

Conceptual Modeling and Referential Control Applied to Batch Distillations

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This paper proposes the combined use of a conceptual modeling method and a referential control strategy to operate batch distillation columns. Using the pinch theory, the modeling method, which is valid for multicomponent mixtures, allows the derivation of a quasi-optimal recipe to guide the operation properly. The referential control implies the empirical determination of reduced-order models for designing and tuning feedback controllers dedicated to tracking nonstationary conditions in batch processes. While defining a feasible recipe implies adjusting the reference trajectory until the desired product purity and recovery is achieved, defining the feedback control system implies first the selection of an appropriate tray-temperature evolution and then the isolation of the dynamics associated with the manipulated variable from the main time-variable behavior desired for the operation. The effectiveness of this combined procedure is illustrated through rigorous simulations where the light component is recovered from binary and ternary mixtures of alcohols.

1. Introduction

Fractionation in batch distillation columns is one of the most common technologies used in pharmaceutical and specialty chemical industries due to its operational flexibility. A batch column can separate multicomponent mixtures with a wide range of feed compositions and different degrees of separation difficulty.

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, conceptual models based on pinch theory have been successfully applied to determine quasi-optimal trajectories intended to obtain products with both purities and recoveries above a certain level while operating the column near the condition of minimum energy demand.^{1,2}

Once the quasi-optimal operation is determined, the practical implementation requires an appropriate control system adjusted to track the desired conditions while maintaining the light-component purity free from unfavorable disturbances. This work proposes a temperature tracking control system composed by an open-loop reflux ratio control plus a closed-loop correction for disturbance rejection. To adjust the closed-loop controller we use the referential dynamic reaction of the process³ and tuning rules⁴ that, though they were originally developed for dynamics valid in the neighborhood of stationary operating points, under this strategy they are useful in the neighborhood of a reference transient evolution like those occurring in batch distillation columns.

Other contributions in this field are summarized in Li and Wozny.⁵ There generalized predictive control (GPC) is used to track both the maximum vapor load and the changing temperature profile of the condenser for multiple-fraction batch distillation. The optimal reflux ratio profile is implemented in an open-loop fashion by moving the set point of a PID flow controller. However, this control structure cannot account for variations in feed compositions.

Barolo and Dal Cengio⁶ have analyzed the problem of uncertainty in the initial condition, and they proposed a three-

step strategy for closed-loop optimization of a batch rectifier operated at a constant reflux ratio. The optimal reflux rates for several different feed compositions are first calculated offline. Then, a correlation between these optimal values and the composition (or temperature) at the end of the start-up phase is derived from previous optimizations. Finally, bottom and distillate product compositions are estimated online via available secondary measurements to establish both the optimal reflux ratio and switching phases in a closed-loop fashion.

Our proposal, on the other hand, lies on the assumption that tracking a selected tray-temperature path corresponding to the nominal case is an appropriate option to handle disturbances in the initial feed composition for feeds located in the neighborhood of that corresponding to the nominal case.

Applied for each case study, the conceptual model based on pinch theory is presented in the second section, and the evolution of the reflux ratio necessary to obtain high purity distillate composition starting from a nominal mixture composition is reported. Then, the referential modeling strategy is outlined. After that, the structure of the control system is presented, and both a feasible recipe and a tray-temperature evolution are obtained from open-loop rigorous simulations of a column having a finite number of separation stages. Two different techniques are tested in selecting the corresponding output-reference trajectory. In section 5, the referential reaction curve is obtained by doing changes to the nominal trajectory of the distillate flow rate to allow the estimation of the parameters needed for tuning the feedback controller. Finally, we show the results of rigorous closed-loop simulations for mixtures with different initial compositions, and the conclusions are presented together with comments about the future work.

2. Conceptual Model

In this work, the quasi-optimal operation of a batch rectifier is defined as the reflux-ratio evolution that allows a distillate stream of constant composition, while the energy demand of the process approaches to the minimum value. The main assumptions of the conceptual model are (i) the rectifier has infinite number of stages and (ii) the instant variations of the molar hold-up in the trays are negligible. Under these assumptions, the following relationship applies for every component in the mixture (see the Appendix)⁷

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$$\frac{d\sigma_i^D}{d\eta} = \frac{x_i^D}{x_{i0}^B} \quad (1)$$

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_{i0}^B is the initial mole fraction of component i in the still. Once the desired distillate composition is selected, the equation system (1) can be integrated to obtain the component recoveries as functions of the rectification advance. All other variables, such as the recoveries of the components in the residue, the residue compositions, and the temperature, can be calculated as a function of the component recoveries and the rectification advance, as explained in the Appendix.

For binary mixtures, the instantaneous minimum reflux ratio $R_{\min}(t)$ to achieve the prefixed distillate composition is estimated from the instantaneous liquid composition in the reboiler x_i^B and the composition of its vapor in equilibrium y_i^B , as follows:

$$R_{\min}(t) = \frac{(x_i^D - y_i^B)}{(y_i^B - x_i^N)} \quad (2)$$

$$x_i^N = x_i^B \quad (3)$$

Equation 2 comes from the instantaneous component balance around the rectifier (i.e., lever arm rule) for known values of the distillate composition x_i^D and vapor feed y_i^B and the unknown value of the composition x_i^N of the liquid stream leaving the rectifier lower end. The main assumption implicit in this equation is that the molar flow rates are constant along the column for both vapor and liquid streams. More important, condition (3) imposes a pinch zone at the lower end of the column, just above the still. This pinch has the same composition as the reboiler content, and it controls the geometry of the internal profiles under the condition of instantaneous minimum energy demand. This assumption in fact requires a rectifier with an infinite number of equilibrium stages. Note that increasing the reflux ratio above $R_{\min}(t)$ leads to distillate purities above the specification, and therefore the instantaneous reflux ratio obtained from solving eqs 2 and 3 represents a minimum. A limiting case is obtained when the distillate flow rate is pure light component, i.e., $x_1^D = 1$. In this case, it is clear that values of the reflux ratio above the minimum maintain the maximum product composition; however, a pinch at the column top (instead of a pinch at column bottom) controls the geometry of the internal profiles indicating a waste of energy. Accordingly, eq 3 is no longer valid. In this situation, the geometry of the internal profiles resembles the behavior during total reflux operation.

For multicomponent mixtures, the estimation of instantaneous minimum energy demand requires either solving an eigenvalue problem of the Jacobian of the equilibrium function in x_i^B or the pinch equations for a limiting distillate composition as explained elsewhere.^{8,9,10} Both methods allow the estimation of the controlling pinch points needed to calculate the composition x_i^N of the liquid stream at the lower end of the column. In this case, the instantaneous minimum reflux ratio to achieve a desired distillate composition at the top is calculated from the lever arm rule (eq 2) with x_i^N located at the intersection of the mass balance line and the hyper-plane formed by the controlling pinch points as shown in Figure 1(a),(b) for a ternary mixture of alcohols and a highly nonideal quaternary system, respec-

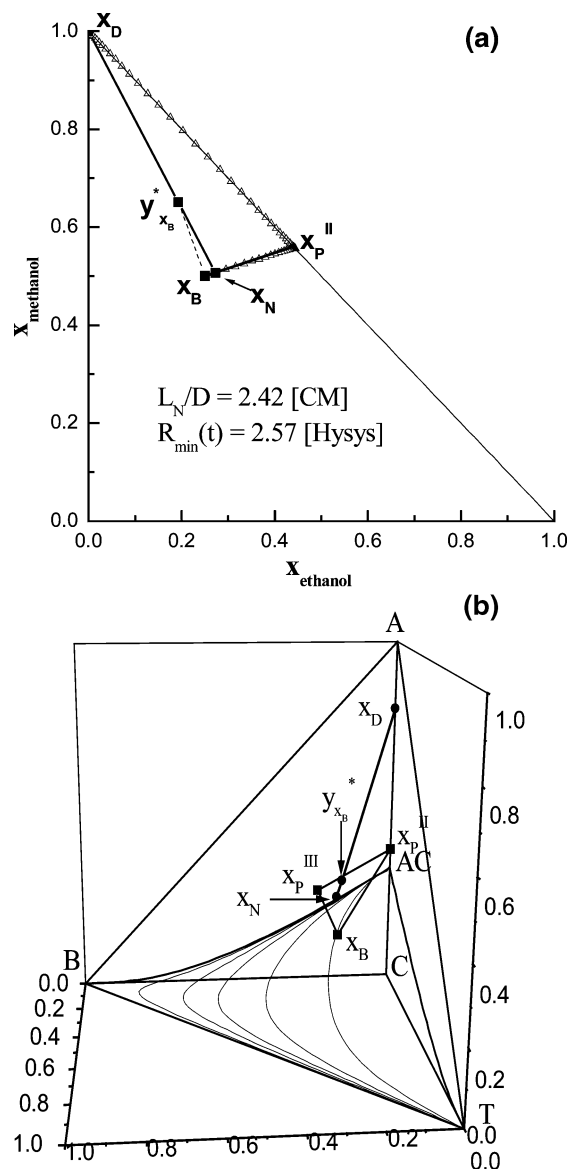


Figure 1. (a). System methanol–ethanol–isopropyl alcohol. Minimum reflux ratio for instantaneous still composition x_B . The composition x_N of the stream leaving the rectifier lower end belongs to the line defined by the controlling pinch points x_B and x_P^{II} . The intersection between the mass balance line $x_D - y_{x_B}^*$ and the line formed by the controlling pinch points $x_B - x_P^{II}$ gives the exact location of x_N . (b). System acetone–chloroform–benzene–toluene. Minimum reflux ratio for instantaneous still composition x_B . The composition x_N of the stream leaving the rectifier lower end belongs to the plane defined by the controlling pinch points x_B , x_P^{III} , and x_P^{II} . Intersection between the mass balance line $x_D - y_{x_B}^*$ and the hyper plane formed by the controlling pinch points gives the exact location of x_N .

tively. Thus, for multicomponent mixtures, eq 3 is replaced by a system of linear equations.

Figure 2(a) shows the evolution of the minimum reflux ratio necessary to have pure methanol at the top of a column having an infinite number of stages. The still is charged with 90 kmol of an equimolar mixture of methanol–isopropyl alcohol. The recovery of methanol at the column top is 94%. The results are first obtained in terms of the rectification advance, and, then, once a value for vapor flow rate V is set (i.e., $V = 30$ kmol/h), they can be seen as functions of the time t . To this end, the following equation is used:

$$t = \frac{1}{V} \int_0^t D(t)[R(t) + 1] dt \quad (4)$$

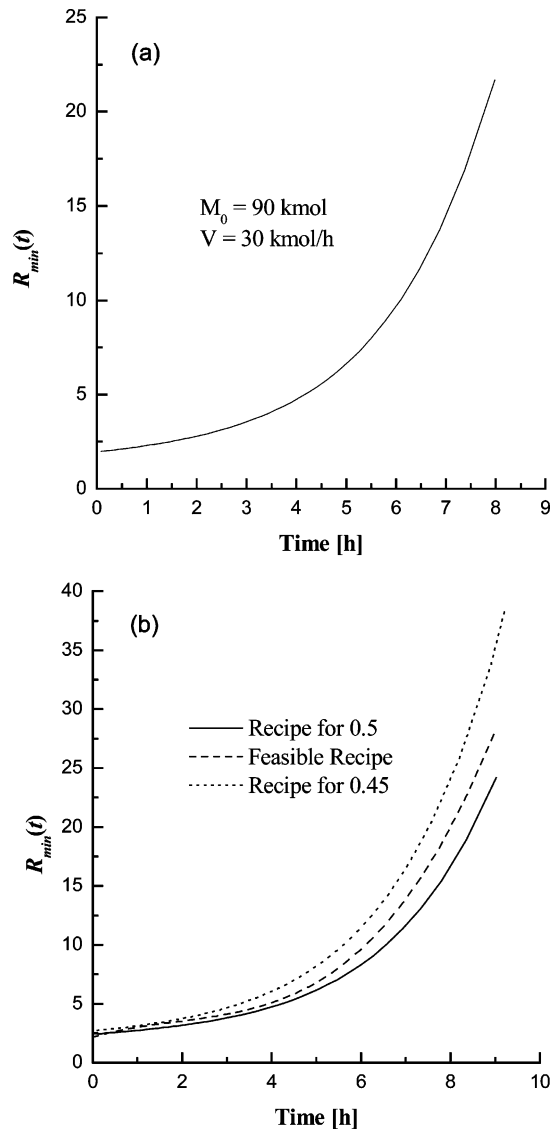


Figure 2. (a). System methanol–isopropyl alcohol. Time evolution of the minimum reflux ratio computed using the conceptual model. (b). System methanol–ethanol–isopropyl alcohol. Time evolution of the minimum reflux ratio computed using the conceptual model.

Table 1. Binary System: Comparison of Minimum Reflux Ratio Values from Hysys and Conceptual Model

$x_B(t)$	$R_{min}(t)$ [Hysys]	$R_{min}(t)$ [conceptual model]
[0.500, 0.500]	1.96	1.985
[0.411, 0.589]	2.43	2.497
[0.346, 0.654]	2.93	3.068
[0.221, 0.779]	4.70	5.014
[0.118, 0.882]	9.01	10.071

Figure 2(b) shows the corresponding minimum reflux ratio trajectory necessary to achieve high purity methanol as a distillate from a ternary mixture formed by methanol–ethanol–isopropyl alcohol. The still is charged with 90 kmol of a mixture with the composition of 0.5 methanol, 0.25 ethanol, and 0.25 isopropyl alcohol, and the vapor flow rate V is 30 kmol/h. The recovery of methanol at the column top is 94%.

For the binary mixture, Table 1 presents a comparison between the values of $R_{min}(t)$ estimated from the conceptual model and from steady-state simulations of a rectifier having 100 stages, which were done with Hysys.¹¹ For each of these simulations, the vapor in equilibrium with the compositions in

Table 1 was fed to the column, and the reflux ratio was changed beginning from a value below 1 until the desired distillate composition is obtained at the top. In this way, good agreements between estimated and simulated values were found. The same results apply for the ternary mixture as shown for the initial composition in the triangular diagram (Figure 1(a)). This figure also shows the internal liquid profile as calculated with Hysys.

3. Referential Modeling

The referential reaction method is a practical controller design and tuning procedure useful for tracking nominal or desired trajectories in batch processes. Specifically, the technical novelty comes up when this desired trajectory is taken as a referential nonlinear model of the overall expected process evolution. In this way, the controller form and its tuning are determined by the residual dynamics associated with the small changes of the main control variable driving the operation.³

The procedure assumes the desired or optimal process trajectory for nominal operating conditions has been previously determined. This means that the nominal time evolution of the main control variable must be known, either as an analytical function of time, a schedule of control actions, or simply as a file of numerical information. In the last case, the numerical information can be originated from rigorous simulations or from data acquisition made on the real process for fairly good runs.

The referential reaction curve necessary to define the controller is determined from the difference between responses of the output obtained from at least two open-loop runs using different input trajectories. One of these trajectories must be associated with the nominal condition where the output follows a behavior relatively close to the desired one. Then, at least one input trajectory must be designed such as to produce a temporary but measurable change in the output trajectory around the nominal evolution. The convenience of using this type of disturbance not only comes from economics reasons (the amount and quality of the product obtained in testing runs should be acceptable) or safety reasons (the operation should remain in a controllable region) but also is aimed to facilitate a linear approach when modeling the relative or referential reaction.

The referential modeling concept can also be introduced as follows: assume that a general nonlinear transient dynamic system is described by the transfer operator N , such that

$$N: u \mapsto y = N(u) \quad (5)$$

Assume also that a nominal output trajectory y_{ref} is associated through this transfer operator to an input sequence u_{ref} . Then, the nonlinear relationship between u_{ref} and y_{ref} can be referred to as

$$N: u_{ref} \mapsto y_{ref} = N(u_{ref}) \quad (6)$$

and consider now the difference

$$y - y_{ref} = N(u) - N(u_{ref}) \quad (7)$$

Assuming that N captures most of the nonlinear transient behavior, small departures from the expected behavior y_{ref} can be described as a family of linear disturbances. Thus, defining new referential variables $\tilde{u} = u - u_{ref}$ and $\tilde{y} = y - y_{ref}$, we may write

$$G: \tilde{u} \mapsto \tilde{y} = G\tilde{u} + O \quad (8)$$

where the aim of the linear transfer operator G is to describe small dynamics around the nominal or desired process trajectory,

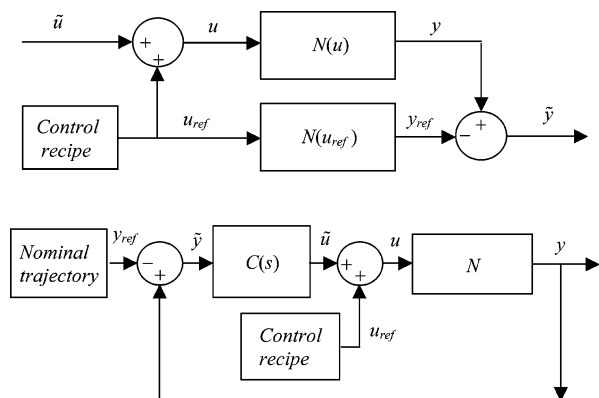


Figure 3. (a). Nonlinear compensation in referential modeling control. (b). Realization structure of referential control.

and O stands for higher order terms. The concept is completed assuming that the residual nonlinearities can be interpreted as bounded disturbances. In this way, referential reaction curves³ obtained by small step changes at different times along the batch progress may help to determine a nominal linear model \tilde{G} plus a global uncertainty Δ such that

$$G = \tilde{G} + \Delta \quad (9)$$

$$\Pi = \{G: |\Delta| = |G - \tilde{G}| \leq \bar{\Delta}\} \quad (10)$$

where Π denotes a family of linear models describing G . Figure 3(a) shows a sketch of this modeling strategy, where the nominal input trajectory is indicated as a control recipe. However, if there are enough data about the desired output trajectory y_{ref} that results from applying the nominal control recipe u_{ref} , then knowing $N(u_{ref})$ is not necessary, and the final control structure takes the form given in Figure 3(b). Hence, under this control structure, the controller $C(s)$ must handle only the residual dynamics G to compensate for disturbances all along the progress of the operation.

The next two sections apply the above conceptual framework to track desired tray-temperature trajectories for the batch distillation of binary and ternary mixtures of alcohols.

4. Open-Loop Rigorous Simulations and Tray-Temperature Selection

Conceptual models based on pinch analysis provide the quasi-optimal evolution of the reflux ratio for a column with an infinite number of stages, which is a good approximation to the variable reflux policy to be followed when considering a column with a finite number of trays and holdup as shown in ref 2 (see Figure 5 of the mentioned paper). For this reason, implementation of the nominal recipe must be analyzed in terms of product purity and recovery through rigorous simulation of the process in order to make changes to it, if necessary.

The simulator used in this analysis is based on a nonlinear dynamic model where each tray is represented by the differential equations for component mole fractions, energy, and total molar balances; the liquid–vapor equilibrium is described using the extended Antoine’s equation, and the tray hydraulics is solved by applying the Francis’ downcomer equation. The column is operated at its maximum feasible vapor flow rate value according to the column diameter, and it is assumed to have a total condenser working at atmospheric pressure with a distillate receiver tank whose level of liquid must be controlled. Table 2 shows the main parameter values used to obtain the results presented in this paper.

Table 2. Main Design Variables Used in the Simulation of the Distillation Column

parameter	binary mixture	ternary mixture
number of trays	20	30
column diameter D	0.47 m	0.47 m
downcomer height h_w	0.04 m	0.04 m
downcomer length L_w	0.329 m	0.329 m
total section A_{tot}	0.1735 m ²	0.1753 m ²
maximum vapor flow rate V	30 Kmol/h	30 Kmol/h
distillate receiver tank vol.	0.1 m ³	0.1 m ³
initial still load	90 kmol	90 kmol

It is noteworthy that vapor–liquid equilibrium calculated from the “extended Antoine equation” with parameters taken from Hysys represents very well the behavior of alcoholic mixtures as the model can handle variations in relative volatilities along the column height and operation time. The approach presented in this work is not limited to a particular method for predicting the thermodynamic behavior of the mixtures; however, it would be always advisable to minimize the effect of uncertainties in the thermodynamic model on the optimal control profile to achieve a better operating performance.¹²

Implementation of the reflux ratio evolution depicted in Figure 2(a) (binary mixture) in an open-loop fashion for a column having 20 stages yields a distillate product with a molar composition above 99%. Therefore, the quasi-optimal recipe derived from the conceptual model is adopted as the feasible one.

On the other hand, open-loop preliminary simulations of a 30 stages column were performed to determine a feasible recipe for the ternary mixture. It has been found that while the open loop implementation of the nominal recipe ($R_{min}(t)$ for 0.5 in Figure 2(b)) produces a large amount of low purity distillate [97.16%, 42.3 kmol], the open loop implementation of the recipe predicted by the conceptual model for composition and holdup in the still corresponding to the end of the start-up phase ($R_{min}(t)$ for 0.45 in Figure 2(b)) gives a reduced amount of high purity distillate [99.99%, 35.40 kmol], and, therefore, it is also discarded. In the last case, a pinch at the column top is maintained through the whole simulation run indicating a waste in energy consumption.

Both trajectories depicted in Figure 2(b) act as limiting curves. While the evolution calculated for the initial still composition does not allow for achieving a high purity product due to both a finite number of stages and column holdup, the trajectory for “0.45” is above the feasible one. Despite in the later case the estimation of the initial minimum reflux ratio uses the still composition at the end of the start-up phase, at this time, all the holdups in column trays and condenser are enriched in the light component, compensating for the lack of separation trays. Therefore, a recipe in between was selected and implemented in the simulation environment. Figure 2(b) shows the feasible recipe adopted due to both its adequate distillate purity and recovery [99.87%, 40.03 kmol]. Figure 4(a) shows the evolution of the light species composition along the column. These profiles indicate that the pinch zone moves from the top down the lower part of the column, behavior that is in stark contrast to that of the second recipe, where the pinch zone remains along 16 stages at the top, as shown in Figure 4(b).

As shown above, open-loop rigorous simulations allow for the correction of the recipe estimated from the conceptual model in order to obtain a high purity product with a recovery near to the maximum value predicted from the conceptual model. Then, the structure of the control system must be designed such to protect these objectives all along the operation. If the actual feed composition coincides with the one assumed by the

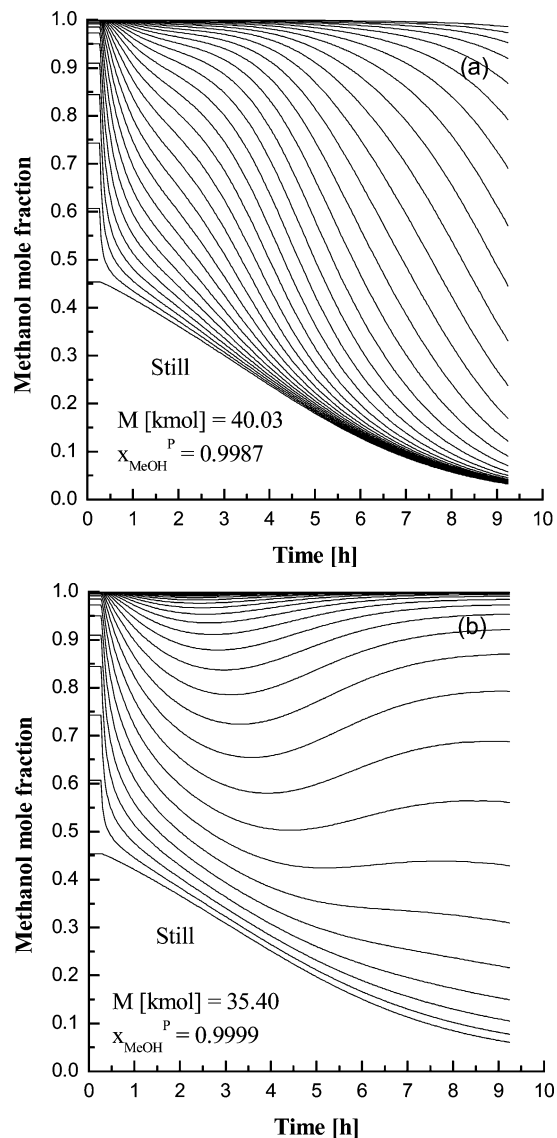


Figure 4. (a). System methanol–ethanol–isopropyl alcohol. Evolution of the light component composition along the column corresponding to the feasible recipe. (b). System methanol–ethanol–isopropyl alcohol. Evolution of the light component composition along the column corresponding to the recipe calculated for composition of the still mixture at the end of the start-up phase.

conceptual model, satisfying the predicted reflux ratio evolution is enough to achieve the desired operation. In this case, the reference trajectory $R_{\min}(t)$ to operate this column is implemented by a simple open-loop ratio controller associated with the condenser-drum level control as shown in Figure 5. The distillate flow rate D (input-reference trajectory, u_{ref}) has to adapt to the flow measures of the reflux stream L_0 , which in turn controls the liquid level in the reflux drum. However, when the feed composition is different from the nominal one, following $R_{\min}(t)$ alone is not sufficient to achieve the above-mentioned objectives. Thus, a temperature feedback control loop is combined with the open-loop structure to track a specific temperature evolution by slightly modifying $R_{\min}(t)$. In order to define this temperature control, the selection of an appropriate tray-temperature evolution (output-reference trajectory, y_{ref}) must be done once the feasible recipe is established.

Figure 6(a) shows the temperature in the condenser and in the first four stages from the top along an 8 h run corresponding to the binary mixture. These evolutions were obtained by charging the still with 90 kmol of an equimolar mixture of

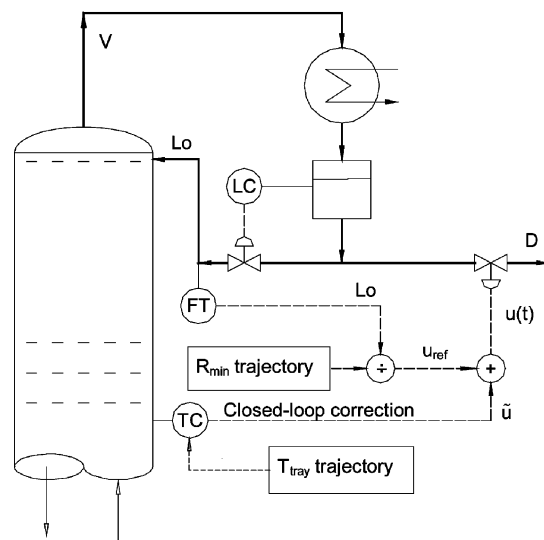


Figure 5. Temperature tracking control system formed by an open-loop ratio controller and a referential closed-loop correction.

methanol and isopropyl alcohol and using, after the start-up period, the open-loop ratio controller shown in Figure 5 without closed-loop correction. Thus, all these are “nominal” temperature evolutions, and, aside from the sensitivity problem, any one could be selected as reference in case that disturbances need to be rejected. In order to select the reference trajectory, two additional open-loop simulations were performed by varying the feed composition (i.e., for feed compositions $x_0^B = [0.45, 0.55]$ and $x_0^B = [0.55, 0.45]$), since we assume x_0^B will be the more frequent perturbation. The difference between the final tray temperature in the composition-perturbed case and the final tray temperature in the nominal case was taken as a decision variable for tray selection. Tray #4 presented the highest absolute difference value, and, therefore, the evolution of the temperature at this stage was selected as the reference trajectory for the closed-loop correction.

Figure 6(b) shows the behavior of several temperatures located immediately above the reboiler when the column, separating the binary mixture, is operated near the condition of minimum energy demand. Note that the temperatures of these trays approach the reboiler temperature indicating the existence of a pinch, whose instantaneous composition approaches the composition of the reboiler content. From the inspection of Figure 6(a),(b), it is clear that the minimum energy demand condition requires a pinch zone at the rectifier lower end, while the top maintains the temperature corresponding to the light-component. This operating condition is the opposite to that of total-reflux during the start-up period, where the energy demand condition is well above the minimum, and the pinch zone is at the top of the rectifier.

For the ternary system, the method used to select the reference temperature was to determine the tray temperature that suffers the most important change when the operation corresponding to the feasible recipe goes from total reflux to the final light component stripping condition. Though alternative techniques can be proposed to select this temperature, the maximum sample variance was successfully used for this purpose. Tray #16 presented the highest value, and, therefore, the evolution of the temperature at this stage was selected as output-reference trajectory.

As described before, the criterion used to determine the controlled temperature in the binary case focus on rejecting the feed composition disturbance, while for the ternary system the

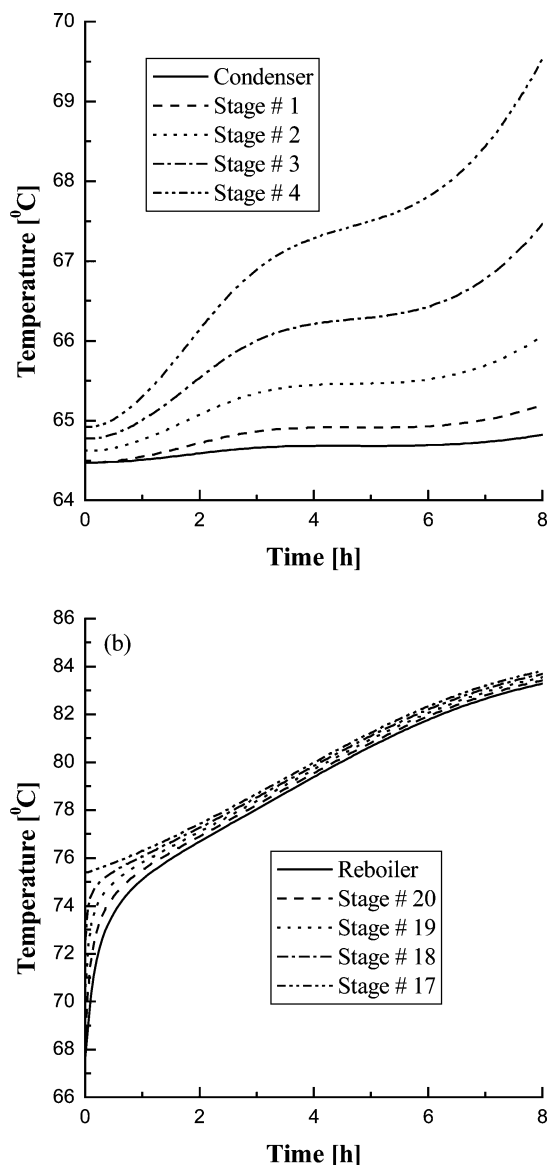


Figure 6. (a). System methanol–isopropyl alcohol. Evolution of temperatures in the condenser and at the column top. (b). System methanol–isopropyl alcohol. Evolution of temperatures in the reboiler and at the column bottom.

maximum nominal variability is observed. Though the problem of temperature selection might deserve a more detailed analysis, the closed loop results obtained in our simulation studies does not show that one method prevails on the other. In part, this could be explained by similarities in the dynamic behaviors of the selected stages and the compensating effect of an appropriate controller tuning for each alternative.

The rigorous nonlinear model of the distillation column used in the simulations of both the binary and ternary mixtures provides a quite realistic dynamic behavior. Besides the above-described control system, this simulation analysis assumes that, in accordance with column diameter, $V = 30$ kmol/h is the maximum vapor load all along the rectification time and that the distillate is maintained under negligible subcooling at the condenser outlet. However, for simplicity and because they are not part of the main issue in this paper, the necessary instrumentation to sustain maximum vapor load and a proper subcooling in the condenser are not indicated in Figure 5.

Summarizing, open-loop simulations with a validated model are used for both selecting the appropriate tray temperature as

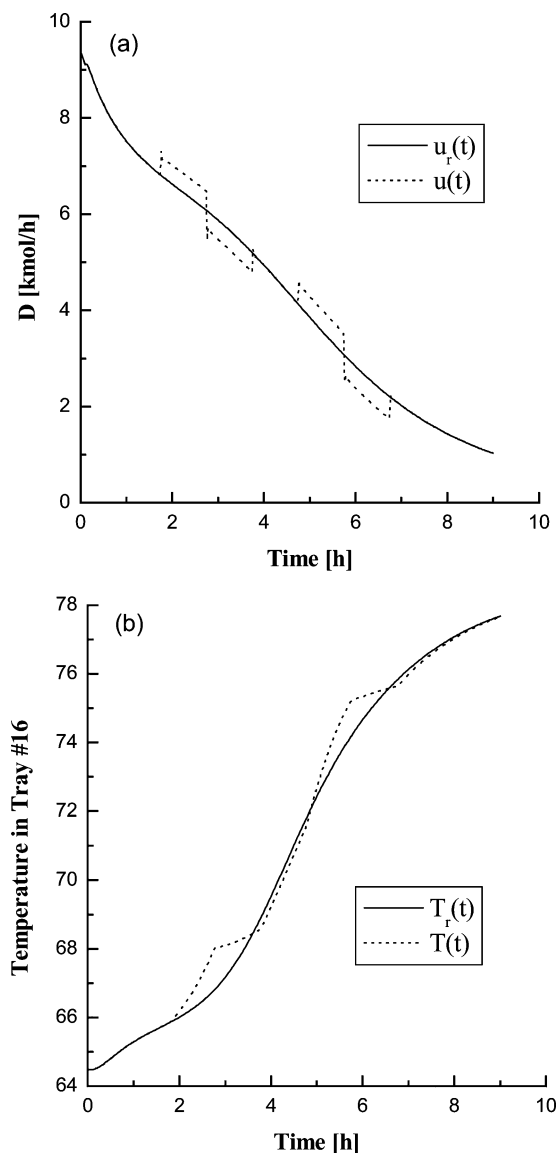


Figure 7. (a). System methanol–ethanol–isopropyl alcohol. Input-reference and input-perturbed trajectories. (b). System methanol–ethanol–isopropyl alcohol. Output-reference and output-perturbed trajectories.

controlled variable and adjusting (if necessary) the reference trajectory until the desired product purity and recovery is achieved.

5. Referential Process-Reaction Curve and Controller Tuning

The referential reaction method basically consists in using a standard or nominal time evolution as reference dynamic to determine by contrast the effect of changing the manipulated variable (the distillate flow rate) on the controlled one (a tray temperature). For the sake of conciseness, only graphics corresponding to results of the ternary mixture will be presented.

Figure 7(a) shows both the input-reference trajectory $u_r(t)$ and the input-perturbed one $u(t)$ made by step changes in the distillate flow rate in such a way that the difference of accumulated amount of distillate is finally compensated. There is an initial step change $+0.5$ kmol/h 2 h after start-up; then, at $t = 3$ h there is a -1.0 kmol/h step change, and finally the distillate flow rate is taken back to the nominal trajectory by another $+0.5$ kmol/h change at $t = 4$ h. The experience is repeated at the time 5, 6, and 7 h, respectively, but just the

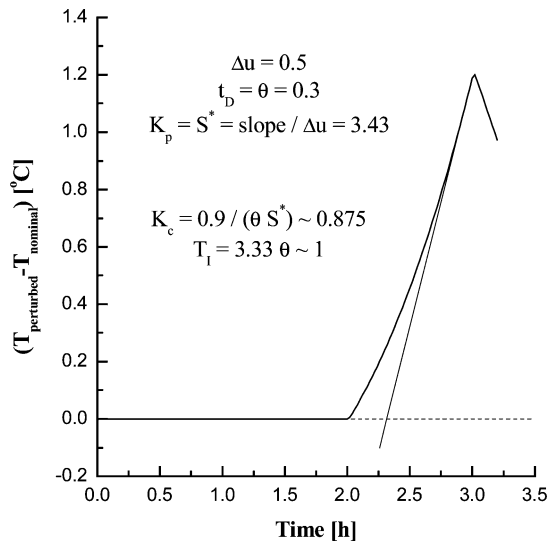


Figure 8. System methanol–ethanol–isopropyl alcohol. Referential process-reaction curve and estimation of parameters.

referential response corresponding to the first disturbance is used because that response exhibited higher gain and time delay and consequently provides a more robust controller tuning.

The corresponding output-reference trajectory $T_r(t)$ and the perturbed response $T(t)$ are shown in Figure 7(b). For each input change, a referential process-reaction curve or referential temperature evolution is determined by the difference between the perturbed response $T(t)$ and the reference $T_r(t)$ on the assumption that no other disturbance has occurred. From this curve it is possible to determine the slope, the normalized slope (i.e., slope/input change), and the time delay and, therefore, to calculate the parameters for a PI controller using the tuning relationships developed by Ziegler and Nichols;⁴ in particular, for PI controllers (note that $S^* = K_p$ and $\theta = T_d$)

$$K_c = \frac{0.9}{\theta \times S^*} \quad (11)$$

$$T_I = 3.33 \times \theta \quad (12)$$

Figure 8 shows the corresponding referential process-reaction curve, together with the necessary data to calculate the controller parameters, $K_c = 0.875 \text{ kmol}/(\text{h } ^\circ\text{C})$ and $T_I = 1.00 \text{ h}$.

In an analogous way, two referential reaction curves were obtained for the binary system: one starting at the operating time 1.75 h and the other one 4.75 h after the start-up. As with the ternary system, when following the $R_{\min}(t)$ trajectory, the pinch zone moves quite rapidly from the top of the column initially working at total-reflux to the desired operating condition which has the pinch zone right above the reboiler. Then, the controller settings are determined using the second referential curve because by that time (4.75 h) the tray #4 is far from the pinch zone, and consequently its temperature shows a faster (higher gain) dynamic response. After obtaining the corresponding process reaction curve, the following values for controller parameters were calculated, $K_c = 3 \text{ kmol}/(\text{h } ^\circ\text{C})$ and $T_I = 0.16 \text{ h}$.

6. Closed-Loop Rigorous Simulation

In order to show the robustness of the tuning approach, closed-loop simulations were performed for different initial feed compositions since the composition of the mixtures charged into the reboiler typically varies from batch to batch.

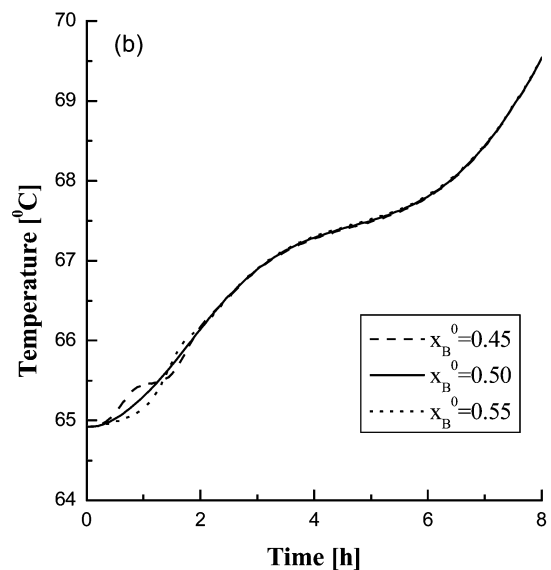
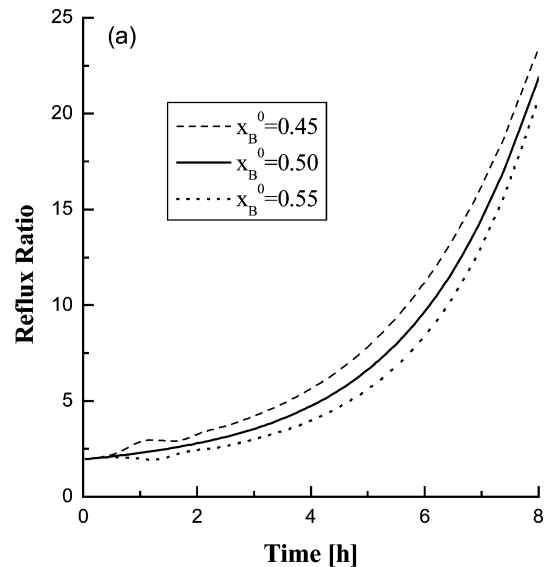


Figure 9. (a). System methanol–isopropyl alcohol. Closed-loop evolutions of the reflux ratio for different initial feed compositions. (b). System methanol–isopropyl alcohol. Closed-loop tracking response obtained for different initial feed compositions.

Binary System. Figure 9(a) shows the evolution of the reflux ratio as a result of control actions on the distillate flow rate for the nominal (in solid line) and perturbed cases, i.e., for $x_0^B = [0.45, 0.55]$ and $x_0^B = [0.55, 0.45]$. In this way, the control system maintains the final mean distillate composition about 99% of methanol.

Note in the same figure that the evolution of the reflux ratio corresponding to $x_0^B = [0.55, 0.45]$ suggests that feed mixtures richer in the light component than the nominal case should be operated at lower reflux ratios, and, hence, a greater amount of distillate should be gained at the distillate product tank and vice versa for mixtures containing lower amounts of the light species. This behavior can be understood by analyzing the results obtained from the conceptual model for separations at a constant reflux ratio as shown in Table 3. The table shows the minimum reflux ratio needed to recover 95% of the light component and 1% of the heavy species in the distillate of a column operated at constant reflux. The lower the amount of light component in the still, the greater the minimum reflux necessary to achieve the prefixed separation. Another way to reach the same conclusion is by calculating the quasi-optimal reflux ratio (and

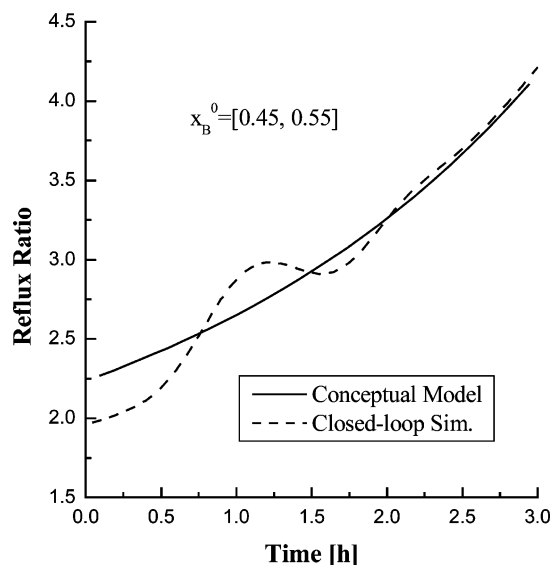


Figure 10. System methanol–isopropyl alcohol. Reflux-ratio evolutions obtained from the conceptual model and the closed-loop simulation for the perturbed initial composition.

Table 3. Binary System: Minimum Reflux Ratio Values for Different Feed Compositions^a

x_B^0	conceptual model	
	R_{\min}	η
[0.45, 0.55]	17.248	0.43
[0.50, 0.50]	14.783	0.47
[0.55, 0.45]	12.716	0.52

$$^a \sigma_{\text{MeOH}}^D = 0.95; \sigma_{2\text{Pr-OH}}^D = 0.01.$$

hence, the distillate flow rate) for the perturbed cases. The evolution corresponding to $x_0^B = [0.45, 0.55]$ ([0.55, 0.45]) is above (below) the evolution corresponding to the nominal case.

Figure 9(b) compares the closed-loop responses of the temperature in tray #4, when the initial feed composition (light component) is 0.45 and 0.55 instead of 0.5. The initial difficulty for tracking the reference temperature appears for any tray and arises from the insensitivity of these variables when a pinch zone is in close proximity. This explains that in spite of selecting the most sensitive tray temperature to disturbances in feed-composition, an important initial lag forces the controller to overactuate when leaving the total reflux condition characterized by a pinch zone at the top.

Figure 10 compares the reflux-ratio evolution followed by the closed-loop simulation when the initial still composition is perturbed to $x_0^B = [0.45, 0.55]$, with the theoretical reflux-ratio trajectory corresponding to that perturbed composition. This last curve was calculated after the closed-loop simulation was performed and is the trajectory that would be implemented if $x_0^B = [0.45, 0.55]$ were the nominal initial composition. In other words, the closed-loop tracking of the reference temperature corresponding to the nominal case $x_0^B = [0.50, 0.50]$ leads to a reflux-ratio evolution that approaches the quasi-optimal reflux-ratio for the actual composition $x_0^B = [0.45, 0.55]$. This result validates the proposed control system as an adequate approach to compensate the effect of working with a different initial composition of binary mixtures. From the inspection of Figure 10 we can also conclude that in this application example, about 2 h are needed for the PI controller with Ziegler-Nichols tuning to sustain the appropriate trajectory. This happens during the same time that the temperature of the selected tray belongs or is close to the initial pinch zone. Similar

Table 4. Ternary System: Composition and Holdup for Nominal and Perturbed Cases

	feed composition	feed amount [kmol]
nominal case	[0.500, 0.250, 0.250]	90.00
case I	[0.525, 0.250, 0.225]	92.32
case II	[0.475, 0.250, 0.275]	89.07
case III	[0.500, 0.275, 0.225]	91.53
case IV	[0.500, 0.225, 0.275]	89.92

Table 5. Ternary System: Performance Comparison between Simulations for Cases I and II and Simulation of the Nominal Case

	product amount [kmol]	product purity [mol %]
case I – open loop	40.03	99.99
case I – closed loop	43.78	99.76
nominal case	40.03	99.87
case II – open loop	40.03	97.61
case II – closed loop	37.19	99.91

conclusions can be reached by analyzing the results obtained for the mixture rich in the light component.

Ternary System. In order to show the robustness of the tuning approach, closed-loop simulations were performed for four different cases. Table 4 shows both the initial still composition and holdup for each case. Each still molar holdup was calculated taking into account a constant value for the volume of the vessel.

An interesting way to evaluate the controller performance is through the analysis of the results of simulations with (closed-loop) and without (open-loop) tracking the temperature in tray #16. An open-loop simulation using the mixture corresponding to case I, for example, means the implementation of the nominal feasible reflux-ratio recipe by the simple open-loop ratio controller associated with the condenser-drum level control. On the other hand, a closed-loop simulation implies that, in addition to the open-loop ratio control, the feedback controller actuating on the distillate flow rate attempts to track the desired nominal trajectory for the temperature in tray #16. It is clear, from inspection of results in Table 5, that this controller is able to differentiate between separations that are easier (case I) and more difficult (case II) than the nominal case, giving rise to high purity products in amounts above (case I) and below (case II) that corresponding to the nominal case, respectively.

Several testing simulations indicated that when the light component concentration is not perturbed, the controller effort to keep the desired operating conditions is not significant as compared with opposite cases. Hence, the closed loop responses obtained from cases III and IV are not reported here since those evolutions show little difference from the nominal one. This basically means that tracking the trajectory for the temperature in tray #16 was not a challenge for the controller in the mentioned cases.

Figure 11(a),(b) summarizes the results obtained for both cases. The very high purity reported in Table 5 for open-loop operation in case I can be explained by analyzing the behavior of the temperature in tray #16. As shown in Figure 11(a), direct implementation of the nominal recipe gives rise to a temperature evolution that is almost constant with a temperature near that corresponding to pure methanol. This behavior remains for the trays above stage #16 and indicates the existence of a pinch zone and, therefore, a waste of energy. Closed-loop operation, on the other hand, ensures a high purity distillate with increased recovery as a result of a reflux ratio evolution below the nominal as depicted in Figure 11(b).

For case II, the temperature evolution for open-loop operation is well above the nominal one. This behavior of the temperature

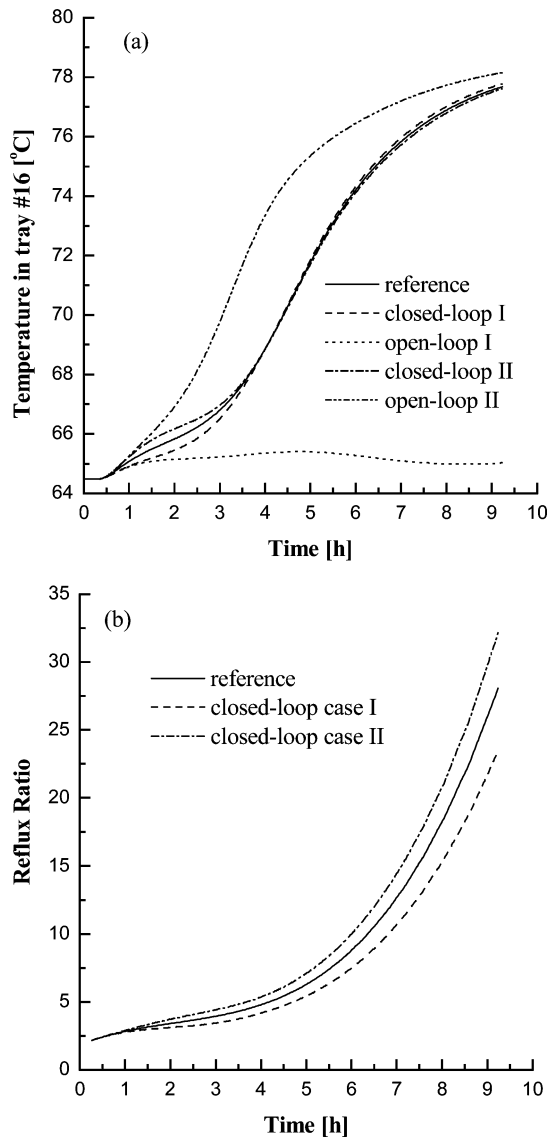


Figure 11. (a). System methanol–ethanol–isopropyl alcohol. Temperature versus time. (b). System methanol–ethanol–isopropyl alcohol. Reflux ratio versus time.

translates into a decrease of product purity with respect to the nominal case. Closed-loop operation prevents such a situation by increasing the reflux ratio above the reference trajectory.

7. Conclusions

Operating batch distillation columns near minimum energy demand conditions is a challenging task due to the inherently high-energy consumption that characterizes the majority of the mixture separations, particularly when obtaining high purity products. As operating near optimal conditions requires a variable distillate flow rate policy, it is advisable to design a control system capable to track the desired operating condition for every batch. This may involve a feedback strategy to absorb perturbations like changes in the initial feed composition from batch to batch.

In this contribution, a novel method is presented that combines the capability of conceptual models based on pinch analysis for predicting the conditions to operate near minimum energy demand, with the simplicity of the referential reaction method as controller tuning technique to track a desired quasi-optimal temperature trajectory.

The results obtained for both binary and ternary mixture of alcohols clearly show the potentiality of the proposed approach and should motivate further research efforts involving problems such as the effect of noise or disturbances in the collected data or the extension to the whole batch operation including both main and intermediate cuts, with or without a chemical reaction in the still.

Acknowledgment

This work received financial support from Agencia Nacional de Promoción Científica y Tecnológica, CONICET and Universidad Nacional del Litoral.

Appendix

Development of Differential Equations To Model the Still.

Assuming negligible changes in tray holdups, the balance of component i , for the whole batch column, is

$$\frac{d(Mx_i^B)}{dt} = -Dx_i^D \quad (\text{A1})$$

where M is the reboiler holdup in number of moles, x_i^B is the instant composition in the reboiler, and x_i^D is the instant composition in the distillate stream. Besides, we define the *fractional recovery* of the i component as

$$\sigma_i^D = \frac{(M_0x_{i0}^B - Mx_i^B)}{M_0x_{i0}^B} \quad (\text{A2})$$

Now, eq A1 can be rewritten as a function of the component recoveries as follows:

$$\frac{d\sigma_i^D}{dt} = \frac{Dx_i^D}{M_0x_{i0}^B} \quad (\text{A3})$$

The total molar balance in the product vessel gives

$$\frac{dn^D}{dt} = D \quad (\text{A4})$$

and defining the *rectification advance* as

$$\eta = \frac{n^D}{M_0} = \frac{M_0 - M}{M_0} \quad (\text{A5})$$

then, an equation in terms of component recoveries and rectification advance is obtained by replacing eqs A4 and A5 into eq A3:

$$\frac{d\sigma_i^D}{d\eta} = \frac{x_i^D}{x_{i0}^B} \quad (\text{A6})$$

Salomone⁷ developed this equation, which has the advantage of considering the component recoveries and the rectification advance instead of the component mole fractions. It is worth recalling that the design specifications in batch distillations are normally given in terms of component recoveries and that the vapor flow rate does not need to be specified at this level.

Note now that for each value of the rectification advance and set of values of component recoveries it is possible to calculate the instantaneous still composition. By reordering eq A2, eq A7 is obtained

$$Mx_i^B = M_0x_{i0}^B(1 - \sigma_i^D) \quad (\text{A7})$$

and using again (A5)

$$x_i^B = x_{i0}^B \frac{(1 - \sigma_i^D)}{(1 - \eta)} \quad (\text{A8})$$

This equation gives the instantaneous reboiler composition needed for calculating the distillate composition (if the column is operated at constant reflux) or for calculating the instantaneous reflux ratio (if the column is operated at constant distillate composition).

Nomenclature

CM = conceptual model
 K_c = controller gain [kmol/(h °C)]
 M, M_0 = still holdup, fresh feed [kmol]
 n_D = product vessel holdup [kmol]
 N = general nonlinear dynamic
 R_{\min}^{inst} = instantaneous minimum reflux ratio
 S^*, K_p = normalized slope or process gain
 t = time [h]
 T_D = time delay [h]
 T_I = integral time [h]
 $T_{\text{ref}}(t), T(t)$ = output-reference trajectory, output-perturbed response
 $u_{\text{ref}}(t), u(t)$ = input-reference trajectory, input-perturbed trajectory, control variable
 V = vapor flow rate [kmol/h]
 x_i^B, x_{i0}^B = instantaneous mole fraction of component i in the still, initial value
 x_B = vector of instantaneous still compositions
 x_D = vector of instantaneous distillate compositions
 x_i^D = instantaneous mole fraction of component i in the distillate
 x_N = vector of instantaneous compositions at lower column end
 x_i^N = instantaneous mole fraction of component i in the liquid stream leaving the rectifier lower end
 x_p = vector of instantaneous pinch compositions

y = controlled output

y_{xB}^* = vector of instantaneous vapor compositions in equilibrium with x_B

y_{ref} = reference trajectory for the output variable

y_i^B = instantaneous vapor mole fraction of component i in equilibrium with x_i^B

Greek letters

η = rectification advance

θ = time delay [h]

σ_i^D = fractional recovery of component i in the distillate

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Received for review January 12, 2007

Revised manuscript received May 11, 2007

Accepted June 18, 2007

IE070075C