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Dynamic behavior of the intensified alternative configurations for quaternary distillation

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ABSTRACT

Process intensification emerges as an important tool in the synthesis of multicomponent distillation configurations aimed at the reduction of the energy use and capital costs. Operational and fixed costs savings coupled with simplicity and controllability design configurations appear as an essential characteristic for industrial acceptance of intensified configurations. Following the aforementioned principles, two intensified configurations for the separation of quaternary mixture, were considered. These configurations were recognized as a valid alternative to overcome the complexity of the quaternary Petlyuk scheme.

The study of the dynamics and control properties represent an important research issue in the analysis of new configurations to prove their effective applicability. The theoretical control properties of the alternative intensified configurations were obtained using the singular value decomposition technique in all frequency domain. In order to complete the control study, the distillation schemes were subjected to closed-loop dynamic simulations. The results show that there are cases in which the intensified sequences do not only provide energy savings, but also may offer dynamic advantages in comparison to the conventional four-component scheme.

1. Introduction

Distillation is the most commonly used separation technique in the chemical and petrochemical process industries but, at the same time, it is also the most energy intensive unit operation. 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 [1]. Data from the United States Department of Energy indicate that distillation columns, in the U.S., consume 5.07 million TJ per year; corresponding to 43% of the total installed net capacity of the 439 nuclear power plants in operation worldwide [2]. To overcome the problem of this high energy consumption, several heat-integrated and fully thermally coupled distillation systems (also called Petlyuk columns) were studied, and it has been proven that thermally coupled configurations are promising alternative energy solutions. Theoretical studies have shown that the Petlyuk scheme, for ternary mixtures separation, can achieve around 30% energy and capital cost savings, compared to conventional distillation systems. Any reduction of energy consumption will bring not only economic benefits but also environmental benefits [3–6]. Reported studies reveal that the ternary-Petlyuk column provides the maximum energy reduction in distillation columns [7]. 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, where all the products are separated in a single shell column.

A number of design and optimization methods for the ternary-Petlyuk column have been proposed by several researchers [8,9]. Despite the energy and capital advantages of ternary-Petlyuk configuration, its industrial application began two decades ago; the world's first ternary-Petlyuk configuration was established by BASF in 1985. In addition, understanding the control and operability issues has improved greatly. Since then, many ternary-Petlyuk arrangements have been established worldwide,

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such as in Europe, South Africa, and the US [10]. Amminudin et al. [11] noted that industrial acceptance and commercialization of Petlyuk columns 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 Petlyuk configuration in the near future, and it might become a standard distillation configuration in chemical process industries over the next 50 years [12]. As industrial separation problems very often involve four or more components, due to complexity, it is impossible to find all the feasible DWC columns by innovative activities. On the other hand, industrial experience shows that the optimal system for a specific application can only be guaranteed by predefining all of the feasible options [13].

It is possible to assert that, for the case of three component separations, the Petlyuk, and its equivalent DWC configuration, has the potential to save a significant amount of energy, different design methods are available, structural complexity may be overcome by using thermodynamically equivalent configurations, and moreover, the control issues may be solved [14]. It is clear that the same results are aimed for a different number of feed components. Moving from three to four components, the complexity of the Petlyuk and DWC structure increases and up to now only a few studies are focused on the possible applications of these configurations [15,16]. The focus of this work is to analyze the dynamic behavior (closed loop control policy, using PI controllers) of alternative configurations to the Petlyuk configuration for a four-component separation. These new distillation configurations were obtained following the systematic method based on four strategies as proposed by Rong [17]. The steady-state study with specific quaternary mixtures showed promising results to save both energy and capital costs when compared to the more complex quaternary Petlyuk scheme [18]. The study of the dynamics and control properties represents an important research problem in the analysis of these new configurations.

2. Alternative intensified configurations

The conventional configurations used to derive the four-component Petlyuk configuration separation are illustrated in Fig. 1. In Fig. 1a) the second and the third columns are connected by a one-way transport for the BC (OW)stream and in Fig. 1b) the BC mixture is connected by a two-way transport stream (TW).

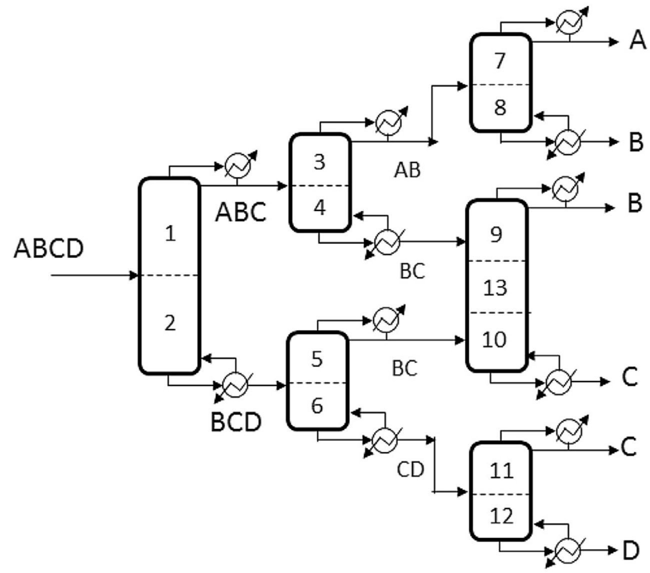

Fig. 2. The simple column configuration (SCC) for the fully sloppy separation sequence of a quaternary mixture.

The number of columns in conventional configurations is N-1, which is equal to the number of columns in any sharp conventional configurations, and which is also the minimum number of columns for any non-sharp conventional configurations [19,20]. However, Rong [21] illustrated that such non-sharp conventional configurations with N-1 columns lost the structural flexibility to produce the intensified distillation configurations with less than N-1 columns. In order to produce the intensified configurations with fewer columns, Rong [17] illustrated that the simple column configuration (SCC) representation for any non-sharp sequence is necessary as the starting point, which keeps all the structural flexibility to derive the intensified configurations. The simple column configuration for the non-sharp sequence of the quaternary intensified columns is illustrated in Fig. 2.

Starting from this SCC representation, a method to derive all the possible intensified distillation configurations with less than N-1 columns was presented; we derived five distinct alternative intensified distillation configurations for the quaternary distillation sequences [22].

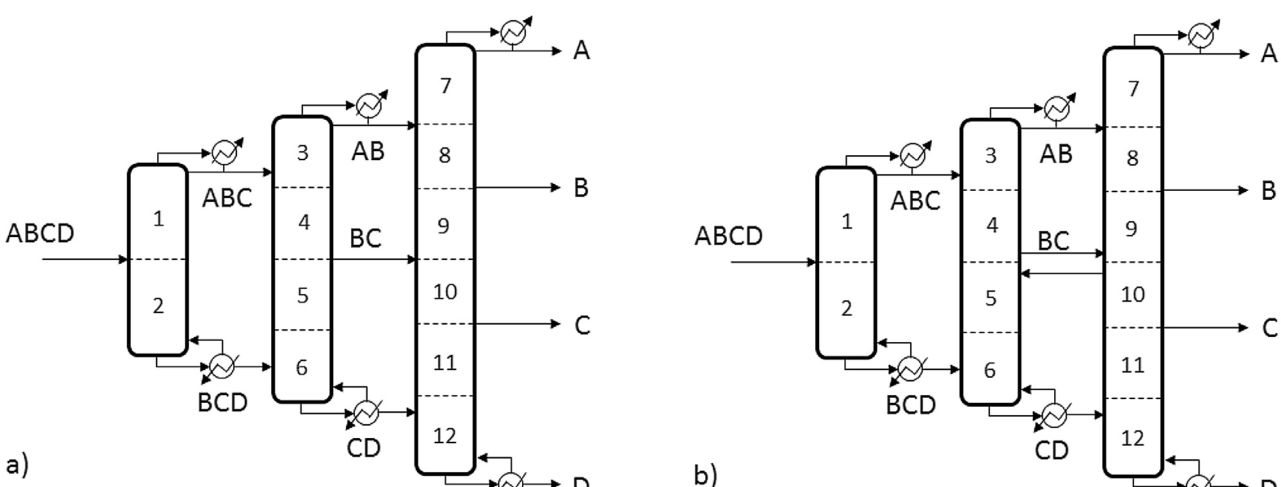

Fig. 1. The conventional configurations used to derive the Petlyuk column for four components separation: a) one-way transport for BC stream (OW); b) two-way transport for BC stream (TW).

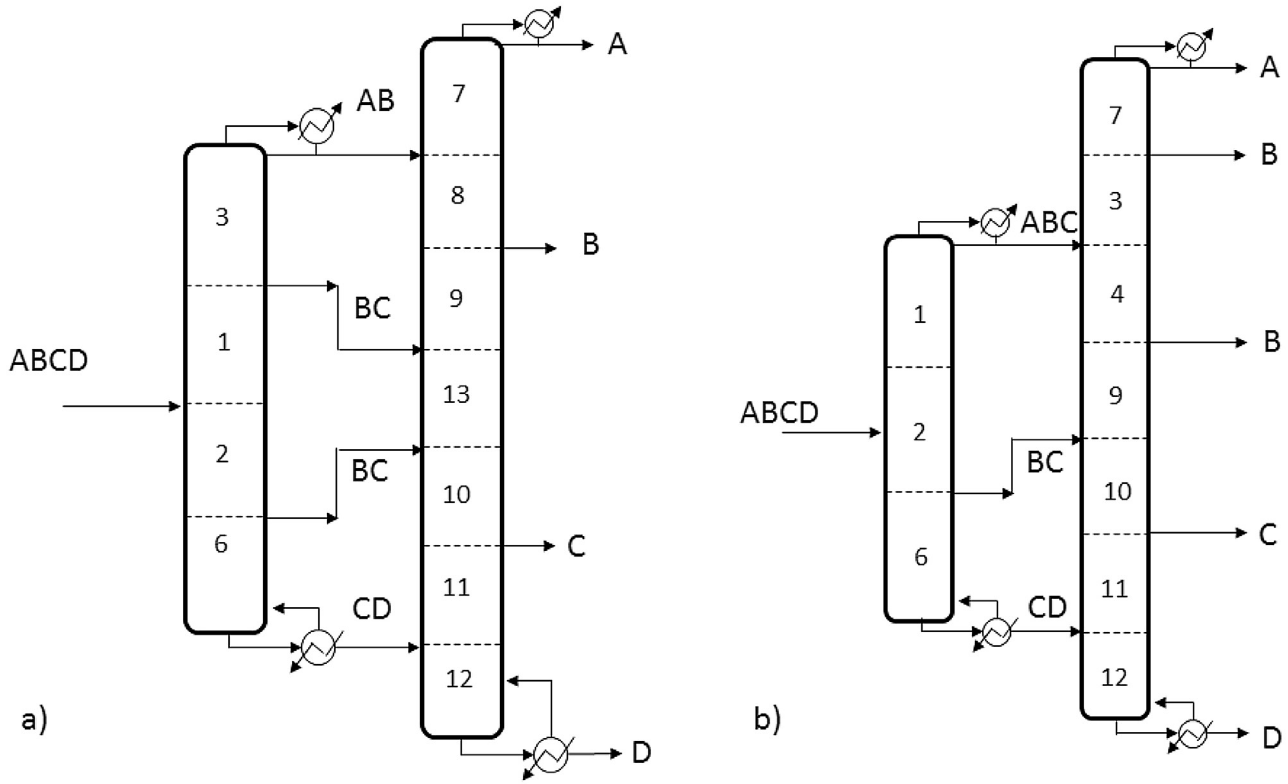

Fig. 3. The alternative intensified configurations for quaternary distillation: a) two intermediate BC transport streams (TSI), b) one intermediate BC transport stream (OSI).

As indicated earlier, the study of the dynamic and control properties represents the essential step in the analysis of such new intensified configurations.

Two intensified alternative configurations are selected as shown in Fig. 3, each with only two columns. Fig. 3a) has two BC streams intermediately transporting between the two columns (TSI), while Fig. 3b) has one BC stream intermediately transporting between the two columns (OSI). Like the conventional configurations in Fig. 1, all the products are obtained in the final column as shown in Fig. 3.

It is useful to clear the structural changes of these intensified configurations while modifying the SCC representation in Fig. 2, which can help to determine the system's parameters for the detailed study of both steady-state and dynamic performance. To systematically derive the intensified configurations from the SCC of Fig. 2, four strategies have been proposed to change the structure and to reduce the number of columns and heat exchangers during the system's derivation. To save space, the four strategies are not repeated here, but can be referred to in earlier work [22]. The two intensified alternatives shown in Fig. 3 are generated following the structural changes in certain steps systematically, which are elaborated as follows.

2.1. Generating alternative intensified configuration TSI

Starting from the SCC in Fig. 2, four steps are needed to achieve the alternative TSI. Step 1: combining the columns co-producing the middle products B and C through strategy 1. Step 2: eliminating condenser ABC and reboiler BCD through strategy 2. Step 3: removing the movable sections 3 and 6 to rearrange the column sections through strategy 3. Step 4: eliminating the single-section-side columns 4 and 5 through strategy 4 to generate the configuration TSI.

2.2. Generating alternative intensified configuration OSI

Starting from the SCC in Fig. 2, four steps are needed to achieve the alternative in Fig. 3 OSI. Step 1: combining the two columns co-producing the middle product C through strategy 1. Step 2: eliminating reboiler BCD, condenser BC, and condenser AB through strategy 2. Step 3: removing the movable sections 6, 7 and 13 + 10 + 11 + 12 together to rearrange the column sections through strategy 3. Step 4: eliminating the single-section-side columns 5, 8 and 9 through strategy 4 to generate the configuration OSI.

Clearly, the two intensified alternatives in Fig. 3 have similar structural features to the conventional configurations shown in Fig. 1. All products are obtained in the final column, and there may be one stream or two streams transporting the intermediate mixture BC. However, as shown in Fig. 3, only two columns are used in the intensified configurations.

3. Dynamic analysis of complex sequences

The calculation of the condition number and minimum singular value have been carried out through the singular value decomposition of the relative gain matrix of the design in the nominal point. For example, considering the mathematical expression of Eq. (1), which represents the relative gain matrix of a linear system:

$$K = W \sum V^T \tag{1}$$

where W and V are unitary matrixes and $\sum$ is a matrix whose diagonal elements are the singular values σ. Assuming that K is not singular, then the condition number of K, is a positive number which relates the minimum singular value (σ_*) and the maximum singular value (σ^*), none of these two being zero, the condition number (γ), can be estimated as in Eq. (2):

$$\gamma = \frac{\sigma^*}{\sigma_*} \qquad (2)$$

Large values of σ_* and small values of σ^* are desirable so that the process may assimilate the perturbations without system destabilization. Therefore, low values of the condition number of design are preferable over high values. In this study the condition number of the relative gain matrix obtained in an open loop control strategy for each design is estimated by generating the relative gain matrix in a nominal state of each distillation sequence design. The elements of this matrix are calculated through the introduction of perturbations in the manipulated variables. The magnitude of the perturbations was defined as a 0.5% positive change in the values of the manipulated variables in the nominal state, the level of these perturbations is low enough so that it is assumed that the response of the system can be approached as a first order response. It is paramount to note that the gain matrix is scaled to take into account variations of the different orders of magnitude of the perturbations.

One drawback of the singular value decomposition is the fact that the singular values depend on the system of units used. Applying the singular value decomposition to the relative gain matrix will include the effects of such units. Therefore it is important to use a scaling method to remove this dependency and provide reliability of the results as well as a physical meaning. In order to approach this issue, different authors have proposed scaling methods for the manipulated variables and the control variables [23–26]. For the distillation configurations examined in this study, the control variables are the molar purities of each component in the corresponding product mixtures, and these are naturally bounded between 0 and 1. Manipulated variables are used for each distillation configuration; they are the reflux ratio, the reboiler duty, and side stream molar flowrates. These manipulated variables are unit dependent and are not bounded. To eliminate this drawback, we propose to limit the manipulated variables considering that the maximum aperture that can be reached by the control valves is twice the nominal value of the steady state; therefore, in principle, the valves are open to 50%. This implies that for the relative gain matrix, the step change is implemented in the manipulated variable and is divided by twice the steady state to have the same range of variation in both the closing and opening operations of the control valves. This allows for a physical interpretation of the way of scaling of the manipulated variables that links the amount of change of the manipulated variables with the magnitude of change of the position corresponding to the valve stem, which can only vary between 0 and 100% (0 and 1). With this form of scaling, dimensionless standardization is achieved simultaneously with the manipulated variables.

For the closed-loop analysis, several issues must be defined first, such as the control loops for each system, the type of process controller to be used, and the values of the controller parameters. This analysis was based on proportional-integral (PI) controllers. In the case of distillation columns, the selection of manipulated variables, for perturbation in the gain matrix, are relatively well established and have been used successfully in practice, at least for conventional columns. The magnitude of the perturbations was defined as a 1% negative change in the values of the manipulated variables in the nominal state. Typically, in a distillation column, there are four control handles: distillate flow rate, reflux flowrate, bottoms flow rate, and the heat rate into the reboiler. However, material balance usually dictates that two of these handles must be used to control the level of the accumulator tank and the level at the base of the column. Therefore, only two variables can be manipulated. The reflux ratio and the heat supplied to the reboiler have been found as some of the best-manipulated variables in control studies [23–25]. The choice of the Proportional-Integral (PI) as a type of controller was due to its wide diffusion in industrial practice. Several alternatives are available for tuning up the controller parameters [26,27]. As shown in the work of Segovia-Hernandez et al. [28] and Ibarra-Sánchez et al. [29], a suitable IAE tuning process (trial and error procedure) was used to maintain a required control performance during severe conditions, and this method provides the lower value. We attempted a common ground for comparison by optimizing the controller parameters, proportional gains (K_C) and reset times (τ_i), for each configuration following the integral of the absolute error (IAE) criterion [23]. Therefore, for each loop, an initial value of the proportional gain was set; a search over the values of the integral reset time was conducted until a local optimum value of the IAE was obtained. The process was repeated for other values of the proportional gain. The selected set of controller parameters was the one that provided the lower value of the IAE. Although the tuning procedure is fairly elaborate, the control analysis is conducted based on a common tuning method for the controller parameters. For the dynamic analysis, individual set point changes for product composition were implemented. One of the key parts of the dynamic analysis is the selection of control outputs and manipulated variables for each control loop. A well-known structure is based on energy balance considerations, which yields to the so-called LV control structure in which the reflux flow rate (L), the vapor buildup rate (V), and side flow rate are used to control the distillate, bottom, and side stream outputs compositions[23]. For the control of the product stream obtained as an overhead product, the reflux flow rate was used, whereas for the control of the product stream that is obtained as a bottom product, the reboiler heat duty was chosen. It should be mentioned that such control loops have been used with satisfactory results in previous studies on thermally coupled systems [30–32].

4. Case study

The hydrocarbon mixture composed of *n*-butane (A), *n*-hexane (B), *n*-octane (C), and *n*-decane (D), was defined as a case study. Two composition cases with the following molar composition were considered; Case I as 0.1, 0.4, 0.4, 0.1 in A–D, respectively; and Case II as 0.4, 0.1, 0.1, 0.4 in A–D, respectively. The feed flow rate was taken as 100 kmol/h as a saturated liquid, and the purities for the product streams were assumed as 99.0 for all components. To avoid the use of refrigerants that would have an adverse impact on the economics of the separation sequence, the design pressure for each column was chosen such that all condensers could operate with cooling water. The thermodynamic properties of the mixture were estimated with the Chao-Seader correlation.

The different column configurations were simulated by means of Aspen Plus. The initial design parameters of the configurations reported in Fig. 1 was obtained using the Underwood-Gilliland-Winn short-cut method then the rigorous tray-by-tray model was used. The design parameters were calculated in order to get the lower value of energy requirements. The intensified configurations of Fig. 3 were designed using the sequential design method based on the correspondence of the column section functionality [14,33]. The trial and error procedure reported by Segovia-Hernandez et al. [34] was used to optimize the thermal coupling flowrates. The trial and error strategy is summarized in Fig. 4 [34,35].

Design parameters for the columns in the configuration OW for Case I are shown in Table 1. In Table 1, N_T is the total number of stages, N_F is the feed stage, N_{INT} is the interlinking stages, N_S is the stage where a side stream is drawn, D_F is the distillate flow rate, FL_1 is the liquid interlinking flow rate from column 2 to column 3, F_S is the molar flow rate of the side stream, R is the reflux ratio, Q is the

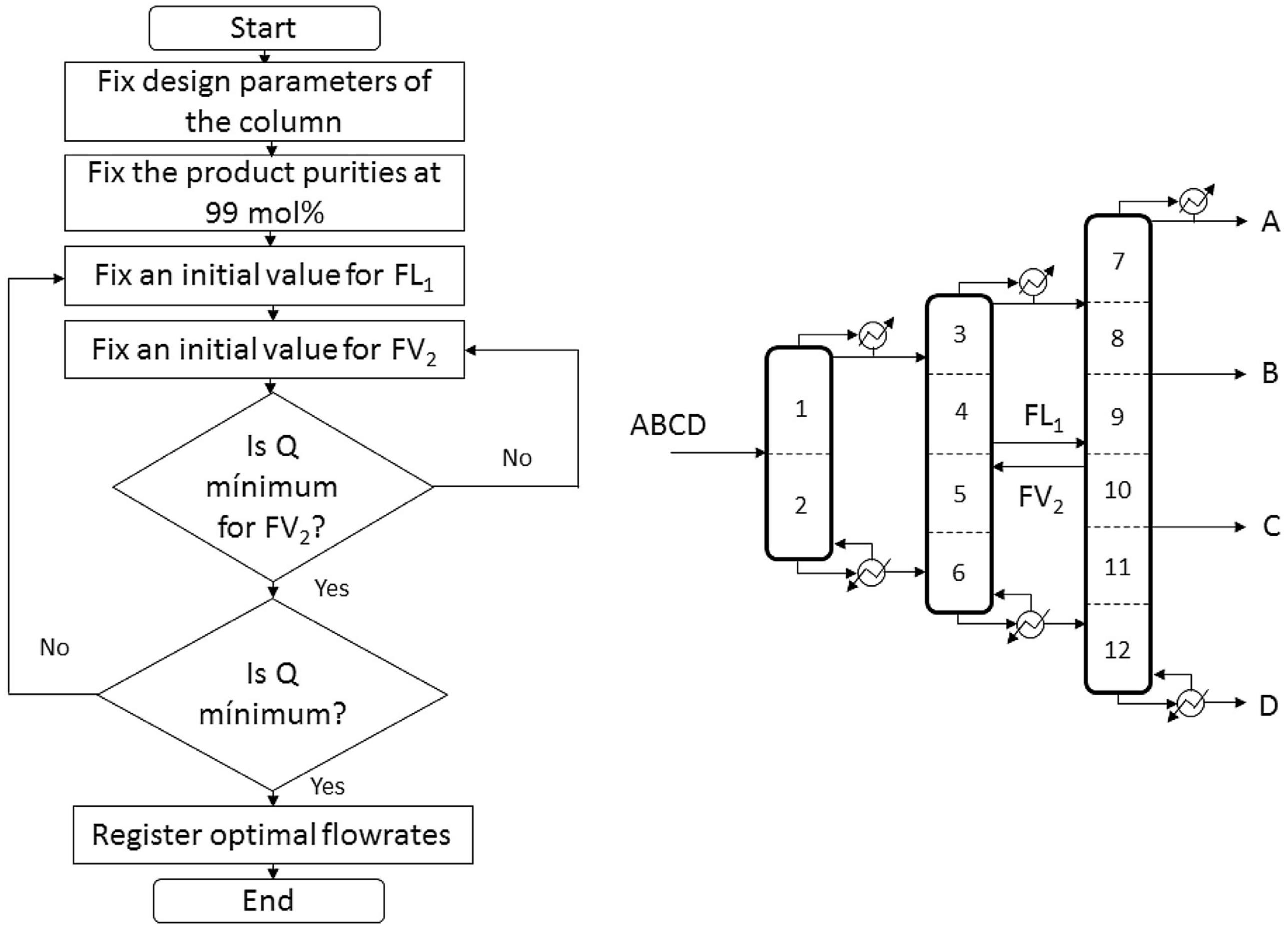

Fig. 4. Methodology for the parametric optimization of the conventional configuration TW.

heat duty and D is the diameter of the column, which is calculated through the Tray Sizing tool of Aspen Plus.

The purities of the components are fixed to their required values through the DesignSpec tool. Table 2 shows the reboiler duty values obtained of the four configurations examined and for both composition cases.

5. Results

The controllability analysis was performed in two parts. The theoretical control properties of the four schemes were first predicted through the use of the singular value decomposition (SVD) technique, and then closed-loop dynamic simulations were conducted to analyze the control behavior of each system and to compare those results with the theoretical predictions provided by SVD.

5.1. Singular value decomposition

The objective is to identify the systems with higher minimum singular values and lower condition numbers; those systems are expected to show the best dynamic performance under feedback control.

Figs. 5 and 6 shows the results obtained from the application of the SVD for each sequence. From Fig. 5a, relative to the composition case I, it can be observed that the intensified configuration OSI has the highest minimum singular value. From Fig. 6a, corresponding to the composition case II, it is evident that the conventional configuration OW has the highest minimum singular value.

These two sequences are expected to show the lowest control effort as confirmed in Figs. 5b and 6b from the condition number evaluation. For the two composition cases we consider that these

Table 1
Design parameters for the conventional configuration OW and composition case I.

	Column 1	Column 2	Column 3
N_T	17	45	49
N_F	9	10, 31	9, 40
N_{INT}	–	13	23
N_S	–	–	15, 30
D_F (kmol/h)	50.00	29	9.99
FL_1 (kmol/h)	–	42	–
F_S (kmol/h)	–	–	40.04, 39.90
R	3	1.33	4.85
Q (kW)	1746.6	491.3	416.2
D (m)	1.20	0.63	0.60

Table 2
Reboiler duty of the considered configurations.

	Case I [kW]	Case II [kW]
OW	2654.277	3553.693
TW	2211.265	3607.315
TSI	2631.991	3096.109
OSI	2494.117	2373.442

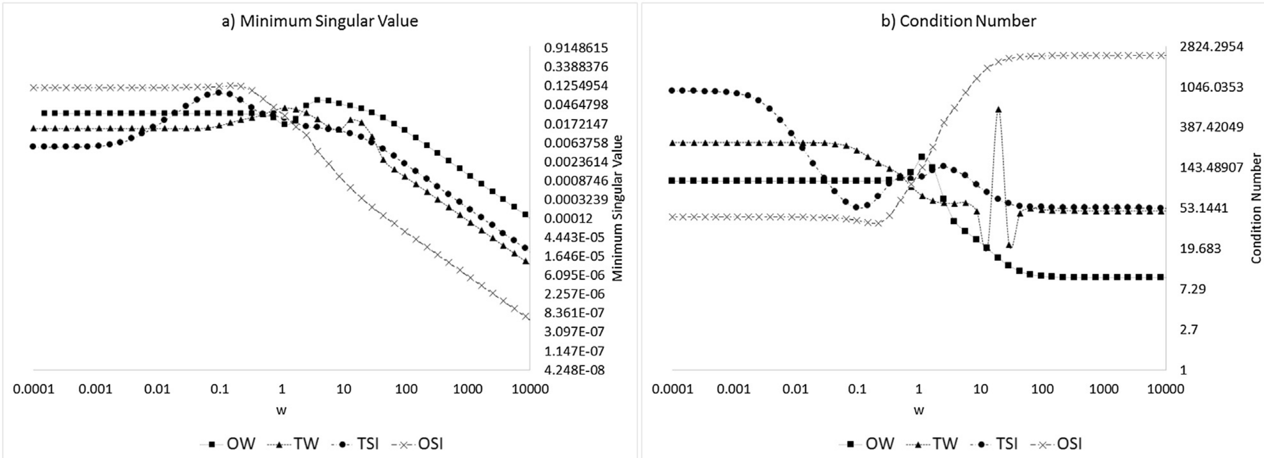

Fig. 5. a) Minimum Singular Value and b) Condition Number, for Case I.

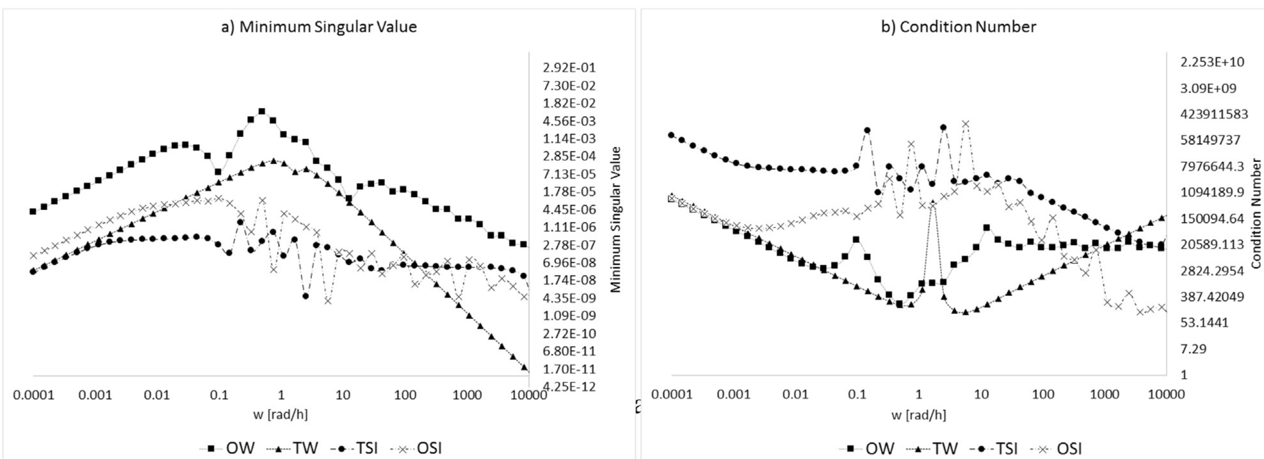

Fig. 6. a) Minimum Singular Value and b) Condition Number, for Case II.

sequences are better conditioned to the effect of disturbance compared to the other alternatives.

Even if it seems that the configurations OW and OSI have good control properties, due to the qualitative nature of SVD, it is necessary to include the closed loop test to know which of those sequences has the best dynamic behavior.

5.2. Closed-loop simulations

The sequences under analysis were compared through the evaluation of IAE obtained following the methodology summarized in Fig. 7. Tables 3 and 4 shows the IAE values obtained for both composition cases considering LV configurations in a closed loop.

5.2.1. Dynamic behavior of the light component

The results of the dynamic test for a negative step change in the set-point of the light component (A) are displayed in Figs. 8 and 9.

From Table 3, when component A and composition case I are considered, it may be observed that the best IAE value is associated to the TSI configuration. Similarly, for composition case II, according to the results reported in Table 4, the OSI configuration has the lower IAE, together with the lower overshoot for both composition cases, may be seen in Figs. 8 and 9.

It is evident that, for the intensified alternatives, the control of the light component is favored. In any case, it is important to highlight that even if the dynamic behavior of intensified designs is relatively better, the difference in the IAE values

Table 3
Optimal values of close loop for Case I.

	N-Butane			*N*-Hexane			*N*-Octane			*N*-Decane		
	K [%%]	Ti [min]	IAE [–]	K [%%]	Ti [min]	IAE [–]	K [%%]	Ti [min]	IAE [–]	K [%%]	Ti [min]	IAE [–]
OW	50	2.1	3.88E-04	250	0.3	5.45E-05	250	0.4	8.96E-05	250	0.25	3.72E-05
TW	250	6	1.13E-03	10	13.5	3.16E-03	250	2.5	5.43E-04	250	0.25	3.82E-05
TSI	45	1.5	2.69E-04	250	0.5	1.06E-04	250	0.2	4.23E-04	250	0.25	3.65E-05
OSI	30	1.5	2.81E-04	250	0.8	1.24E-04	250	0.15	4.20E-04	250	0.25	3.63E-05

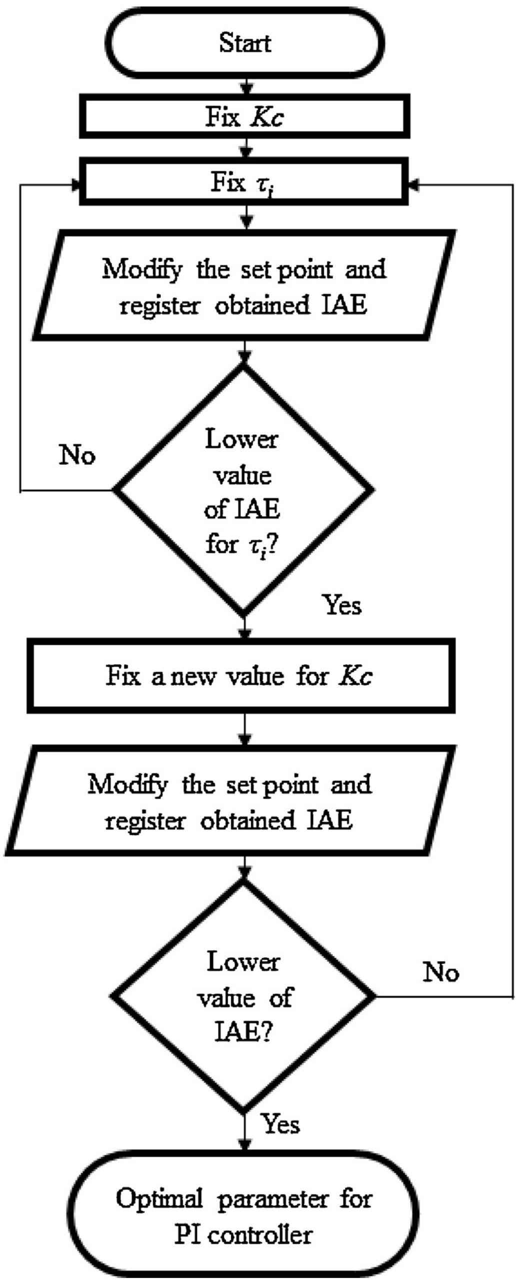

Fig. 7. Methodology to get the lower value of IAE.

Table 5
Simulation results with continuous disturbances.

N-Butane	Kc [%%]	Ti [min]	IAE 0.5% + [–]	IAE [–]	IAE 0.5% – [–]	IAE [–]
OW	50	2.1	3.20E-04	6.08E-04	8.26E-04	2.73E-03
TW	250	6	7.82E-04	1.44E-03	1.98E-03	2.53E-03
TSI	45	1.5	1.43E-04	2.89E-04	4.13E-04	7.32E-04
OSI	30	1.5	1.85E-04	4.91E-04	7.18E-04	9.62E-04

between conventional configuration OW and intensified cases are small.

5.2.2. Dynamic behavior of the intermediate components

In Figs. 10–13 we report the dynamic responses obtained when the set-point of the intermediate components are changed. For both composition cases and for both the intermediate components, the conventional configuration OW exhibited a faster answer compared to the other alternatives with minor overshoots in the responses. This result was also confirmed by the analysis of the IAE values reported in Tables 3 and 4.

On the other hand, the conventional configuration TW showed the worst dynamic behavior for both composition cases.

5.2.3. Dynamic behavior of the heavy component

The dynamic responses reported in Figs. 14 and 15 were obtained for a negative step change in the set-point equal to 1%.

All the configurations considered showed a good dynamic response, with relatively low values of settling times. In addition to the visual observations of the dynamic responses, the quantitative measure of the IAE is used to identify the best configuration. According to the data reported in Tables 3 and 4, for both composition cases, the intensified configuration OSI has the best dynamic behavior, this configuration shows the minor overshoots in the responses as shown in Figs. 14 and 15. For the heavy components, the intensified configurations are favored.

To test the dynamic stability of all schemes, a study was performed by disturbing the stream composition of *n*-butane relative to the composition case I. Positive and negative 0.5% disturbances were applied. The first disturbance was positive and the second negative in order to return at the initial *n*-butane concentration. The third disturbance was negative, and finally the fourth was positive, setting the *n*-butane composition to its initial value. The results are reported in Fig. 16.

As also reported in Table 5, it is evident that all the sequences are able to stabilize the system to various disturbances in the composition of *n*-butane.

As summary, for the lightest component, for both composition cases, the intensified configurations have better control properties, this can be shown in Figs. 8 and 9 we can see a higher overshoot for *n*-butane and *n*-hexane for the TW configuration and less oscillation in the response for intensified configurations, which can help us to say that the greater number of columns penalize the control behavior for the light components. For the intermediate components, the conventional configuration OW reported better performances and the best responses are reported in Figs. 10–13 where less overshooting is observed. Finally, even if the IAE values are quite similar for the heaviest components, the intensified alternative OSI showed the best control properties, this is shown in Figs. 14 and 15, where we can see a minor oscillation for the intensified responses, a reduced number of columns, and an

Table 4
Optimal values of close loop for Case II.

	Case II											
	N-Butane			*N*-Hexane			*N*-Octane			*N*-Decane		
	K [%%]	Ti [min]	IAE [–]	K [%%]	Ti [min]	IAE [–]	K [%%]	Ti [min]	IAE [–]	K [%%]	Ti [min]	IAE [–]
OW	50	1.5	2.71E-04	250	1	2.01E-04	250	0.4	2.14E-04	250	0.25	3.75E-05
TW	250	1.5	6.43E-04	45	15.8	3.04E-03	250	0.8	1.59E-03	250	0.25	3.98E-05
TSI	250	0.8	1.43E-04	250	2.25	6.21E-04	250	2	7.79E-03	250	0.25	3.67E-05
OSI	250	0.6	1.19E-04	250	2	8.39E-04	250	1.25	1.98E-02	250	0.25	3.64E-05

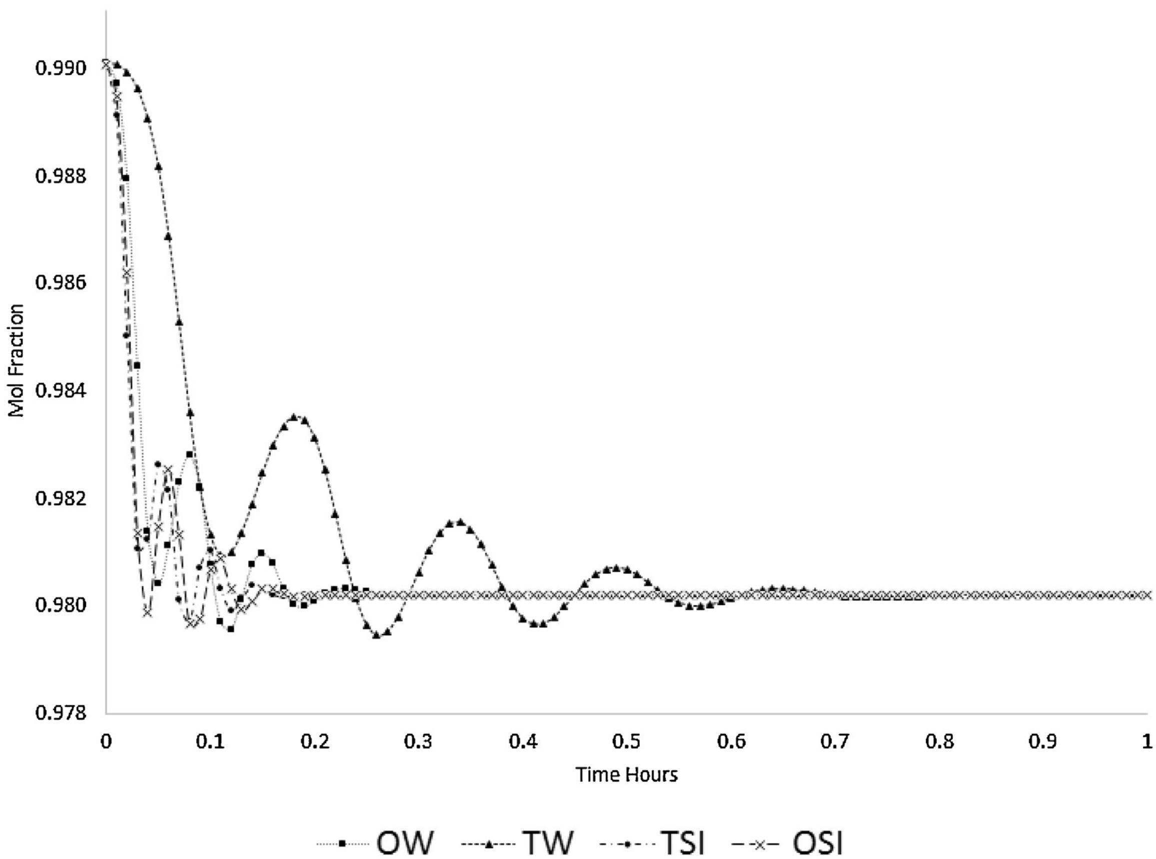

Fig. 8. Dynamic responses for *n*-butane in Case I.

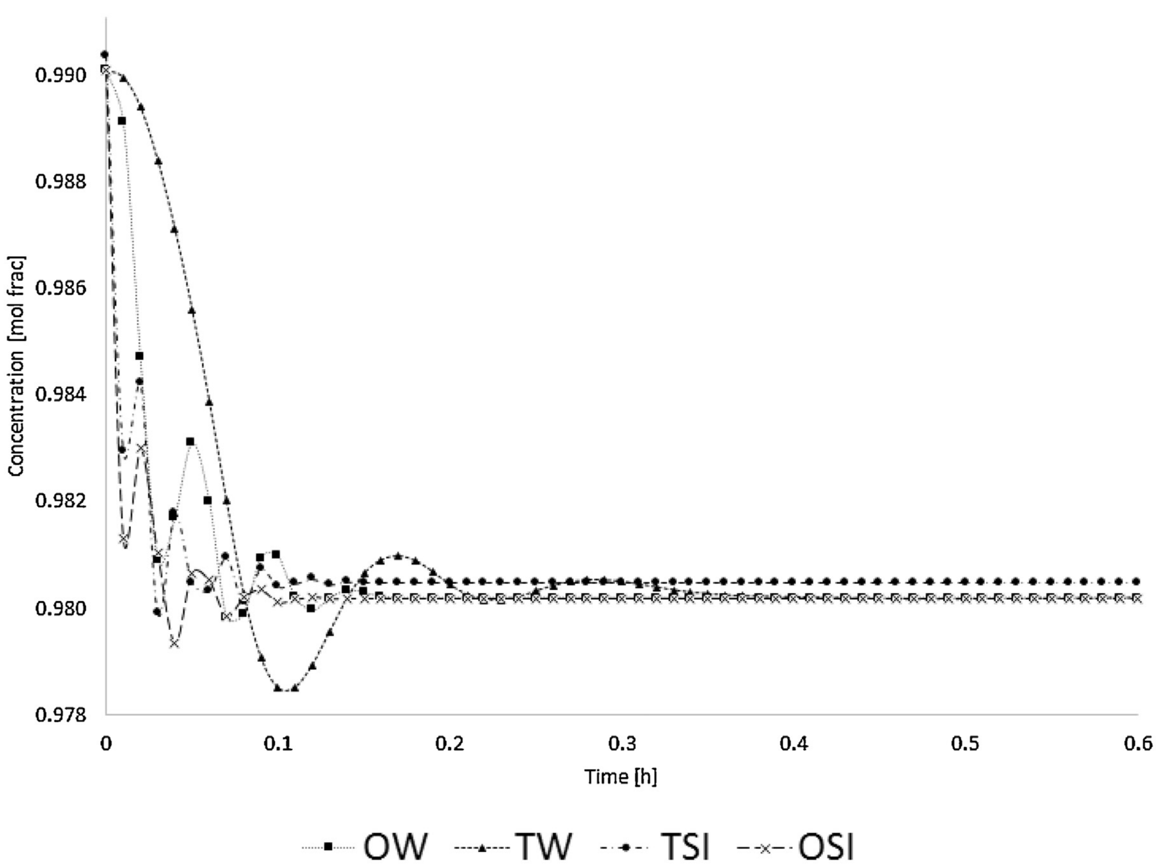

Fig. 9. Dynamic responses for *n*-butane in Case II.

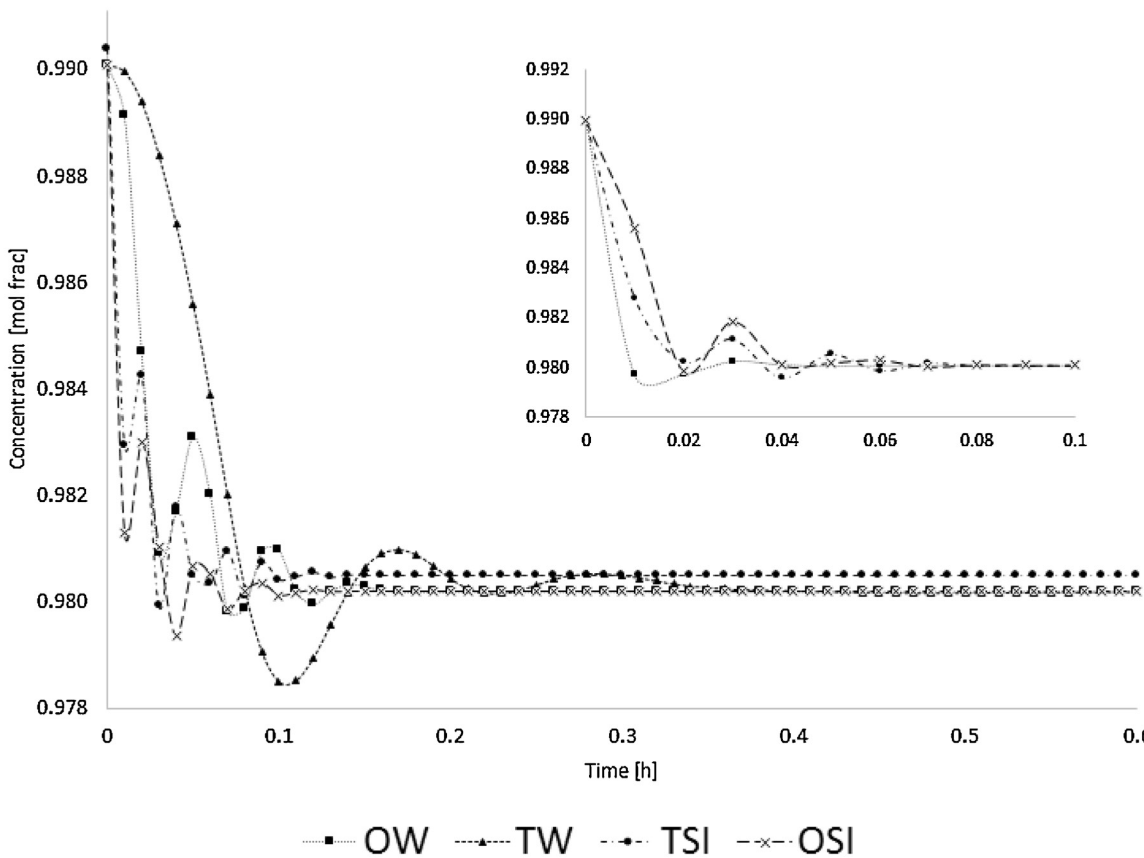

Fig. 10. Dynamic responses for *n*-hexane in Case I.

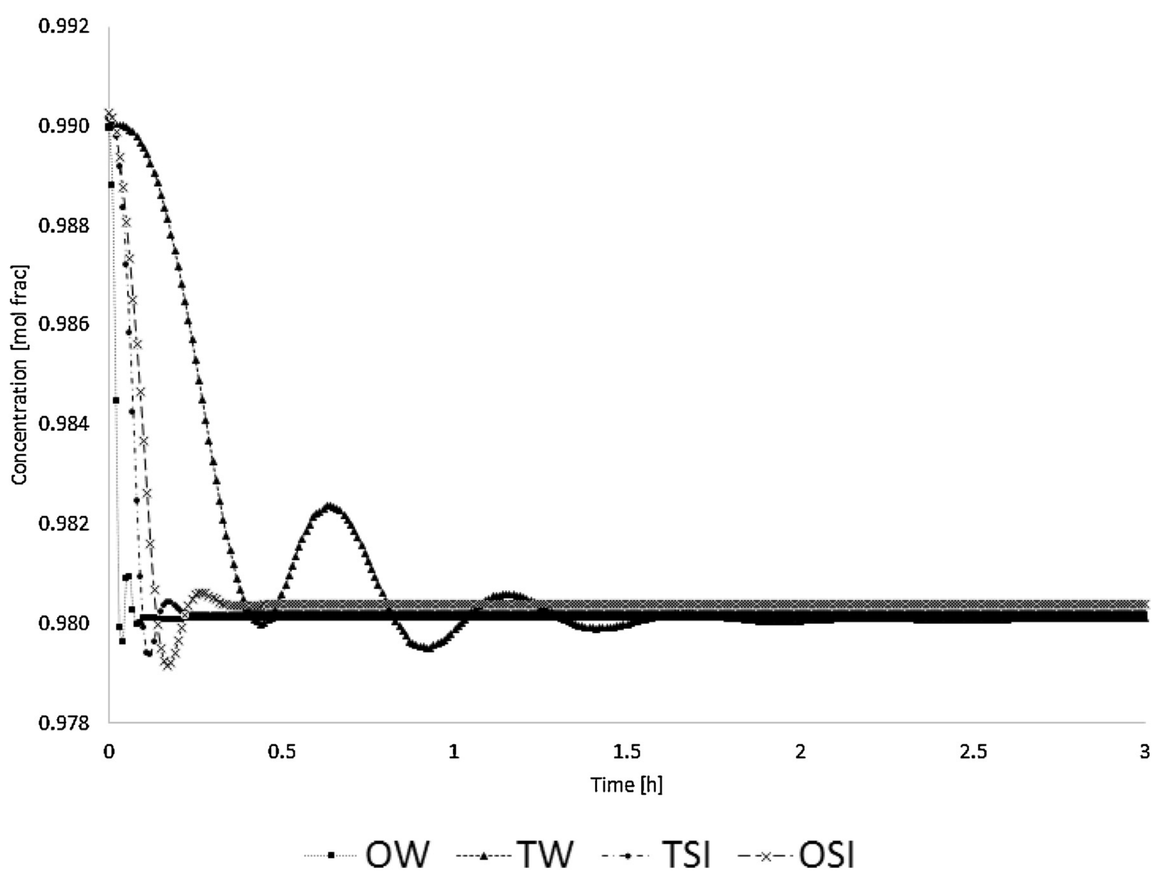

Fig. 11. Dynamic responses for *n*-hexane in Case II.

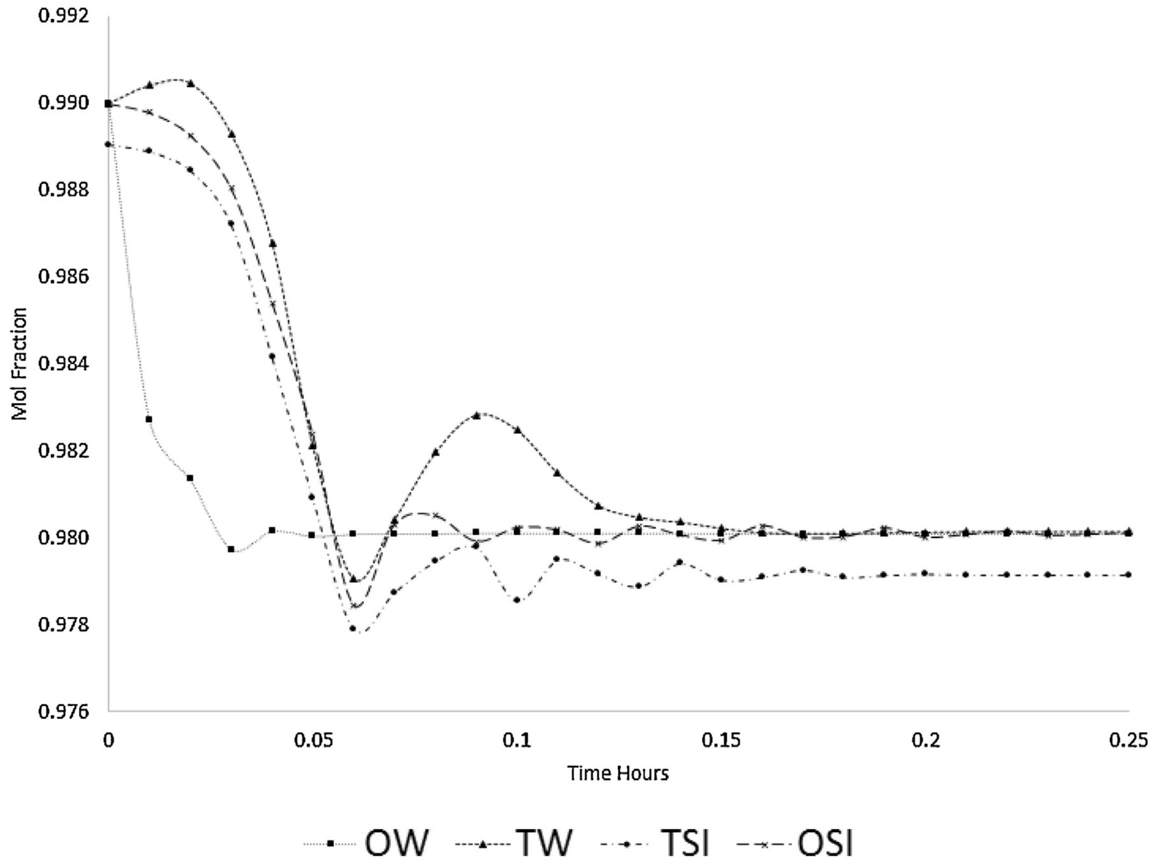

Fig. 12. Dynamic responses for *n*-octane in Case I.

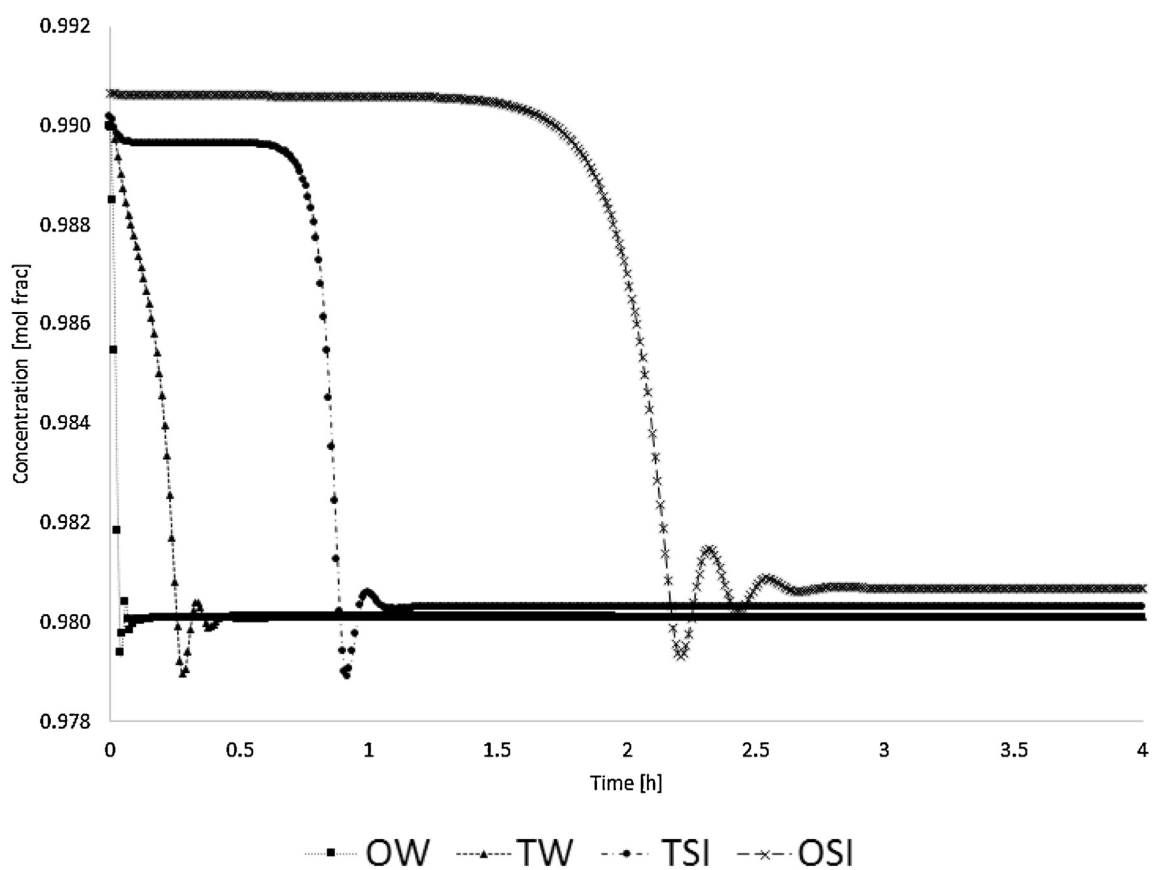

Fig. 13. Dynamic responses for *n*-octane in Case II.

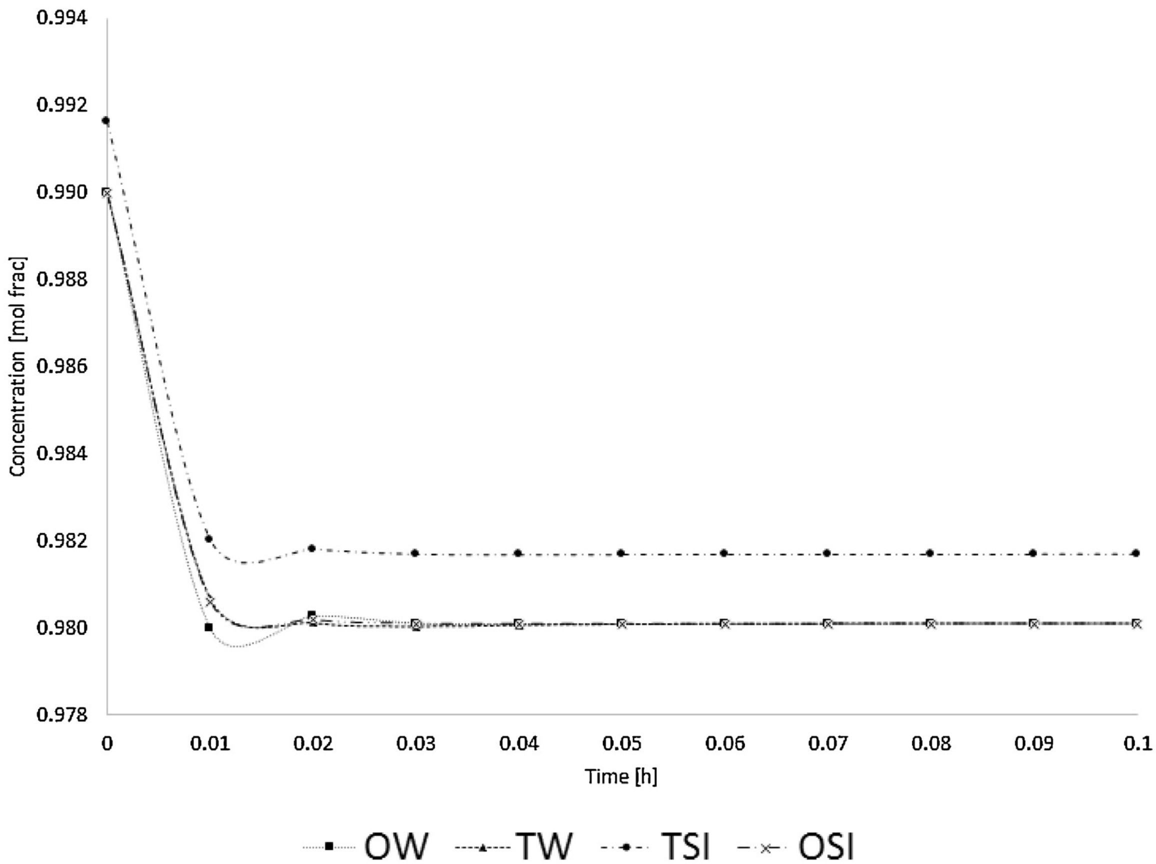

Fig. 14. Dynamic responses for *n*-decane in Case I.

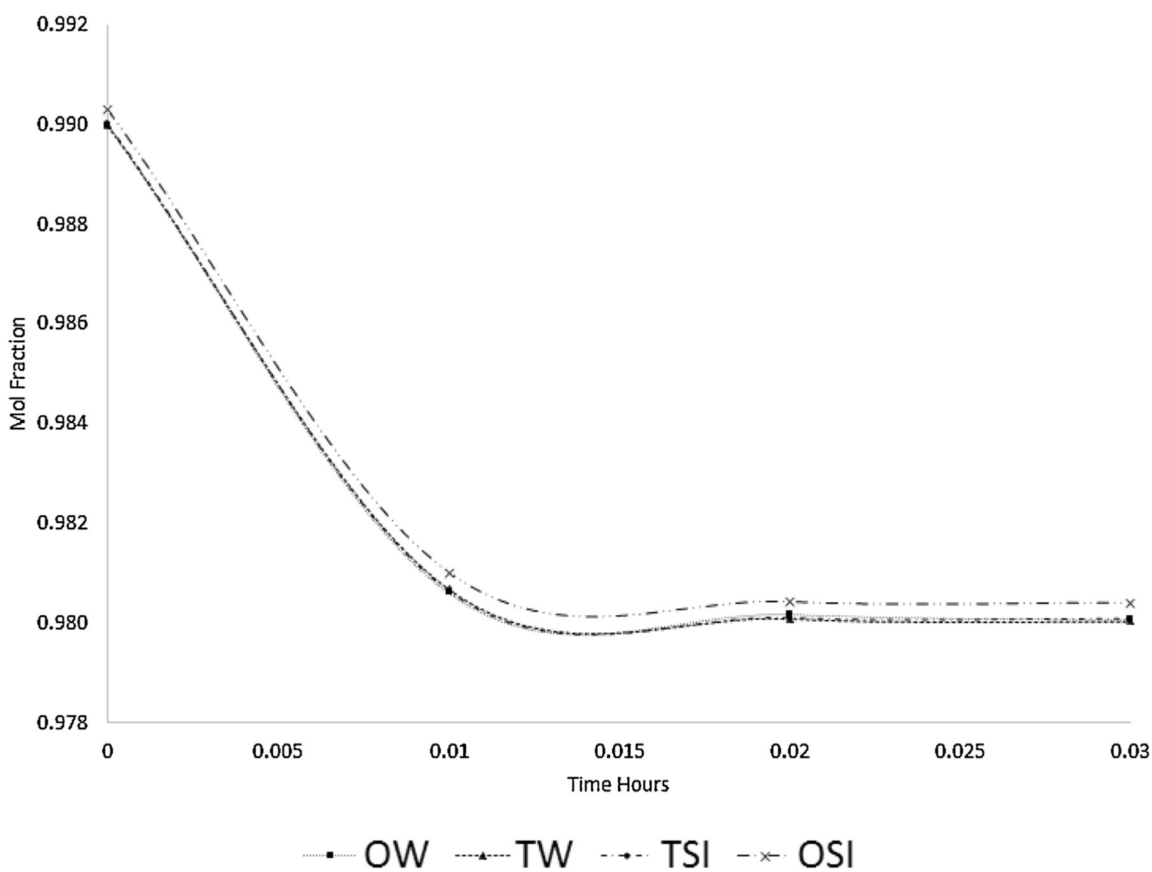

Fig. 15. Dynamic responses for *n*-decane in Case II.

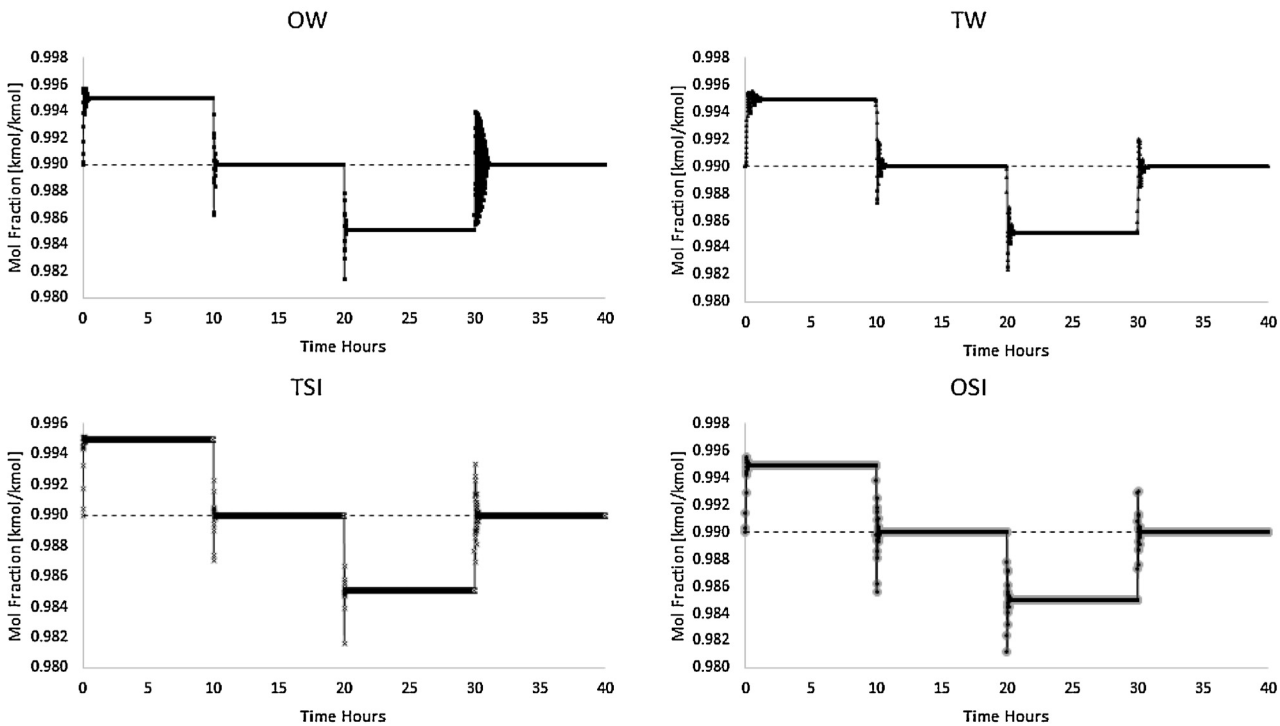

Fig. 16. Disturbance of the composition of *n*-butane for composition case I in all sequences.

improvement in the control behavior for the heaviest components. In general, the intensified alternative OSI and conventional configuration OW showed the best control properties.

6. Conclusions

Recently proposed intensified configurations for the separation of four-component mixtures were analyzed for their dynamic performances. The dynamic analysis was based on SVD technique and PI controllers, for which the parameters were tuned through a trial and error procedure to get the lower value of the integral of the absolute error. Clearly, for light and heavy components the use of intensified configurations is favored, but the IAE values obtained show that the conventional OW design has a similar dynamic performance reducing the oscillation of the response. Also, showing that a major number of columns and minor number of interconnection flows improve the control behavior. For intermediate components, the conventional configuration OW exhibits a better dynamic response, with a major number of columns and only one interconnection flow. In general, intensified designs have better control properties when the components of interest are the lightest and heaviest. So, we highly recommended using the conventional configuration OW when the intermediate compounds are the most valuable ones, in general, the major number of columns and a minor number of interconnection flows showed better control properties.

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