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Multi-objective optimization of intensified processes for the purification of levulinic acid involving economic and environmental objectives. Part II: A comparative study of dynamic properties

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A B S T R A C T

In the continuous search for the improvement of processes, recently, alternative schemes have been proposed to separate and purify levulinic acid produced from the acid hydrolysis of biomass. These alternatives offer both energy and economic savings, and reduction of environmental impact; however, their control properties have not been considered for possible industrial implementation. In this work, a controllability analysis was carried out of previously optimized designs using the relative gain array (RGA), the total condition number (TCN) and a sensitivity index for performance assessment in open-loop. Finally, the dynamic performance in closed-loop is evaluated using PI controllers having as criteria the integral absolute error (IAE). Through analysis, the best scheme obtained presented a low total condition number, however, its sensitivity index is below the average of all the designs analyzed. Additionally, the RGA analysis shows a stable structure in the frequencies studied keeping low values of eco-indicators 99 and total annual cost. The general topology consists of a liquid-liquid extraction column, a conventional distillation column and a dividing wall column with a decanter. On the other hand, by means of this robust analysis of the control properties was proposed some guidelines between the structure for improving control properties in intensified distillation schemes.

1. Introduction

Process intensification (PI) has been defined as a design philosophy that aims to enhance process performance by improving phenomena that limit it at different scales [1]. Then, process intensification consists of the development of new technology and techniques that, compared to those currently commonly used, are expected to provide dramatic improvements in manufacturing and processing. In this way, PI may substantially reduce: the relationship between the size of the equipment and production capacity, the energy consumption or the production of waste, and ultimately, translate into cheaper and more sustainable technologies [2]. Therefore, through intensification, greater efficiency, lower expenses, operations that are more respectful with the environment, reduction in size or any combination of the above are achieved [3]. On the other hand, intensification shows some disadvantages, such as the loss of manipulated variables as a result of equipment integration, a higher level of interactions and faster response as a result of equipment size reduction. This would imply that uncommon control options should be considered. One of the most challenging aspects of controlling intensified schemes is the pairing of the n-control variables (interesting for production purposes) with a set of $m < n$ manipulated variables [4].

Therefore, it is recommended that the process design, optimization, and control system design of intensified processes (and any other) must be carried out simultaneously, due to the control problems caused during integration/intensification.

The process intensification in distillation can be implemented in different ways [5], for example:

- Combining multiple process tasks or equipment into a single unit (e.g., membrane distillation [6], HiGee distillation [7], extractive dividing wall column [8–11]).
- Material and/or energy integration (e.g., cyclic distillation [12], dividing wall column (DWC) [13–16]).

Complex column configurations have shown to provide great energy savings, but they are not commonly used in industrial practice because of potential process control issues, due to a more challenging process understanding of robust control structure [17,18]. Consequently, it is relevant to investigate the operability of intensified separation schemes during the design process [19], in the case of DWC (one or more walls), operational difficulties may arise due to having fewer degrees of

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Nomenclature

A	Conventional sequence
B	Dividing wall sequence and decanter
C	Dividing wall sequence, decanter and thermal coupling
D	Sequence with column of double dividing wall
DDWC	Double dividing wall column
DETL	Differential Evolution with Tabu List
DWC	Dividing wall column
EI99	Eco-indicator 99
G	Singular square matrix
Kc	Proportional gain
LA	Levulinic acid
MIMO	Multiple input-multiple output systems
MSV	Minimum singular value
PI	Process intensification
RGA	Relative gain array
SVD	Singular value decomposition
TAC	Total annual cost
TCN	Total condition number

Greek letters

γ	Condition number
σ^*	Maximum singular value
σ^*	Minimum singular value
τi	Reset time
ω	Frequency

freedom. In addition, it is important to understand the relationship between the degree of intensification and the control properties.

It is desirable a process with good control properties since, in terms of operability, it is preferable a robust system that rejects the possible disturbances because it requires a lesser control effort to stabilize it [20]. Including the study of operability properties in an early stage of process design is important since there may be control problems due to an inadequate or inflexible design that generates dynamic restrictions in the process. As a consequence, the controllers do not guarantee optimal performance and the system will not meet the design requirements [21]. The study of the operability of intensified schemes is complex if the integration of processes causes propagation of disturbance, making local controllers insufficient to control inventories [22]. This is because the dynamic and static behavior of the system is characterized by high sensitivity, sudden changes in time constants, multiplicity of steady states, multiplicity of inputs and outputs, unrealizable arrangements, among others [23–25].

In a broader vision, going beyond the economic and environmental design objective, accounting for security objectives is taking a step forward in the continuous search for sustainable processes. For example, Jimenez et al. [26,27] list a series of characteristics that a process must have to be evaluated within a green process framework. They recommend that some metrics in the processes must be jointly evaluated, highlighting the analysis of the life cycle of the process, its controllability, its economic impact as well as the risks associated with this process.

Contrary to what can be inferred from the complex structure of the intensified distillation systems [18], their control properties might not differ significantly and even could be better than conventional distillation sequences [28–31]. Hernández et al. [28] have studied the control properties of thermally coupled systems by using condition number (γ) and minimum singular value (MSV). Rodriguez et al. [29] show through closed-loop analysis and RGA, that the use of Petlyuk columns has a positive impact on the process economy as well as on the improvement of control properties. Additionally, Wolff et al. [31] demonstrated through an RGA study that the control of Petlyuk columns is affected by increasing the number of outlet variables. Serra et al. [32] reported that the control capability of the DWC can be improved when DWC works relatively far from the minimum energy consumption. For the closed-loop control of DWC, different alternative schemes have been proposed [33–38] based on the manipulation of the manipulated and controlled variables. Studies have also been proposed in DWC that use the RGA to identify the best pairing between variables. All mentioned DWC control papers, only columns with only one wall were studied. Gómez-Castro et al. [20] conducted a study of the intensification effect on the control, reducing their system to a double dividing wall column (DDWC). In this research, the control properties were assessed by γ and MSV, finding that in this case, the increase in process intensification has a negative impact on control properties. Therefore, it is important to analyze whether the control properties in intensified systems, as well as their relationship with the degree of process intensification.

Taking this into account, particularly, intensified alternatives for the separation and purification of levulinic acid (LA) have recently been proposed. Within these alternatives, separation schemes are used with dividing wall [39], double dividing wall [40] and reactive extraction [41]. The LA is considered among the main products coming from biomass in terms of market potential, so it is obtaining great interest. The LA is obtained from the acid hydrolysis of lignocellulosic biomass, recognized as one of the renewable resources most abundant and economic in the world [42]. In this work, an evaluation of the dynamic performance of the purification schemes of levulinic acid previously obtained is carried out [40]. These schemes were optimized, using the stochastic hybrid optimization method of Differential Evolution with Tabu List (DETL), taking as objective functions the total annual cost (TAC) and the Eco-indicator 99 (EI99). The optimization technique with this hybrid algorithm, Differential Evolution with tabu List (DETL), has proved to be able of solving complex non-linear and potentially non-convex problems. This optimization method has been successfully applied in complex models such as: thermally coupled systems [43,44], reactive distillation [45,46] and divided wall columns [47,48]. Due to the complex nature of these systems, a convergence failure may occur during the optimization process, however, this problem is solved by implementing a penalty on such designs monitoring its run-status state. The objective then is determining the dynamic process properties as a tool for predicting possible controllability problems in studied schemes. This work was selected due to the current importance of production and purification of LA, in addition, it is important the control evaluation in these intensified schemes for future industrial implementation. Finally, this work aims to process understanding the evaluation of control properties in an early stage of designing distillation schemes.

The operability analysis will be made to a set of different designs, which will allow the evaluation of the dynamic properties with different trade-offs between the optimization variables. Additionally, it will allow us to identify the design variables to improve the control properties. Note, there are different degrees of intensification within these studied designs, so the control indexes behavior is also be evaluated in terms of the degree of intensification of the separation process. This is relevant since there are many implementations of intensification in distillation schemes, but little has been studied regarding how intensification favors or not control properties. Then, we are aiming to propose a separation scheme for the purification of levulinic acid in line with current trends in green chemistry (minimizing the process cost and environmental impact) as well as having good control properties. The latter to guarantees both the operability and safety of the process. This is particularly relevant when dealing with systems with high energy demand, potential unstable steady states or little controllable system that could cause a catastrophe. Finally, this study contributes to providing a better understanding of the dynamic properties of intensified schemes.

1.1. Control indicators and case study

The use of process operability indexes in an early stage of process design helps us to generate economically optimal chemical processes, with the best potential to run in an efficient dynamic mode within an operating window around a nominal operating point [49]. Among these indicators, the most commonly used in separation processes are: RGA and singular value decomposition (SVD) though the Condition number (γ) [50]. Jørgensen et al. [51] presented theoretical control studies, identifying the best control structure through RGA. Luyben et al. [52,53] present studies that identify the best control properties of distillation reactor-column systems and distillation systems, through RGA and γ. Zheng et al. [54] presented a control study of a distillation column using RGA, analyzing the influence of the design variables in the system. Palazoglu et al. [55,56] have carried out theoretical control studies using SVD and MSV to evaluate the flexibility of a plant against disturbances. Studies including conventional distillation systems [57], thermally coupled [28,57,58], Petlyuk [59], DWC y DDWC [60] have used as indices the γ and the MSV, as a measure of the control properties of those processes. Moreover, γ and RGA are indices used to formulate optimization problems for simultaneous design and control [49,61].

In our previous work, four separation schemes have been proposed to separate and purify levulinic acid (shown in Fig. 1): conventional sequence (A), dividing wall sequence and decanter (B), dividing wall sequence, decanter and thermal coupling (C) and a sequence with column of double dividing wall (D). These schemes were optimized, using the stochastic hybrid optimization method of Differential Evolution with Tabu List (DETL), taking as objective functions the total annual cost (TAC) and the Eco-indicator 99 (EI99) [40]. The results showed that by implementing the intensification through the use of dividing wall columns (DWC), costs and energy requirements were reduced. The dividing wall and decanter scheme (B) showed a decrease in both objective functions: the TAC and EI99. The selected optimal design achieved a saving of 8.42 % in TAC and 10.94 % in EI99 compared to the best design of the conventional scheme.

From each Pareto front reported by Alcocer-Garcia et al. [40], 5 designs were selected per scheme for further investigation in this following up work. For those designs, operability was analyzed as a function of both the TAC (cost influenced by the size of the equipment and/or cost of services) and the EI99 (reboiler duty associated with the amount of steam used). The selected designs per scheme are: (1) the design with the lowest environmental impact but the most expensive (this is located at the upper end of the Pareto front), (2) and (3) for study how the control properties are modified by decrease of TAC and increase of EI99, and vice versa (these are located arbitrarily in several places within Pareto front, taking into account that design 2 must be more expensive than design 3), the optimum point (4) where a tradeoff of both objective functions is found and (5) the most polluting and cheapest design (this is located at the lower end of the Pareto front), see Fig. 2. These points were selected since the control properties are affected if exist a variation in the optimum [62].

2. Methodology

Based on previous efforts, in this work, the open-loop analysis was performed using 3 indicators to compare the dynamic performance of the selected designs, referred to as: two measures that are used to quantify the degree of directionality and the level of interactions (bidirectional) in multiple input-multiple output systems (MIMO), (i) condition number (γ) and arrangement of (ii) RGA [50], respectively, and (iii) a sensitivity index (SI) proposed by Prado-Rubio et al. [24] with the objective of evaluating the sensitivity of the system to disturbances in the feed input.

The performance of the closed-loop was evaluated for setpoint tracking for outlet composition. This analysis is useful for validating the prediction of the operability indexes as those performed in other publications [60,63]. This analysis was carried out in the levulinic acid

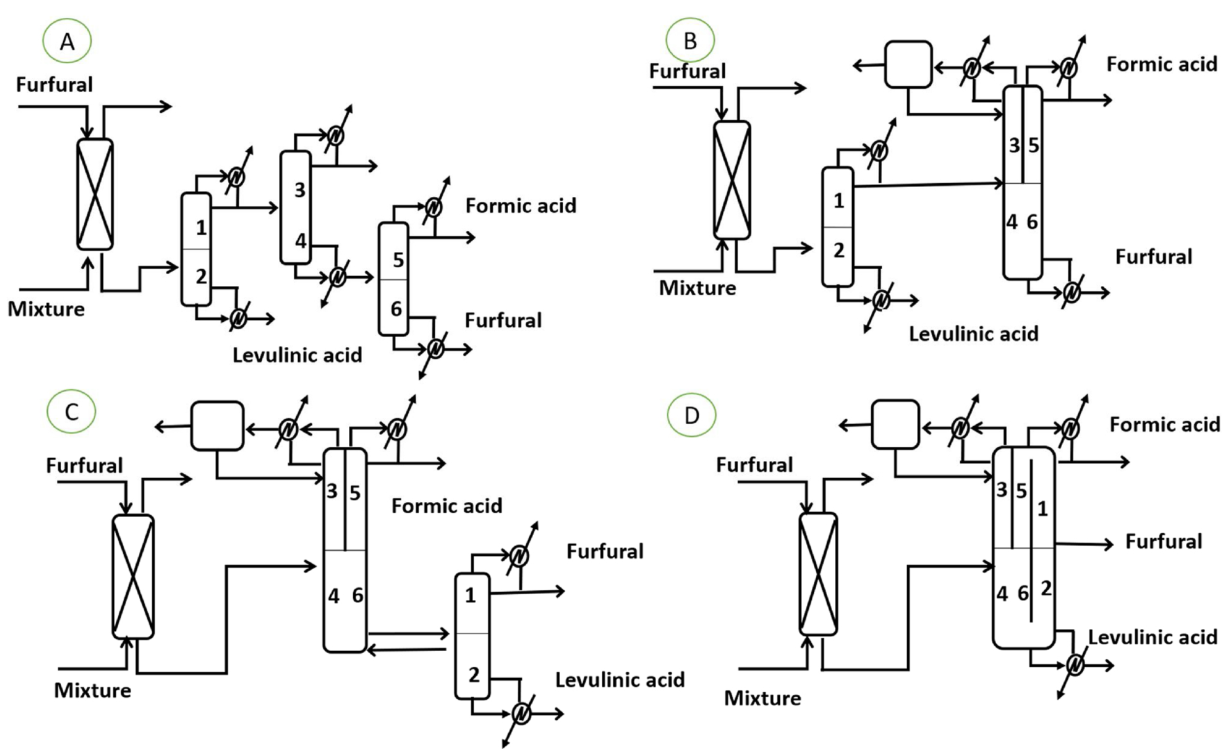

Fig. 1. Optimized schemes in the work of Alcocer-García et al. [40].

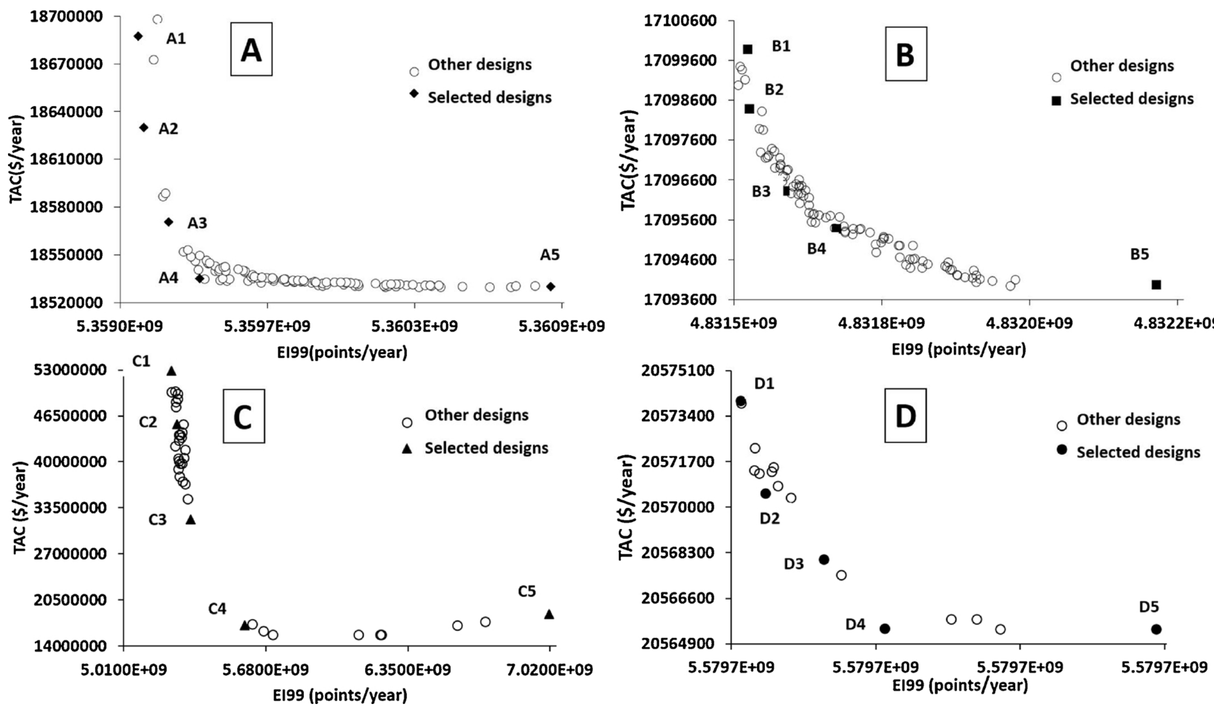

Fig. 2. Selection of designs to study from each Pareto front.

stream output because levulinic acid is the product of main interest in this case study.

2.1. Open-loop analysis methodology

Due to the complexity of the optimal design models, the linear models required for the indexes calculation are obtained using system identification of the dynamic responses in open-loop of each optimized from the Aspen Dynamics 8.8 simulator. A step disturbance was implemented around the nominal operating point. The magnitude of the step change was +0.5 % of each potential variable manipulated, selected by means of a sensitivity analysis of the system, taking as criterion that with a minor disturbance, the influence of non-linearities is avoided [52]. In addition, similar studies [64–66] have shown that intensified systems are sensitive to these small disturbances.

Based on the optimized designs reported by Alcocer-García et al. [40] the simulation of those schemes in Aspen plus were exported to Aspen Dynamics, to the latter with the aim of performing disturbances in the system and obtaining dynamic responses. For inventories in dynamic distillation columns, the dimensions for reboiler and reflux drum were 3 ft in length and 2 ft in diameter, according to reboiler and drum sizes reported by Buckley et al. [67]. Once dynamic designs and responses to disturbances are obtained, the responses are adjusted to a transfer function, which is used for the calculation of γ and RGA. For the calculation of SI, only the dynamic responses obtained when disturbing the feeding are taken, then its calculation is made by finite differences and obtained the norm. The open-loop methodology is illustrated in Fig. 3.

2.1.1. Relative gain array

The calculation of RGA is used to design control structures from a single input-single output perspective. This analysis was performed aiming to find the best control pairing between manipulable and controlled variables of the system. The RGA calculation is based on the system gain array, which is obtained through the transfer functions resulting from the disturbances. The RGA of a not singular square matrix G is a square matrix defined as [50]:

$$RGA(G) = \Lambda(G) \triangleq G \otimes (G^{-1})^T \tag{1}$$

Where ⊗ denotes an element multiplication per element (Schur product). Bristol [68] originally introduced RGA as a stable state of

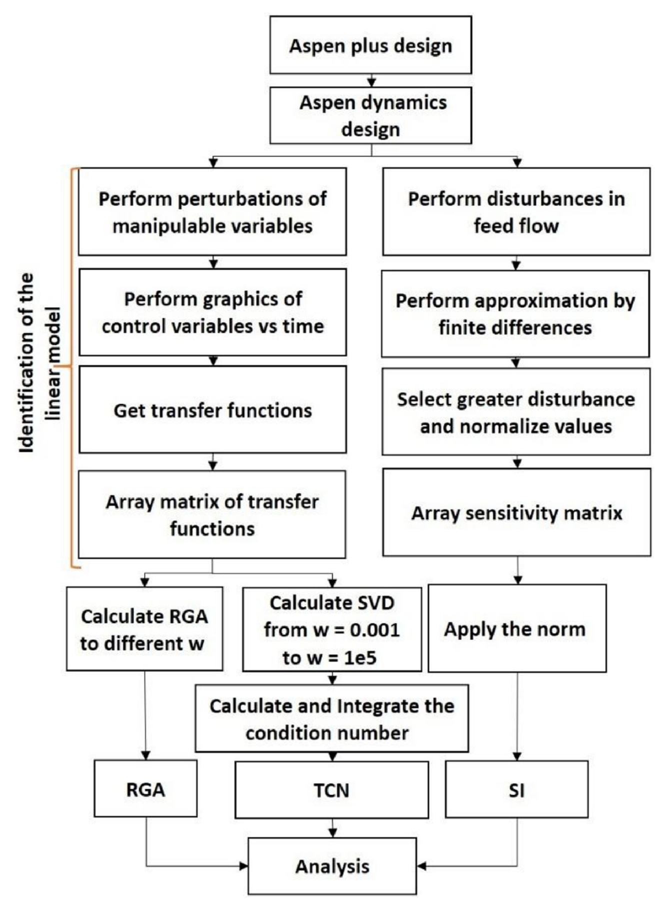

Fig. 3. Dynamic and operability analysis open-loop methodology.

interactions measure for decentralized control structures. Unfortunately, according to the original definition, many people have ruled out that RGA is only significant at zero frequency (steady-state). On the contrary, in most cases the value of RGA phase crossover frequency is more important, since it would indicate us that the control structure can vary to different frequency values (ω) [50], so in this study RGA was obtained for 3 different frequencies to analyze if the control structure is maintained, the frequencies studied were: 0, 0.001 and 1e5 (rad/h), which are representative to steady-state, close to steady-state and high frequencies.

The RGA results in an array containing the manipulable variables of the system and which one controls each output of interest. For conventional distillation columns LV configuration is chosen (the distillate composition is controlled with the reflux and the bottom composition with the reboiler heat duty), because the framed literature has proved its economical viability because of the proximity of the control loop and is, therefore, the most used in distillation columns [67,69], see Fig. 4. However, through the calculation of RGA, different configurations can be found to the LV (DV), which means that there can be more pairings between the manipulable and the output variables, which comply with a stable control structure.

In this case of study, existing schemes with dividing wall columns, so their control structure depends not only on the reflux and the reboiler heat duty. Halvorsen y Skogestad [70] established five manipulated variables for a DWC, which are the reflux ratio, the overheating service, the flow rate of the side stream, flow rate of steam flow and flow rate of liquid flow, these two last being translated as interconnection flows.

For our case of study, the calculation of the sensitivity indexes considers LV configuration (for the reasons mentioned above) for the designs of the schemes A, B and C, controlling the distillation flow with the reflux and the bottom flow with reboiler thermal load. For scheme D, Fig. 1(D), the composition in the distillate flow was controlled with the reflux of its section, the composition in bottom flow with reboiler head duty and the lateral composition with the flow rate of the same side stream. In the extractive column of all designs, the composition of the bottom flow was controlled with the extractant flow. The control variables considered in this study are the composition of: levulinic acid, water, formic acid, and furfural. The purities of those components were considered since the compositions are considered as control variables. The minimum purity objectives resulting from the optimization were at 98 % (%wt) for levulinic acid, 85 % for formic acid and 99.9 % for furfural. The purities for levulinic acid and formic acid were considered according to industrial uses and the purity of the furfural for being used and recycled in the same process [71,72]. Usually, the control composition in industrial practice requires chromatographs on-line for composition measurement.

2.1.2. Condition number

The condition number has been used as an input-output controllability measure, assessing the influence of uncertainties in process parameters and modelling errors on the system performance. In addition, it indicates the difficulties to decouple the interaction of the control loops that can occur for systems with high values of the condition number [73]. The condition number is obtained by SVD methodology, and it is a very useful tool in the theory of linear systems and plays an important role in the analysis and design of control systems for real processes in the industry [74].

For the calculation of γ, linear models are obtained for the system using the same disturbances as in RGA, using the same manipulative variables and output variables. Once all dynamic responses were obtained by using the Aspen Dynamics simulator, the transfer function matrices (G) were collected and subjected to SVD. It is important to emphasize that the transfer function matrices contain all the dynamic of the plant. The calculation of the SVD was carried out as follows:

$$G = V \sum W^H \tag{2}$$

Where $\sum = diag(\sigma_1, \sigma_2....\sigma_n)$, $\sigma_1 =$ singular value of $G = \frac{\lambda 1}{2}(GG^H)$, $V = (V_1, V_2, ...V_n)$ singular matrix of left vectors and $W = (W_1, W_2, ...W_n)$ singular matrix of right vectors. From the SVD of G, the two parameters of interest are the minimum singular value σ_* and the ratio of the maximum to minimum singular values σ^*, called the condition number which can be determined at the frequency of interest [50],

it is calculated as follows:

$$\gamma = \frac{\sigma^*}{\sigma^*} \tag{3}$$

Recently, Santaella et al. [66], proposed the calculation of the condition number as a function of the frequency and then determine an accumulated index called total condition number (TCN), calculating the area generated by the condition and frequency number curve:

$$\mathrm{TCN} = \int_{\omega 1}^{\omega 2} \gamma d\omega \tag{4}$$

For this study, the frequeny response of the condition number was calculated and the integral of the curve generated from condition number vs frequency was performed. The frequency range studied was from 0.001 to 1e5, this range was chosen based on works performing condition number analysis and using this range [64,65].

The comparison between condition numbers shows whether one system may be more likely to have control problems than another, given a control structure. So, if the condition number is large then this may indicate control problems. A large condition number can be caused by a small value of σ*, which is generally undesirable (on the other hand, a high value of σ* should not necessarily be a problem). A large condition number implies that the system is sensitive to input uncertainty, but this type of uncertainty often does not occur in practice. Therefore, we cannot conclude that a system with a large number of conditions is sensitive to uncertainties [50]. In general, schemes with lower status values would be expected to show better dynamic performance under the proposed feedback control structure [75].

2.1.3. Sensitivity index

The sensitivity index (SI) is based on the disturbances in the input variables. This index was proposed by Prado-Rubio et al. [24] to evaluate the dynamic sensitivity of the control variables to the input manipulable variables:

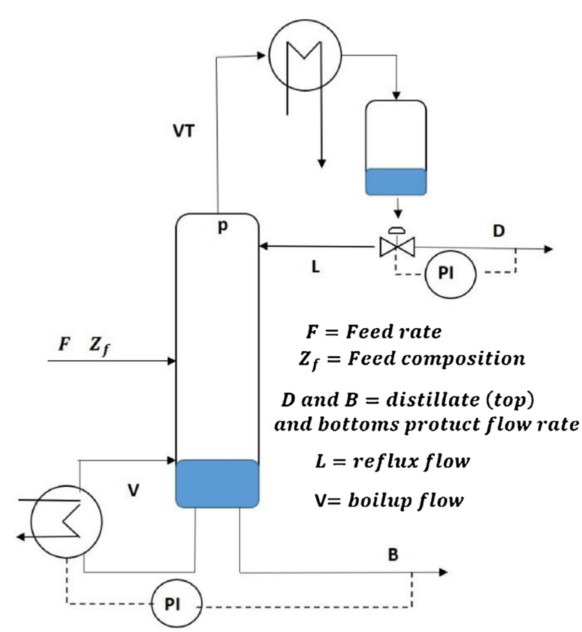

Fig. 4. Conventional distillation column with a control LV structure.

$$SI(t) \approx \frac{X[F_{in}(t) + \Delta F_{in}(t)] - X(F_{in})}{\Delta F_{in}} \frac{Fin(0)}{X(0)} \quad (5)$$

Where X means the state of interest to evaluate the sensitivity. The dimensionless sensitivity is selected to do a fair comparison between the two variables of the system since the measurement will not be affected by the selection of units and the scale of the variables. The accuracy of the approximation of the finite quotient of the derivatives is proportional to the square root of the increment used for the evaluation. The resulting sensitivities will be nonlinear functions of time from a t = 0 to the stationary state. This index contains important dynamic information that would otherwise be difficult to obtain for this system. The scale values chosen are the initial conditions before the perturbation is applied, that is, in t0 = 0 [24].

In order to calculate the sensitivity index, the same Aspen Dynamics file generated for the calculation of TCN and RGA is used. The output and input variables are then identified. In this case, the output variables were the same as those considered for the calculation of RGA and TCN, and the manipulable variables were the composition of levulinic acid and the flow of levulinic acid in the feed, corresponding to the most likely disturbances. These manipulative variables were considered because levulinic acid is the interest product. The other input variables required to solve the model are fixed to constant values. The purpose of this step is to determine the operation at nominal values of the input variables [76]. Once the variables have been identified, disturbances of +0.5 % are performed, analogously to the other indices. In this case with two manipulable variables and four outputs, at the end of the disturbances, we must have 8 data sets. Using Eq. (5) for each data set (concentration vs time) the approximation by finite differences is performed, it is normalized using the initial values of the manipulable variables, then chose the maximum value of all-time range. With the standard values or each perturbation, the sensitivity matrix (P) is assembled and its norm is evaluated, obtaining a single value for both disturbances.

If $P = [a_{ij}]$ is a mxn matrix of real or complex elements, the norm of P, designated by $\|P\|$, is defined as the nonnegative number given by:

$$\|P\| = \sum_{i=1}^{m} \sum_{j=1}^{m} |aij| \quad (6)$$

That is, the norm of P is the sum of the absolute values of all its elements [77]. Being the value of the norm, the index that tells us how sensitive the system is to the disturbances in the input, so low values of this indicates that the system is more robust to disturbances in the feed.

2.2. Closed-loop analysis methodology

Starting from the design in Aspen Dynamics 8.8, the closed-loop test was performed as follow: (i) a step change was induced in the setpoint for the levulinic acid composition under single-input single-output feedback control at each output flow rate; and (ii) 1 %, 5 %, 10 % and 30 % changes in the composition of one component (adjusting the proportion in the composition of other components) was implemented as feed disturbance. For the closed-loop control evaluation, proportional–integral (PI) composition controllers were implemented according to the determined control structure, in this case, the control variable for levulinic composition is the reboiler duty for the four schemes. The reason for using composition controllers is simply that a 'back-off' from the purity specifications makes composition control simpler [50]. PI controller was chosen because of its wide use in industrial practice. In this study, a common strategy was considered to compare and optimize the controller parameters. Because we considered PI controllers, the proportional gain (Kc) and the reset times (τi) were tuned for each scheme studied here [78], the tuning criterion was the minimization of the IAE, Fig. 5.

3. Results

This section presents the results of open-loop and closed-loop control analysis, potential difficulties to control the system, and how sensitive is the systems to input disturbances. All designs were modeled and simulated in Aspen plus using the rigorous RADFRAC unit. Therefore, all designs were designed robustly considering the MESH equations (mass balance, balance ratios, sum of constraints and matter balance). Subsequently, they were exported to a dynamic model in Aspen Dynamics, disturbances were performed and control indicators were calculated.

3.1. Open-loop control analysis

Table 1 shows the results of the 3 performance indices for the 20 designs studied. In the RGA study it was found that some designs presented an LV control structure at all frequencies studied; other designs presented a different pairing than the LV control structure. (DLV) remained constant at the frequencies studied, this implies that the distillate is not necessarily controlled with the reflux ratio and that the bottoms are not necessarily controlled by reboiler heat duty. Also, in some designs, their control structure was not the same in the frequencies studied, which implies that presents different control arrangement (DCA) in each frequency evaluated. It is interesting that more than 50 % of the schemes studied present a structure of control DCA, see Table 1, seeing here the importance of studying the

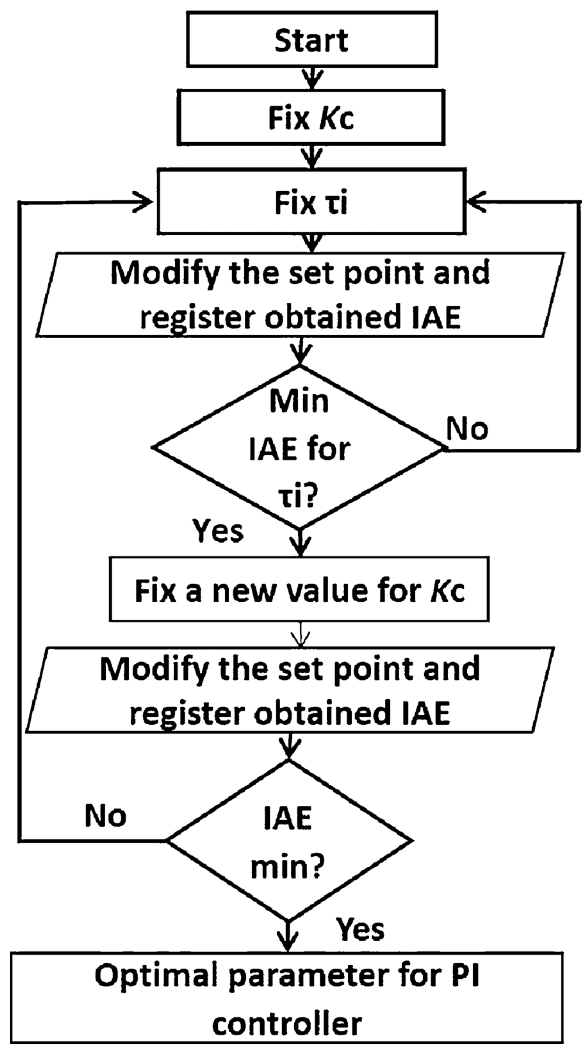

Fig. 5. Flowsheet for tuning PI controllers.

Table 1
Summary of Results for all designs studied.

	TAC	EI99	TCN	SI	RGA
A1	1.8687E+07	5.3591E+09	2.77E+07	8.11	DLV
A2	1.8630E+07	5.3591E+09	1.16E+08	14.17	LV
A3	1.8571E+07	5.3592E+09	2.01E+09	10.30	DCA
A4	1.8535E+07	5.3594E+09	5.87E+09	13.37	DCA
A5	1.8530E+07	5.3608E+09	1.01E+11	15.22	DCA
B1	1.7100E+07	4.8316E+09	9.65E+07	12.36	DCA
B2	1.7098E+07	4.8316E+09	6.52E+05	11.98	DCA
B3	1.7096E+07	4.8316E+09	6.34E+05	11.98	LV
B4	1.7095E+07	4.8317E+09	4.35E+06	12.38	DLV
B5	1.7094E+07	4.8322E+09	1.46E+09	11.98	DLV
C1	5.2888E+07	5.2347E+09	4.34E+08	5.70	LV
C2	4.5247E+07	5.2608E+09	1.02E+10	4.90	DCA
C3	3.1841E+07	5.3251E+09	3.39E+11	2.89	DCA
C4	1.6939E+07	5.5807E+09	2.87E+09	22.06	DLV
C5	1.8509E+07	7.0155E+09	1.87E+11	28.36	DCA
D1	2.0574E+07	5.5797E+09	5.88E+06	12.56	DCA
D2	2.0571E+07	5.5797E+09	8.35E+06	4.56	DCA
D3	2.0568E+07	5.5797E+09	1.48E+06	8.85	DLV
D4	2.0565E+07	5.5797E+09	5.25E+05	6.90	LV
D5	2.0565E+07	5.5797E+09	5.15E+05	11.76	DLV

Table 2
Influence of the amount of extractant and the dimensions of the first distillation column in the SI.

	A4	B4	C4	D4
Extractant (kg/h)	106722.836	107975.270	115139.838	107752.080
Number of stages	28	28	25	16
Diameter (m)	3.642	3.577	1.437	0.573
SI	13.367	12.381	22.06	6.90

controllability of these schemes.

From conventional scheme (A), design A1 design has the lowest sensitivity to disturbances of the manipulable variables of the system, with the lowest values of TCN and SI. The robustness to the disturbances in this design is due to its has the largest diameters compared to the other conventional designs increasing its holdup. Design A2 is the second design with larger dimensions of conventional designs, which favors a low value of TCN. Both designs A1 and A2, have a stable structure in the whole range of frequencies studied, DLV and LV, respectively. Note that a stable structure favors stability of dynamic behavior. On the other hand, design A4, considered optimal of this scheme, shows a reduction in the size of its equipment, which favors its values of TAC and EI99. However, it has consequences at the control level since this design exhibits a changing control structure at different frequencies and large TCN values. Based on these results, we can evidence that having greater dimensions favors obtaining better control properties. Then, it can be inferred that an improvement in the control properties respect to the optimal design of the conventional scheme (A4) with the A1, produce an increase in the size of the equipment. Consequently, it is presented a penalty of 0.821 % in TAC and its EI99 value remains almost equal.

In scheme B, design B3 presents the best control properties of these designs, since it has the smallest TCN and a stable LV control structure at the frequencies studied. In this case, the diameter of the first column in the conventional distillation has a great influence on the dynamic behavior of the system, since the designs with larger diameters in this column result in lower TCN values. The SI is affected by the dimensions of the first two columns: the liquid-liquid extraction column and the conventional column. For this scheme, design B3 and B4 present similar values of TAC and Eco-99, this because they have very similar design variables. However, the B3 design has larger diameters in all the columns with respect to the B4 design and this is what favors improving its control properties.

Scheme C, presents a thermal coupling between the DWC and a conventional column, following the trend where larger dimensions favor the control properties. However, the trend of SI is not clearly reflected in these schemes, the latter being attributed to recirculation having an effect on input disturbances. It is important to visualize that in this case the design considered optimal (C4) presents the second-best value in TCN in this scheme, but presents bad values of SI, and a control structure different to LV for all frequencies. It is even more important to analyze that in this scheme, if you wanted to select the best design based on the best control (design C3) is necessary an increase in the TAC of 46.8 % respect the design with the best TAC (C4), which would be equivalent to $14,902,341 USD, which for practical purposes in engineering would not be viable.

The diameter of column 4 of the schemes with double dividing wall (D), represents the size of the total diameter of the column and it is this diameter that directly affects the TCN, since based on the results, the bigger the diameter gives the better values of TCN and the designs present a stable structure through RGA. For these designs, the variable with the greatest impact on SI is the extractor flow used, since the designs D2 and D4 present the smallest flows and the lowest values of SI, this is because of the greater amount of flow, the greater the disturbance in the system.

Therefore, for the sensitivity index, it is seen that for all studied designs, increasing the amount of extractant has a negative effect on SI,

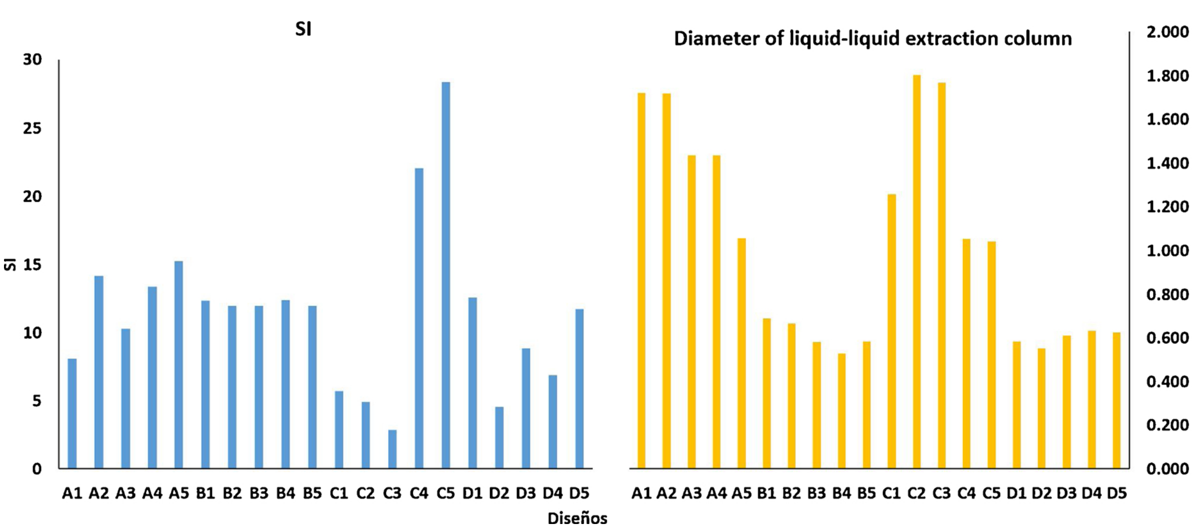

Fig. 6. Influence of the diameter of the liquid-liquid extraction column in the SI.

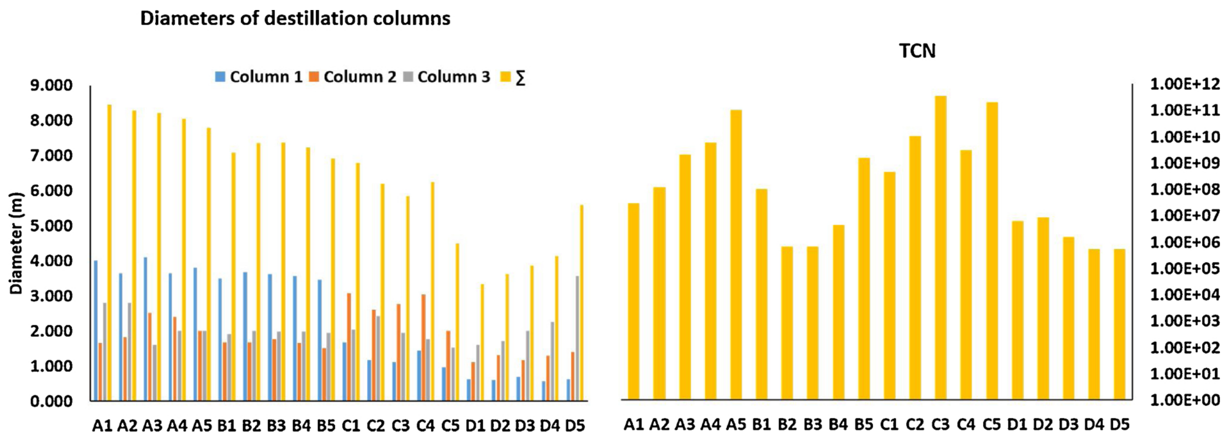

Fig. 7. Influence of diameter on total condition number.

Table 3
LV control structure for design B3.

Frequency (w)		Bottom flow of levulinic acid, column 2	Flow of water, column 3	Distillation Flow of formic acid, column 4	Bottom Flow of furfural, column 4
0	RHD- column 2	1.097	0.000	0.000	−0.097
	RR- column 3	0.000	0.999	0.001	0.000
	RR - column 4	0.000	0.001	0.999	0.000
	RHD- column 4	−0.097	0.000	0.000	1.097
0.001	RHD- column 2	**0.995**	0.000	0.000	0.005
	RR- column 3	0.000	**0.999**	0.001	0.000
	RR - column 4	0.000	0.001	**0.999**	0.000
	RHD- column 4	0.005	0.000	0.000	**0.995**
1E+05	RHD- column 2	**0.987**	0.000	0.000	0.013
	RR- column 3	0.000	**1.000**	0.000	0.000
	RR - column 4	0.000	0.000	**1.000**	0.000
	RHD- column 4	0.013	0.000	0.000	**0.987**

Table 4
Design parameters of B3 desings.

		B3
Column 1	Number of stages	21
	Extractant (kg/h)	107991.982
	Diameter (m)	0.581
	Operative pressure (kPa)	101.325
Column 2	Number of stages	28
	Feed stage	11
	Reflux	0.069
	Reboiler heat duty(KW)	23188.496
	Diameter (m)	3.623
	Operative pressure (kPa)	101.325
Column 3	Number of stages	20
	Feed stage	4
	Reboiler heat duty (KW)	0.000
	Diameter (m)	1.760
	Operative pressure (kPa)	101.325
Column 4	Number of stages	63
	Feed stage	20
	Reflux	8.186
	Reboiler heat duty (KW)	4740.007
	Steam flow (Kg / h)	30023.902
	Steam outlet stage	21
	Diameter (m)	1.994
	Operative pressure (kPa)	101.325
Purity	Levulinic Acid %w/w	0.980
	Formic Acid %w/w	0.952
	Furfural %w/w	0.999

see Table 2. This increase in the extractant in some designs is due to the fact that as the amount of levulinic acid is disturbed, more extractant will be required, and if extractant is in excess it will generate a disturbance that spread throughout the system. On the other hand, by increasing the size of both the extraction columns, these disturbances are damped, improving the performance of the system in the event of disturbances in the feed. Note Fig. 6, all designs with a smaller diameter have the highest SI values in each set of designs.

In addition, it is important to visualize the relationship that exists between a stable control structure (from RGA), the values of TCN and the diameter of the columns, since the designs with greater diameter, have the lowest values of TCN and stable structures of control. Fig. 7 shows the relationship between the condition number and the diameter of all distillation columns of the designs. Analyzing the behavior of each scheme, in the 4 schemes it is clear the tendency that when increasing the value of the diameter of the columns, the natural dynamics of the process is favored, this is because the system have a larger hold-up/inertia, then it mitigates disturbances.

Interestingly, the most intensified designs belonging to D schemes have low TCN values and SI values below the average in comparison with all the designs studied. Contrarily to what is stated in literature where control problems are associated with process intensification [18], the implementation of a double dividing wall may favor the control properties. For example, the results of the designs of scheme B and C, it would be expected that the control properties favored the more intensified scheme, in this case to C. However, when comparing TCN values, designs B have lower values than designs C. This may be due to the fact that C designs in their structure have a thermal and mass coupling more than designs B, losing degrees of freedom and causing both a more restricted and more sensitive system. On the other hand, the recycles caused by this extra coupling in designs C, cause a decrease in the sensitivity index, dampening the disturbances in the system caused by perturbing the feed. However, in this case study it is important to emphasize that, by means of the control study, it was found that it is possible to obtain better control properties of intensified

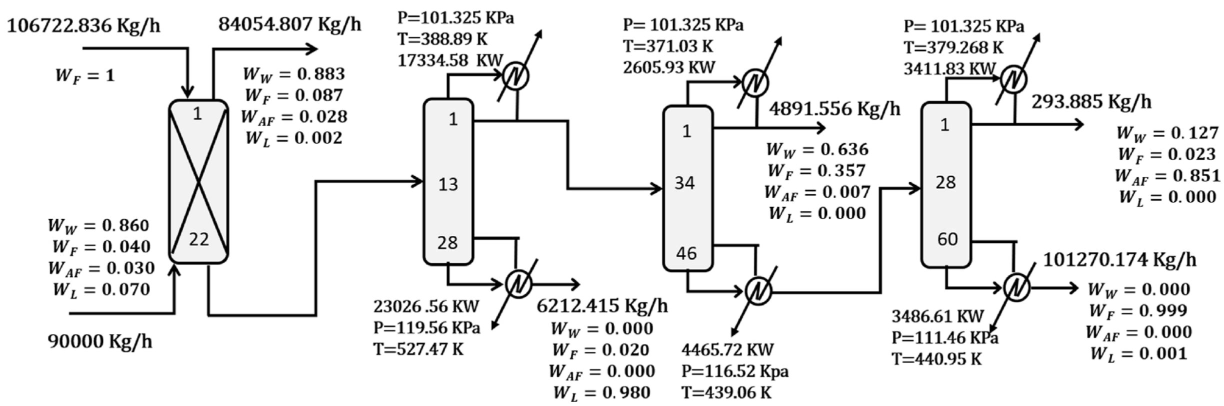

Fig. 8. Design specification of conventional sequence (A4).

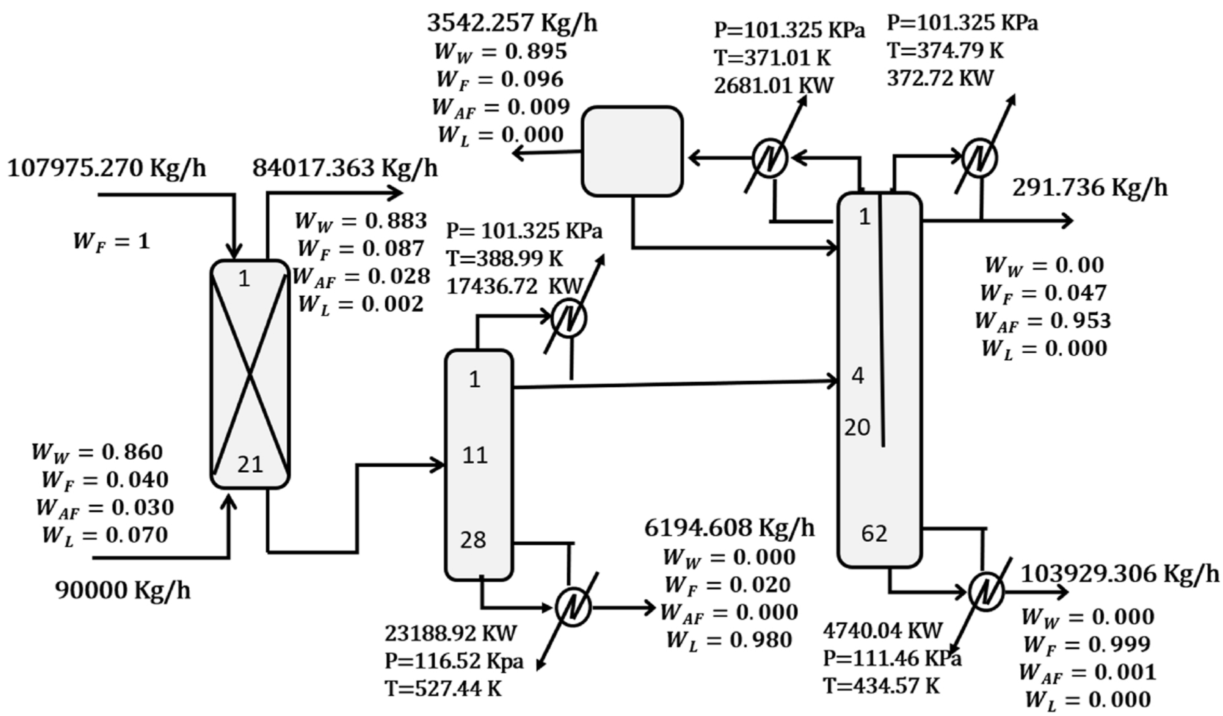

Fig. 9. Design specification of dividing wall sequence with decanter (B4).

schemes in comparison to conventional schemes. Note within the schemes studied, there are alternatives that present economic-environmental savings through intensification and at the same time control properties are favored.

For schemes with a stable control structure, low values of SI, TCN, TAC and EI99, the best designs are: from scheme C the design C4 and scheme B the designs B3 and B4. The design C3 presents the best value of TAC and also good value of EI99, which also presents a stable control structure. However, design C3 presents greater sensitivity to both feed disturbances and process variables, through its high values of SI and TCN, respectively, compared to designs B3 and B4. The design B4 shows savings in both TAC and Eco-indicator 99 compared to the design B3. On the other hand, the scheme B3 presents lower values of TCN and SI, this can be attributed to has larger columns than the B4 design. In addition, the design B3 has a stable LV structure, as shown in Table 3. So, the design B3 has the best control properties of the schemes studied and has good values of TAC and EI99, which consists of a liquid-liquid extraction column, a conventional column and a dividing wall column with a decanter, its design parameters are shown in Table 4. It is worth mentioning that in this design, column 3 represent one internal section in dividing wall column and column 4 represents the shell of the dividing wall column.

3.2. Closed-loop control analysis

This analysis was extended to the designs considered optimal in each Pareto: A4, B4, C4 and D4, these designs were showed in Figs. 8–11. Only these designs were considered because the objective of the closed-loop analysis is to compare the results with those obtained in the open-loop. The closed-loop responses of the four designs analyzed are shown in Fig. 12, and the results of tuning the PI controller in Table 5. Based on the results obtained in open-loop, we would expect the D4 design to present the lowest values of IAE, which is consistent with the data shown in Table 5. In addition, the other designs follow the behavior found with the TCN, since the values found in IAE are consistent with the values found in TCN. This behavior is consistent with all set point tracking from nominal point studied. Obtaining with this that through an open-loop analysis, an approximation of the dynamic behavior of closed-loop can be obtained. Through the analysis at different step changes in the nominal set point at closed-loop, its observed that regardless of size step, the behavior obtained by the TCN remained. At minor step changes (1 %) similar dynamic responses were presented

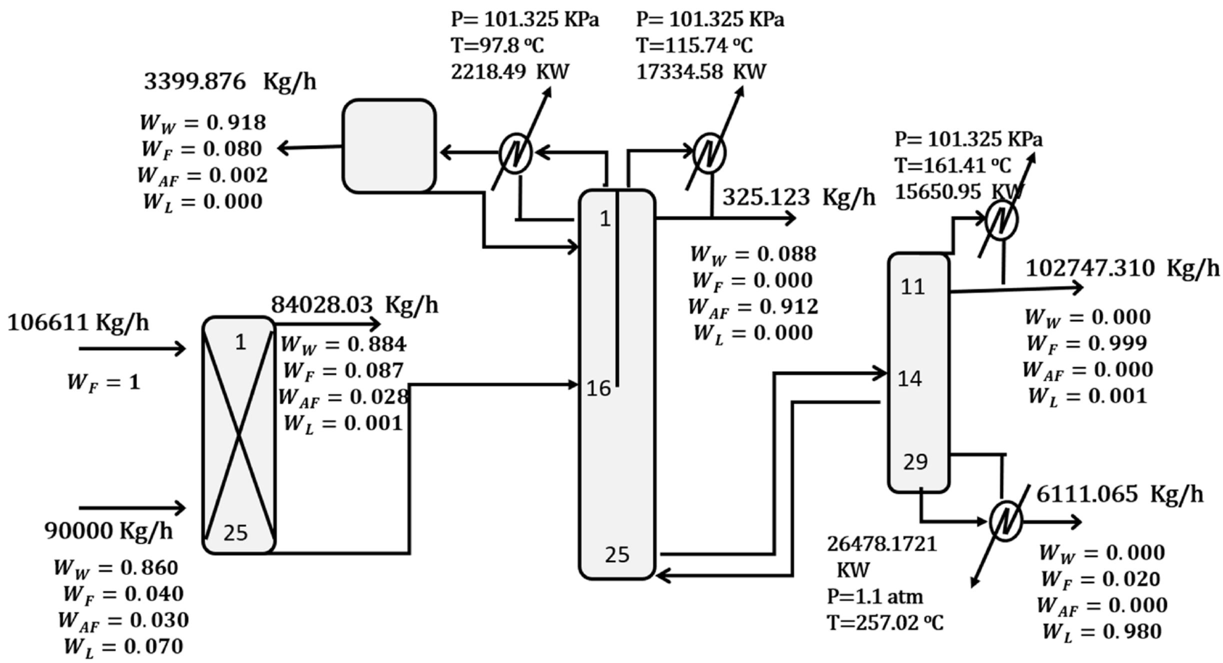

Fig. 10. Design specification of dividing wall sequence with decanter and thermal coupling (C4).

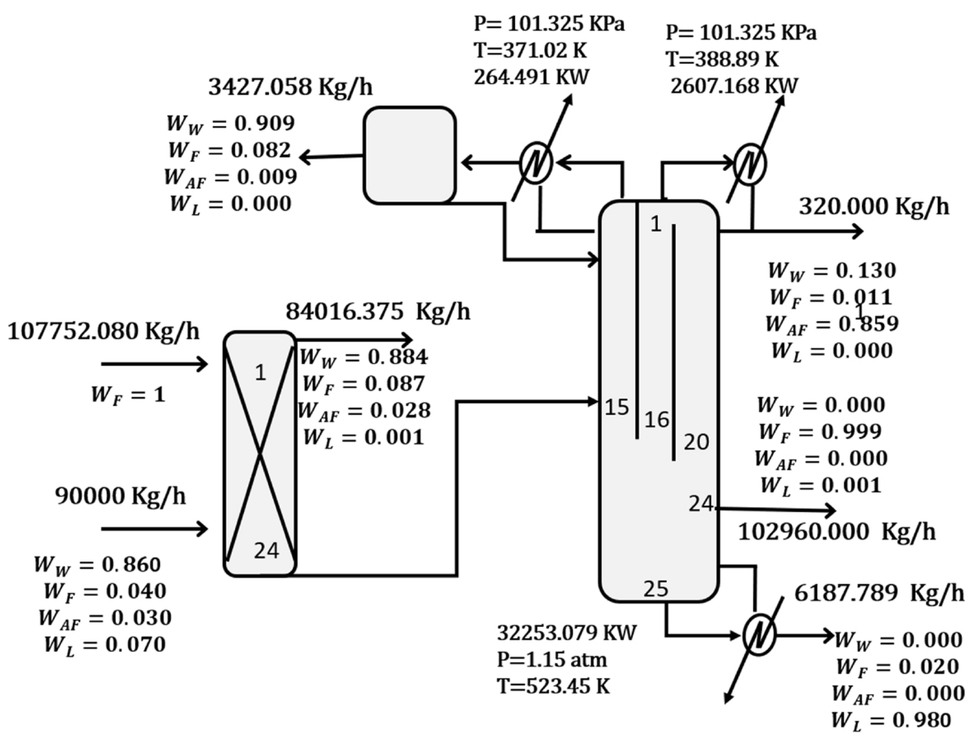

Fig. 11. Complete mass balance of schematic diagram of dividing wall sequence with column of double dividing wall and decanter (D4).

(Fig. 12, case a), this was due to the fact that at small disturbances, it was very close to the nominal value. However, when studying major changes (5 %, 10 % and 30 %), it was clearly differentiated the behavior of each design, (Fig. 12, case b, c and d).

4. Conclusions

In this work, an operability analysis was performed on 20 designs of 4 schemes for obtaining levulinic acid (LA). Operative performance indexes are used as criteria to both assess and discard process designs with good economics and environmental properties. The relationship that exists between the RGA, the TCN and the diameter of the columns was notorious, since the designs that presented a constant arrangement in the RGA, present the best values of TCN and the largest diameters in the schemas studied. This relationship exists due to changing control structure will generate greater sensitivity thus TCN, and the increase in diameter favors the buffering of disturbances of system variables. In general terms, the control properties of a system are benefited by the size of the equipment, for example: the diameter and the type of pairing in the control structure.

Interestingly, this case study has shown that intensification favors the control properties of most designs compared to conventional

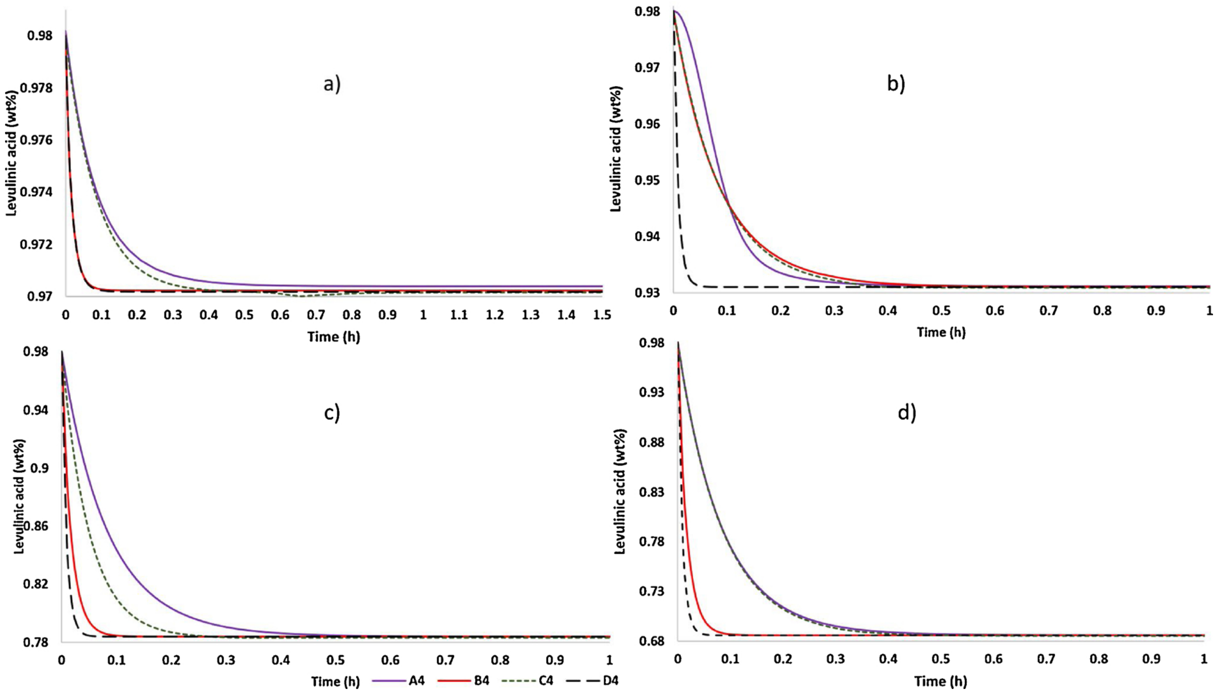

Fig. 12. Set point tracking in representative designs for the levulinic stream. At 1 % (a), 5 % (b), 10 % (c) and 30 % (d).

Table 5
PI controller tuning results, IAE and TCN values.

Step change	Design A4 IAE	Kc (%%)	T (min)	Design B4 IAE	Kc (%%)	τ (min)	Design C4 IAE	Kc (%%)	T (min)	Design D4 IAE	Kc (%%)	T (min)
1 %	8.11E-04	245	5	1.58E-04	250	1	8.63E-04	250	5	1.58E-04	240	1
5 %	4.12E-03	250	5	4.06E-03	250	5	4.10E-03	250	4	3.81E-04	250	5
10 %	3.26E-02	240	5	1.58E-03	250	2	3.28E-02	250	1	9.38E-04	245	4
30 %	2.43E-02	250	4	4.74E-03	245	4	2.44E-02	250	5	2.29E-03	250	1
TCN	5.87E+09			4.35E+06			2.87E+09			5.25E+05		

schemes. It was also observed that the degree of intensification and improvement in the control properties does not occur in a linear manner, since schemes with a lower degree of intensification may have better control properties than a more intensified one. Through this study the importance of the evaluation of the dynamic performance of intensified schemes is observed, since, being complex configurations, these may or may not have an appropriate control structure. In other words, the control arrangement in intensified schemes is not necessarily the LV control structure, and this is not possible to know a priori without an analysis of its control properties.

Looking for a design that meets the economic-environmental objectives and good control properties. Design B3 has small values of TAC and EI99, and show good dynamic behavior. The last one because this design has a sensitivity index below the average this due to its large flow of extractant, its control structure remained stable at the frequencies studied and it has a value of the smallest of the TCN designs studied this due to the dimensions of its columns.

The designs in scheme D, which consists of a liquid-liquid extraction column and a double-wall dividing column, which showed greater intensification, presented the best TCN values, within the configurations evaluated, but these does not present low values of TAC and EI99. This is due to the fact that the increase of its internal flows decreases the perturbations of the variables of the system, with respect to the output variables of the system. This guideline was reflected in all the schemes studied, since the higher the degree of intensification the TCN was lower. Additionally, the results obtained in closed-loop are consistent with the open-loop study, these results indicate that the degree of intensification may favor the control properties.

In summary, it is possible to obtain designs that meet economic, environmental and control objectives, although, these are not necessarily the most intensified designs.

Author contributions

All authors have participated in (a) conception and design, or analysis and interpretation of the data; (b) drafting the article or revising it critically for important intellectual content; and (c) approval of the final version.

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Declaration of Competing Interest

None.

References

[1] P. Lutze, A. Román-Martinez, J.M. Woodley, R. Gani, A systematic synthesis and design methodology to achieve process intensification in (bio) chemical processes, Comput. Chem. Eng. 36 (2012) 189–207.
[2] A. Stankiewicz, J.A. Moulijn, Process Intensification: Transforming Chemical Engineering vol. 96, (2000), pp. 22–33.
[3] C.E. Torres-Ortega, C. Ramírez-Márquez, E. Sánchez-Ramírez, J.J. Quiroz-Ramírez,

J.G. Segovia-Hernandez, B.-G. Rong, Effects of intensification on process features and control properties of lignocellulosic bioethanol separation and dehydration systems, Chem. Eng. Process. Process. Intensif. 128 (2018) 188–198.
[4] J.G. Segovia-Hernández, A. Bonilla-Petriciolet, Process Intensification in Chemical Engineering, (2016).
[5] Y. Tian, S.E. Demirel, M.M.F. Hasan, E.N. Pistikopoulos, An overview of process systems engineering approaches for process intensification: state of the art, Chem. Eng. Process. Process. Intensif. 133 (2018) 160–210.
[6] G. Lewandowicz, W. Białas, B. Marczewski, D. Szymanowska, Application of membrane distillation for ethanol recovery during fuel ethanol production, J. Memb. Sci. 375 (1) (2011) 212–219.
[7] G.E. Cortes Garcia, J. van der Schaaf, A.A. Kiss, A review on process intensification in HiGee distillation, J. Chem. Technol. Biotechnol. 92 (6) (2017) 1136–1156.
[8] C.E. Torres-Ortega, B.-G. Rong, Synthesis and simulation of efficient divided wall column sequences for bioethanol recovery and purification from an actual lignocellulosic fermentation broth, Ind. Eng. Chem. Res. 55 (27) (2016) 7411–7430.
[9] M. Errico, C. Ramírez-Márquez, C.E. Torres Ortega, B.-G. Rong, J.G. Segovia-Hernandez, Design and control of an alternative distillation sequence for bioethanol purification, J. Chem. Technol. Biotechnol. 90 (12) (2015) 2180–2185.
[10] M. Errico, B.-G. Rong, G. Tola, M. Spano, Optimal synthesis of distillation systems for bioethanol separation. Part 1: extractive distillation with simple columns, Ind. Eng. Chem. Res. 52 (4) (2013) 1612–1619.
[11] H. Luo, C.S. Bildea, A.A. Kiss, Novel heat-pump-assisted extractive distillation for bioethanol purification, Ind. Eng. Chem. Res. 54 (7) (2015) 2208–2213.
[12] V.N. Maleta, A.A. Kiss, V.M. Taran, B.V. Maleta, Understanding process intensification in cyclic distillation systems, Chem. Eng. Process. Process. Intensif. 50 (7) (2011) 655–664.
[13] I. Dejanović, L. Matijašević, I.J. Halvorsen, S. Skogestad, H. Jansen, B. Kaibel, Ž. Olujić, Designing four-product dividing wall columns for separation of a multicomponent aromatics mixture, Chem. Eng. Res. Des. 89 (8) (2011) 1155–1167.
[14] I. Dejanović, L. Matijašević, Ž. Olujić, Dividing wall column—a breakthrough towards sustainable distilling, Chem. Eng. Process. Process. Intensif. 49 (6) (2010) 559–580.
[15] M. Errico, G. Tola, B.-G. Rong, D. Demurtas, I. Turunen, Energy saving and capital cost evaluation in distillation column sequences with a divided wall column, Chem. Eng. Res. Des. 87 (12) (2009) 1649–1657.
[16] Ö. Yildirim, A.A. Kiss, E.Y. Kenig, Dividing wall columns in chemical process industry: a review on current activities, Sep. Purif. Technol. 80 (3) (2011) 403–417.
[17] G. Dünnebier, C.C. Pantelides, Optimal design of thermally coupled distillation columns, Ind. Eng. Chem. Res. 38 (1) (1999) 162–176.
[18] M. Baldea, From process integration to process intensification, Comput. Chem. Eng. 81 (2015) 104–114.
[19] R. Nadendla, A. Venu Vinod, Controllability of Distillation Sequences for the Separation of Ternary Mixture vol. 6, (2011).
[20] F.I. Gómez-Castro, M.A. Rodríguez-Ángeles, J.G. Segovia-Hernández, S. Hernández, C. Gutiérrez-Antonio, A. Briones-Ramírez, A.R. Uribe Ramírez, Analysis of dynamic performance for multiple dividing wall distillation columns, Ind. Eng. Chem. Res. 52 (29) (2013) 9922–9929.
[21] A. Malcolm, J. Polan, L. Zhang, B.A. Ogunnaike, A.A. Linninger, Integrating systems design and control using dynamic flexibility analysis, AIChE J. 53 (8) (2007) 2048–2061.
[22] A. Faanes, S. Skogestad, pH-neutralization: integrated process and control design, Comput. Chem. Eng. 28 (2004) 1475–1487.
[23] D.E. Seborg, D.A. Mellichamp, T.F. Edgar, F.J. Doyle III, Process Dynamics and Control, John Wiley & Sons, 2010.
[24] O.A. Prado-Rubio, S.B. Jørgensen, G. Jonsson, pH control structure design for a periodically operated membrane separation process, Comput. Chem. Eng. 43 (2012) 120–129.
[25] L.A. Ricardez-Sandoval, H.M. Budman, P.L. Douglas, Simultaneous design and control: a new approach and comparsons with existing methodologies, Ind. Eng. Chem. Res. 49 (6) (2010) 2822–2833.
[26] C. Jiménez-González, D.J. Constable, C.S. Ponder, Evaluating the “Greenness” of chemical processes and products in the pharmaceutical industry—a green metrics primer, Chem. Soc. Rev. 41 (4) (2012) 1485–1498.
[27] D.J.C. Constable, A.D. Curzons, V. Cunningham, Metrics to ‘Green’ chemistry—which are the best? Green Chem. 4 (2002) 521–527.
[28] S. Hernández, A. Jiménez, Controllability analysis of thermally coupled distillation systems, Ind. Eng. Chem. Res. 38 (10) (1999) 3957–3963.
[29] J.P. Rodríguez, E. Moreno, J.G. Segovia-Hernández, A. Jiménez, R. Maya-Yescas, Control and energy savings of the Petlyuk distillation system, IFAC Proc. Vol. 40 (5) (2007) 195–200.
[30] J.G. Segovia-Hernández, S. Hernández, V. Rico-Ramırez, A.J. Jiménez, A comparison of the feedback control behavior between thermally coupled and conventional distillation schemes, Comput. Chem. Eng. 28 (5) (2004) 811–819.
[31] E.A. Wolff, S. Skogestad, Operation of integrated three-product (Petlyuk) distillation columns, Ind. Eng. Chem. Res. 34 (6) (1995) 2094–2103.
[32] M. Serra, A. Espuña, L. Puigjaner, Control and optimization of the divided wall column, Chem. Eng. Process. Process. Intensif. 38 (1999) 549–562.
[33] C. Buck, C. Hiller, G. Fieg, Technology, applying model predictive control to dividing wall columns, Chem Eng. Techol. 34 (5) (2011) 663–672.
[34] D. Dwivedi, J.P. Strandberg, I.J. Halvorsen, H.A. Preisig, S. Skogestad, Active vapor split control for dividing-wall columns, Ind. Eng. Chem. Res. 51 (46) (2012) 15176–15183.
[35] R. Ignat, A. Woinaroschy, Dynamic analysis and controllability of dividing-wall distillation columns using a four points control structure, UPB Sci. Bull. Ser. B: Chem. Mater. Sci. 73 (2011) 71.
[36] A. Kiss, C. Bildea, A control perspective on process intensification in dividing-wall columns, Chem. Eng. Process. Process. Intensif. 50 (2011) 281–292.
[37] H. Ling, W.L. Luyben, New control structure for divided-wall columns, Ind. Eng. Chem. Res. 48 (13) (2009) 6034–6049.
[38] H. Ling, W.L. Luyben, Temperature control of the BTX divided-wall column, Ind. Eng. Chem. Res. 49 (1) (2010) 189–203.
[39] L.C. Nhien, N.V.D. Long, S. Kim, M. Lee, Design and assessment of hybrid purification processes through a systematic solvent screening for the production of levulinic acid from lignocellulosic biomass, Ind. Eng. Chem. Res. 55 (18) (2016) 5180–5189.
[40] H. Alcocer-García, J.G. Segovia-Hernández, O.A. Prado-Rubio, E. Sánchez-Ramírez, J.J. Quiroz-Ramírez, Multi-objective optimization of intensified processes for the purification of levulinic acid involving economic and environmental objectives, Chem. Eng. Process. Process. Intensif. 136 (2019) 123–137.
[41] T. Brouwer, M. Blahusiak, K. Babic, B. Schuur, Reactive extraction and recovery of levulinic acid, formic acid and furfural from aqueous solutions containing sulphuric acid, Sep. Purif. Technol. 185 (2017) 186–195.
[42] T.A. Werpy, G. Petersen, Top Value Added Chemicals From Biomass, National Renewable Energy Laboratory, 2004.
[43] J. Martinez-Gomez, E. Sánchez-Ramírez, J.J. Quiroz-Ramírez, J.G. Segovia-Hernandez, J.M. Ponce-Ortega, M.M. El-Halwagi, Involving economic, environmental and safety issues in the optimal purification of biobutanol, Process. Saf. Environ. Prot. 103 (2016) 365–376.
[44] M. Errico, E. Sanchez-Ramirez, J.J. Quiroz-Ramìrez, J.G. Segovia-Hernandez, B.-G. Rong, Synthesis and design of new hybrid configurations for biobutanol purification, Comput. Chem. Eng. 84 (2016) 482–492.
[45] G. Contreras-Zarazúa, J.A. Vázquez-Castillo, C. Ramírez-Márquez, J.G. Segovia-Hernández, J.R. Alcántara-Ávila, Multi-objective optimization involving cost and control properties in reactive distillation processes to produce diphenyl carbonate, Comput. Chem. Eng. 105 (2017) 185–196.
[46] C. Márquez, G. Contreras-Zarazua, M. Martín, J. Segovia-Hernandez, Safety, economic, and environmental optimization applied to three processes for the production of solar-grade silicon, ACS Sustain. Chem. Eng. 7 (2019).
[47] M. Errico, E. Sanchez-Ramirez, J.J. Quiroz-Ramìrez, B.-G. Rong, J.G. Segovia-Hernandez, Multiobjective optimal acetone–butanol–ethanol separation systems using liquid–liquid extraction-assisted divided wall columns, Ind. Eng. Chem. Res. 56 (40) (2017) 11575–11583.
[48] E. Sánchez-Ramírez, J.J. Quiroz-Ramírez, S. Hernández, J.G. Segovia Hernández, G. Contreras-Zarazúa, C. Ramírez-Márquez, Synthesis, design and optimization of alternatives to purify 2, 3-Butanediol considering economic, environmental and safety issues, Sustain. Prod. Consum. 17 (2019) 282–295.
[49] Z. Yuan, B. Chen, G. Sin, R. Gani, State-of-the-art and progress in the optimization-based simultaneous design and control for chemical processes, Process Syst. Eng. 58 (6) (2012) 1640–1659.
[50] S. Skogestad, I. Postlethwaite, Multivariable Feedback Control: Analysis and Design vol. 2, Wiley, New York, 2007.
[51] J.B. Jørgensen, S.B. Jørgensen, Towards automatic decentralized control structure selection, Comput. Chem. Eng. 24 (2) (2000) 841–846.
[52] M.L. Luyben, C.A. Floudas, Analyzing the interaction of design and control—1. A multiobjective framework and application to binary distillation synthesis, Comput. Chem. Eng. 18 (10) (1994) 933–969.
[53] M.L. Luyben, C.A. Fluodas, Analyzing the interaction of design and control—2. Reactor-separator-recycle system, Comput. Chem. Eng. 18 (10) (1994) 971–993.
[54] A. Zheng, R.V. Mahajanam, A quantitative controllability index, Ind. Eng. Chem. Res. 38 (3) (1999) 999–1006.
[55] A. Palazoglu, Y. Arkun, A multiobjective approach to design chemical plants with robust dynamic operability characteristics, Comput. Chem. Eng. 10 (6) (1986) 567–575.
[56] A. Plazoglu, Y. Arkun, Design of chemical plants with multiregime capabilities and robust dynamic operability characteristics, Comput. Chem. Eng. 11 (3) (1987) 205–216.
[57] J.G. Segovia-Hernandez, M. Vázquez-Ojeda, F.I. Gómez-Castro, C. Ramírez-Márquez, M. Errico, S. Tronci, B.-G. Rong, Process control analysis for intensified bioethanol separation systems, Chem. Eng. Process. Process. Intensif. 75 (2014) 119–125.
[58] S. Robles-Zapiain, J. Segovia-Hernandez, A. Bonilla-Petriciolet, R. Maya-Yescas, Energy-efficient complex distillation sequences: control properties, Can. J. Chem. Eng. 86 (2008) 249–259.
[59] J. Alcantara-Avila, J. Cabrera Ruiz, J. Segovia-Hernandez, S. Hernández, B.-G. Rong, Controllability analysis of thermodynamically equivalent thermally coupled arrangements for quaternary distillations, Chem. Eng. Res. Des. 86 (2008) 23–37.
[60] F.I. Gómez-Castro, J.G. Segovia-Hernández, S. Hernández, C. Gutiérrez-Antonio, A. Briones-Ramírez, Dividing wall distillation columns: optimization and control properties, Chem. Eng. Technol. 31 (9) (2008) 1246–1260.
[61] G.R. Salehi, M. Amidpour, K. Hassanzadeh, M.R.J.C. Omidkhah, C. Engineering, Controllability analysis of heat integrated distillation systems for a multicomponent stream, Comput. Chem. Eng. 36 (2012) 282–293.
[62] J.G. Segovia-Hernández, E.A. Hernández-Vargas, J.A. Márquez-Muñoz, Control properties of thermally coupled distillation sequences for different operating conditions, Comput. Chem. Eng. 31 (7) (2007) 867–874.
[63] J.G. Segovia-Hernández, A. Bonilla-Petriciolet, L.I. Salcedo-Estrada, Dynamic analysis of thermally coupled distillation sequences with undirectional flows for the separation of ternary mixtures, Korean J. Chem. Eng. 23 (5) (2006) 689–698.
[64] A.D.A. Acosta-Solórzano, O. Guerrero-Farfán, C. Ramírez-Márquez, F.I. Gómez-Castro, J.G. Segovia-Hernández, S. Hernández, C. Gutiérrez-Antonio, A. Briones-

Ramírez, Controllability analysis of distillation sequences for the separation of bio-jet fuel and green diesel fractions, Chem. Eng. Technol. 39 (12) (2016) 2273–2283.
[65] E. Sánchez-Ramírez, H. Alcocer-García, J.J. Quiroz-Ramírez, C. Ramírez-Márquez, J.G. Segovia-Hernández, S. Hernández, M. Errico, A.J. Castro-Montoya, Biotechnology, control properties of hybrid distillation processes for the separation of biobutanol, J. Chem. Technol. Biotechnol. 92 (5) (2017) 959–970.
[66] M.A. Santaella, L.E. Jiménez, A. Orjuela, J.G. Segovia-Hernández, Design of thermally coupled reactive distillation schemes for triethyl citrate production using economic and controllability criteria, Chem. Eng. J. 328 (2017) 368–381.
[67] P.S. Buckley, J.P. Shunta, W.L. Luyben, Design of Distillation Column Control System, Edward Arnold, 1985.
[68] E. Bristol, On a new measure of interaction for multivariable process control, IEEE Trans. Automat. Control 11 (1) (1966) 133–134.
[69] S. Skogestad, M. Morari, Control configuration selection for distillation columns, AIChE J. 33 (10) (1987) 1620–1635.
[70] I.J. Halvorsen, S. Skogestad, Optimizing control of Petlyuk distillation: understanding the steady-state behavior, Comput. Chem. Eng. 21 (1997) S249–S254.
[71] D. Hayes, S. Fitzpatrick, M.H.B. Hayes, J. Ross, The Biofine Process – Production of Levulinic Acid, Furfural, and Formic Acid from Lignocellulosic Feedstocks vol. 1, (2008), pp. 139–164.
[72] R.A. Burke, Hazardous Materials Chemistry for Emergency Responders, CRC Press, 2013.
[73] O. Santolík, M. Parrot, F. Lefeuvre, Singular value decomposition methods for wave propagation analysis, Radio Sci. 38 (1) (2003).
[74] C. Moore, Application of singular value decomposition to the design, analysis, and control of industrial processes, 1986 American Control Conference, IEEE, 1986, pp. 643–650.
[75] S. Skogestad, The dos and don'ts of distillation column control, Chem. Eng. Res. Des. 85 (1) (2007) 13–23.
[76] O.A. Prado-Rubio, S.B. Jørgensen, G. Jonsson, Control system development for integrated bioreactor and membrane separation process, Computer Aided Chemical Engineering vol. 28, Elsevier, 2010, pp. 289–294.
[77] T.M. Apostol, Calculus vol. 2, (1969).
[78] S. Skogestad, M. Morari, Effect of disturbance directions on closed-loop performance, Ind. Eng. Chem. Res. 26 (10) (1987) 2029–2035.