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Assessment of control properties in intensified processes for levulinic acid purification☆

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ABSTRACT

Currently, the aim is for the processes to consume as little energy as possible and to use raw materials of renewable origin, this to guarantee their sustainability. Recently, proposals have emerged through a methodical synthesis process aimed at purifying levulinic acid, a bioproduct derived from lignocellulosic biomass. These proposals manage to reduce energy consumption and therefore the environmental impact. However, an analysis of the control properties of these designs has not been conducted. Therefore, in this work an evaluation of the control properties was implemented. This with the aim of identifying designs with the best control properties, which translates into more sustainable, safe and operable designs. In the first part, an open loop analysis was carried out to analyze the relationship of the manipulable variables with the output variables, using the total condition number, a sensitivity index, and a relative gain array analysis. In a second stage, the dynamic behavior at closed loop was analyzed, using as a criterion the minimization of the integral absolute error. The results reveal that the design consisting of a liquid-liquid extraction column, three distillation columns and thermal coupling between the last two columns, exhibits the best dynamic performance. Presenting a lower value of total condition number, a sensitive index value below the average, a stable control structure and low values of the absolute integral of the error. In addition, this design presents superior cost and environmental impact indicators, which makes it the best option among the proposed designs.

1. Introduction

Sustainability has become a potential means of combating the harmful effects of anthropogenic development and must therefore be adapted as a new way of life [1]. The chemical industry is adopting various strategies to improve its sustainability, including the use of renewable raw materials, improving energy efficiency, and reducing CO_2 emissions, through the implementation of circular economy and green chemistry principles [2]. In addition, more sustainable processes and products are being developed, and Industry 4.0 technologies are being used to optimize process control and monitoring [3,4]. As fossil-based industry is revealed to be unsustainable and an unviable option, our current society must immediately implement alternative energies and valuable chemical resources [5]. One of these alternatives is the use of lignocellulosic biomass, which is recognized as one of the most abundant and renewable resources on earth. Lignocellulosic biomass has the potential to promote an alternative platform to fossil resources, by producing more than two hundred value-added compounds, such as biofuels, bio-products, and biopolymers [5]. The production of these high-value compounds demands the development of new chemical processes that offer high yields, as well as effective purification methods to achieve the desired purity and recoveries [6]. Purification operations represent a challenge to achieve sustainable processes due to their high energy consumption. One of the alternatives to reduce energy consumption and with it the environmental impact is through the Process Intensification (PI) [7].

PI seeks to improve processes through the manipulation of the phenomena involved in it, with the aim of minimizing the size of the equipment and its energy cost, in some cases by making a combination of unitary equipment [8]. The application of PI will bring with it an improvement in both technology and techniques that will maximize yields, while reducing operability costs. For example, considering that distillation represents about 50 % of the overall energy used in

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Nomenclature

A	Conventional Sequence 1
B	Conventional Sequence 2
C	Conventional Sequence 3
H	Design with n-1 Columns
DETL	Differential Evolution with Tabu List
I	Design with Dividing Wall Column
DWC	Dividing Wall Column
EI99	Eco-indicator 99
IAE	Integral Absolute Error
MSV	Minimum Singular Value
MIMO	Multi-input Multi-output
MPC	Multi-Parametric Control
NMPC	Nonlinear Model Predictive Control
PI	Process Intensification
PIC	Proportional-Integral Controllers
RHD	Reboiler Heat Duty
RR	Reflux Ratio
RGA	Relative Gain Array
SI	Sensitivity Index
SVD	Singular Value Decomposition
SDGs	Sustainable Development Goals
G	Thermally Equivalent Scheme
D	Thermal Coupled Sequence 1
E	Thermal Coupled Sequence 2
F	Thermal Coupled Sequence 3
TAC	Total Annual Cost
TCN	Total Condition Number

Greek letters

γ	Condition number
σ^*	Maximum singular value
σ_*	Minimum singular value
ω	Frequency

separation operations [9], the PI used in distillation systems contemplates the hybrid nonreactive separation as dividing wall columns (DWC) [10], Petlyuk columns [11] and thermally coupled columns [12] present significant energy savings, obtaining savings of more than 25 % [13,14].

PI is a useful and powerful strategy for more efficient, cheaper, safer, and more environmentally friendly processes. However, one of the challenges when designing intensified processes is their industrial operability, which is determined by the controllability of the process [15]. The increased complexity of an intensified process could result in a process that is difficult to control or requires a more sophisticated or novel control structure [16]. In other words, highly efficient but difficult to operate intensified processes can be generated. The study of control in intensified processes can be complex, some studies [17–20] showing that intensified designs may exhibit improved control properties compared to conventional schemes and other studies [15,21] claiming that increased control does not necessarily improve control properties. Therefore, the search for intensified processes of utility in industry requires an in-depth analysis of their control properties [22], this is because each process is different, and its dynamic sharing cannot be intuited. Process control enables safe, economical and environmentally optimal performance of intensified processes. Nikačević et al. [23] provide an overview of process control in process intensification, highlighting the orientation of current research towards advanced model-based control and optimization, and the specific challenges of intensified process control:

- Modeling and parameter tuning: It requires great effort, is expensive, and time-consuming. The models are obtained through rigorous first-principles modeling or system identification techniques [24].
- Complex dynamic behavior: Intensified processes are highly nonlinear, integrating multiple physical and chemical phenomena into smaller devices, making online applications difficult and requiring advanced developments such as nonlinear model predictive control (NMPC) and multi-parametric (MPC) [24].
- Reduced degrees of freedom: Intensified systems have fewer degrees of freedom due to the combination of unit operations in a single device, which limits the possibilities of measurement and manipulation [25].

These challenges highlight the importance of an in-depth analysis of control properties to develop effective strategies and contribute to more sustainable and efficient processes. By developing processes with good control properties, it is possible to contribute to the Sustainable Development Goals (SDGs) by generating a process with greater energy efficiency, lower emissions of pollutants and waste, and optimized use of resources. In addition, processes with superior control characteristics are inherently safer and support the transition to Industry 4.0, enabling more precise and sustainable production [26]. Therefore, a simultaneous analysis between design and control when building intensified processes allows for a more efficient, sustainable, and profitable process [27].

Incorporating process operability indices during design allows us to develop chemical processes that are efficient, economically viable, and dynamically stable near the point of operation [28]. To analyze the control of distillation systems, as well as other industrial systems, various techniques and indices have been developed to understand their dynamic behavior. Molina-Guerrero et al. [29] performed an analysis of the open-loop control of an extractive distillation system, analyzing the condition number (γ) and the minimum singular value (MSV) and the minimum integral absolute error (IAE) in the closed loop. Li et al. [30] performed a closed-loop analysis minimizing the IAE for distillation systems with thermally coupled columns. Pushkala et al. [31] Conducted theoretical studies to determine the optimal control structure for a reactive distillation column using the relative gain array (RGA). Several studies have used γ to evaluate the control properties in conventional and intensified distillation systems such as: DWC, thermally coupled columns, and Petlyuk columns [21,31–33]. These studies show that despite the complex structure of intensified distillation systems, their control properties may not only be comparable to those of conventional distillation sequences but could potentially be better. Alcocer-García et al. [21] conducted a control study of distillation schemes with different degrees of intensification, using the RGA, γ, a feeding sensitivity index (SI) and minimization of the IAE, finding that not necessarily the schemes with greater intensification presented the best control properties. Therefore, performing dynamic analyzing processes is essential to understand their behavior, make a better selection in the design stage and thus build control strategies that allow the operation of the process.

Therefore, in this work, the analysis of the control properties of a set of processes for the purification of levulinic acid, a bioproduct of commercial interest, is proposed. The analysis was conducted with a closed loop and an open loop. The open loop analysis was performed using the γ, RGA and SI as indicators. On the other hand, the closed loop was analyzed taking as a criterion the minimization of the IAE through the tuning of gain and integral time. The objective of this analysis is to determine the dynamic properties of the schemes, which will allow us to predict control problems and develop strategies to solve or avoid them.

Information gathering on total annual cost (TAC), environmental impact through the eco-indicator 99 (EI99) and control properties will allow for a thorough understanding of the schemes studied and identify those that could be viable for the industry.

2. Problem statement

Control in intensified processes is crucial to ensure operational stability, optimize resources, guarantee process safety, and maintain product quality. Additionally, it allows for higher energy efficiency, reduces emissions and waste, and facilitates integration into automated systems, supporting the transition to Industry 4.0. Effective control in these schemes contributes to more sustainable, efficient, and adaptable processes, essential for industrial modernization and environmental sustainability.

Recently, Alcocer-Garcia et al. [34] developed a systematic synthesis of intensified alternatives for the purification of levulinic acid, the designs consist of a liquid–liquid extraction column followed by distillation columns, shown in Fig. 1: conventional sequences (A, B, C), thermal coupled sequences (D, E, F), thermally equivalent schemes (G), designs with n-1 columns (H) and DWC (I).

These schemes were optimized following the stochastic hybrid method of Differential Evolution with Tabu List (DETL). This optimization used the TAC as an economic criterion and the minimization of EI99 as an environmental objective. Although these designs present savings of TAC and EI99, it is important to analyze the control properties, since as mentioned above it is essential to know their dynamic behavior, to determine if the process is operable, safe, and digitizable. A similar study on the design of levulinic acid purification processes [21] used the same control property indicators as those proposed in this work. However, the designs considered in the present work are different. This is the first time that an analysis of control properties has been performed on these designs.

3. Methodology

In this work, an analysis of the control properties in the schemes previously optimized by Alcocer-García et al. [34], which are detailed in Figs. 2-10. The annual production of levulinic acid considered is 5x10^7 kg/year for designs. The feed used for the mixture to be purified is 90000 kg/h, with a mass composition of 86 % water, 7 % levulinic acid, 4 % furfural, and 3 % formic acid [35].

The analysis was conducted with open loop and closed loop, to have a better understanding of the dynamic behavior of each of the schemes. This study considers the control variables to be the compositions of levulinic acid, water, formic acid, and furfural. Therefore, the control loops were closed in the streams that present the compounds of interest at higher concentrations.

3.1. Open-loop analysis methodology

Open loop analysis was performed using three indicators: γ which measures the degree of directionality in multiple input multiple output (MIMO) systems, the RGA which evaluates interactions between output

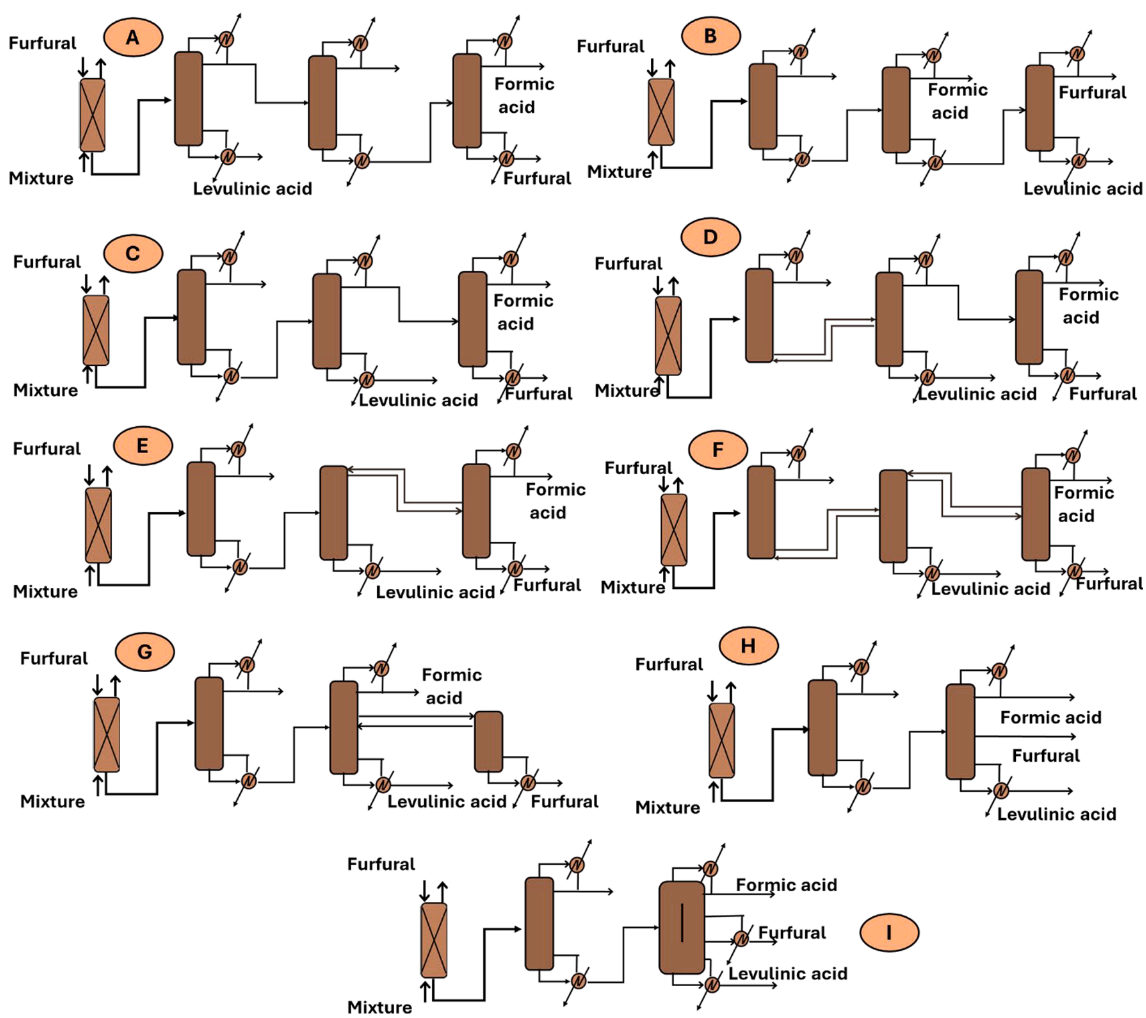

Fig. 1. Schemes analyzed in this study, proposed by Alcocer-García et al. [34].

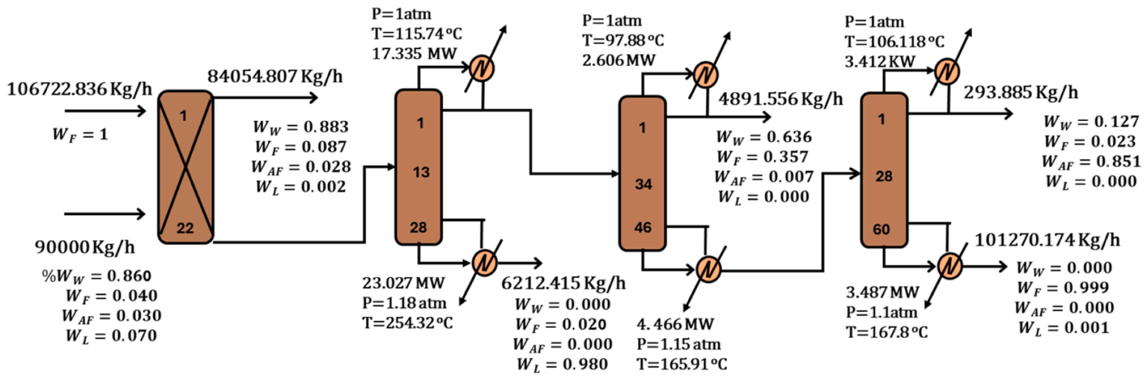

Fig. 2. Design specifications of conventional sequence 1 (A).

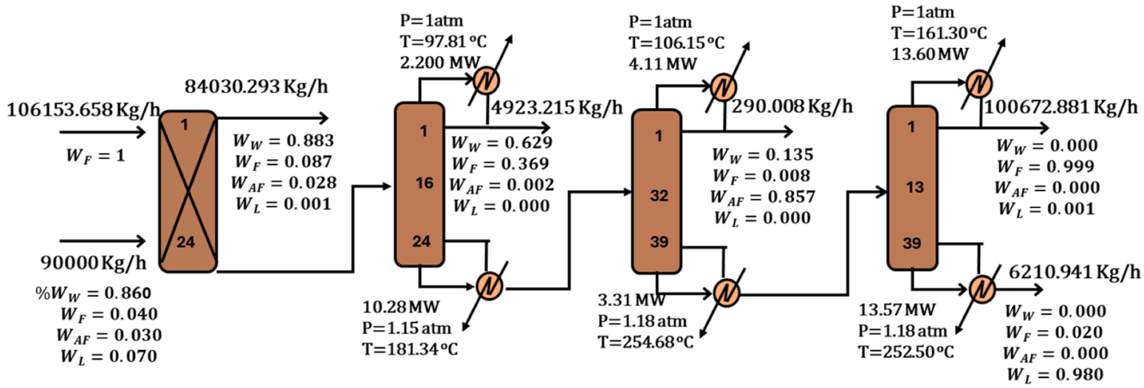

Fig. 3. Design specification of conventional sequence 2 (B).

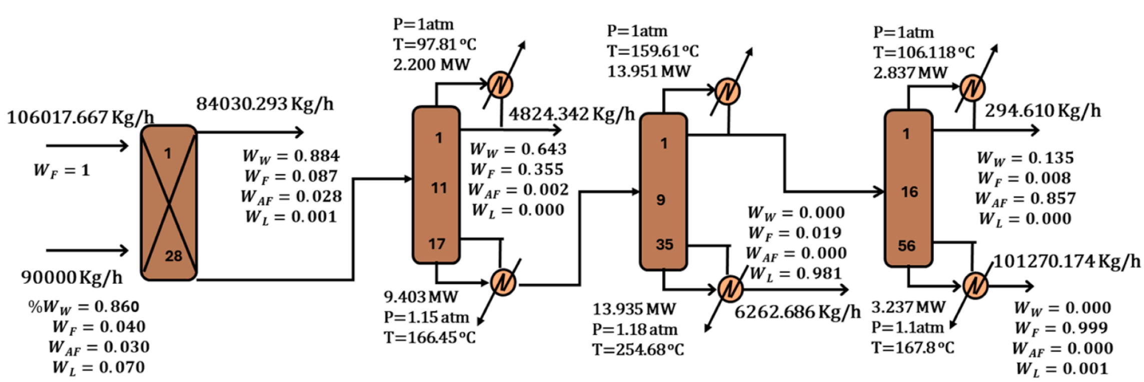

Fig. 4. Design specification of conventional sequence 3 (C).

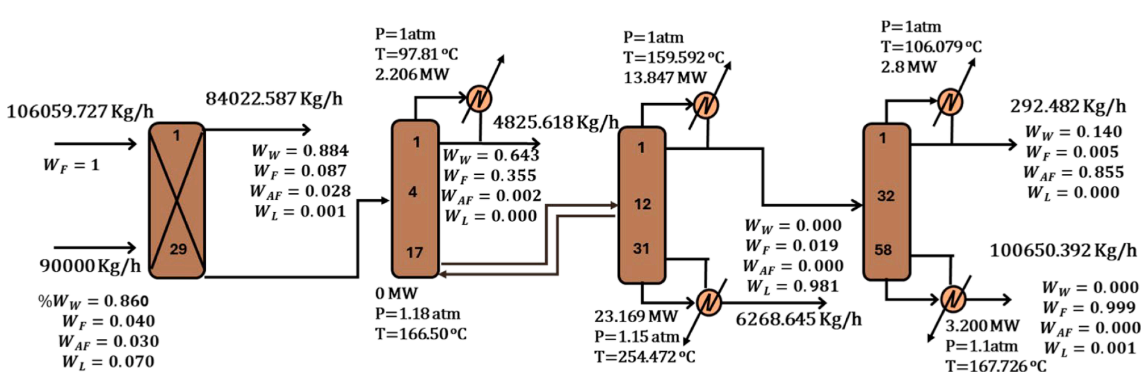

Fig. 5. Design specification of thermally coupled sequence 1(D).

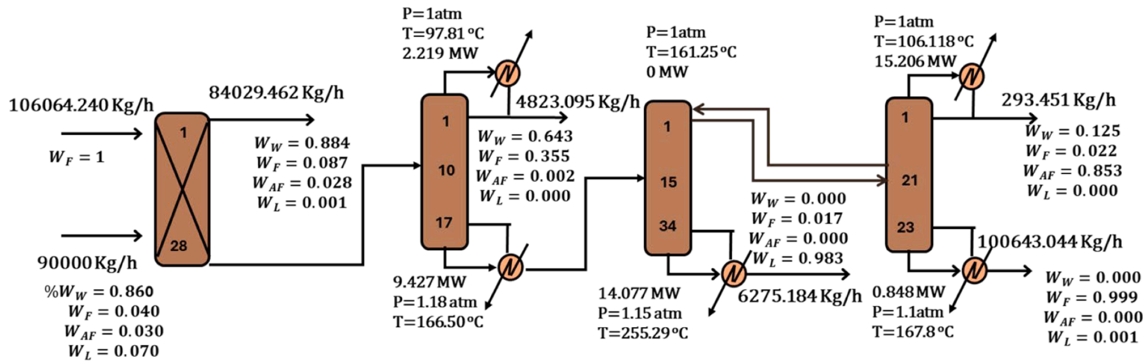

Fig. 6. Design specification of thermally coupled sequence 2 (E).

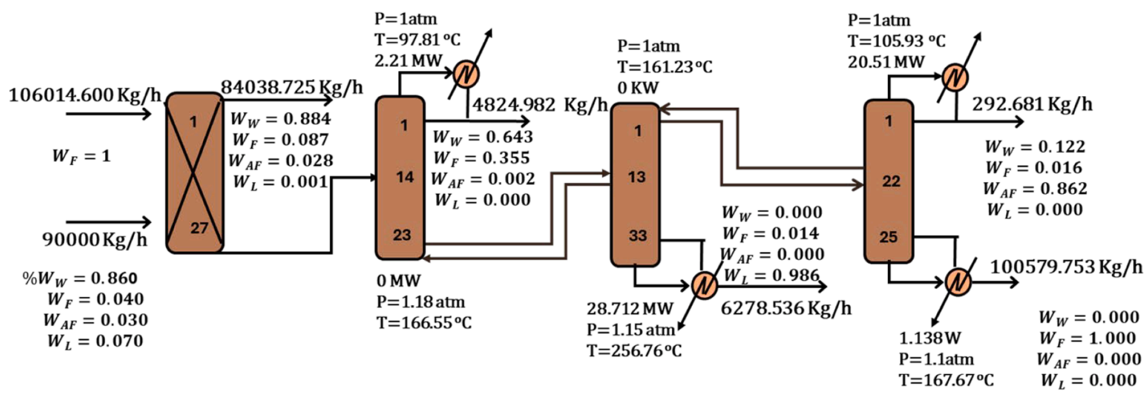

Fig. 7. Design specification of thermally coupled sequence 3 (F).

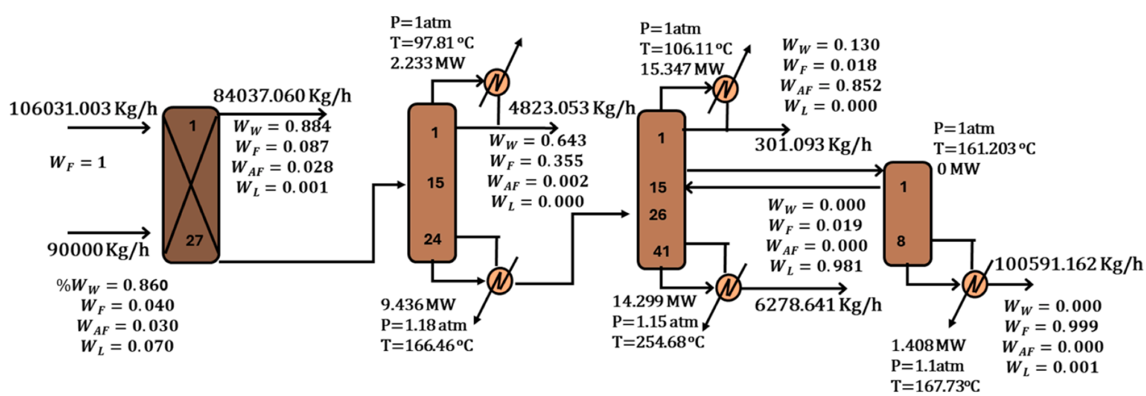

Fig. 8. Design specification of thermodynamic equivalent sequence (G).

variables and manipulable variables, and a SI which evaluates the sensitivity of the system to disturbances in the feeding.

Considering the complexity of the designs to study, the linear model required for indicator calculations was obtained by identifying the open-loop dynamic response of the system using the Aspen Dynamics simulator. Therefore, the schemes proposed by Alcocer-García et al. [34] were simulated in Aspen plus, for subsequent export to Aspen Dynamics, with the objective of performing perturbations in the system and obtaining dynamic responses. Because the components of the mixture are organic acids and polar compounds, the NRTL-HOC thermodynamic model was used [35,36]. For its part, the Non-Random Two-Liquid (NRTL) model is recommended for highly non-ideal chemical systems and can be used to calculate the liquid–vapor equilibrium. On the other hand, the Hayden-O′Connell (HOC) equation of state calculates the thermodynamic properties of the vapor phase of organic acids.

Step perturbations were applied around the nominal operating point, with a step change of + 0.5 % for each manipulated variable. This amplitude is chosen based on a sensitivity analysis to minimize the effect of nonlinearity using small perturbations [37]. This disturbance is made with the aim of being able to analyze the behavior of the systems, monitoring the behavior of the composition when performing a perturbation in the manipulable variables. After performing the perturbations and getting their responses (composition vs. time), they are adjusted to a transfer function in the time domain using MATLAB. The data is adjusted by linear regression to different transfer functions and the one with the best fit is selected. Then, each transfer function is transformed to the frequency domain. With the obtained transfer functions, the matrix of transfer functions is constructed, which serves as the basis for calculating the γ and the RGA.

To calculate the SI, only the dynamic responses obtained by disturbing the feeding are taken, then their calculation is conducted by finite differences and the norm is obtained. The general methodology for

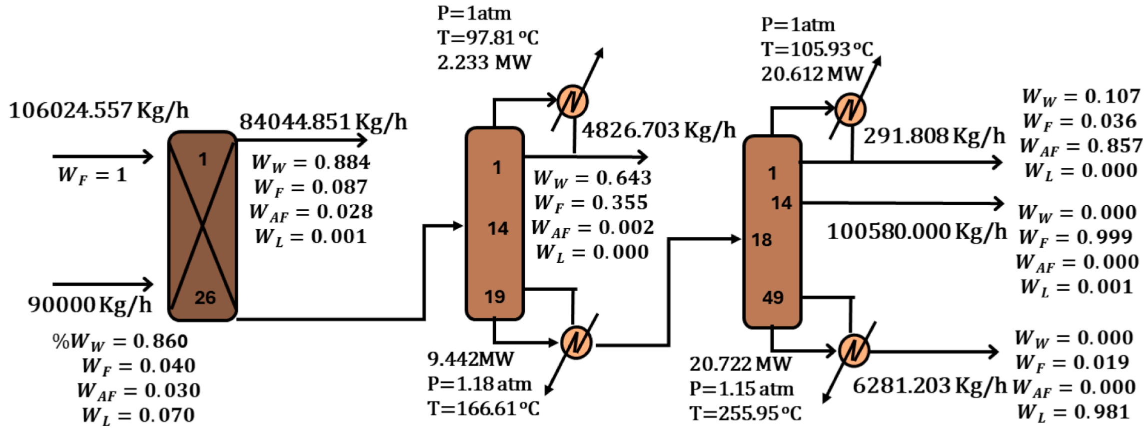

Fig. 9. Design specification of n-1 columns sequence (H).

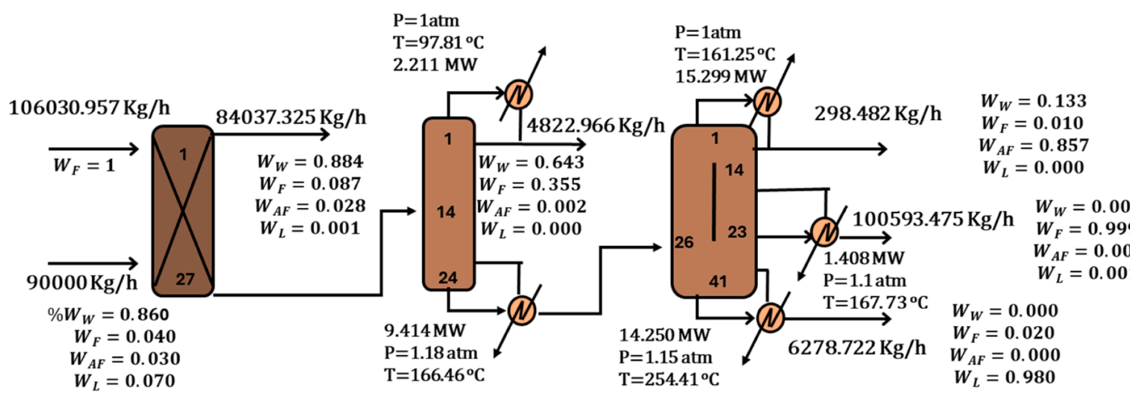

Fig. 10. Design specification of sequence with DWC (I).

open loop analysis is shown in Fig. 11.

3.1.1. Relative gain array

RGA provides a jointly-conditioned relative measure of input–output interactions among a MIMO system [38]. The RGA is a function of a matrix, which typically is interpreted as a plant/gain matrix G for a set of input parameters that control a set of output parameters, and is defined for nonsingular G as [39]:

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

Where $\otimes$ represents the elementwise Hadamard matrix product [39]. Originally RGA was believed to be only relevant in the steady state (zero frequency). However, the phase crossover frequency of RGA is more important, as it tells us how the control structure will vary at different frequencies [40]. In this study, RGA was calculated at three distinct frequencies 0, 0.001, and 1e5 (rad/h) to assess whether the control structure remains consistent. These frequencies represent steady state, near steady state, and high frequency conditions, respectively. When a control structure in a RGA study varies at different frequencies, it indicates that the behavior of the control system is frequency-dependent, suggesting nonlinearity and variations in interactions between process variables. This can affect the effectiveness of control at different frequencies, requiring adaptive or robust control strategies. Evaluating these properties is essential to designing a control system that works efficiently under all operating conditions.

The RGA analysis produces a matrix that shows how the manipulable variables of the system affect the outputs. For distillation columns, the most used control structure involves controlling the distillate composition with the reflux ratio (RR) and the bottom composition with the reboiler heat duty (RHD) [41,42]. This control structure is known as LV, where the L represents the reflux flow and the V boilup flow, see Fig. 12. For this case study, the LV configuration corresponds to a Type 1 control structure.

However, the RGA can identify different configuration than LV, which can also achieve stable control in different frequencies, control structure Type 2. In addition, of the Type 1 and Type 2 structures, there may be a Type 3 structure, in which for each frequency studied there may be an LV or DVL array. These distinct types of configurations are summarized in Table 1.

In this study, an LV configuration was chosen for the calculation of the different indices for the designs: A, B, C, D, F, G, and I. For the scheme H, for the first distillation column an LV arrangement is used, for the second distillation column with side outlet, the composition in the distillate and bottoms flow is controlled using the LV configuration and the composition of the side stream with the flow rate of the same side stream. The bottom composition of the liquid–liquid extraction column, present in all designs, is controlled by the extractant flow.

3.1.2. Condition number

The γ is used to measure controllability between inputs and outputs, evaluating the impact of uncertainties in process parameters and modeling errors on system performance. Furthermore, this number points out the difficulties in decoupling the interaction between control loops in systems with high values of γ [43]. Calculated using the Singular Value Decomposition (SVD) methodology, the γ is a key tool in linear systems theory and plays a fundamental role in the analysis and design of control systems for industrial processes [44].

To calculate γ, linear models of the system were generated using the same perturbations as in the RGA analysis and the same manipulable and output variables. After obtaining all dynamic responses with the

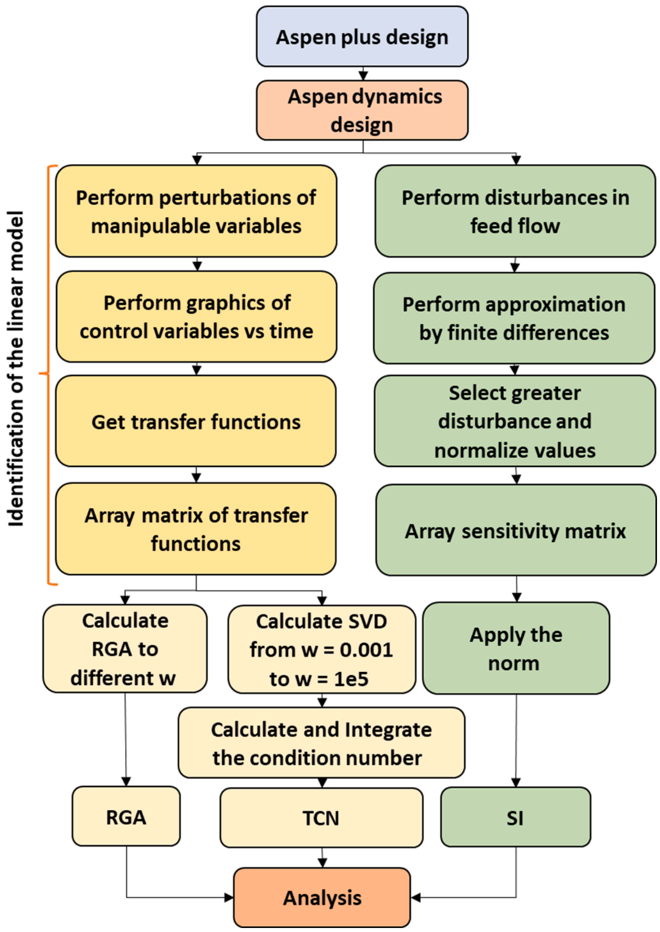

Fig. 11. Open-loop methodology.

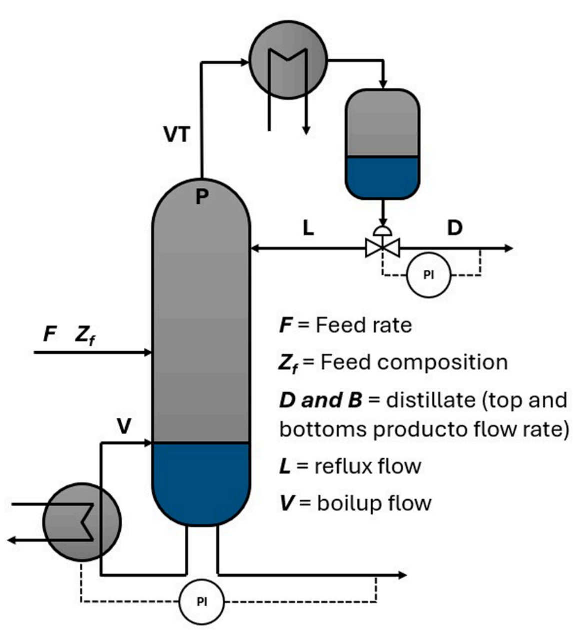

Fig. 12. Conventional distillation column with a control LV structure.

Aspen Dynamics simulator, transfer function matrices (G) were collected and subjected to SVD analysis. Importantly, these transfer function matrices reflect the entire dynamics of the plant. The SVD analysis was performed as follows:

Table 1
Types of arrangements possible in the study of the RGA.

Type of arrangement	Description
Type 1	LV Configuration
Type 2	Different configuration from the LV but constant at different frequencies
Type 3	Changing configuration at different frequencies

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

Where $\sum = diag(\sigma_1, \sigma_2 \cdots . \sigma_n)$, σ_1 = singular value of $G = \frac{\lambda 1}{2}(GG^H)$, $V = (V_1, V_2, \cdots V_n)$ singular matrix of left vectors and $W = (W_1, W_2, \cdots W_n)$ is the matrix of the right singular vectors. From the SVD of G, the two main parameters of interest are the minimum singular value σ_* and the ratio between the maximum σ^* and maximum singular value, known as the γ, which can be calculated at the frequency of interest [40]:

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

Comparing γ helps determine which system is more likely to encounter control issues given a specific control structure. A high γ can indicate potential problems, often associated with a low value of σ_*, although a high value of σ^* is not necessarily problematic. A high γ implies that the system is sensitive to input uncertainties, although such uncertainties often do not occur in practice. Therefore, it cannot be concluded that a system with a high γ is necessarily more sensitive to uncertainties. Systems with lower γ are expected to show better dynamic performance under the proposed feedback control structure [45]. Therefore, systems with a lower condition number tend to show better dynamic performance and are generally easier to control, since they are less sensitive to disturbances. This allows the implementation of simpler and more direct control strategies, such as standard Proportional-Integral-Derivative control, without the need for constant adjustments or advanced techniques.

Although the γ provides information at a certain frequency, it is complex to be able to analyze its behavior in a frequency range. Santaella et al. [46] proposed determining the γ as a function of frequency and then calculating an accumulated metric called the total condition number (TCN). Therefore, the area under the curve of the γ as a function of frequency is evaluated:

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

For this study, the frequency range studied for the TCN and the frequency range was between 0.001 and 1e5, this is based on similar works that perform analysis of the γ within this range [21,47].

3.1.3. Sensitivity index

The SI evaluates how input disturbances affect the system. Prado-Rubio et al. [48] proposed the SI and evaluated the dynamic sensitivity of the control variables to variations in the manipulable input variables.

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

Where X represents the variable of interest to evaluate sensitivity. The index is dimensionless so as not to be influenced by the selection of units or the scale of the variable. The finite difference approximation is used for the derivatives and is proportional to the square root of the increment used. The responses to the perturbation are nonlinear functions of time, from t = 0 to the steady state. This index provides valuable dynamic information that would otherwise be difficult to obtain for this system [48].

For the SI calculation, the output variables were the same as those used for the RGA and TCN calculations, while the manipulated variables were the composition of levulinic acid and the flux of levulinic acid in the diet. Once the variables were identified, perturbations of + 0.5 % were applied, like those used for other indices in Aspen Dynamics. In this case, there are two manipulable variables and four outputs, this results in eight sets of data after performing the perturbations. Using equation (5) for each data set (concentration versus time), a finite difference approximation was performed, normalized with the initial values of the manipulated variables, and the maximum range value always was selected. Finally, the sensitivity matrix (P) was assembled with the standard values for each perturbation and its norm was evaluated, resulting in a single value for both perturbations.

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:

$$\|P\| = \sum_{i=1}^{m}\sum_{j=1}^{m}|aij| \tag{6}$$

The norm of P is the sum of the absolute values of all its elements [49]. This value indicates the sensitivity of the system to input disturbances. Therefore, lower values of this norm suggest that the system is more stable under feeding disturbances. A high SI value suggests a high sensitivity of the system to disturbances in the feed, which would require implementing robust control strategies, such as predictive control to anticipate and correct disturbances, and adaptive control to adjust parameters in real time to maintain process stability and efficiency.

3.2. Closed-loop analysis methodology

The closed-loop performance for tracking the setpoint of the output composition was assessed. This evaluation helps validate the operability indices predicted in other studies [60,63]. The analysis specifically targeted the output of levulinic acid, as it is the main product of interest in this case study.

The closed loop analysis was conducted in Aspen dynamics. Step changes were made in the composition of levulinic acid, with variations of 1 %, 5 %, 10 %, and 30 %. These changes were implemented to evaluate both small and large perturbations in the system. By making these step changes, the goal was to observe how the system responds to different magnitudes of disturbance, which is essential for understanding its dynamics and robustness. Small perturbations, such as the 1 % and 5 % changes, allow for the analysis of the system's sensitivity to minimal variations, while larger perturbations, such as the 10 % and 30 % changes, help evaluate its response to significant changes. This information is crucial for designing effective control strategies that ensure the stability and efficiency of the process under variable operating conditions. Proportional-integral controllers (PIC) were implemented to control the composition, following the established control structure. In this case, the reboiler duty of the reboiler was used as a control variable for the composition of levulinic acid in all schemes. Composition controllers were selected because they facilitate composition control, especially in theoretical studies. However, it is important to mention that at an industrial level, the use of temperature controllers is chosen as these are usually cheaper [50]. For PIC, proportional gain (Kc) and reset times (τi) were adjusted in each analyzed scheme. Taking as tuning criterion the minimization of IAE, as shown in Fig. 13.

The IAE It is a measure of the controller's tracking accuracy by integrating the absolute error (e) over time between the reference value and the output composition:

$$IAE = \int_0^\infty |e(t)|dt \tag{7}$$

A high IAE indicates that the system is performing less efficiently, with larger, longer-lasting errors compared to a lower IAE number. This suggests that the PI controller is not correcting errors effectively, resulting in a longer settling time and possibly a less stable system. In contrast, a low IAE reflects more efficient performance of the control system, with smaller and more briefly sustained errors, indicating a better ability of the PI controller to keep the system close to its set point.

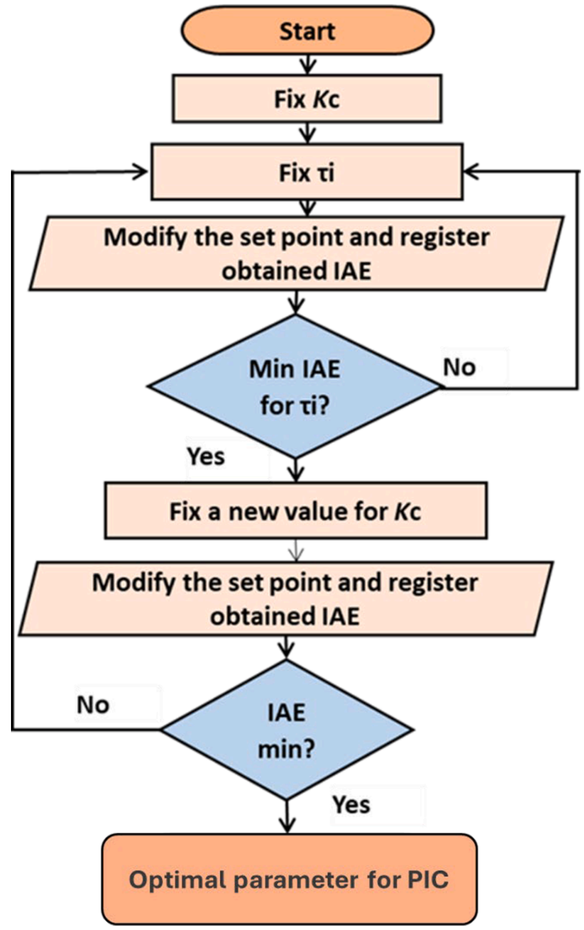

Fig. 13. Process diagram for tuning PIC.

4. Results

4.1. Results of open-loop control analysis

The analysis of the RGA was implemented, and the results are shown in Table 2. It was obtained that designs B, D and E present a Type 1 configuration in all the frequencies studied, which is the most used type in distillation columns and corresponds to a stable LV configuration at different frequencies. These designs have better dynamic performance, from steady-state behavior to response to fast disturbances. It is

Table 2
Results of open loop indices.

Design	TAC	EI99	TCN	SI	RGA
A	1.85E + 07	5.35E + 09	5.87E + 09	13.37	Type 3
B	1.56E + 07	4.70E + 09	1.04E + 07	6.68	Type 1
C	1.49E + 07	4.60E + 09	1.95E + 08	12.37	Type 2
D	1.64E + 07	4.56E + 09	4.68E + 09	0.50	Type 1
E	1.39E + 07	4.21E + 09	6.44E + 06	11.28	Type 1
F	1.90E + 07	5.16E + 09	1.78E + 07	33.15	Type 3
G	1.43E + 07	4.35E + 09	1.18E + 07	11.50	Type2
H	1.77E + 07	5.22E + 09	6.30E + 07	7.56	Type 3
I	1.43E + 07	4.34E + 09	1.23E + 07	11.49	Type 2

important to visualize that the E design also presents the best values of TAC and EI99, which makes it an interesting alternative since apart from being the cheapest and economical, it presents a stable control structure.

On the other hand, designs C, G and I present a Type 2 configuration in all the frequencies studied, which implies that about 30 % of the designs studied present a configuration in which a manipulable variable different from the one established in the LV configuration has a greater influence on the output variable. This configuration, although stable, differs from the LV and may require more specific control. Design G, which represents the thermodynamically equivalent design, and design I, which corresponds to the design with the DWC, have slightly higher values of TAC and EI99 compared to design E. Despite having slightly higher values in these metrics, both G and I designs show a stable control structure. This means that, although they are not the most economical or have the least environmental impact, their control configurations are robust and effective. Therefore, designs G and I could represent viable alternatives to design E. The choice between these designs could depend on specific priorities, such as minimizing operating costs and environmental impact, versus the need for a highly stable and effective control system.

Finally, designs A, F and H present a Type 3 structure, which is unstable in all the frequencies studied. This means that the system responds differently to control signals from different frequencies, which may require sophisticated control strategies to maintain optimal performance. These strategies could include predictive control or supervisory control models. The use of these advanced techniques would make it possible to anticipate and correct disturbances before they significantly affect system performance. However, it is important to note that implementing these advanced control strategies can be costly due to the need for specialized software, high-capacity hardware, and staff training. Despite these costs, these approaches are crucial to ensure the operability and safety of systems with Type 3 control structures, where unstable response to various frequencies could lead to unpredictable and potentially detrimental behavior to the process. Although advanced control strategies could improve stability and performance, designs A, F, and H have high values in TAC and EI99, making the implementation of these techniques inconvenient due to high costs and the need for specialized training. It is wiser to consider designs with a better balance between cost, environmental impact, and system stability.

As for the TCN, design A presents the worst value, this can be attributed to the fact that, in this sequence, water is not removed in the first distillation column, unlike the other sequences. This causes the system to become more unstable as it is subject to disturbances in the controllable variables due to the presence of water in the system. In addition, design A features the largest dimensions of equipment, as shown in Fig. 14a, which makes the disturbance propagate over a larger volume and is more complex to stabilize.

One of the variables that has a significant impact on the value of the TCN is the diameter of the columns. In general, it is observed that, the smaller the dimensions of the columns, the lower the TCN. This is because smaller diameter columns have a lower mass and energy holding capacity, which facilitates more effective disturbance control and faster system response to changes in control variables. Designs B and E have the best TCN values, which can be attributed to the fact that these designs have smaller diameters compared to the rest of the configurations analyzed. Reducing column diameter not only improves TCN but can also lead to a decrease in capital and operating costs, because smaller equipment typically requires less material and energy to operate.

Another factor that impacts the behavior of TCN is the number and location of thermal couplings in the distillation columns. For example, design D presents a thermal coupling between the first two distillation columns, which causes the perturbations in the manipulable variables to propagate to the other columns, thus increasing the TCN. In contrast, the F design also includes the thermal coupling between the first two columns, like the D design. However, the F design features a second thermal coupling between the last distillation columns, which helps to reduce the impact of disturbances. This additional coupling improves the distribution of disturbances and contributes to keeping the system more stable, resulting in a lower TCN value.

In addition, the number of stages in distillation columns has a direct effect on TCN, as designs featuring columns with the highest number of stages have the worst TCN values, as shown in Fig. 15b. This is because a greater number of stages can increase the complexity of the control and the sensitivity of the system to disturbances. On the other hand, designs that have better TCN values tend to have the most efficient energy consumptions (Fig. 15a), which reaffirms that a process with efficient dynamic control operates closer to its optimal conditions, resulting in lower energy consumption [18]. It is interesting to note that variables such as liquid–liquid extraction column dimensions, extractant flow, and reflux ratio did not show any significant trends in performance indicators. This suggests that although these variables are important for the overall process design, they do not have a direct impact on the TCN and therefore on the stability and efficiency of the system's dynamic control.

Design D has a SI close to zero. Interestingly, this same design exhibits high TCN values, which indicates that, although the thermal coupling between the first two columns provides greater stability against disturbances in the feed, it also makes the system more unstable against disturbances coming from controllable variables. This suggests

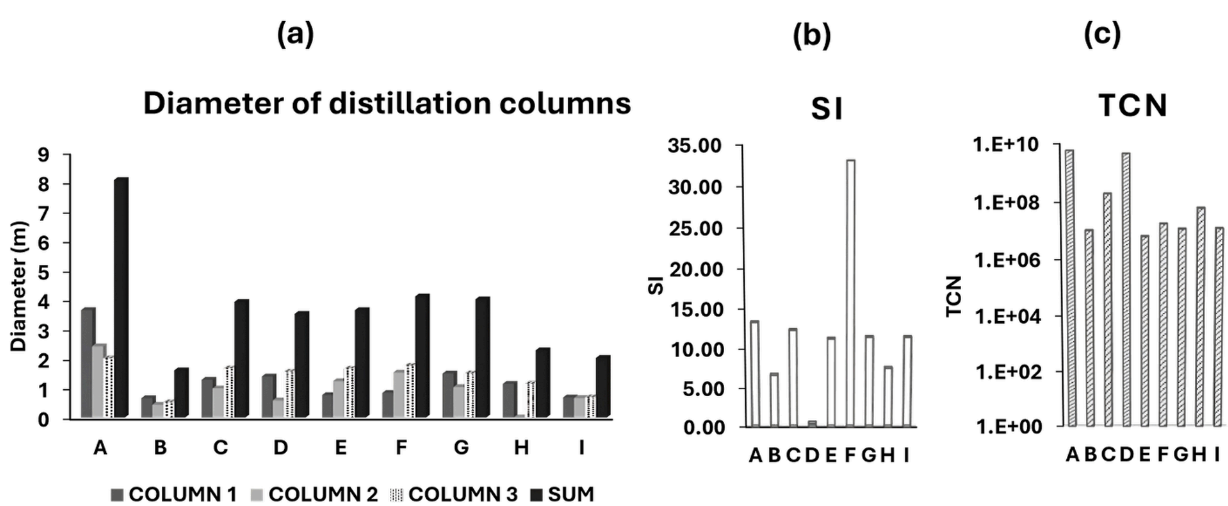

Fig. 14. Influence of the diameter of the distillation columns (a) on the SI (b) and TCN (c).

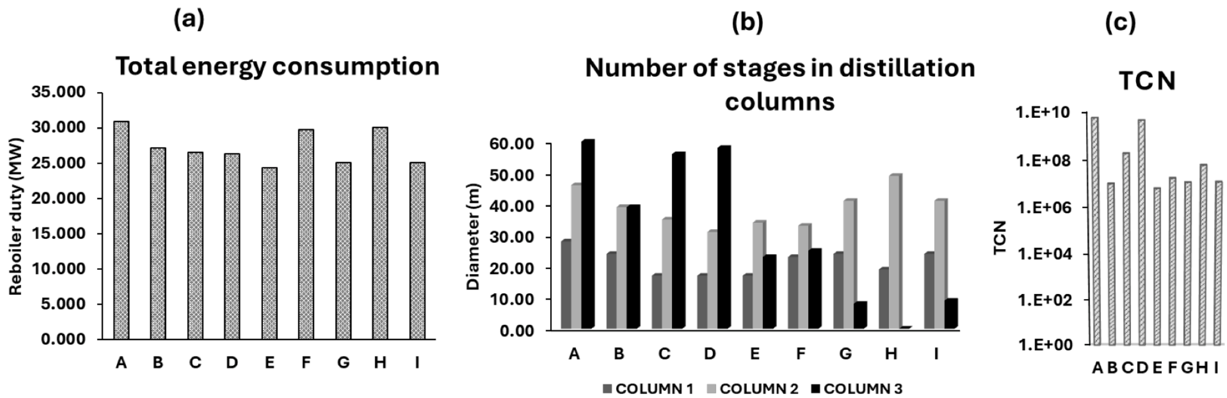

Fig. 15. Relationship between the total energy consumption (a), the number of stages of the distillation columns (b) and TCN (c).

that design D, while efficient in handling variations in feeding, may be less robust when it comes to controlling other process variables. The F design, on the other hand, has the highest SI value. This behavior can be attributed to the fact that it is the only design that has two thermal couplings. The presence of these additional couplings causes disturbances in the feed to propagate more aggressively through the system, increasing the sensitivity of the F design to these changes. This high sensitivity can be a disadvantage in terms of stability, but it can also offer opportunities for more precise control if managed properly.

In general, a system with a high TCN also tends to have a high SI, as both indicators reflect the sensitivity and stability of the system. If a system is very sensitive to disturbances in manipulable variables (high TCN), it is likely to be sensitive to disturbances in the feed (high SI). Systems with low TCN and SI values tend to be more stable and less sensitive to disturbances, which is desirable to maintain efficient and stable control. In contrast, systems with high TCN and SI values require more sophisticated and often expensive control strategies to maintain stability.

4.2. *Results of closed-loop control analysis*

The responses obtained in the closed-loop analysis are shown in Fig. 16, and the results of the PIC tuning are presented in Table 3. The starting composition in designs is different, this is because during the optimization process, a minimum purity of 98 % by weight was taken as a criterion, however, several of these designs managed to achieve a higher purity, with design F reaching the highest of 98.6 %.

In all perturbations, design D presents the lowest values of SAI as shown in Fig. 16. This rapid response to all disturbances is due to the recycling that exists between the first two distillation columns, which helps to dampen disturbances. Also, it is important to note that this design features the smallest Kc (proportional driver gain) values. Small Kc values suggest that the driver's responses are smooth, which improves stability, reduces oscillations, and prevents an overly aggressive response [51].

On the other hand, design A has the highest IAE values, which can be attributed to the fact that it is the only design that recovers levulinic acid in the first distillation column. This means that the water is removed in

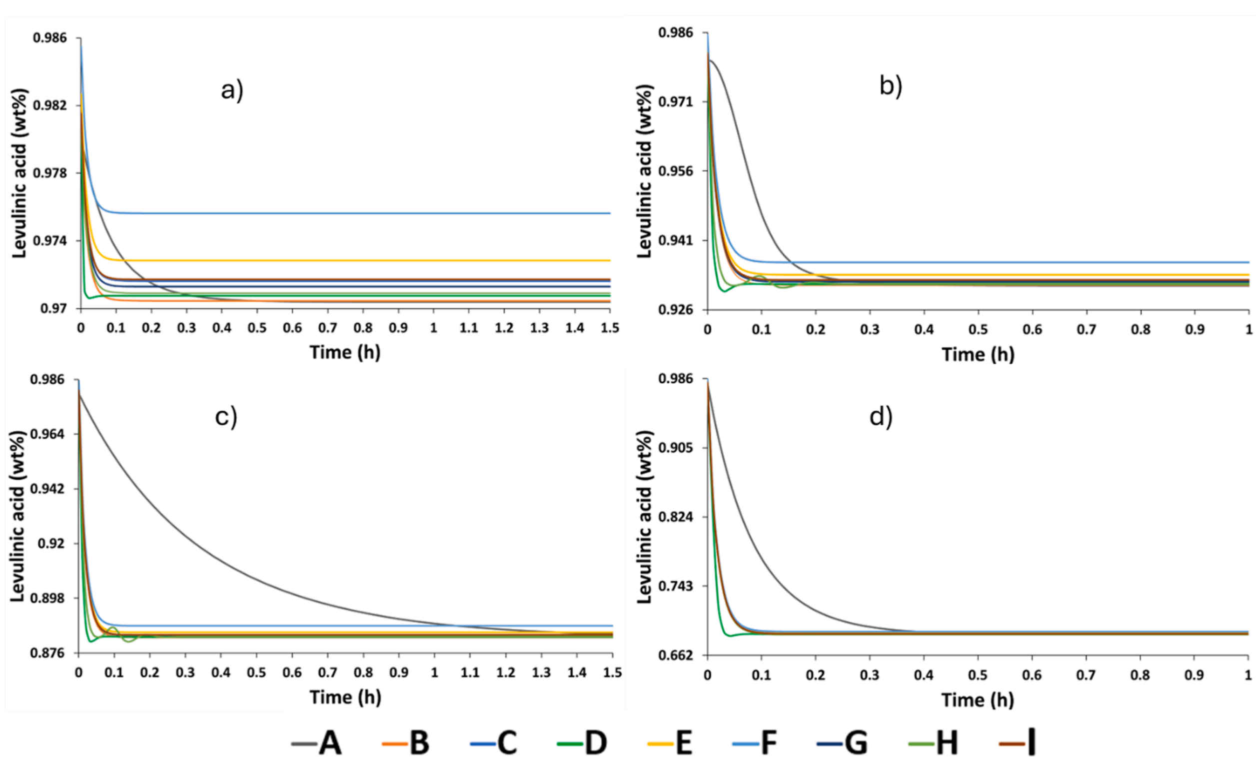

Fig. 16. Set point tracking at 1%(a), 5%(b), 10%(c) and 30%(d).

Table 3
PIC tuning results.

Design	Step change	Kc (%/%)	τi (min)	IAE	Step change	Kc (%/%)	τi (min)	IAE
A	1 %	245	5	8.11E-04	5 %	250	5	4.12E-03
B	1 %	250	1	1.61E-04	5 %	250	1	7.93E-04
C	1 %	249	1	1.58E-04	5 %	250	1	7.91E-04
D	1 %	76	1	1.75E-05	5 %	19	1	2.79E-04
E	1 %	250	1	1.58E-04	5 %	250	1	7.92E-04
F	1 %	250	1	1.59E-04	5 %	250	1	7.94E-04
G	1 %	250	1	1.59E-04	5 %	250	1	7.93E-04
H	1 %	250	1	1.58E-04	5 %	21	1	4.92E-04
I	1 %	250	1	1.59E-04	5 %	250	1	7.93E-04
A	10 %	250	5	3.26E-02	30 %	250	4	2.43E-02
B	10 %	250	1	1.58E-03	30 %	250	1	4.74E-03
C	10 %	250	1	1.58E-03	30 %	250	1	4.75E-03
D	10 %	12	1	7.45E-04	30 %	6	1	3.03E-03
E	10 %	250	1	1.58E-03	30 %	250	1	4.75E-03
F	10 %	250	1	1.59E-03	30 %	250	1	4.76E-03
G	10 %	250	1	1.59E-03	30 %	250	1	4.75E-03
H	10 %	15	1	1.27E-03	30 %	250	1	4.75E-03
I	10 %	250	1	1.59E-03	30 %	250	1	4.76E-03

the subsequent distillation column, leading to thermodynamic complications that hinder its stability. These complications make the system more sensitive to perturbations in controllable variables, which is reflected in the high values of IAE.

Designs B, C, E, F, G, H and I stand out for having similar IAE values in all perturbations, revealing homogeneous dynamic behavior. This uniformity suggests that, despite variations in the individual configurations and features of each design, their response to disturbances remains consistently balanced.

From the perspective of control theory, a system that exhibits similar dynamic behavior under different perturbations indicates that its transfer function is robust and well-tuned.

This consistency in performance facilitates the implementation of more predictable control strategies, allowing the system's response to be predicted and managed efficiently and consistently. In addition, consistent performance can reduce complexity in adjusting controller parameters, as variations in system behavior under different disturbances are minimal.

4.3. *General results*

Designs B, D and E feature a Type 1 configuration at all frequencies, standing out for their superior dynamic performance and stability. Designs C, G, and I have a Type 2 configuration, which, while stable, may need more specific control. Despite having slightly higher values of TAC and EI99, the G and I designs are still good alternatives.

Designs A, F and H, with Type 3 structure, are unstable and require advanced control strategies to maintain stability. Design A shows the highest values of TCN and IAE due to the presence of water in the system and larger dimensions of the equipment.

Factors such as column diameter and location of thermal couplings significantly impact TCN. Smaller diameter columns facilitate more effective control and reduce operating costs. Additional thermal couplings, as in the F design, improve disturbance distribution and stability.

The number of stages in the columns also affects TCN. Designs with more stages have greater control complexity. Design D shows stability against disturbances in the feed but is less robust against manipulable variables. However, it has the lowest SI values.

In summary, systems with low TCN and SI values are more stable and less sensitive to disturbances, while systems with high values require more sophisticated and expensive control strategies to maintain stability. Balance in the design of control systems, both open-loop and closed-loop, is essential to guarantee stability, robustness, efficiency, quality, safety and sustainability of the process. By optimizing key indicators such as RGA, TCN, SI, IAE, TAC, and EI99, it ensures that the system responds predictably and stably to disturbances, minimizing operating and maintenance costs, improving product quality, and reducing environmental impact. A well-balanced design not only provides precise and efficient control, but also contributes to a safe and economically viable operation, which is essential for the long-term competitiveness and sustainability of industrial operations.

Taking this into account, the E design is the one that best balances

Table 4
LV control structure for design E.

ω		Distillation Flow of water, column 2	Bottom Flow of levulinic acid, column 3	Distillation Flow of formic acid, column 4	Bottom Flow of furfural, column 4
0	RR-column 2	1.00	0.00	0.00	0.00
	RHD-column 3	0.00	1.00	0.00	0.00
	RR – column 4	0.00	0.00	1.00	0.00
	RHD-column 4	0.00	0.00	0.00	1.00
0.001	RR-column 2	1.00	0.00	0.00	0.00
	RHD-column 3	0.00	1.00	0.00	0.00
	RR – column 4	0.00	0.00	1.00	0.00
	RHD-column 4	0.00	0.00	0.00	1.00
1E + 05	RR-column 2	1.00	0.00	0.00	0.00
	RHD-column 3	0.00	1.00	0.00	0.00
	RR – column 4	0.00	0.00	1.00	0.00
	RHD-column 4	0.00	0.00	0.00	1.00

these indicators since it presents a Type 1 RGA configuration (shown in Table 4), which indicates a stable and predictable control configuration. In addition, it has the best values of TAC and EI99, which suggests that it is the most economical option with the lowest environmental impact. The low values of TCN and SI in Design E indicate a lower sensitivity to disturbances, both in manipulable variables and in the feed, providing greater stability of the system. Likewise, the IAE values are consistent and low, ensuring a smooth and efficient response to disturbances.

5. Conclusions

In this work, the control properties of nine different schemes for the purification of levulinic acid are analyzed. More than 50 % of designs do not feature an LV array, so they may require advanced control techniques to maintain stability. Designs with larger diameter in the first distillation column have the highest TCN values. In addition, a higher number of thermal couplings increases the SI, especially if they are present in the first distillation column. In general, intensified schemes show better open-loop control properties than conventional schemes.

In the closed-loop control, all designs presented similar responses, except for designs A and D. Design A, a conventional scheme, showed the worst results due to the extraction of water in the second distillation column. Design D, with thermal coupling, presented the lowest values of EAI, associated with its low values of Kc.

Overall, design E, which includes a liquid–liquid extraction column, three distillation columns, and a thermal coupling between the last two columns, presented the best dynamic behavior with the lowest TCN values, a below-average SI, a stable LV control structure, and small IAE values. In addition, it is the most efficient in terms of cost and environmental impact, making this design the best alternative proposed.

The present work shows the need to study control properties at an early design stage, since intensified designs the control structure is not necessarily the best known. And there may be designs with economic and environmental savings, such as designs G and I, that require an advanced control strategy. Identifying and optimizing these aspects from the beginning not only ensures greater stability and operational efficiency, but also allows for the development of more sustainable and economically viable solutions. In this way, the implementation of more advanced and robust technologies in industrial environments is promoted, aligning with the principles of Industry 4.0 and green chemistry, which is essential for the long-term competitiveness and sustainability of industrial operations.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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