optimization method and with less than a 2% difference in both cases. It is not conclusive to say that the control properties for the design yielded by the optimization method are better, on the contrary, the intermediate loop is better for MT. This would, as it was stated by Lucero-Robles et al.[41] represent better control properties for the design that is provided by MT. In this specific case, the results also match those shown in the open-loop control study where MT has a slight advantage in the control properties of the system. Figure 7 displays the behavior pointed out. Similar responses can also be observed in Figure 7A–C with a slight stabilization in the intermediate loop for the MT design. The steady-state design of the system in Montonati et al.[11] has approximately illustrated a 1% higher reboiler duty and an 8% higher TAC for MT design compared to the optimization method. In the case of Mixture 3, the responses stabilize before 40 min of perturbation.

It is important to point out that all the designs obtained by the MT method are larger, both in height and in diameter (as can be seen in the Supporting Information Material). This, according to authors such as Luyben et al.[42] presents an advantage in the dynamic properties of the systems, making them more resilient to the perturbations that the system may present. Although the MT method shows slight advantages in the control properties, it is also similar in design characteristics to other methods, yet with a shorter time.

The PI parameters and IAE values for the perturbation of the medium boiling component feed composition are shown in Table 8. Overall, better control dynamics are shown in the designs obtained with the MT method, since it presents the lowest IAE values for each perturbation case. With regards to the set-point changes and feed composition of the medium boiling component we have observed that in the case with a set point change (−5 wt% of medium boiling component) in the *n*-pentane, isopentane and in the *n*-hexane streams, the MT method presented the lowest IAE values, (fastest dynamic responses). These results are consistent with those observed in Figures 5B–7B, where the intermediate components by the MT method showed the lowest response time to reach the new steady-state (less than 0.5 h). The optimization method showed the longest settling time and high drift before reaching the new set point.

Note that, the maximum limits for the gain and time integral values in a PI controller depend on the specific process being controlled and the desired performance specifications. In general, the gain should be high enough to provide sufficient control action to meet the desired performance objectives, but not so high that the system becomes unstable or exhibits excessive overshoot or oscillation. Similarly, the time integral (also known as the reset time or integral time) should be set to a value that allows the controller to respond

TABLE 8 IAE values for the perturbation of the medium boiling component feed composition.

	Mix 1 (molecular tracking)			Mix 1 (optimization method)		
	K	Ti	IAE	K	Ti	IAE
Isopentane	250.00	1.00	1.1116E−04	250.00	1.00	2.4889E−04
n-Pentane	250.00	1.00	9.6374E−04	250.00	1.00	1.7048E−03
n-Hexane	250.00	1.00	1.0433E−04	250.00	1.00	2.4159E−04
	Mix 2 (molecular tracking)			Mix 2 (optimization method)		
	K	Ti	IAE	K	Ti	IAE
n-Butane	250.00	1.00	2.0713E−04	250.00	1.00	3.7337E−04
Isopentane	250.00	1.00	2.5941E−03	250.00	1.00	3.0089E−03
n-Pentane	250.00	1.10	2.2468E−04	250.00	1.00	4.1072E−04
	Mix 3 (molecular tracking)			Mix 3 (optimization method)		
	K	Ti	IAE	K	Ti	IAE
n-Pentane	250.00	1.00	1.9090E−04	250.00	1.00	2.0253E−04
n-Hexane	250.00	1.00	1.0732E−03	170.00	7.00	3.3672E−03
n-Heptane	250.00	1.00	1.7547E−04	250.00	1.00	1.9364E−04

Abbreviation: IAE, integral of the absolute error.

appropriately to changes in the process while avoiding excessive overshoot or instability. In some cases, the time integral may need to be adjusted dynamically based on the changing process conditions.

In the particular case of separation processes such as those studied in this work, it has been observed that the gain values of 250 is the maximum limit that allows to generate a sufficient control action to stabilize the process and in parallel to adequately model the opening of the control valve. Similarly, it has been observed that 1 is the minimum integral time limit to adequately eliminate the offset at the new set point.[32,38,43]

Once both open-loop and closed-loop control studies have been carried out, it is possible to generate the following preliminary conclusions related to the design of the equipment with both strategies. The equipment size, including the diameter and internal flows, can significantly impact the controllability of a distillation column. Larger-diameter columns generally have more stable dynamics and are easier to control than smaller-diameter columns. Internal flows, such as tray spacing and hole size, can also affect the column's dynamics and may need to be adjusted to achieve optimal control.

Moreover, the equipment size can impact the time constants of the system, which affect the dynamic response of the process to changes in the input and disturbance variables. For instance, a smaller diameter column may have a faster response to changes in input and disturbance variables but may also be more difficult to control due to its faster dynamics. Conversely, a larger diameter column may have a slower response but may be easier to control because of its slower dynamics. In Tables S1–S3 of the Supporting Information Material, it can be observed that, in all cases, the DWC designs using the MT methodology have a larger diameter. Table S1 demonstrates that the column diameter is 1.87 m when the design is obtained using the MT method, whereas it is 1.10 m for the design obtained using the optimization method. Similarly, Table S2 shows values of 2.02 and 1.74 m for the designs obtained by MT and the optimization method, respectively. Finally, in Table S3, the diameters are 1.78 and 1.52 m for the designs obtained by MT and the optimization method, respectively. As mentioned earlier, the column diameter is one of the most critical factors in terms of the controllability of a column. This is because smaller-diameter designs are more susceptible to perturbations that they may encounter. It can be observed that the MT methodology aims to strike a balance between the diameter and height of the column. As a result, the established designs are superior in terms of control and cost-effectiveness.

The MT methodology presented in comparison with other design and control integration methodologies available in the literature such as Flores-Tlacuahuac and Biegler[44] and Moon et al.[45] is that the latter approaches are limited by the complexity of the resulting optimization problems and the computational effort they represent. In comparison, the MT methodology is a robust technique for tracking controllability properties in the integrated design problem formulation. Moreover, it is a fast and computationally efficient methodology.

We would like to emphasize that the primary objective of the present study is to conduct a preliminary investigation into the control properties of the different designs presented. Specifically, it aims to efficiently evaluate and compare the control properties of these designs. While comprehensive control studies often involve extensive frequency analysis and validation, our study provides initial insights into the performance of various design methodologies. By focusing on the open-loop and closed-loop responses, we can promptly assess which design exhibits superior control properties. Despite its preliminary nature, this study holds significant value. It allows for a comparison of different design approaches and facilitates a better understanding of their relative strengths and weaknesses. Moreover, it provides a foundation for further exploration and refinement of control methodologies, enabling researchers to make more informed decisions in future studies. While acknowledging the limitations of the study and its preliminary nature, we underscore the importance of its contribution to evaluating and comparing design methodologies based on their controllability outcomes.

4 | CONCLUSIONS

With the use of both the SVD analysis and the closed-loop control methodology, the dynamic properties of six different DWC designs were obtained. Overall, all the studied designs display acceptable controllability and closed-loop performance for the investigated steady-state methodologies. According to SVD, the designs obtained by the MT method have slightly better controllability as it presents lower values of the condition number and higher values in the minimum singular value. The closed-loop study presented similar quantitative results with a slight advantage in the case of the MT method in two of the three intermediate loops. In general terms, we could say that the control properties obtained with both DWC design methods are very similar (with a slight advantage in the case of MT) yet this does not make the designs obtained by the optimization method inoperable. Rather, under the characteristics of DWC dimensions (such as diameter and height) which, in the case of the sequences that are obtained by the MT method, are slightly larger, it is helpful to possess enhanced control properties that better withstand the disturbances of the process. Although the obtention of designs through the MT method (which reduces the time to find an optimal solution) is favorable, there is also a slight advantage in control properties.

NOMENCLATURE

DWC	divided-wall columns
IAE	integral of the absolute error
K_c	proportional gain
MRI	Morari Resilience Index
SVD	singular value decomposition
t	time (h)
U	matrix of right singular vectors
V	matrix of left singular vectors
$y(t)$	value of the measured variable in a given time
y_d	Set point
$\varepsilon(t)$	Function of integral time
Σ	Matrix of singular values
τ_i	integral time constant of the controller (min)
γ	condition number
σ_*	minimum singular value
K	steady-state gain matrix

AUTHOR CONTRIBUTIONS

Seyed Soheil Mansouri: Conceptualization (equal); funding acquisition (equal); investigation (equal); methodology (equal); supervision (equal); writing – review and editing (equal). **Jens Abildskov:** Investigation (equal); methodology (equal); supervision (equal); writing – review and editing (equal). **Thomas Bisgaard:** Formal analysis (equal); investigation (equal); methodology (equal); project administration (equal); supervision (equal); writing – review and editing (equal). **Nima Nazemzadeh:** Formal analysis (equal); investigation (equal); methodology (equal); writing – review and editing (equal). **César Ramírez-Márquez:** Formal analysis (equal); investigation (equal); methodology (equal); writing – review and editing (equal). **Eduardo Sánchez-Ramírez:** Formal analysis (equal); investigation (equal); methodology (equal); writing – review and editing (equal). **Juan Gabriel Segovia-Hernández:** Formal analysis (equal); investigation (equal); methodology (equal); project administration (equal); supervision (equal); writing – review and editing (equal).

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflict of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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