Research Article

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Control properties of hybrid distillation processes for the separation of biobutanol

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Abstract

BACKGROUND: Butanol produced from fermentation has attracted the interest of research groups because its physicochemical properties show several enhancements over bioethanol. Recent studies have proposed alternative methods to separate and purify biobutanol from a fermentation broth. These alternatives offer energy and economic savings; in addition, a reduction in environmental impact is observed. However few studies have analyzed the control properties of the process which involves separation of an acetone–butanol–ethanol (ABE) mixture.

RESULTS: A controllability analysis using the singular value decomposition technique and a closed-loop dynamic analysis was performed on several hybrid distillation processes including conventional, thermally coupled, thermodynamically equivalent and intensified designs. The results indicated that under the closed-loop control policy, an intensified design which is integrated for only two distillation columns instead of three distillation columns, showed good dynamic properties. In addition, thermally coupled sequence A showed better control properties under open-loop analysis.

CONCLUSIONS: Using both SVD analysis and closed-loop tests the dynamics properties were obtained for several hybrid processes to separate an effluent produced by fermentation. It was possible to control all schemes under both methodologies and it was clear that when the base case became more complex with thermal coupling, section movement or elimination of a column section improved the control properties.

Keywords: control properties; thermal couplings; ABE fermentation; biobutanol

INTRODUCTION

At the end of the Second World War in 1945, nearly 66% of butanol consumed was produced by fermentation through ABE (acetone–butanol–ethanol) fermentation. After this, butanol production was completely supplied by the petrochemical industry through the oxo process. However, the necessity for renewable energy sources, the volatility in crude oil prices, environmental pollution and greenhouse gas emissions have become major issues.

Over the last two decades, society and research groups have focused their efforts on exploring options that could either replace or be blended with petroleum fuels. Among several biofuels, biobutanol has shown properties such as energy density (27.8 MJ L^{-1}), a low vapor pressure at ambient temperature (5.6 hPa) and a higher flash point (35 °C).[1] Furthermore, engine modifications are unnecessary to completely replace fossil fuels with biobutanol.[1] Currently, biobutanol produced from fermentation broth is attracting the attention of research groups due to its potential for reducing the dependence on crude oil as a main energy source. Nevertheless, the main hurdle with biobutanol fermentation is the use of dilute sugar solutions, because of the toxicity/inhibition to the culture and the highly demanding energy process for separating and purifying the biobutanol produced from fermentation broth.[2] Under this scenario, a solution to these issues could lead to the use of engineering techniques in fermentation cultures. Furthermore, the recovery technique should show high selectivity and high energy savings.[3] To separate the ABE mixture, several operations have been proposed; some do not show promising results because the presence of two azeotropes renders the ABE mixture difficult to handle, i.e. some adsorbent materials have been tested with poor results in industrial applications.[4] Gas stripping is the usual technique applied to processes where low yields are present.[3] When the distillation process is considered, several hurdles must be overcome. Errico *et al.*[5] presented several

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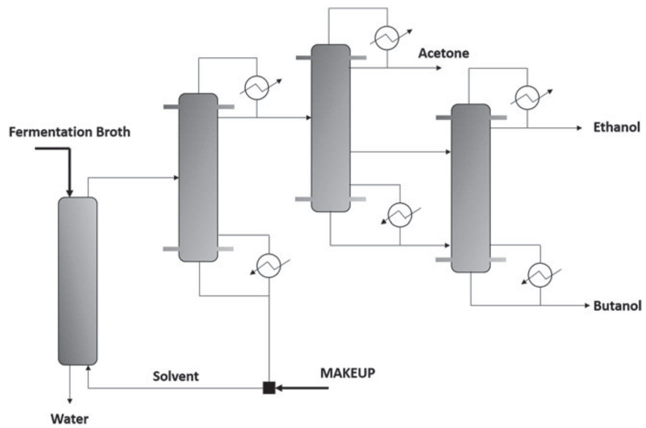

Figure 1. Hybrid L–L scheme taken as a reference configuration.

hybrid designs containing a liquid–liquid extractive column for separating both homogenous and heterogeneous azeotropes. In addition, they reported that the designs considering thermal coupling and intensified designs exhibit promising energy savings and reductions in environmental impact.

The aim of this work was to conduct an analysis of the theoretical control properties of 10 schemes (Figs 1–4), that exhibit promising economic and environmental results. These designs were proposed by Errico *et al.*[5] to separate a broth produced from fermentation through hybrid fermentation using n-hexyl acetate as extractant agent. We considered the schemes that exhibited lower energy consumption in comparison with conventional distillation schemes. The designs are hybrid processes because a liquid–liquid column is included with the distillation columns. The analysis was conducted through application of the singular value decomposition technique followed by a set of dynamic tests under closed-loop control considering set point changes and feed composition disturbance with proportional–integral (PI) controllers.

Despite ABE fermentation being a well-known process since Louis Pasteur reported microbial fermentation in 1861,[6] the dynamic scenario of the control properties in the separation of ABE mixtures has not been fully explored. Luyben[7] studied the control of an n-butanol/water mixture; however, only the interaction between these two components was considered, and a more complete mixture was not contemplated. Mariano *et al.*[8] presented a mathematical model to assess the dynamic behavior of a flash fermentation process for the production of biobutanol; nevertheless, a comprehensive plant control test was not performed. A controllability study of a more complete scenario is required.

Configuration analyzed and case study

Ten hybrid designs have been considered and evaluated under open-loop and closed-loop control strategies (Figs 1–4). The ten schemes are hybrid processes because a liquid–liquid extraction column is included, and all of them showed high energy savings in comparison with conventional schemes.[5] Note that the designs were developed addressing the design problem as a retrofit design. The first configuration considered is shown in Fig. 1. This design comprises a liquid–liquid extraction column where n-hexyl-acetate is used as an extractant agent to break both the homogenous and heterogeneous azeotropes; in addition, this configuration is considered the reference configuration for producing the other designs. Three conventional distillation

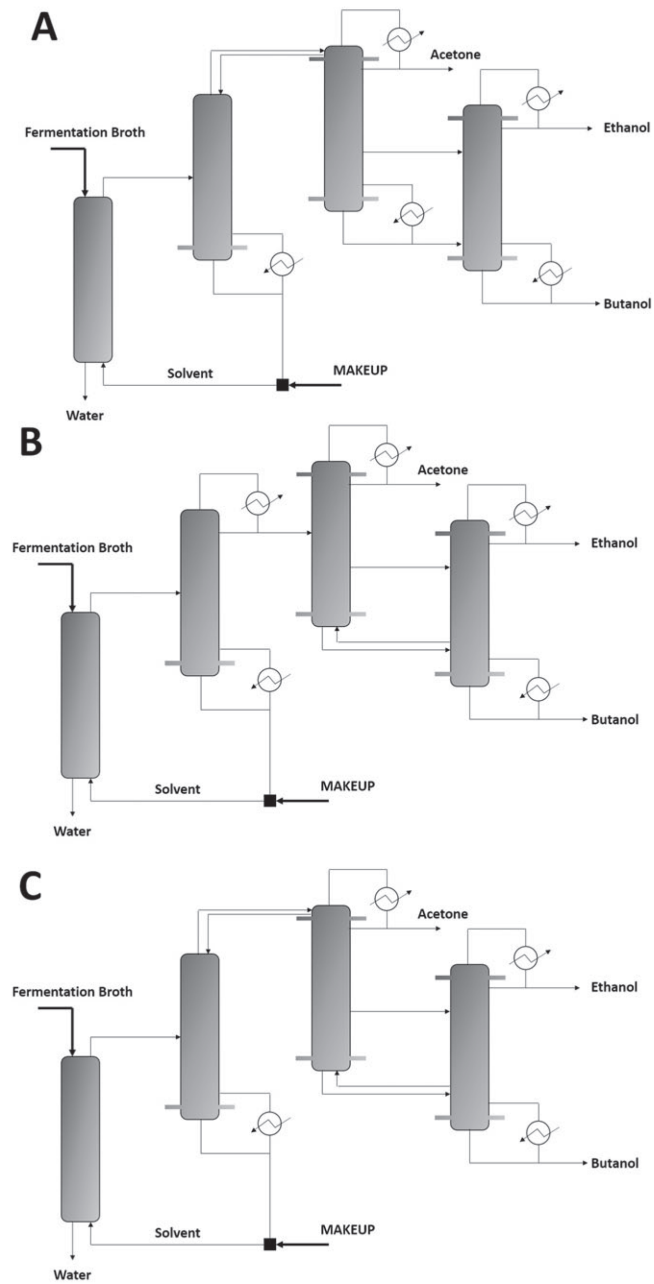

Figure 2. Thermally coupled alternatives.

columns performed purification of the ABE mixture and recovery of the extractant agent. Considering the design contained in Fig. 1 as a reference, thermal couplings were introduced corresponding to the condenser or reboiler associated with the non-product streams, producing the schemes depicted in Fig. 2. Because thermal couplings were introduced into the reference design and several sections supplied a common reflux ratio/boil-up ratio, it was possible to move those sections to effect the thermodynamically equivalent alternatives presented in Fig. 3. Figure 4 presents some intensified designs. The intensified designs were produced using the thermodynamically equivalent designs as a base followed by the elimination of single column sections.[9]

Before the dynamic tests, all of the designs were firstly simulated in Aspen Plus; the feed composition and physical parameters

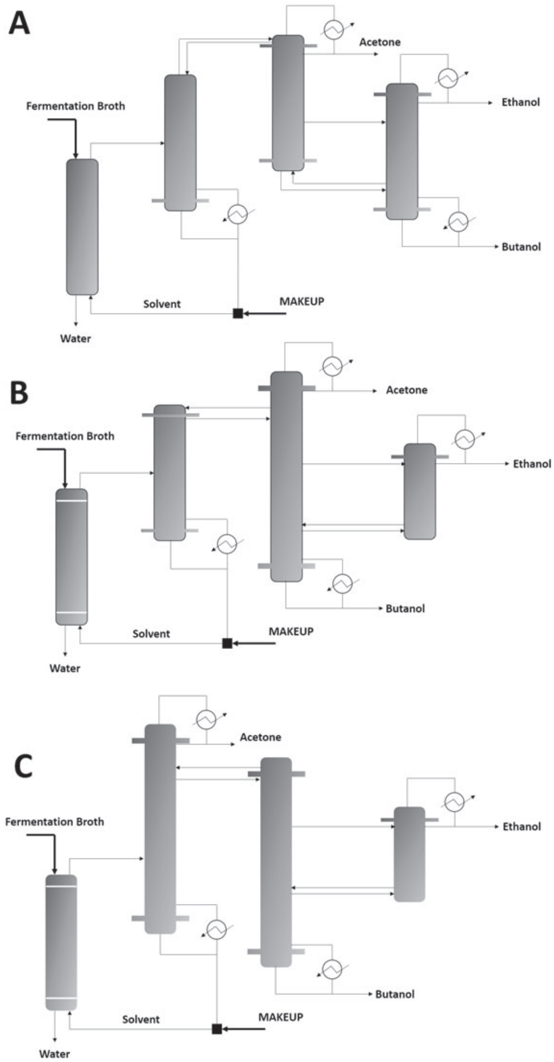

Figure 3. Thermodynamically equivalent configurations.

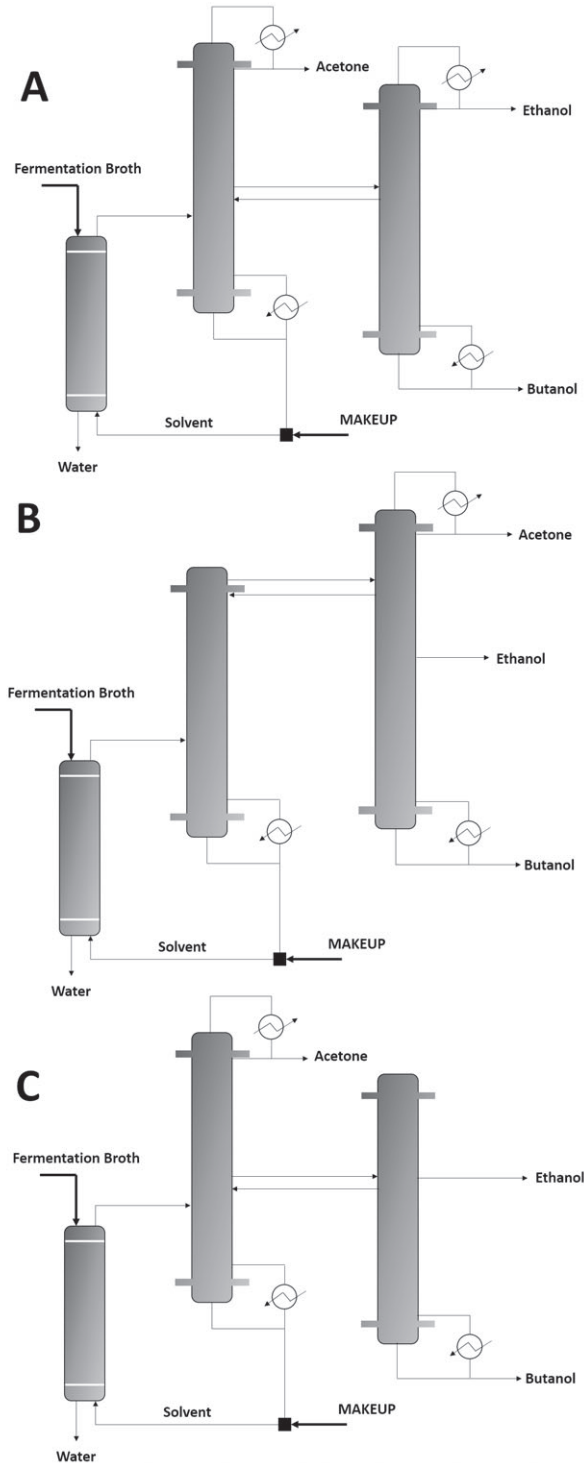

Figure 4. Intensified configurations.

are shown in Table 1 according to Wu *et al.*[10] The NRTL-HOC thermodynamical model was chosen to model the interaction among all components.[11] N-hexyl-acetate was chosen as an extractant in the liquid–liquid extraction column. The acetone purity was set to 0.996 (%wt), the butanol purity to 0.995 (%wt) and the ethanol purity to 0.95 (%wt). The capital cost and environmental impact were measured through the Total Annual Cost and the eco-indicator 99; obtained as results by Errico *et al.*[5] and presented in Table 2.

DYNAMIC ANALYSIS

To compare the control properties of the 10 designs, the control analysis was conducted in two parts. First, the singular value decomposition (SVD) technique was performed to obtain a comparative framework on the control properties of the schemes. Singular value decomposition (SVD) is a very useful tool in linear systems theory. It also plays an important role in analysis and design of control systems for real processes in industry.[12] SVD determines the rank and the condition of a matrix and is quite useful to chart geometrically the strengths and weaknesses of a set of equations.[12] The closed-loop control policy was performed under composition disturbance scenarios. This type of analysis is quite useful for investigating the theoretical properties and the dynamic behavior under feedback control such as those performed by several authors.[13,14] In addition, the control analysis revealed the best structures from a dynamic point of view, and which of those schemes show better dynamic behavior in separating and purifying a fermentation broth.

Table 1. Feed characterization[10]	
Temperature (K)	322
Vapor fraction	0
Flow rate (kg h^{-1})	45.3592
Composition (mol %)	
Biobutanol	0.1128
Acetone	0.0808
Ethanol	0.0043
Water	0.80198

Table 2. Total annual cost and eco-indicator 99 for the studied configurations[5]

Scheme	**TAC (\$ yr^{-1})**	Eco-Ind. (Points yr^{-1})
Reference case	128785	13016
Thermally coupled A	117503	12461
Thermally coupled B	118767	13350
Thermally coupled C	103083	11641
Thermodynamically equivalent C	104353	11814
Thermodynamically equivalent D	104215	11570
Thermodynamically equivalent E	104576	11893
Intensified C	119839	19684
Intensified D	101012	16680
Intensified E	99110	15594

Singular value decomposition

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

$$G = V\Sigma W^H \tag{1}$$

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

$$\gamma^* = \frac{\sigma^*}{\sigma_*} \tag{2}$$

To obtain the open-loop dynamic responses, a step change around the nominal operation point was implemented. The magnitude of the step change was 0.5% of the manipulated variable. Each manipulated variable was chosen according to each product stream, i.e. when a component was purified in the top of a distillation column, the manipulated variable was the reflux ratio; however, if the component purified remained in the distillation column as a bottom product, the manipulated variable was the reboiler heat duty, and so on.

The engaging aspect of the SVD study regarding the process control is that when applied to a matrix which describes the steady-state characteristics of a multivariable process, the singular values have a strong physical interpretation. In practice, the minimum singular value measures the invertibility of the evaluated

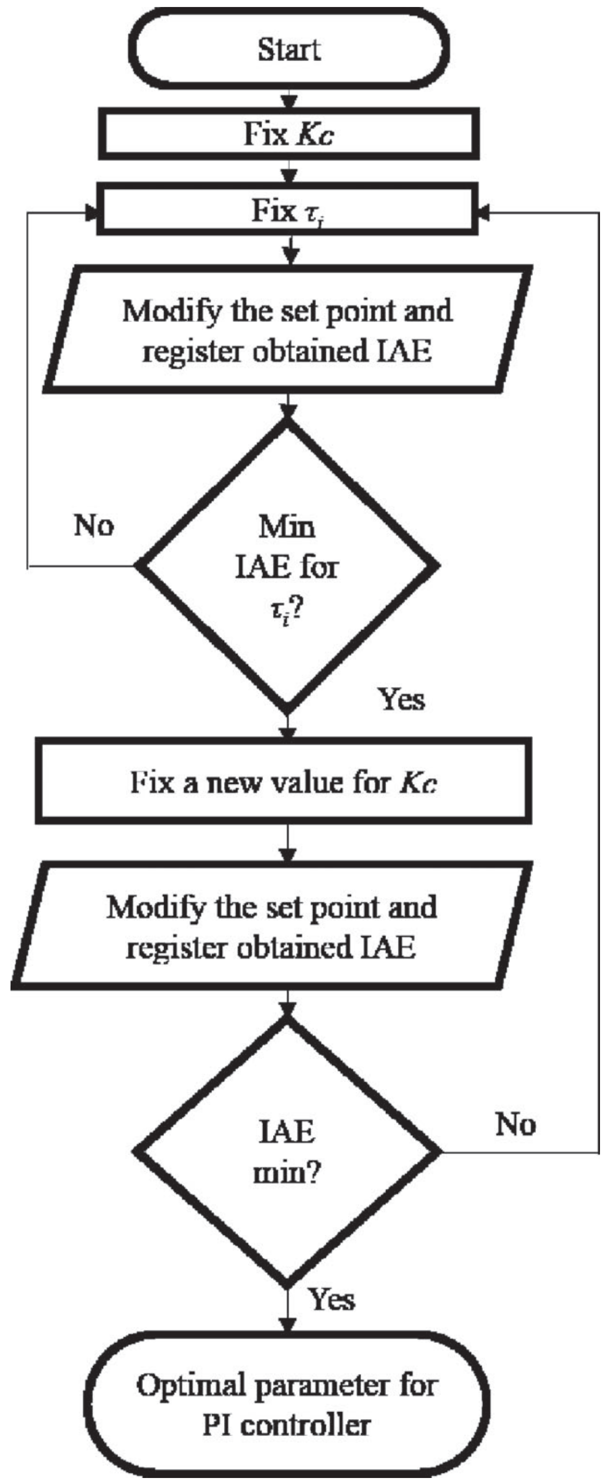

Figure 5. Flowsheet for tuning PI controllers.

scheme, and it also measures the potential problems of the system under feedback control. Very small singular values could indicate that in spite of a good condition number, the system is simply not sensitive enough to control. On the other hand large singular values indicate a practical control problem.[12] Furthermore, the condition number could be interpreted as the sensitivity of the system under uncertainties and modeling errors. However, the condition number only provides a qualitative assessment of

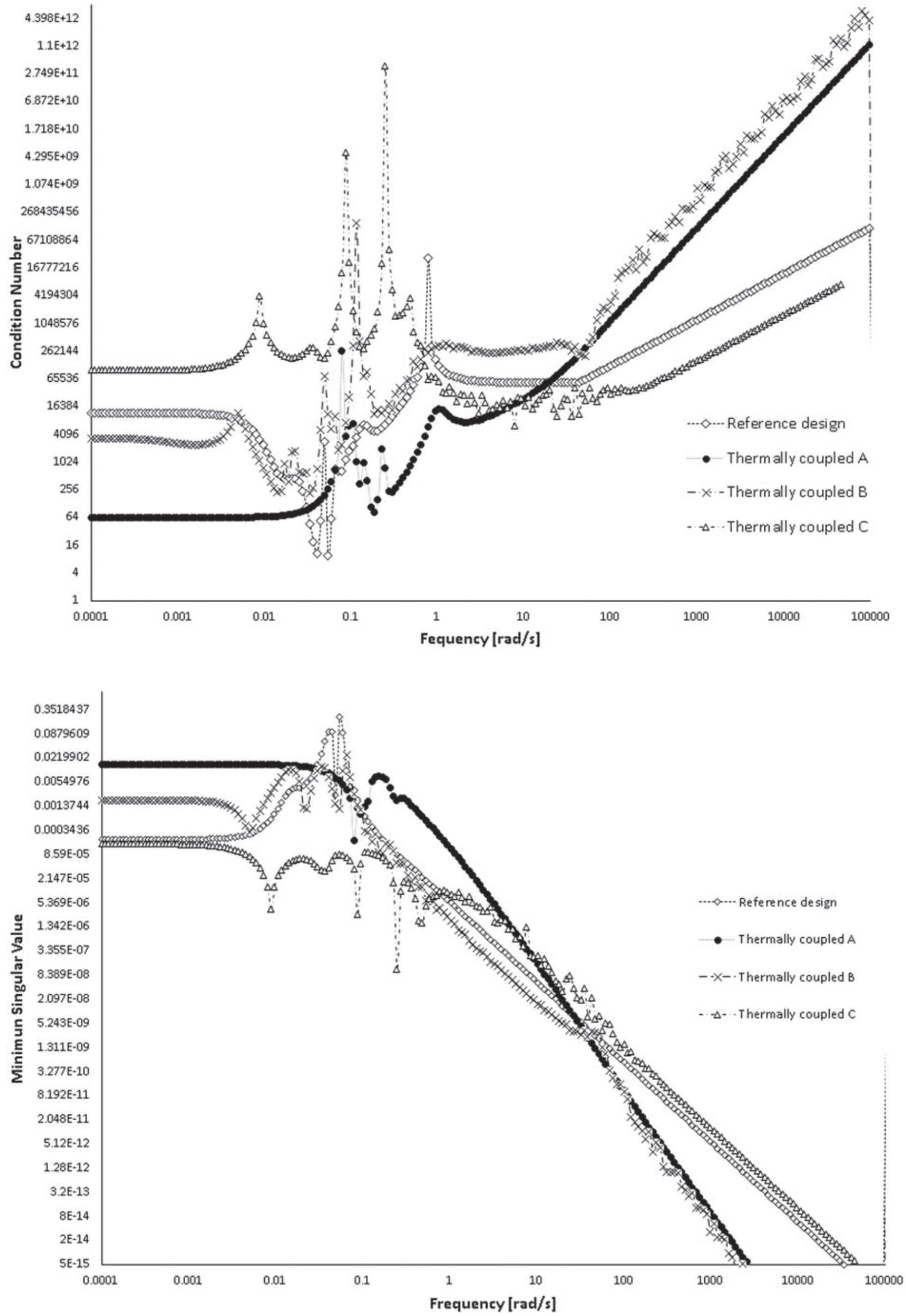

Figure 6. Minimum singular value and condition number for the base case and the thermally coupled designs.

the theoretical control properties of the schemes under analysis. In general, schemes presenting lower values of the condition number were expected to show a better dynamic performance under feedback control.[15] In physical terms the condition number represents the ratio of the maximum and minimum open-loop, decoupling gains of the system. A large condition number indicates that the relative sensitivity of the system in one multivariable direction is very weak.[12] SVD analysis does not solve all the control problems which may be found in industrial multivariable control, however, it is relatively easy to understand and identify basic control difficulties.[12] The SVD technique has been used by several authors to study the dynamic properties of complex designs.[13,14]

Closed-loop analysis

The second control test was performed as follow: (i) a step change was induced in the setpoint for each product composition under single-input single-output feedback control at each output flow rate; and (ii) a 0.5% change in the composition of one component (adjusting the proportion in the composition of other components) was implemented as feed disturbance in the reference configuration and the most promising design regarding control properties. For the closed-loop control policy, the analysis was based on proportional–integral (PI) composition controllers. The reason for using composition controllers is simply that a ‘back-off’ from the purity specifications makes composition control simpler.[15] This type of controller was chosen because of its

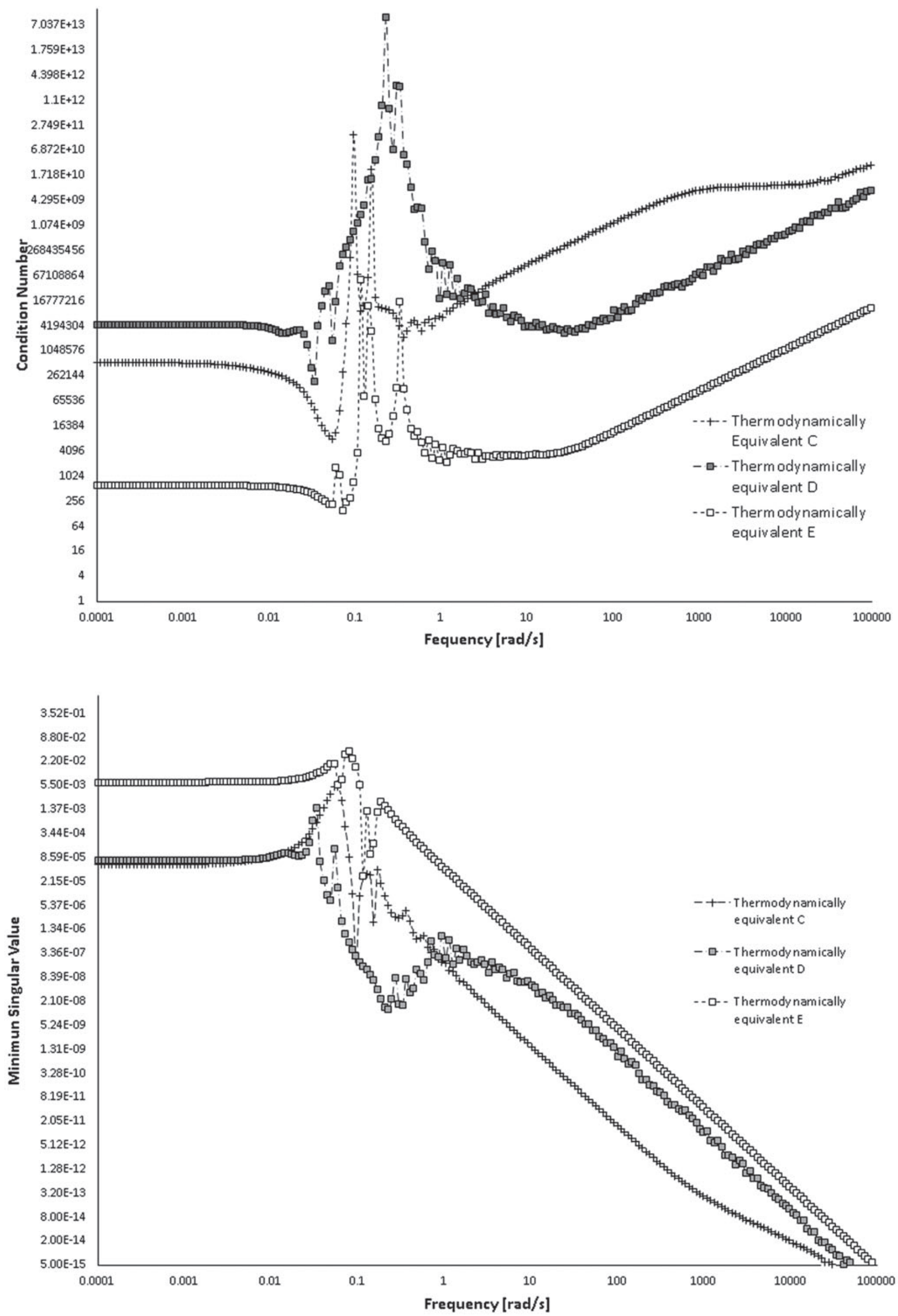

Figure 7. Minimum singular value and condition number for the thermodynamically equivalent designs.

wide use in industrial practice. When a controller is used, a main issue is tuning the controller. 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 (τ_i) were tuned for each scheme studied here; in addition, we compared the dynamic performance by using the integral of the absolute error (IAE) criterion.[16,17] A key part for this dynamic analysis of each loop was the selection of control outputs and their respective manipulated variables. In this manner, to control the distillate and bottom output compositions, structures based on energy balance considerations were used; this structure yields the so-called LV control structure, which uses the reflux flow rate L and the vapor boil up rate V as the manipulated variables.[18] In other words, we chose the corresponding reflux flow rate for the top of the column, the reboiler heat duty at the bottom of the column and the side stream flow rate for the side streams as the manipulated variables. In general, for feedback control a model is necessary that describes the effect of the inputs (flows) on the

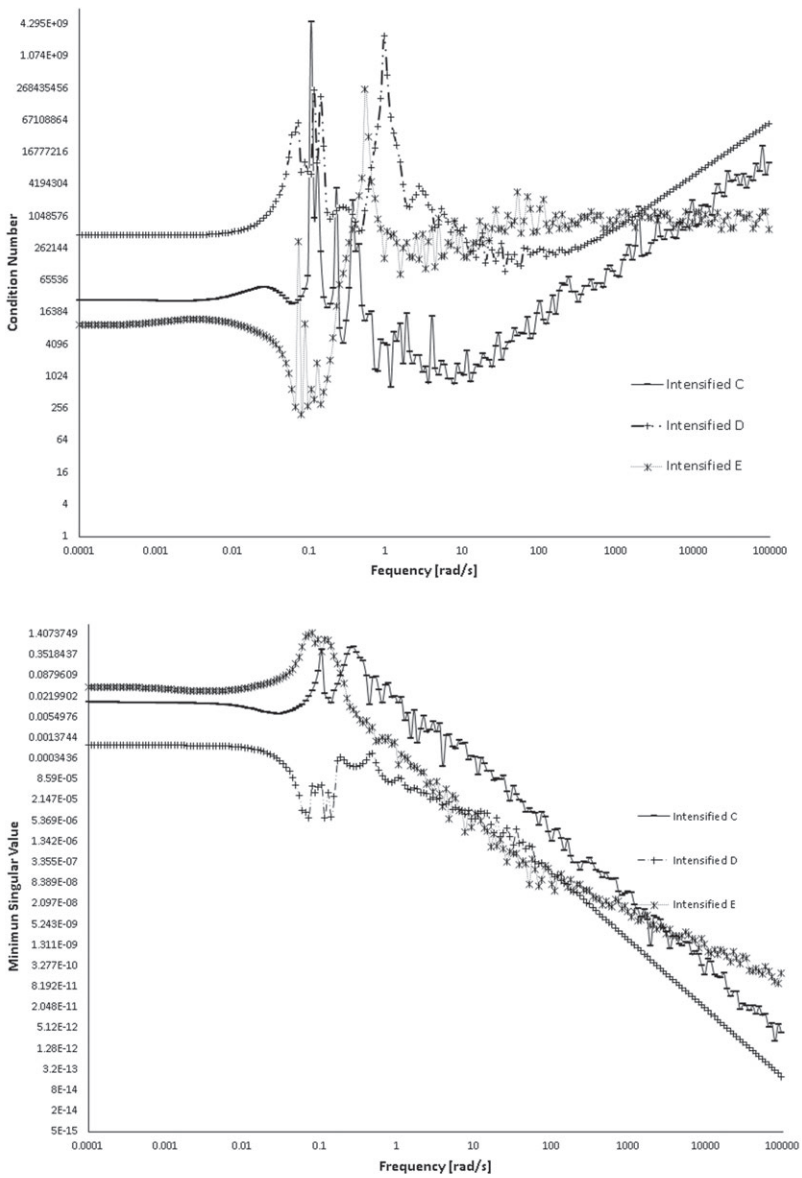

Figure 8. Minimum singular value and condition number for the intensified designs.

Table 3. Design parameters and comparison indexes for the intensified design E

	Extractor	C1	C2
Number of theoretical stages	5	58	20
Reflux ratio	---	27.182	---
Feed stage	1	45	---
Solvent feed stage	5	---	---
Side stream stage	---	---	12
Diameter (m)	0.335	0.323	0.324
Operative pressure (kPa)	1013.53	1013.53	1013.53
Distillate flow rate (kg h^{-1})	---	7.711	---
Thermal coupling flow rate (kg h^{-1})	---	118.621	---
Side stream flow rate (kg h^{-1})	---	---	0.336
Solvent flow rate (kg h^{-1})	708.289	---	---
Solvent makeup (kg h^{-1})	0.684	---	---
Condenser duty (kW)	---	31.094	0.000
Reboiler duty (kW)	---	65.642	24.517
TAC (\$ y^{-1})	99110		
Eco-Indicator 99 (points y^{-1})	15594		

outputs (product composition). This does not imply that the LV control structure is the preferred selection for control tests, the choice is made because L and V have a direct influence on composition and their effect is consequently only weakly dependent on the tuning of the level loops. This also makes it natural to consider the column model in terms of L and V as manipulated inputs.[19] This type of control loop has been applied with satisfactory results in industry and also to study thermally coupled schemes.[20,21]

In brief, to tune each controller, an initial value of proportional gain was set, and a range of integral reset times was tested with this fixed value until a local optimum in the IAE value was obtained. This methodology was repeated with other proportional gain values until a global minimum in the IAE value was detected (see Fig. 5). Note that this procedure was conducted considering one control loop at a time until all control loops were considered. For the dynamic analysis, individual set point changes of −0.5% for product composition were implemented in the product streams of acetone and butanol.

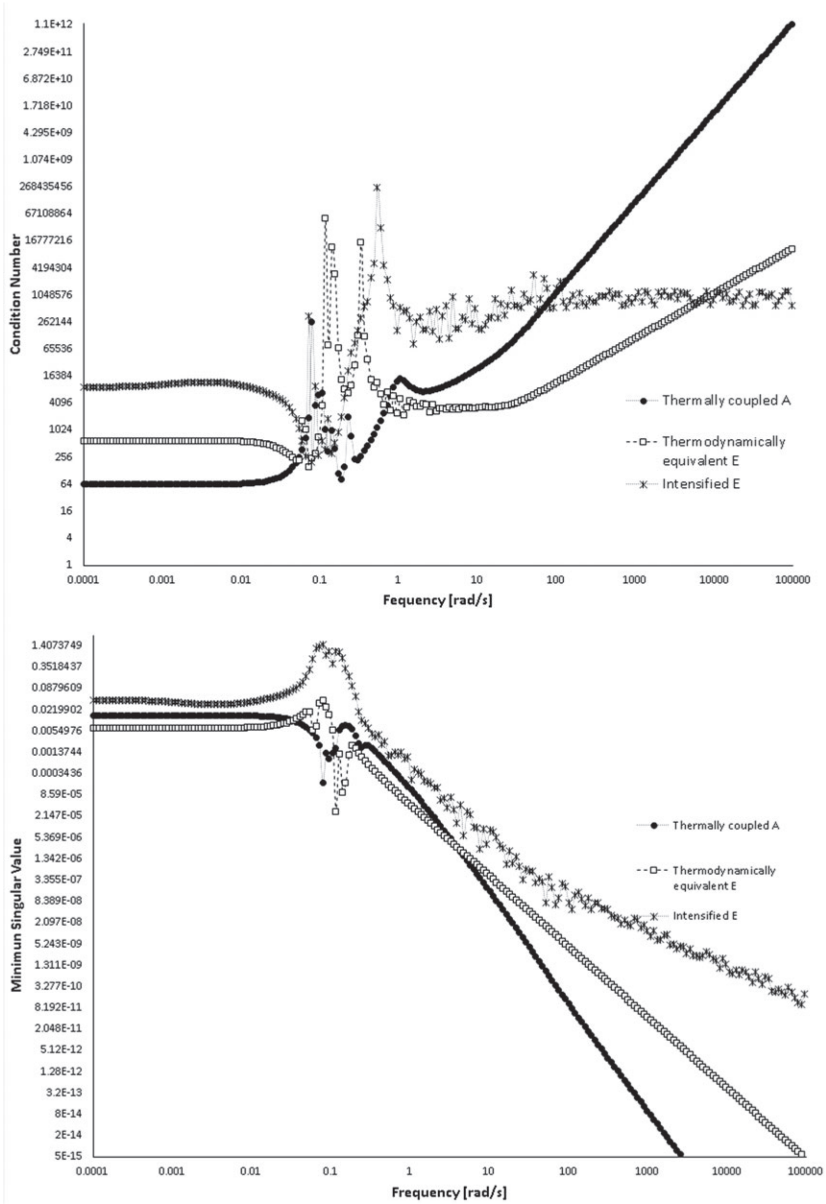

Figure 9. Minimum singular value and condition number for the best design of each category.

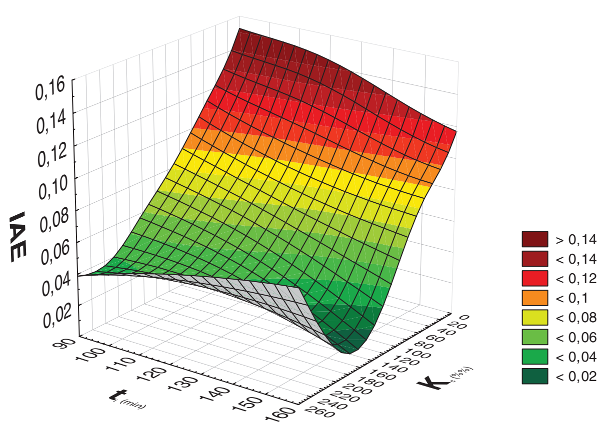

Figure 10. Surface obtained through tuning process of K_c and t_i parameters.

RESULTS

Open-loop analysis

First, the theoretical control properties obtained through the SVD technique were analyzed. As mentioned previously, the main parameters in the open-loop analysis were the condition number and the minimum singular value. Figure 6 shows the $\gamma*$ and σ_* for the reference design and the thermally coupled designs. Considering the designs which exhibited the lowest values in the condition number and the highest minimum singular values as the best, it is clear that thermally coupled sequence A is better conditioned to the effect of disturbances than the reference case and the other thermally coupled designs. However, this good dynamic behavior of thermally coupled sequence A was only observed at low frequencies. Because this behavior was observed in the open-loop control policy, it is expected that under a feedback control this design would show better control

Table 4. K_c, τ_i and IAE values for the tuning process for all study cases

	Acetone			Butanol		
Set point change	k_c **(%%)**	τ_i **(min)**	**IAE**	k_c **(%%)**	τ_i **(min)**	**IAE**
Reference case	250	150	0.13939675	250	150	0.04471066
Thermally coupled A	220	150	0.01573976	250	150	0,02187934
Thermally coupled B	20	150	0,0521022	140	150	0.0857721
Thermally coupled C	154	150	0.09061534	250	150	0,04583352
Thermodynamically equivalent C	220	150	0.02913265	250	150	0.03255766
Thermodynamically equivalent D	60	150	0.02531811	250	20	0.00166647
Thermodynamically equivalent E	120	150	0.06801718	250	100	0.00911842
Intensified C	250	30	0.00254760	250	140	0.00315114
Intensified D	200	80	0.0070438	140	20	0.00165158
Intensified E	230	150	0.00499379	210	150	0.03124394
Composition feed disturbance						
Reference case	235	150	0.00521375	205	150	0.00029494
Intensified E	210	140	0.00158174	225	150	0.00024387

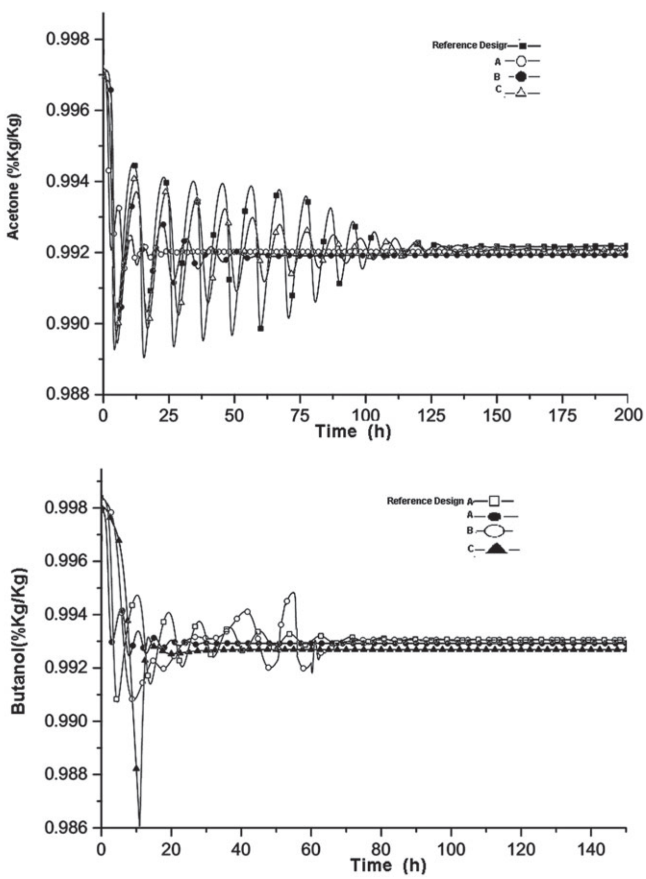

Figure 11. Closed-loop dynamic response for the reference design and the thermally coupled designs for the acetone and butanol streams.

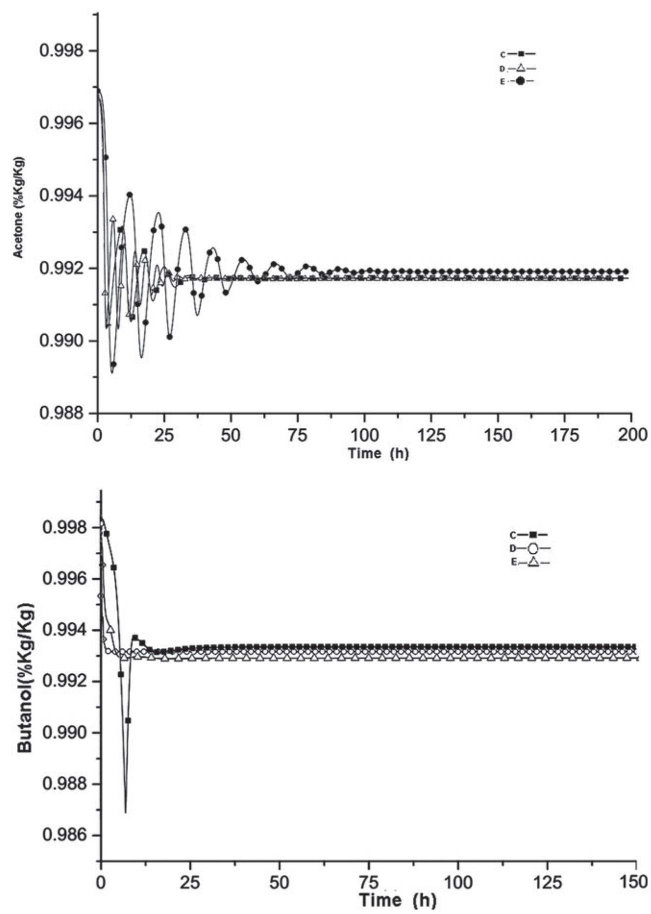

Figure 12. Closed-loop dynamic response for the thermodynamically equivalent designs for the acetone and butanol streams.

properties among the thermally coupled designs. Furthermore, considering the TAC and eco indicator 99 values as shown in Table 2, thermally coupled sequence A is not actually the cheapest design. In other words, the thermally coupled sequence A did not show sufficient energy savings to compete with thermally coupled sequence B and thermally coupled sequence C. Concerning the thermodynamically equivalent designs, Fig. 7 shows the γ^* and σ_* values in all frequency domains. It is clear that at all ranges of frequency, thermodynamically equivalent sequence E showed the best dynamic behavior. However, in this case, a relationship among TAC and dynamic behavior was not as evident as in the case of the thermally coupled sequences because the differences in TAC and eco indicator 99 among them was not so large. The worst of those designs, showing poor dynamic behavior

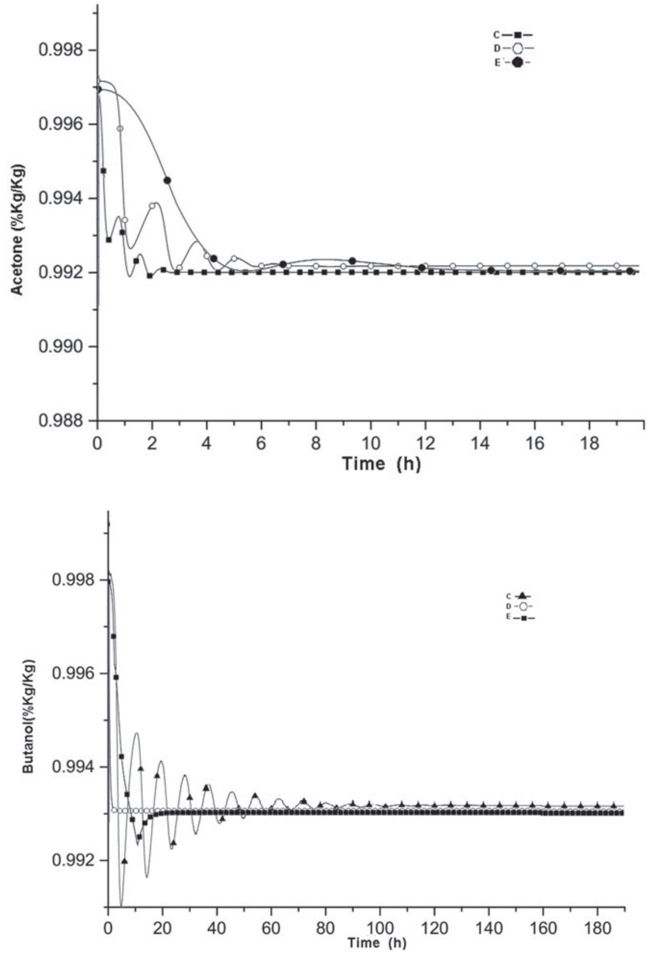

Figure 13. Closed-loop dynamic response for the intensified designs for the acetone and butanol streams.

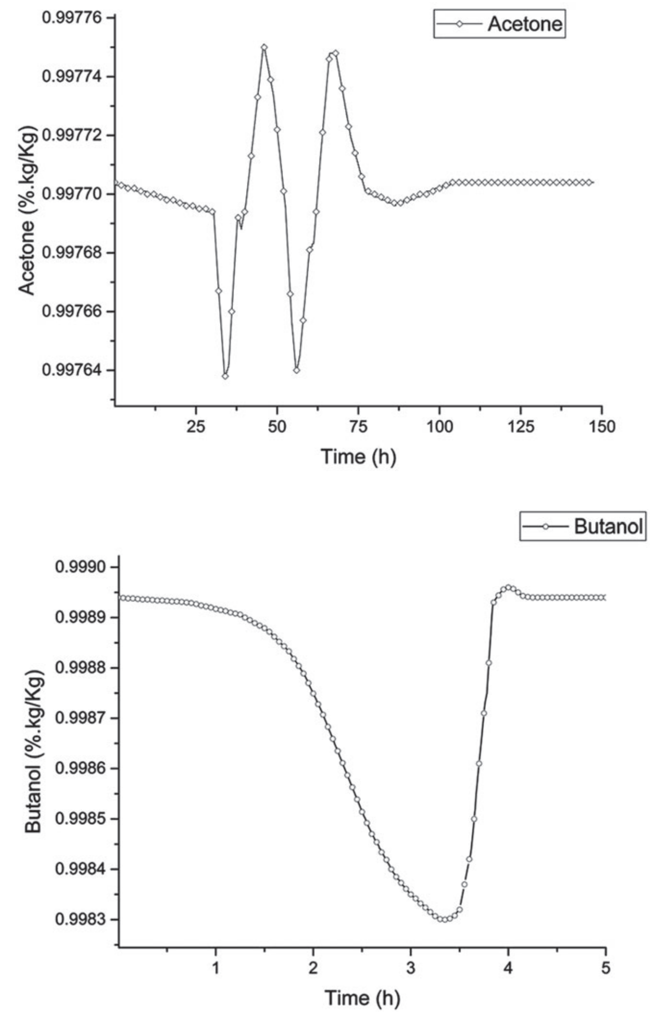

Figure 14. Closed-loop dynamic response under a feed composition disturbance for the acetone and butanol outputs in the intensified design E.

at all ranges of frequency, was the thermodynamically equivalent sequence D.

Figure 8 shows γ^* and σ_* of the intensified designs. Generally, the intensified design E was best-conditioned to disturbances in all frequency domains; however, examining the middle part of Fig. 8, in a frequency range between 0.2 and 21 the intensified design C showed the best dynamic behavior among all three intensified designs. In this manner, when only low and high frequency were considered, the intensified E sequence showed better dynamic behavior. All design parameters of the intensified design E are shown in Table 3.

Finally, in Fig. 9, the best design of each category (thermally coupled, thermodynamically equivalent and intensified designs) are compared. Regarding the minimum singular value, the intensified design E showed the largest value. This value is an indication of the sensitivity of the associated sensor to its manipulated variable; in other words, good dynamic behavior is expected under a closed-loop control policy. On the other hand, regarding the condition number, the thermally coupled designs showed the lowest value, representing a naturally and easily controlled system. As a preliminary conclusion of the open-loop analysis, using the reference design as a base, an improvement in control properties was observed when thermal coupling was introduced. In addition, when some column sections of the thermally coupled designs were moved, from a retrofit point of view, an improvement in control properties measured through a minimum singular value was produced. Furthermore, when some column sections were eliminated from the thermally equivalent designs, the control properties were once again improved; thus, reduction in the number of sections provided the expected operational advantages with respect to the reference case.

Closed-loop analysis

Prediction of the transient response of a process is highly important because the effective control of the process must be known. As mentioned before, closed-loop simulations were performed introducing a step change in the set point for the product composition of acetone and butanol under a single-input/single-output feedback control. All simulations were performed in Aspen Dynamics, and PI controllers were considered. The parameters of the controllers were tuned to minimize the integral of absolute error (IAE) as criterion.[16,17] Under this tuning methodology it is clear that the minimum IAE value is not guaranteed as could be claimed under a rigorous optimization strategy, however this parametric methodology has produced an IAE surface which

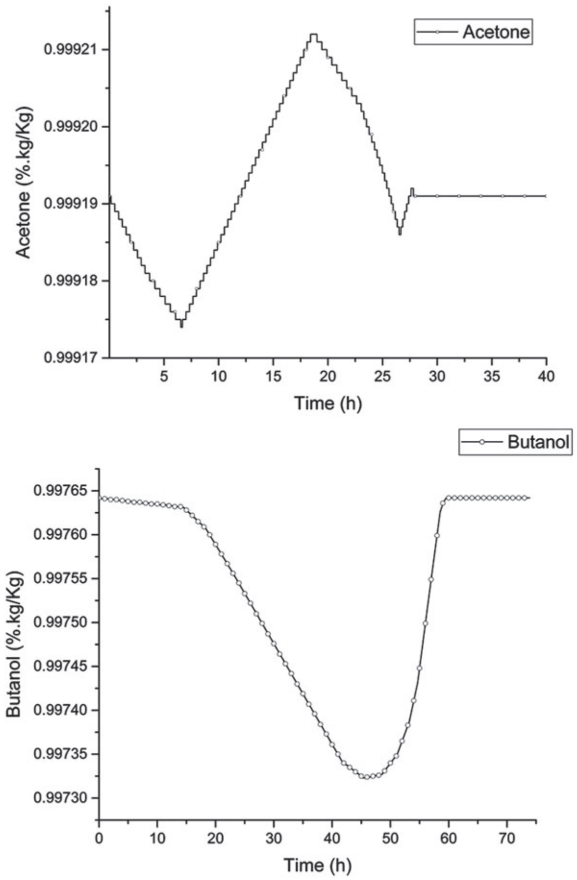

Figure 15. Closed-loop dynamic response under a feed composition disturbance for the acetone and butanol outputs in the reference design.

shows that the IAE value obtained is totally located in the zone where the minimum IAE values are situated (see Fig. 10).

The results from the individual servo test applied to the reference case and the thermally coupled designs are shown in Fig. 11 only for components of industrial applications for this case study: butanol and acetone (high compositions in the fermentation broth). It can be observed that, considering both components, thermally coupled sequence A exhibited the best dynamic behavior because the settling time was the lowest in comparison with the base case and all the thermally coupled designs; in addition, the IAE value in Table 4 was the lowest. These results are consistent with those obtained through the SVD analysis, where the thermally coupled sequence A also exhibited the best control properties and good behavior is expected under feedback control. On the other hand, thermally coupled sequence C and thermally coupled sequence B exhibited longer settling times. In particular, when the acetone loop was evaluated, the thermally coupled sequence C design was nearly as poor as the reference design, and when the butanol loop was evaluated, thermally coupled sequence B showed a behavior just slightly better than the reference case. In general, the inclusion of some thermal coupling improves the dynamic behavior tested under a closed-loop control policy.

Regarding the thermodynamically equivalent designs, Fig. 12 shows the behavior of those designs through time after the step change. It is easy to note that considering both loops, thermodynamically equivalent sequence D showed the best dynamic behavior, which was quantitatively confirmed through the IAE values. Considering the acetone loop, thermodynamically equivalent sequence D was nearly followed by thermodynamically equivalent sequence E; however, considering the butanol loop, the thermodynamically equivalent sequence D was followed very closely by thermodynamically equivalent sequence C. However, as previously mentioned, the thermodynamically equivalent sequence D was the best in both loops.

In Fig. 13 when the intensified designs were evaluated, the intensified design E showed the best dynamic behavior in controlling acetone; however, when the butanol loop was closed, the intensified design D design exhibited the best dynamic behavior. This behavior was corroborated on examination of the IAE values. Defining the best among the intensified designs could be difficult because there was no design that showed the best behavior in both closed loops. However, because butanol was our compound of interest, it is possible that the intensified design D was actually the best option. From the IAE values in Table 4 through all designs evaluated, it is clear when the reference case become more complex because of thermal coupling, column section movement or the intensification process, the IAE value decreased. Moreover, as predicted by the minimum singular value, when thermal coupling is included and/or some column sections are eliminated, better behavior is expected under feedback control, as demonstrated in this study.

In a further study, a change in feed composition was applied trying to keep the same composition in the output product. This study was applied to the best design under a set point change in the closed-loop control policy, the intensified design E and the reference design. This test gives a similar situation to the set point change. The intensified design showed better dynamic behavior in comparison with the reference case (see Figs 14 and 15 and Table 4).

CONCLUSIONS

Several hybrid designs were evaluated through an SVD analysis and a closed-loop control policy. When the SVD analysis was conducted, the thermally coupled sequence A design was selected as the best of the thermally coupled designs. The thermodynamically equivalent sequence E design showed the best dynamic behavior in the evaluation of thermodynamically equivalent designs. Finally, the intensified design E showed the best dynamic behavior among all intensified designs. Considering all designs, the intensified design E exhibited a better minimum singular value, with good behavior expected under feedback control. The closed-loop test was in accordance with the open-loop test; when the thermally coupled sequences were analyzed in comparison with the SVD analysis, thermally coupled design A exhibited the lowest IAE value. Among the thermodynamic equivalents, design D showed the lowest IAE values. However, considering all designs, the intensified designs showed the lowest IAE values, indicating better control properties under feedback control. In this study, since all the schemes considered in this work were designed from a retrofit point of view, it is difficult to relate the dynamic properties with some design variable because all process routes involve the same diameter value and all process routes but the intensified processes consider the same number of theoretical stages. However, a variable that impacts directly on dynamic properties and is totally measurable is the thermal coupling flow rate. In this scenarios the intensified designs C, D and E showed a thermal flow coupling rate of $FL = 76.50\ \mathrm{kg\ h^{-1}}$ and $FV = 62.47\ \mathrm{kg\ h^{-1}}$; $FL = 18.38\ \mathrm{kg\ h^{-1}}$ and $FV = 40.13\ \mathrm{kg\ h^{-1}}$; $FL = 118.62\ \mathrm{Kg\ h^{-1}}$ and

FL = 118.62 Kg h^{-1}, respectively.[5] In comparison, the best thermally equivalent and thermally coupled designs had thermal coupling flow rates of FL = 9.139 kg h^{-1} and FV = 15.050 kg h^{-1}, respectively; these designs, in general terms, showed worse dynamic properties than the intensified designs. Further, the reference design which does not have thermal coupling, exhibited the worst dynamic behavior of all the designs.

This relation among thermal coupling flow rate is not new. Indeed a similar behavior has been reported by Segovia-Hernandez *et al.*[22] which describes in detail the decrease of numerical control indexes such as condition number and minimum singular value when the thermal coupling flow rates increases.

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