

Computer-Aided Conceptual Design of Batch Distillation Systems

José Espinosa,* Enrique Salomone, and Oscar Iribarren

INGAR–CONICET, Avellaneda 3657, S3002 GJC, Santa Fe, Argentina

During the past decade, several authors worked in the development of methods to cope with different steps of the design and synthesis of batch distillation systems. Among these methods, the conceptual design is based both on the thermodynamic analysis of residue curve maps and on the behavior of composition profiles in batch columns operating at limiting operating conditions, i.e., infinite number of stages and total reflux. In this work, we apply the conceptual design approach to design the recovery of 2-propanol from a mixture containing water by using cyclohexane as entrainer. All the computations were performed through an integration of these methods in a software tool for conceptual design.

Introduction and Short Review on Conceptual Models

Fractionation in batch distillation columns is one of the most common technologies used in pharmaceutical and specialty chemical industries due to its operational flexibility. As organic solvents are widely used for carrying out the process reactions and crystallizing the product, batch distillation is mainly used for solvent recovery in order to minimize raw material costs while complying with environmental regulations.

Since typical solvents are likely to form azeotropic mixtures, the design of recovery strategies based in distillation needs to be supported by conceptual design tools capable to reproduce the behavior of nonideal and azeotropic mixtures.

As was pointed out by Bernot et al.,¹ a conceptual design and synthesis procedure is very valuable for the development of batch processes because it provides a means for evaluating design alternatives quickly. Furthermore, conceptual design permits gaining insights into the design problem, to pose it correctly.

The first step of the conceptual design consists of determining the maximum feasible separation for a given initial mixture since the attainable cuts depend on the feed composition due to the existence of different distillation regions.² As a result of this step, the maximum recovery of each species as pure component or as a part of one or more azeotropes can be estimated for a column operating at infinite separation power (i.e., infinite number of stages and “near” total reflux operation).

By using the information of this first step, synthesis strategies can be applied to break the azeotropes and analyze different separation alternatives, including the selection of entrainers and the use of other equipment configurations (e.g., middle vessel distillation columns or batch strippers). As this step may involve the generation of new mixtures, feasibility and synthesis steps are very coupled.³

After a feasible sequencing alternative is chosen, the next step consists of evaluating how much separation power (number of stages and reflux ratio) is needed to

perform the separation, which in turn depends on the sequencing. In this phase of the conceptual design, the issue is the tradeoff between separation costs associated with the number of stages and the amount of material that must be evaporated to perform the separation, which in turn depends on the reflux ratios used and the amounts and compositions of the intermediate recycle cuts generated. In addition, a preliminary economic evaluation of each of the process alternatives can be done.

Several methods are needed to deal with the different steps of the conceptual design. Numerical methods to calculate azeotropes from thermodynamic models, such as homotopy continuation methods,^{4,5} global optimization techniques,^{6,7} and, more recently, interval Newton with generalized bisection^{8,9} can be used to reproduce/find all the azeotropes of a multicomponent mixture. We use an adaptation of the method presented by Maier et al.,⁸ which combines the sequential formulation processed in a *given order* with the Zharov–Serafimov topological index theory as explained in Salomone and Espinosa.⁹

Once azeotropic compositions and temperatures are calculated, pure node and azeotrope stabilities need to be known to determine distillation regions and boundaries. For this issue, several algorithms are at hand.^{10–13} The methods in Poellmann et al.¹⁰ and in Rooks et al.¹² calculate distillation regions and separating spaces. Safrit and Westerberg¹¹ and Ahmad et al.¹³ established algorithms to generate batch distillation regions. Other works in this field have been developed by researchers of the former Soviet Union.^{14–19} A survey of these contributions is done in Safrit and Westerberg.¹¹ As proposed by Poellmann et al.¹⁰ and Rooks et al.,¹² we calculate both the adjacency matrix and the reachability matrix to determine distillation regions with their corresponding natural and distillation boundaries. Using piecewise linear approximations for each boundary, a mathematical representation is automatically generated.²⁰

With this information, feasible cuts at infinite separation power can be calculated for a given feed composition following the ideas introduced in Bernot et al.^{1,2} Methods based in determining batch distillation regions were developed by Safrit and Westerberg²¹ and Ahmad et al.¹³

* To whom correspondence should be addressed. Phone: +(54 342) 453 4451. Fax: +(54 342) 455 3439. E-mail: destila@ceride.gov.ar.

The algorithm that we implemented²⁰ follows the guidelines introduced in Bernot et al.^{1,2}

To cope with the design step, dynamic representations of limiting operation modes for batch columns are necessary: namely, columns with infinite stages operating at a given reflux ratio and columns with a given number of stages operating at total reflux. For the infinite stage limiting condition and ideal systems, a method developed by Salomone et al.²² can be used. For azeotropic multicomponent mixtures, methods based on the estimation of the instantaneous minimum reflux²³ and the instantaneous top composition²⁴ without resorting to multistage calculations were developed. By using linear approximations of the composition profile in the neighborhood of the pinch points, the model developed by Espinosa and Salomone²⁴ and Salomone and Espinosa²⁵ identifies the geometry of the profiles and the controlling pinch corresponding to the present still composition and reflux ratio. To cope with different operation policies (such as operation at constant distillate composition followed by cuts at constant reflux ratio) we implemented both methods.

The total reflux limiting condition, on the other hand, can be adequately described by solving a sequence of equilibria and mass balance computations for a given still composition in order to predict the instantaneous distillate composition, assuming a differential rate of product withdrawal from the column at any time. The method that we use to get the minimum number of stages is an iterative procedure where an initial guess of N is refined until the specified recovery fractions of two components are reached in the distillate.²²

As the different methods developed for each one of the steps of the conceptual design are used several times (e.g., information about unstable boundaries is used for determining maximum feasible separation, simulation runs, and minimum reflux calculations), all the methods were integrated in a software tool for conceptual design (Conceptual Batch Distillation Toolkit²⁶).

The conceptual design of a plant for recovering 2-propanol is presented next, to illustrate the methodology proposed here.

Preliminary Analysis

The mixture 2-propanol (IPA)/water (W) presents a homogeneous minimum-boiling azeotrope, which must be broken in order to recover water-free alcohol. A way to break the azeotrope is by using a dewatering entrainer like cyclohexane (CyH), which also forms an azeotrope with IPA and water but separates into two immiscible phases. Figure 1 shows the results of the azeotrope calculation together with pure component and azeotrope stabilities as calculated by using the Wilson model. The results obtained are in agreement with experimental data from Gmehling et al.²⁷ Liquid-phase separation data for the ternary heteroazeotrope were taken from Smallwood²⁸ and Gmehling et al.²⁷

Figure 1 presents the different distillation regions and "unstable" boundaries as calculated from pure component and azeotrope stabilities. The system has one unstable node (heteroazeotrope CyH-IPA-W, light) and three stable nodes (pure IPA, pure water, and pure CyH, heavies). Therefore, three distillation regions completely describe the behavior of any mixture of the ternary system. Feed compositions to a batch rectifier lying in the region formed by the ternary azeotrope, IPA-water azeotrope, CyH-IPA azeotrope, and IPA

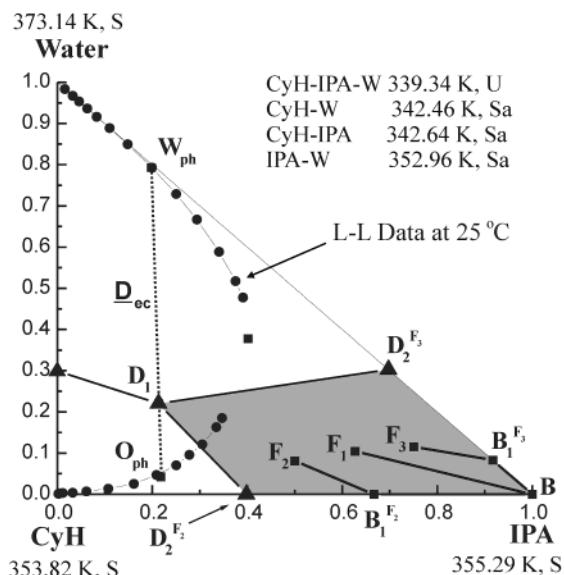


Figure 1. Distillation regions and feasible cuts at infinite separation power for three different initial feeds. (The reflux established during the first cut is formed by a mixture of heteroazeotropic composition.) U, unstable node; Sa, saddle node; S, stable node.

will produce the alcohol as residue in the reboiler at the end of a batch rectification because the alcohol is the stable node in this region. As the feed to the process is a binary mixture IPA-W whose IPA mole fraction is richer in IPA than the one corresponding to the homogeneous azeotrope IPA-W, we will focus our attention on feeds to a batch rectifier lying in the region where IPA is the stable node. Feeds in this region produce three different sequences of cuts: namely, CyH-IPA-W/IPA-W/IPA, CyH-IPA-W/CyH-IPA/IPA, and CyH-IPA-W/IPA, giving rise to two "batch" distillation regions. Figure 1 shows the calculated maximum feasible cuts corresponding to three different initial still composition belonging to the different "batch" distillation regions (F_2 , F_3) and to the internal boundary (without a physicochemical meaning) between the "batch" regions (F_1). The feed compositions in Figure 1 were calculated by adding different amounts of the entrainer-rich phase (O_{ph}) to the process feed with the composition [0.868 IPA, 0.132 W]. These feasible cuts correspond to a reflux policy such that the heteroazeotrope is returned as the column reflux during the first cut. This means that both the CyH-rich and W-rich phases (O_{ph} and W_{ph}) are refluxed to the column with an overall composition corresponding to that of the ternary azeotrope. It must be noted that in some systems it may be convenient that the reflux to the column be formed by only one of the phases from the condenser drum/decanter.²⁹

Note that an initial still composition lying in the line between the ternary azeotrope and pure IPA avoids the appearance of homogeneous azeotropic cuts. As a result of this, F_1 achieves the maximum alcohol recovery in the still at the end of a batch rectification. The calculated maximum IPA recoveries in the still for [F_2 , F_1 , F_3] in Figure 1 are [56.36%, 83.87%, 73.84%], respectively. Therefore, it would be interesting to investigate feasible sequences with ternary separations with compositions lying in the line connecting the ternary azeotrope with pure 2-propanol. Düssel³⁰ arrived at the same conclusion for the system 1-propanol/water/ethyl acetate.

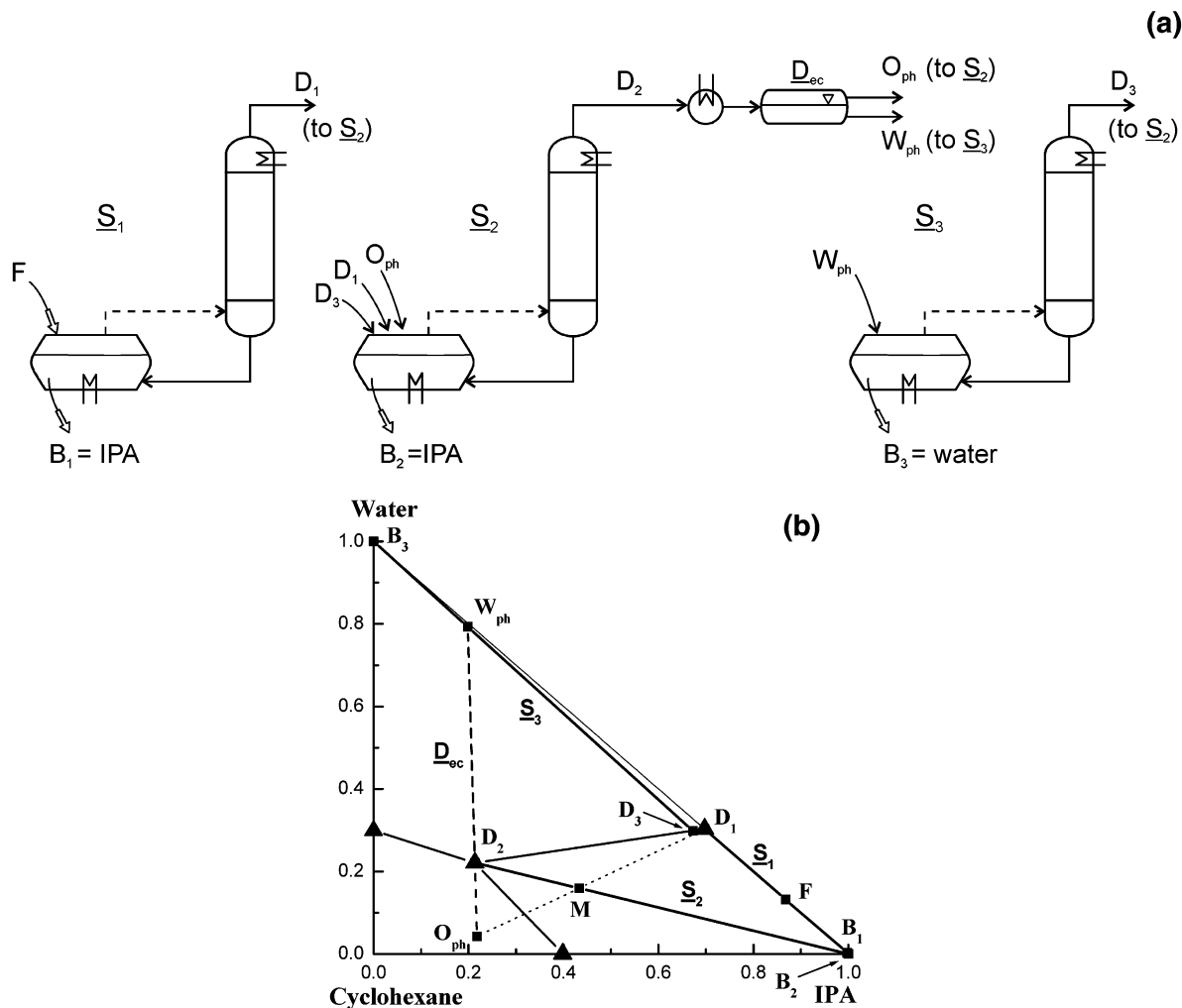


Figure 2. (a) Operation sequence corresponding to alternative I. (b) Overall mass balances in composition simplex for the batch process for recovering IPA from a binary mixture IPA–W with cyclohexane as dewatering agent (alternative I).

As the selected reflux policy gives rise to feasible cuts such as those found in homogeneous distillation, we use as a first approach of column performance the homogeneous model developed in Espinosa and Salomone²⁴ and Salomone and Espinosa.²⁵

Analysis of Two Different Recovery Alternatives

Definition of Tasks. Alternative I is presented in Figure 2 and requires three separation steps, which are performed in the same batch rectifier at different periods of time, and one decantation step.

The process cycle can be described as follows:

(i) The feed to the process F is charged in the reboiler of a batch rectifier. As a result of the distillation task S_1 , a distillate amount D_1 with composition close to that of the binary azeotrope IPA–W and a bottom fraction B_1 mainly formed by IPA is obtained. Task S_1 provides an IPA recovery in the bottom close to 65% of the alcohol contained in the feed F .

(ii) Distillation task S_2 processes the distillate fraction D_1 (S_1) and the distillate amount D_3 (S_3) together with the organic phase O_{ph} from the decantation task D_{ec} . Both D_3 and O_{ph} come from the previous cycle. The still content changes its composition from M to B_2 (IPA in specification) while the ternary azeotrope is removed as distillate by the rectifying column. D_2 represents the

overall amount of distillate collected in a product vessel after separation task S_2 is completed. In this step, an almost complete recovery of the alcohol initially contained in feed F is achieved.

(iii) Decantation step D_{ec} is done after separation task S_2 to split D_2 into a cyclohexane rich-phase O_{ph} and a water rich-phase W_{ph} . The organic phase supplies the cyclohexane necessary for the second distillation task S_2 in the next cycle, while the water rich-phase is driven to the batch rectifier (distillation task S_3) in order to obtain a wastewater amount B_3 in the residuum and a distillate amount D_3 , which is mainly formed by IPA–W at its azeotropic composition. D_3 is recycled to separation task S_2 in the next cycle.

Table 1 presents the approximate overall mass balances corresponding to a simplified state-task network representation of alternative I, where the tasks are perfect splits and the smaller cyclohexane makeup stream was not taken into account.

The alternative II is presented in Figure 3 and requires two distillation steps that are performed in the same batch rectifier at different periods of time and one decantation step.

The process cycle can be described as follows:

(i) The feed to the process F together with distillate amount D_2 (S_2) and organic phase O_{ph} (D_{ec}) are charged in the reboiler of a batch rectifier. Both D_2 and O_{ph} come from the previous cycle. The still content changes its

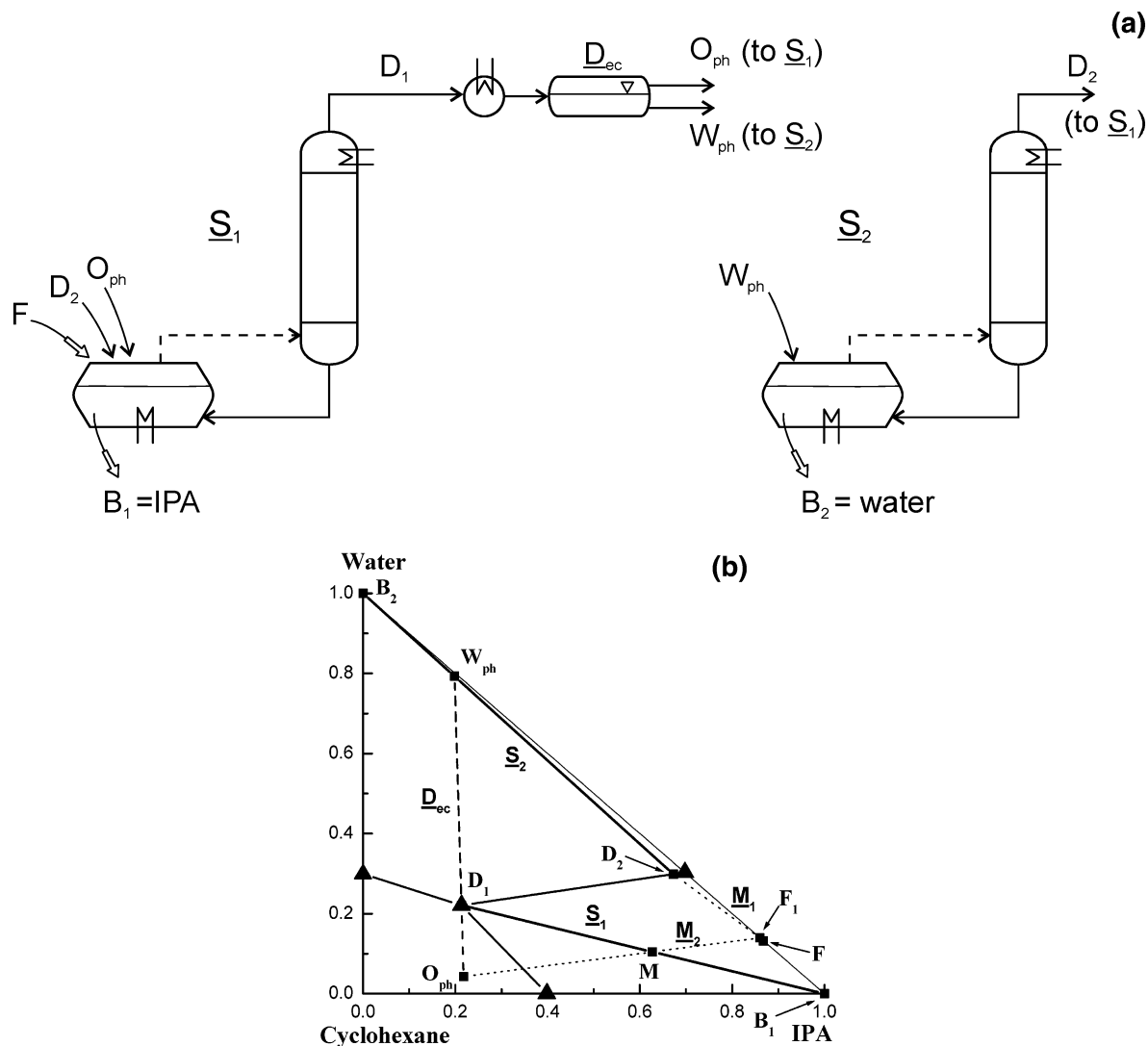


Figure 3. (a) Operation sequence corresponding to alternative II. (b) Overall mass balances in composition simplex for the batch process for recovering IPA from a binary mixture IPA–W with cyclohexane as dewatering agent (alternative II).

Table 1. Overall Mass Balances of the Batch Process for Recovering IPA from a Binary Mixture IPA–W with Cyclohexane as Dewatering Agent (Alternative I)

distillation task S_1	distillation task S_2	decantation task D_{ec}	distillation task S_3
F (1000 kmol)	M (1090.1 kmol)	D_2 (785.5 kmol)	W_{ph} (186.9 kmol)
D_1 (436.2 kmol)	D_2 (785.5 kmol)	O_{ph} (598.6 kmol)	D_3 (53.10 kmol)
B_1 (563.8 kmol)	B_2 (304.6 kmol)	W_{ph} (186.9 kmol)	B_3 (133.8 kmol)

Table 2. Overall Mass Balances of the Batch Process for Recovering IPA from a Binary Mixture IPA–W with Cyclohexane as Dewatering Agent (Alternative II)

mixing task	distillation task S_1	decantation task D_{ec}	distillation task S_2
F (1000 kmol)	M (1651.2 kmol)	D_1 (785.1 kmol)	W_{ph} (187 kmol)
D_2 (53.1 kmol)	D_1 (785.1 kmol)	O_{ph} (598.1 kmol)	D_2 (53.1 kmol)
O_{ph} (598.1 kmol)	B_1 (866.1 kmol)	W_{ph} (187 kmol)	B_2 (133.9 kmol)
M (1651.2 kmol)			

composition from M to B_1 (IPA in specification) while the ternary azeotrope is removed as distillate by the rectifying column. D_1 represents the overall amount of distillate collected in a product vessel after separation task S_1 is completed. Task S_1 provides an almost complete recovery of the alcohol contained in the feed F .

(ii) Decantation step D_{ec} is done after separation task S_1 to split D_1 into a cyclohexane rich-phase O_{ph} and a water rich-phase W_{ph} . The organic phase supplies the cyclohexane necessary for the distillation task S_1 in the next cycle, while the water rich-phase is driven to the

batch rectifier (distillation task S_2) in order to obtain a wastewater amount B_2 in the residuum and a distillate amount D_2 , which is mainly formed by IPA–W at its azeotropic composition. D_2 is recycled to separation task S_1 in the next cycle.

Table 2 shows the pseudo-steady-state overall mass balances obtained for the cyclic operation after a few cycles, consisting of recycling the amount D_2 of separation task S_2 and fraction O_{ph} of decantation step D_{ec} to the reboiler in distillation task S_1 .

Calculation of Minimum Reflux and Minimum Number of Stages. To evaluate how much separation

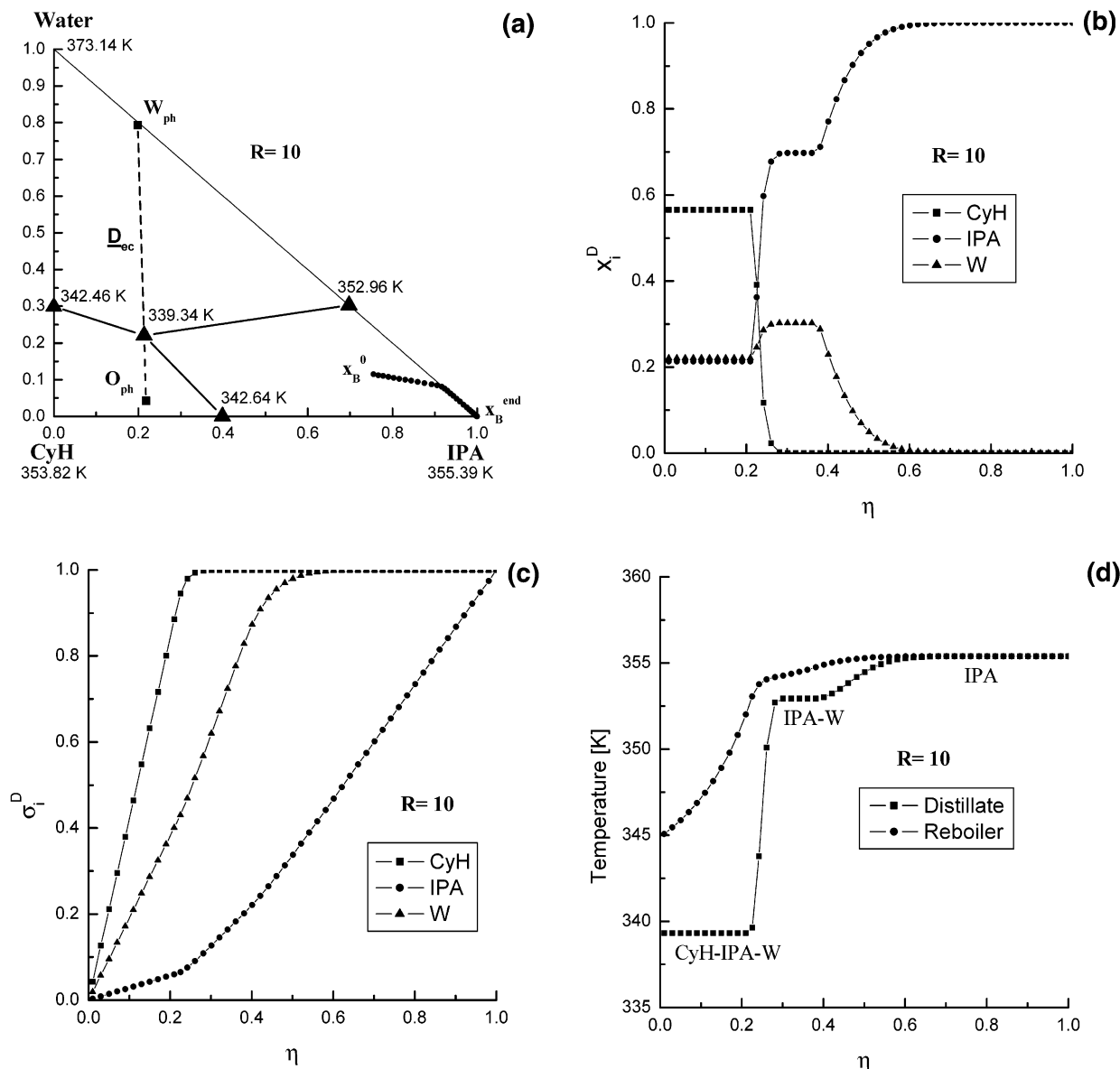


Figure 4. (a) Evolution of the still path from a dynamic simulation of the conceptual model. (b) Distillate composition from a dynamic simulation of the conceptual model. (c) Component recoveries from a dynamic simulation of the conceptual model. (d) Top and bottom temperatures from a dynamic simulation of the conceptual model.

power (number of stages and minimum reflux) is needed for a given purity requirement for each of the tasks belonging to an alternative, simulation runs of the dynamic conceptual model developed in Espinosa and Salomone²⁴ and Salomone and Espinosa²⁵ were performed.

As two design variables have to be selected in addition to the feed composition to the batch rectifier in order to enable simulation runs of a batch rectifier with an infinite number of stages, we select the distillate recovery of a given component plus the reflux ratio. The recoveries of the components in the distillate as a function of rectification advance are estimated through integration of the following equations:

$$\frac{d\sigma_i^D}{d\eta} = \frac{x_i^D}{x_i^0} \quad (1)$$

where σ_i^D is the fractional recovery of component i in the distillate, η is the rectification advance, x_i^D is the

mole fraction of component i in the distillate, and x_i^0 is the initial mole fraction of component i in the still.

All the other variables such as recoveries of the components in the residue, distillate, and residue compositions and top and residue temperatures can be calculated as a function of component recoveries in the distillate and rectification advance. While reflux ratio is used to estimate the instantaneous distillate composition x_D starting from the instantaneous still composition x_B , the pre-fixed final recovery of a given species in the distillate (or alternatively, the final rectification advance) is used as integration stop criterion.

To facilitate comprehension, the results of a simulation run with the conceptual model for a mixture with composition [0.1343 CyH, 0.75 IPA, 0.1157 W] are shown in Figure 4. The graphics correspond to total rectification of the mixture ($\eta = 1$) at an operating reflux ratio of 10. The model calculates the controlling pinch points, and therefore, a column with an infinite number of stages is considered.

Table 3. Maximum Feasible Separation at Infinite Separation Power

	composition (mol/mol) [CyH, IPA, W]	bubble and dew T (K)	amount (kmol)
feed	[0.1343, 0.7500, 0.1157]	[344.91, 351.10]	1.0000
cut 1	[0.5656, 0.2138, 0.2206]	[339.31, 339.31]	0.2374
bottom 1	[0.0000, 0.9170, 0.0830]	[354.01, 354.46]	0.7626
cut 2	[0.0000, 0.6966, 0.3034]	[352.92, 352.92]	0.2087
residue	[0.0000, 1.0000, 0.0000]	[355.39, 355.39]	0.5539

By analyzing the results of the simulation, it is clear that the separation becomes more difficult during the second cut, where a distillate with composition close to the IPA–W azeotrope is obtained. The selected reflux ratio is not able to maintain the distillate composition at the azeotropic composition, and hence, an extra loss of alcohol in the distillate occurs. For this reflux ratio, a final fractional recovery of IPA in the residue of ~ 0.45 mol/mol is reached. By increasing the reflux ratio, the alcohol loss in the distillate could be decreased, and hence, IPA recovery in the residue could be increased.

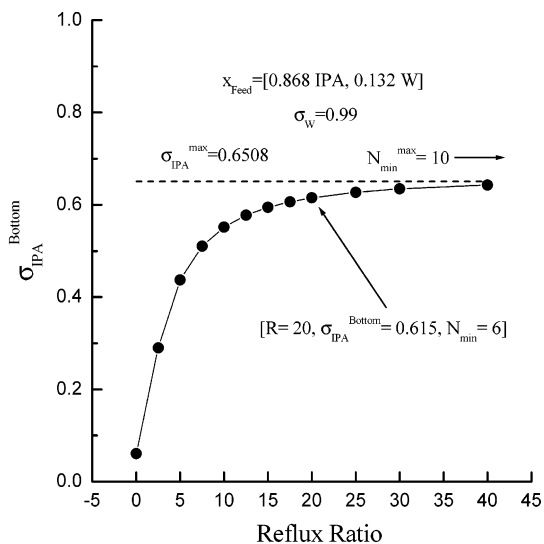
As already mentioned, all the predicted variable trajectories will be good approximations of the first part of the process only if the liquid reflux to the column has a global composition corresponding to that of the ternary azeotrope.

When the recovery of a given component is used as the criterion for stopping the integration, the information about maximum feasible separation is necessary to specify feasible separations. Table 3 shows the results obtained for a mixture with composition [0.1343 CyH, 0.75 IPA, 0.1157 W]. The calculation method is explained in Espinosa et al.²⁰ and follows the guidelines introduced in Bernot et al.^{1,2}

Table 3 tells us for example that the maximum feasible recovery of alcohol in the residue is $(1 \times 0.5539) / (0.7500 \times 1) \times 100 = 73.85\%$ (26.15% is lost in both cuts 1 and 2). This recovery needs infinite separation power, and therefore, it is a limit that cannot be achieved in real practice. However, recoveries close to the limit can be approached by operating at finite reflux ratios.

In this work, each of the separation tasks was rated by finding a finite value of the reflux ratio as a measure of the degree of difficulty of the task. The value of this characteristic reflux enables a separation close to the maximum feasible one. To compute it, several simulation runs are performed, varying the reflux ratio while maintaining at a constant value the recovery of a prefixed species. As an example, Figure 5 presents a graphic of the recovery of the alcohol in the residuum $\sigma_{\text{IPA}}^{\text{B}}$ at the end of the separation task versus the reflux ratio for a given recovery of water in the distillate $\sigma_{\text{W}}^{\text{D}}$. This plot allows us to select a characteristic reflux ratio for tasks involving the separation of the alcohol. Furthermore, if the separation is difficult, an operation with two or three reflux levels can be proposed to reduce the energy requirement of the distillation task. This point will be discussed in more detail at the end of the paper.

The minimum number of stages is also computed for each simulation run at constant reflux. Once the recovery of a given component and the reflux ratio are chosen as design variables, all the remaining recoveries are determined after integration of eqs 1. The minimum number of stages for the separation is then calculated by defining the recoveries in distillate of both the light and heavy components. As was mentioned, the total reflux limiting operating condition is adequately de-

**Figure 5.** Separation task S_1 [I]: recovery of IPA in the residue at rectification end as a function of the reflux ratio.**Table 4. Maximum Feasible Separation at Infinite Separation Power (S_1 [I])**

	composition (mol/mol) [CyH, IPA, W]	bubble and dew T (K)	amount (kmol)
feed	[0.0000, 0.8680, 0.1320]	[353.52, 353.95]	1.0000
cut 1	[0.0000, 0.6966, 0.3034]	[352.92, 352.92]	0.4351
residue 1	[0.0000, 1.0000, 0.0000]	[355.39, 355.39]	0.5649

scribed by solving a sequence of equilibria and mass balance computations from a given still composition in order to predict the instantaneous distillate composition and by assuming a differential rate of product withdrawal from the column at any time. The method that we use to get the minimum number of stages is an iterative procedure where an initial guess of N is refined until the specified recovery fractions of two components are reached in the distillate. The simulations are stopped when the amount of the light key in the distillate corresponds to the specified component recovery, $\sigma_{\text{hk}}^{\text{D}}$. Then, the amount of the heavy key in the distillate is used to drive the iteration on N until this amount corresponds to the specified fractional recovery, $\sigma_{\text{hk}}^{\text{D}}$.

Finally, a comparison of the energy requirements of each alternative is performed in terms of the overall vapor generated in order to select the best one. To illustrate the approach, consider the analysis below, which corresponds to a process feed with composition [0.000 CyH, 0.868 IPA, 0.132 W] mol/mol.

Distillation Task S_1 [I]. Table 4 shows the maximum feasible separation at infinite separation power. This situation is equivalent to an alcohol recovery in the residue of 65.08%. Figure 5 shows the alcohol recovery in the residue as a function of the reflux ratio. Each of the simulation runs corresponds to a water recovery of 0.99 mol/mol. From Figure 5, it is clear that a rectifier operating at a reflux ratio of 20 closely approaches the maximum feasible separation because the IPA recovery in the residuum amounts to 61.5%. A minimum number of six stages is needed for this separation.

It is noteworthy that the calculation of the minimum energy demand in a batch distillation consists of estimating the minimum constant value of the reflux ratio necessary to achieve a given separation (in terms of distillate recoveries of both light and heavy compo-

Table 5. Comparison between Two Different Reflux Policies To Recover 61.5% of IPA Contained in the Feed (S₁[I])

different reflux policies to achieve 61.5% recovery of IPA in S ₁		
amount (kmol)	mole fraction [IPA, water]	reflux ratio R (component recovery, σ or rectification advance, η) and boiler temperature T_b
Operation at Constant Reflux		
F	100.00	$R = 20,$
D	46.45	$\sigma_W = 0.99,$
B	53.55	$T_b = 355.34 \text{ K}$
Operation at Two Different Reflux Ratio Levels		
F	100.000	$R_1 = 10,$
D_1	30.000	$\eta = 0.30,$
B_1	70.000	$T_b = 354.33 \text{ K}$
D_2	16.443	$R_2 = 20,$
B_2	53.557	$\sigma_W = 0.9679, T_b = 355.34 \text{ K}$

Table 6. Comparison between Two Different Reflux Policies To Recover 62.7% of IPA Contained in the Still (S₂[I])

different reflux policies to achieve 62.7% recovery of IPA in S ₂		
amount (kmol)	mole fraction [CH, IPA, water]	reflux ratio R (component recovery, σ or rectification advance, η) and boiler temperature T_b
Operation at Constant Reflux		
F	100.00	$R = 12.5,$
D	72.66	$\sigma_W = 0.99,$
B	27.34	$T_b = 355.15 \text{ K}$
Operation at Two Different Reflux Ratio Levels		
F	100.00	$R_1 = 5,$
D_1	50.00	$\eta = 0.50,$
B_1	50.00	$T_b = 342.36 \text{ K}$
D_2	22.66	$R_2 = 12.5,$
B_2	27.34	$\sigma_W = 0.9673, T_b = 355.15 \text{ K}$

nents). As an example, consider the following separation specifications: $[\sigma_W^D = 0.99 \text{ mol/mol}; \sigma_{\text{IPA}}^D = 0.385 \text{ mol/mol}]$. The minimum reflux ratio for this separation is 20.

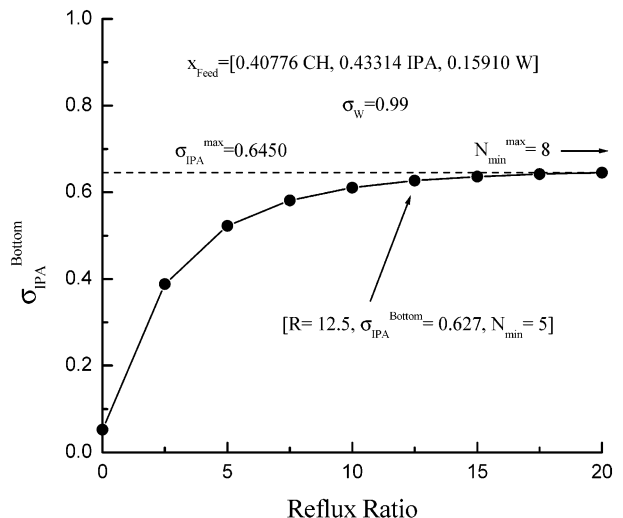
To reduce the energy demand of the task, it is possible to replace the operation at a constant value of the reflux ratio with an operation with two or more reflux levels. The idea is based on the concept that the separation is easier at the beginning of the operation because the reboiler contains the component to be separated (water in this case) in large amounts and the separation becomes more difficult at the end of the operation when the amount of the key species in the still diminishes (i.e., water in traces). Table 5 compares the initial single-reflux option with a more economic one in terms of energy demand. Both reflux policies result in the same amount and composition of the product. Note that in the operation with two different reflux levels, the first part of the process is characterized by a distillate of azeotropic composition. Only near the end of the operation with a reflux of 20, this reflux level is not able to maintain the distillate composition at a constant value, giving rise to an extra loss of IPA in the top product.

Distillation Task S₂[I]. In this case and considering a feed with composition M [0.40776 CyH, 0.43314 IPA, 0.15910 W] (see Figure 2b), the limiting alcohol recovery in the residue is 64.50%. Taking into account the mass balances around the whole process cycle, the limiting recovery in this task amounts to 34.92% of the IPA contained in feed F , and therefore, a total recovery of alcohol is achieved once separation tasks S₁[I] and S₂-[I] are performed.

Figure 6 presents the results of simulation runs for this mixture. An operation at a reflux ratio of 12.5 produces a recovery of 62.7% of the alcohol charged in the still at the beginning of separation task S₂[I], a value that is close enough to the maximum feasible recovery. This separation requires a minimum number of stages of 5.

In a way similar to separation S₁[I], a more economic alternative in terms of energy demand is presented in Table 6. The variable reflux policy could be implemented by first operating the column at constant distillate composition ($R = 5$) until rectification of 50% of the initial mixture. The first part could be followed by operating the column at variable distillate composition ($R = 12.5$). The second part is equivalent to operating the column until achieving a water recovery in the distillate amounting to 96.73% of the remaining water at the end of the first operation step.

Distillation Task S₃[I]. The goal of this separation task is the purge of the water contained in the feed to F . Water is obtained in the still as a residue of distillation task S₃[I] (see Figure 2). The distillate obtained at the end of rectification is mainly formed by IPA–W at a composition close to that of the correspond-

**Figure 6.** Separation task S₂[I]: recovery of IPA in the residue at rectification end as a function of the reflux ratio.

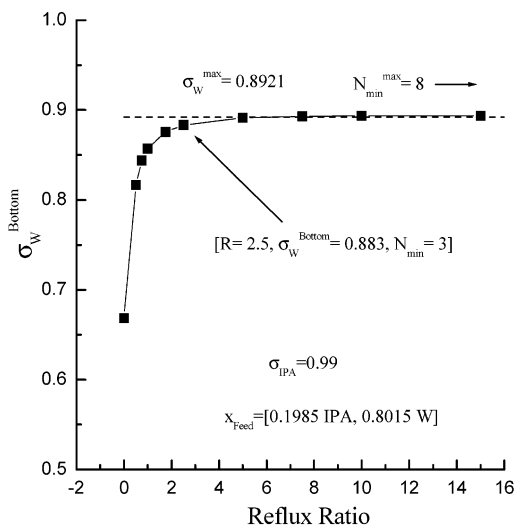


Figure 7. Separation tasks S_3 [I] and S_2 [II]: recovery of IPA in the residue at rectification end as a function of the reflux ratio.

Table 7. Comparison between Two Simulations Runs To Recover 99% and 99.9% IPA of the Alcohol Contained in the Still, as IPA in the Distillate (both S_3 [I] and S_2 [II])

operations to achieve 88.3% and 85.74% recovery of water at constant reflux		
amount (kmol)	mole fraction [IPA, water]	reflux ratio R (component recovery, σ or rectification advance, η) and boiler temperature T_b
Operation at Constant Reflux		
F	100.00 [0.1985, 0.8015]	$R = 2.5$,
D	28.99 [0.67779, 0.32221]	$\sigma_{IPA} = 0.99$,
B	71.01 [0.00279, 0.99721]	$T_b = 371.63$ K
Operation at Constant Reflux		
F	100.00 [0.1985, 0.8015]	$R = 2.5$,
D	31.26 [0.63436, 0.36564]	$\sigma_{IPA} = 0.999$,
B	68.74 [0.00029, 0.99971]	$T_b = 372.97$ K

ing binary azeotrope. This distillate is recycled to separation S_2 [I] of the next processing cycle.

Figure 7 shows the different water recoveries in the residue as a function of the reflux ratio together with the maximum feasible water recovery in the residue at rectification end. All the simulations were done up to an alcohol recovery in the distillate of 99%. A reflux ratio as low as 2.5 enables a water recovery of 88.3% that is very close to the maximum value of 89.21%. A minimum number of three stages is necessary to perform this separation at operation near total reflux.

Table 7 shows the overall balances for this operation. In addition, the mass balances of an operation at reflux ratio of 2.5 and alcohol recovery of 99.9% in the distillate are presented. In this case, the distillate composition moves a little away from the azeotropic composition while the water recovery in the residue amounts to 85.74%.

As this separation task remains the same for the second processing alternative, the analysis above is also valid for S_2 [II].

Distillation Task S_1 [II]. Table 8 shows the maximum feasible separation for the mixture of composition M [0.2680 CyH, 0.6274 IPA, 0.1046 W] in Figure 3b. The maximum alcohol recovery in the residue is 83.8% of the IPA charged in the still at the beginning of task S_1 [II] and represents all the alcohol contained in the initial feed F .

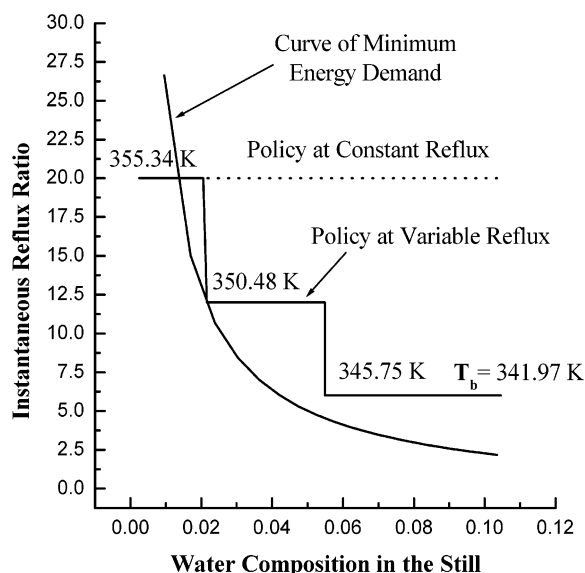


Figure 8. Minimum “instantaneous” reflux ratio to achieve the ternary azeotropic composition versus water mole fraction in the still. Water mole fraction corresponds to the still path depicted from the line CyH–IPA–W/IPA.

Table 8. Maximum Feasible Separation at Infinite Separation Power (S_1 [II])

	composition (mol/mol) [CyH, IPA, W]	bubble and dew T (K)	amount (kmol)
feed	[0.2680, 0.6274, 0.1046]	[341.97, 347.95]	1.0000
cut 1	[0.5656, 0.2138, 0.2206]	[339.31, 339.31]	0.4738
residue 1	[0.0000, 1.0000, 0.0000]	[355.39, 355.39]	0.5262

By comparing the initial compositions in separation tasks S_2 [I] and S_1 [II] (Figures 2b and 3b) it seems that S_1 [II] is more difficult because the representative point M [II], located in the line CyH–IPA–W/IPA, is closer to pure IPA than the initial still composition corresponding to the first alternative. The reason of this can be explained by analyzing Figure 8, where the minimum “instantaneous” reflux that is necessary to achieve the ternary azeotrope at the top of the column is plotted against the water composition in the still. To obtain this curve, both the distillate composition and a component recovery or the rectification advance must be fixed. Then, eqs 1 are integrated by using the selected component recovery or rectification advance as integration stop criterion. At each instant of time, the minimum reflux ratio is estimated in order to achieve the prefixed distillate composition. Figure 8 shows the evolution of the reflux ratio for still compositions belonging to the line CyH–IPA–W/IPA departing from composition M [II] in Figure 3b. From this figure it is clear that the separation becomes more and more difficult as the composition in the still path approaches the alcohol vertex.

Figure 9 confirms the ideas above. An approximated reflux ratio of 20 (the dent in the graphic is due to a numerical inaccuracy) and a minimum number of five stages is required to achieve an alcohol recovery in the bottom close to 80% of IPA charged in the still at the operation beginning. Table 9 presents the results for operation at three different reflux ratio levels in order to reduce operating costs. Figure 8 points out the meaning of the variable reflux policy. Each of the two first reflux levels are enough to maintain the distillate composition at a constant value (ternary azeotropic composition) because the selected reflux policy does not

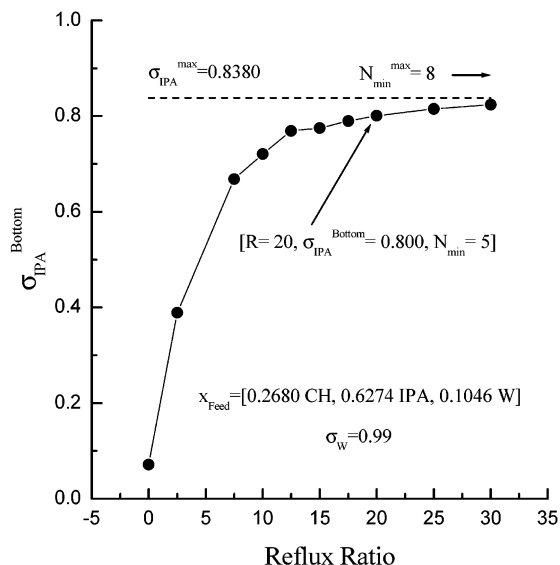


Figure 9. Separation task S_1 [III]: recovery of IPA in the residue at rectification end as a function of the reflux ratio.

Table 9. Comparison between Two Different Reflux Policies To Recover 80.0% of IPA Contained in the Still (S_1 [III])

different reflux policies to achieve 80% recovery of IPA in S_1		
amount (kmol)	mole fraction [CH, IPA, water]	reflux ratio R (component recovery, σ or rectification advance, η) and boiler temperature T_b
Operation at Constant Reflux		
F 100.00	[0.2860, 0.6274, 0.1046]	$R = 20$,
D 49.66	[0.53959, 0.25189, 0.20852]	$\sigma_w = 0.99$
B 50.34	[0.00006, 0.99786, 0.00208]	$T_b = 355.34$ K
Operation at Three Different Reflux Ratio Levels		
F 100.0000	[0.2860, 0.6274, 0.1046]	$R = 6$,
D_1 30.0000	[0.56594, 0.21325, 0.22081]	$\eta = 0.3$,
B_1 70.0000	[0.14031, 0.80489, 0.05480]	$T_b = 345.75$ K
D_2 11.6410	[0.56594, 0.21325, 0.22081]	$R = 12$,
B_2 58.3590	[0.05542, 0.92289, 0.02169]	$\sigma_w = 0.67$, $T_b = 350.48$ K
D_3 8.0244	[0.40268, 0.45260, 0.14472]	$R = 20$,
B_3 50.3346	[0.00006, 0.99786, 0.00208]	$\sigma_w = 0.9174$, $T_b = 355.34$ K

intersect the curve of minimum energy demand during the first two steps. During the last step, however, a variable distillate composition policy occurs until the pre-fixed IPA bottom recovery is achieved, and therefore, an intersection between the reflux policy and the minimum energy demand curve takes place. Therefore, it is clear that the curve of minimum "instantaneous" reflux acts as a limiting curve and, used together with the minimum reflux calculated for a column operating at constant reflux, permits a first "optimization" of the reflux levels for a given distillation task. To decide about the extension of each separation in Table 9, the minimum energy demand curve as a function of the rectification advance instead of water composition was used.

Note that although the minimum energy demand curve could be totally approached from operation beginning until the value of the minimum instantaneous reflux ratio reaches the value of the minimum reflux corresponding to operation at constant reflux ($R_{\min} = 20$ in Figure 8), this would require a very elaborate control mechanism. Therefore, we approach the minimum energy curve by selecting one or two reflux levels because it is very easy to implement in practice even if some energy is wasted. In this case, the temperature

Table 10. Overall Amount of Vapor Generated in S_1 [I]

V (kmol/h)	50	50	50	
	policy 1		policy 2	
reflux ratio	20	10	20	
D (kmol)	46.45	30	16.45	
D (kmol/h)	2.381	4.545	2.381	
time (h)	19.509	6.600	6.909	overall amt
V (kmol/h)	975.45	330.00	345.45	675.45

Table 11. Overall Amount of Vapor Generated in S_2 [I]

V (kmol/h)	50	50	50	
	policy 1		policy 2	
reflux ratio	12.5	5	12.5	
D (kmol)	72.66	50	22.66	
D (kmol/h)	3.704	8.333	3.704	
time (h)	19.618	6.000	6.118	overall amt
V (kmol)	980.91	300.00	305.91	605.91

Table 12. Overall Amount of Vapor Generated in S_1 [II]

V (kmol/h)	50	50	50	50	
	policy 1		policy 2		
reflux ratio	20.0	6	12	20	
D (kmol)	49.66	30	11.99	7.21	
D (kmol/h)	2.381	7.143	3.846	2.381	
time (h)	20.857	4.2	3.118	3.028	overall amt
V (kmol)	1042.86	210.00	155.88	151.42	517.30

in the reboiler or the temperatures in some rectifier stages could eventually be used in the control strategy to establish the different reflux levels in the task recipe.

We select, on the other hand, to operate the column at its "minimum reflux ratio at constant reflux" only at the end of the operation in order to achieve the pre-fixed component recovery.

Selection among Alternatives. Once estimations of both reflux levels and minimum number of stages for each of the distillation tasks were done, the selection of the best in terms of energy demand was performed.

As the amount of organic phase is dictated by the amount of water in the original feed, the separation task corresponding to the purge of water is common to both alternatives, and therefore, this separation can be put aside from the analysis.

To evaluate the best alternative, an arbitrary value for the maximum allowable vapor flow rate is selected, and then, the overall amount of vapor generated for each separation task is computed. After that, the corresponding heat duty is estimated by using the enthalpy of vaporization of the corresponding feed. Feed enthalpies are simply computed from component mole fractions and latent heats. Tables 10–12 show the results for both alternatives. Policy 1 corresponds to tasks operation at constant reflux ratio while policy 2 accounts for the energy consumption of tasks operated with variable reflux levels. The feed amount to the batch process considered in calculations is 100 kmol and the vapor flow rate is 50 kmol/h.

Taking into account that the computation of vapor generated by each alternative reported in Tables 10–12 is on a 100 kmol basis, we also resort to the ratio M/F reported in the overall mass balances (Tables 1 and 2), to calculate the overall amount of vapor and heat duty of each alternative: amount of vapor generated in alternative I = $675.45 + 1.0901 \times 605.91 = 1335.95$ kmol; heat duty in alternative I = $675.45 \times \Delta H^v[F(S_1, I)] + 1.0901 \times 605.91 \times \Delta H^v[F(S_2, I)]$; heat duty in alternative I = $675.45 \times 40032 + 1.0901 \times 605.91 \times 35953 = 5.08 \times 10^7$ kJ; amount of vapor generated in

alternative II = $1.6512 \times 517.30 = 854.17$ kmol; heat duty in alternative II = $1.6512 \times 517.30 \times \Delta H^F[F(S_1, \text{II})]$; heat duty in alternative II = $1.6512 \times 517.30 \times 37320 = 3.19 \times 10^7$ kJ.

Therefore, alternative II saves ~37% energy and it is the alternative selected for the IPA recovery process.

Conclusions

Along this work, all the steps of conceptual design were applied to solve a solvent recovery problem.

By using a software tool that integrates all the algorithms necessary to approach the conceptual design of batch distillations, we were able to analyze this case study all the way from azeotrope prediction and determination of distillation regions to the calculation of minimum reflux, minimum number of stages, and maximum feasible separation for each separation task belonging to a given separation alternative.

To select among different alternatives, the maximum feasible separation of each task was first calculated and then approximated through a finite value of the reflux ratio of a column with an infinite number of stages. The minimum number of stages for each separation task was also calculated. Operation of each task at different reflux levels was also explored to reduce energy costs.

Based on the minimum energy demand of each separation task and taking into account the overall mass balances of both studied alternatives, the overall amount of energy consumed by each alternative was considered in order to select the best one.

The contribution of this paper is, we think, to present an articulation of existing methodologies of conceptual design, to solve a practical design problem.

Finally, the minimum number of stages and minimum reflux of each distillation task calculated in this approach could be used as bounds in a refined design through a dynamic optimization of a detailed model of the separation network.

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Nomenclature

B = amount of residue collected at the end of rectification in the rectifier still (kmol)

CyH = cyclohexane

D = amount of distillate collected at the end of rectification (kmol)

D_{ec} = decanter task

F = amount of feed (kmol)

IPA = 2-propanol

M = initial composition for separations $S_2(\text{I})$ and $S_1(\text{II})$

N = number of stages

Q_{ph} = organic phase amount in the decanter (kmol)

R = reflux ratio

S = separation task batch wise performed

T = temperature (K)

V = vapor amount (kmol), vapor flow rate (kmol/h)

W = water

W_{ph} = water phase amount in the decanter (kmol)

x = liquid composition

y = vapor composition

Greek letter

σ_i^D = recovery of component i in distillate product

σ_{lk}^D = recovery of light key component in distillate product

σ_{hk}^D = recovery of heavy key component in distillate product

σ_i^B = recovery of component i in the residue

η = rectification advance

ΔH^F = enthalpy of vaporization (kJ/kmol)

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