

COMBINING CONCEPTUAL AND REFERENTIAL REACTION METHODS FOR BATCH DISTILLATION CONTROL

Jacinto L. Marchetti
José Espinosa

INTEC-CONICET-UNL, Güemes 3450, 3000 Santa Fe, Argentina.
INGAR-CONICET, Avellaneda 3657, S3002 GJC, Santa Fe, Argentina.

Abstract: The referential reaction empirically determines reduced-order models for designing and tuning feedback controllers dedicated to tracking non-stationary conditions in batch processes. Defining a feedback control system for a batch distillation implies first the selection of an appropriate tray temperature evolution. Then, the dynamics associated to the manipulated variable (distillate flow rate) is isolated from the main time-variable behavior desired for the operation, and used with available tuning rules for integrating systems. The effectiveness of this procedure is illustrated by implementing a feasible quasi-optimal recipe derived from pinch theory and valid for multicomponent mixtures.
Copyright © 2007 IFAC

Keywords: Computer-aided design, batch control, distillation columns.

1. INTRODUCTION

During the last decade, several authors worked in the development of methods to cope with the 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 (Espinosa, *et al.*, 2004; Brüggemann, *et al.*, 2004).

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 process and tuning rules (Ziegler and Nichols, 1942) 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 (see the Appendix for more details), like those occurring in batch distillation columns. The present application is partially motivated by a previous successful experience in tracking a desired temperature evolution of a bioreactor (Marchetti, 2004). Here, the separation of the light component from its ternary mixture of alcohols is selected as case study.

2. CONCEPTUAL MODEL

In order to perform a simulation run of a batch rectifier with an infinite number of stages, two design variables have to be selected in addition to the feed composition. We select the distillate composition plus the final rectification advance. Then, the recoveries of the components in the distillate as a function of rectification advance are estimated by integrating 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 other variables such as recoveries of the components in the residue, residue compositions and temperature can be calculated as a function of component recoveries in the distillate and rectification advance. The instantaneous minimum reflux ratio $R_{min}(t)$ to achieve the pre-fixed distillate composition is estimated from linearization of column profiles at instantaneous still composition x_B , which requires solving an eigenvalue problem of the Jacobian of the equilibrium function in x_B as explained elsewhere (Offers, *et al.*, 1995; Espinosa and Salomone, 1999).

The key ingredient of the model is illustrated in Figure 1(a) for the mixture methanol-ethanol-isopropanol. Figure 1(a) shows the mass balance line given by the desired distillate composition x_D (pure methanol), the vapor feed to the rectifier (vapor y_{xB}^* in equilibrium with the instantaneous still composition x_B) and the composition x_N of the liquid leaving the rectifier lower end. The last composition is calculated as the intersection between the mass balance line and the line formed by the two controlling pinch points; i.e; x_B and x_P^H . Figure 1(a) also shows the internal profile calculated through simulation in Hysys (1999). The simulated internal profile approximating x_B is contained in a line very close to that estimated by linearization of column profiles at instantaneous still composition (Espinosa and Salomone, 1999) and therefore, good agreement between rigorous and simplified simulation is found.

Figure 1(b) shows the evolution of the minimum reflux ratio necessary to achieve high purity methanol at the top of a column having an infinite number of stages. The still is charged with 90 kmol of a mixture with composition 0.5 methanol, 0.25 ethanol, 0.25 isopropanol and the vapor flow rate V is 30 kmol/h. The predicted recovery of methanol at column top is 94 %.

3. 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 first approximation to the variable reflux policy to be followed when considering a column with a finite number of trays and holdup as shown in Brüggeman, *et al.* (2004) (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. Three open-loop simulations of a column with 30 stages were performed to determine a feasible recipe. Whilst implementation of the nominal recipe (“recipe for 0.5” in Figure 1(b)) produced a low purity distillate in maximum amount [97.16%, 42.3 kmol], the reflux ratio evolution predicted by the conceptual model for composition and holdup in the still corresponding to the end of the start-up phase (“recipe for 0.45” in Figure 1(b)) gives rise to a high purity distillate with minimum amount [99.99%, 35.40 kmol]. In the last case, a pinch at column top is maintained through the whole simulation indicating a waste in energy consumption.

Both trajectories act as limiting curves. Whilst the evolution calculated for the initial still composition does not allow to achieve a high purity product because the column has a finite number of stages, the trajectory for “0.45” is above the feasible one because at this time all holdups in column trays and condenser are enriched in the light component. Therefore, a recipe in between was selected and implemented in the simulation environment. Figure 1(b) shows the feasible recipe adopted due to both its adequate distillate purity and recovery [99.87%, 40.03 kmol]. Figure 2(a) shows the evolution of the light species composition along the column. The behavior of the compositions in Figure 2(a) is in stark contrast to that of the second recipe, where 16 stages form a pinch zone, as shown in Figure 2(b).

In order to define a feedback control system, the selection of an appropriate tray temperature evolution (output-reference trajectory) must be done once the feasible recipe is established. The reference trajectory to operate this column is implemented by a simple open-loop ratio controller associated to the condenser-drum level control as shown in Figure 3. The distillate flow rate D (input-reference trajectory) has to adapt to the flow measures of the reflux stream L_0 , which in turn controls the liquid level in the reflux drum.

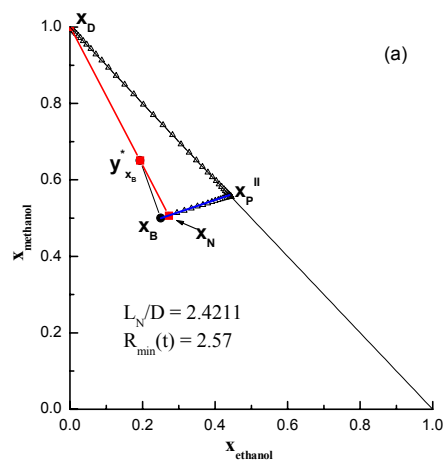


Fig. 1. System MeOH-EtOH-IPA. (a) Instantaneous minimum reflux

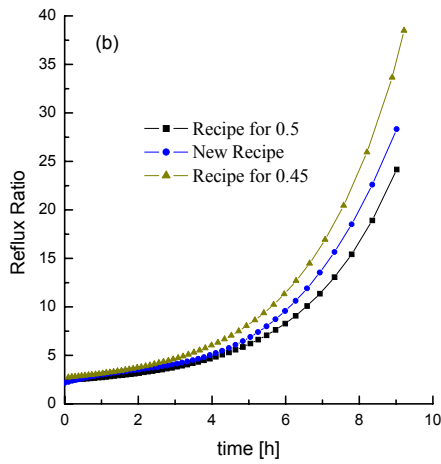


Fig. 1. System MeOH-EtOH-IPA. (b) reflux ratio versus time.

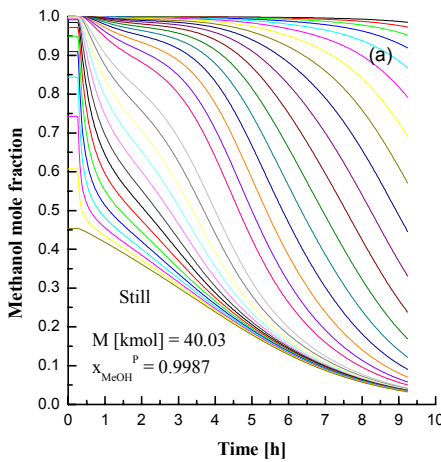


Fig. 2. (a) Evolution of the light component composition along the column corresponding to the feasible recipe.

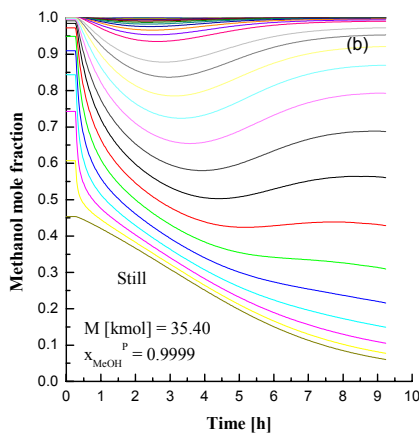


Fig. 2. (b) Evolution of the light component composition along the column corresponding to the recipe calculated for composition of the still mixture at the end of the start-up phase.

In this work, the method used to select the reference temperature was to determine the tray temperature that suffers the most important change when the operation 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 (see Figure 4(b)).

The rigorous nonlinear model of the distillation column used in these simulations provides a quite realistic dynamic behavior. Besides the above described control system, this simulation analysis assumes that $V = 30$ kmol/hr is the maximum vapor load all along the rectification time, and that the distillate is maintained under negligible sub cooling 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 sub cooling in the condenser are not indicated in Figure 3.

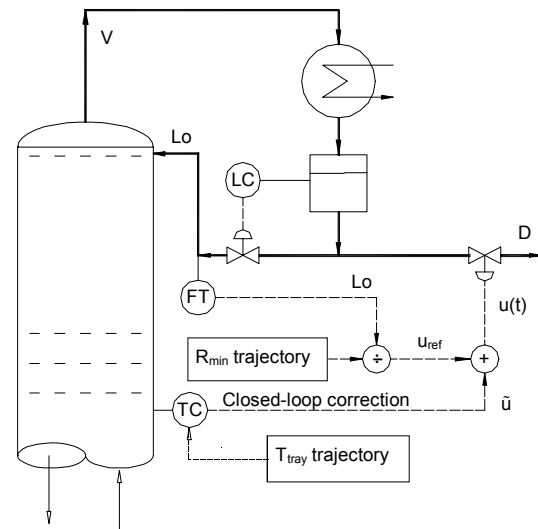


Fig. 3. Temperature tracking control system.

4. REFERENTIAL PROCESS-REACTION CURVE AND CONTROLLER TUNING

The method basically consists of using a standard or nominal time evolution as reference dynamic to determine by contrast the effect of changing the manipulated variable (distillate flow rate) on the controlled one (temperature in Tray #16). Figure 4(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. The corresponding output-reference trajectory $T_r(t)$ and the perturbed response $T(t)$ are shown in Figure 4(b). For each initial input change, a referential process-reaction curve or referential temperature evolution can be 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. See Figure 5. Then, the parameters of an integrating system can be estimated from this curve, i.e., the normalized slope (slope / input change) and the time delay, and therefore, this allows the estimation of appropriate parameters for a

PI controller using the tuning relations developed by Ziegler and Nichols (1942).

Figure 4(a) shows the way the nominal distillate flow-rate trajectory was perturbed. There is an initial step change +0.5 kmol/hr 2 hours after start-up; then, at $t = 3$ hr there was a -1.0 kmol/hr step change and finally the distillate flow rate was taken back to the nominal trajectory by another +0.5 kmol/hr change at $t = 4$ hr. The experience was repeated at the times 5, 6 and 7 hr respectively, but just the referential response corresponding to the first disturbance was used to adjust the controller. Figure 5 shows the referential process-reaction curve corresponding to the first step change only, together with the necessary data to calculate the controller parameters, $K_c = 0.875$ °C/kmol and $T_I = 1.00$ hr.

Since different referential reaction curves can be obtained at different points in the trajectory, a family of models (see A6) and a set of different controller parameters can be determined. Thus, selecting the lowest controller gain and the highest integral time should provide robustness along the entire trajectory.

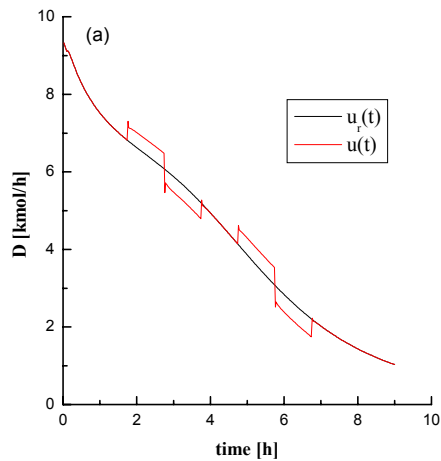


Fig. 4. (a) Input-reference and input-perturbed trajectories.

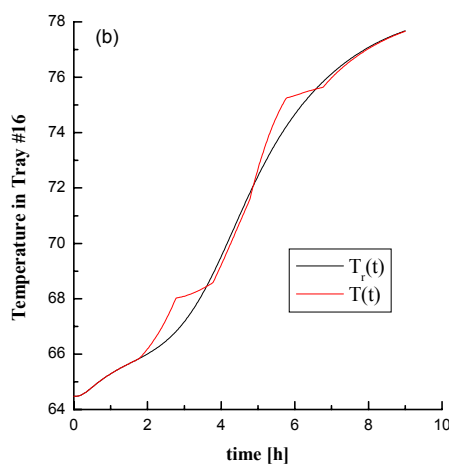


Fig. 4 (b) Output-reference and Output-perturbed trajectories.

5. CLOSED-LOOP SIMULATION

In order to show the robustness of the tuning approach, closed-loop simulations were performed for four different cases. Table 1 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. As expected, results of closed-loop simulations of the first cut for cases III and IV did not present any noticeable deviation with respect to the nominal case and therefore, only cases I and II will be analyzed in detail.

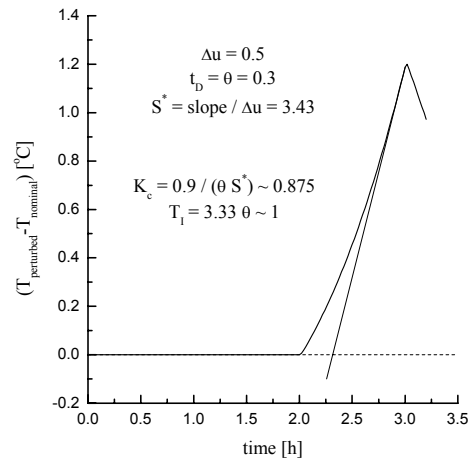


Fig. 5. Referential process-reaction curve and estimation of parameters.

An interesting way to evaluate the performance of the controller is through the analysis of the results of simulations with (closed-loop) and without (open-loop) the tracking of the temperature in tray #16. An open-loop simulation of the mixture corresponding to case I, for example, means the implementation of the nominal feasible recipe by the simple open-loop ratio controller associated to the condenser-drum level control. On the other hand, a closed-loop simulation implies a continuous change in the manipulated variable (distillate flow rate) to track the desired trajectory for the temperature in tray #16. It is clear, from inspection of results in Table 2, that the controller is able to differentiate between separations that are more 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) the corresponding to the nominal case, respectively.

Figures 6(a) and (b) summarize the results obtained for both cases. The very high purity reported in Table 2 for open-loop operation in case I can be explained by analyzing the behavior of the temperature in tray #16. As shown in Figure 6(a), direct implementation of the nominal recipe gives rise to a temperature evolution that is almost constant with a temperature near the 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 6(b).

Table 1 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 2 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 | 99.989 |
| Case I – Closed loop | 43.78 | 99.763 |
| Nominal case | 40.03 | 99.872 |
| Case II – Open loop | 40.03 | 97.606 |
| Case II – Closed loop | 37.19 | 99.913 |

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

5. CONCLUSIONS AND FUTURE WORK

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 the first cut of a 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.

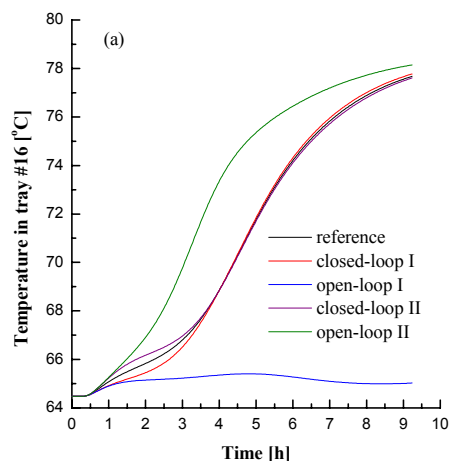


Fig. 6. Simulations results (a) Temperature versus time.

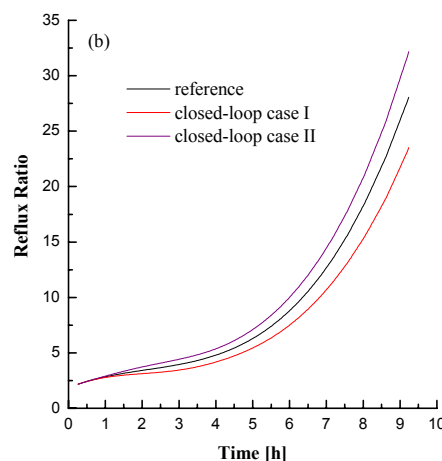


Fig. 6. Simulations results (b) Reflux ratio versus time.

6. ACKNOWLEDGEMENTS

The authors would like to acknowledge the financial support provided by ANPCyT, CONICET and UNL, Argentina.

REFERENCES

- Brüggemann, S., J. Oldenburg and W. Marquardt (2004). Combining conceptual and detailed methods for batch distillation process design. In: *FOCAPO 2004*, 247-250.
- Espinosa, J. and E. Salomone (1999). Minimum reflux for batch distillations of ideal and nonideal mixtures at constant reflux. *Ind. Eng. Chem. Res.*, 38 (7), 2732-2746.
- Espinosa, J., E. Salomone and O. Iribarren (2004). Computer-Aided Conceptual Design of Batch Distillation Systems. *Ind. Eng. Chem. Res.*, 43, 1723-1733.
- Hysys User Manual (1999). *Hyprotech Ltd.*, Calgary, Canada.

- Marchetti, J. L. (2004). Referential process-reaction curve for batch operations. *AIChE J.*, 50, 3161-3168.
- Offers, H., R. Düssel and J. Stichlmair (1995). Minimum energy requirement of distillation processes. *Comput. Chem. Eng.*, 19, S247-S252.
- Ziegler, J. G. and N. B. Nichols (1942). Optimum Settings for Automatic Controllers. *Trans. ASME*, 64, 759-768.

APPENDIX: REFERENTIAL MODELLING

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 tuning are determined by the residual dynamics associated to small changes of the main control variable driving the operation.

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 to 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 to produce a temporary but measurable change in the output trajectory around the nominal evolution. The convenience of using this type of disturbance 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 it 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

$$N : u \mapsto y = N(u) \quad (A1)$$

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 as

$$N : u_{ref} \mapsto y_{ref} = N(u_{ref}). \quad (A2)$$

Consider now the difference

$$y - y_{ref} = N(u) - N(u_{ref}). \quad (A3)$$

Assuming that N captures most of the non-linear 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

$$G : \tilde{u} \mapsto \tilde{y} = G\tilde{u} \quad (A4)$$

where the aim of the linear transfer operator G is to describe small dynamics around the nominal or desired process trajectory due to a residual non linearity or to bounded disturbances. In this way, referential reaction curves (Marchetti, 2004) obtained at different batch progress may help to determine a nominal linear model \tilde{G} plus a global uncertainty Δ such that,

$$G = \tilde{G} + \Delta \quad (A5)$$

$$\Pi \triangleq \{G : |\Delta| = |G - \tilde{G}| \leq \bar{\Delta}\} \quad (A6)$$

where Π denotes the family of models describing G . Figure A1 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 it is not necessary knowing $N(u_{ref})$, and the final control structure takes the form given in Figure A2.

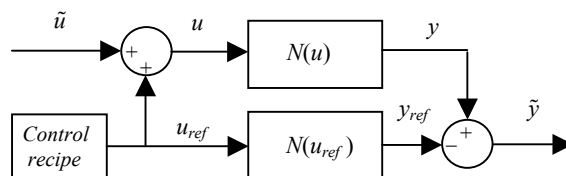


Figure A1: Non linear compensation in referential modelling control.

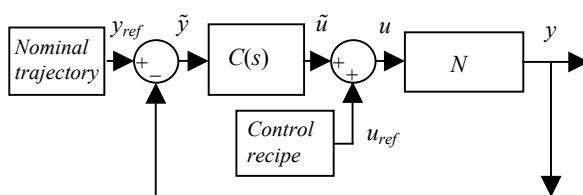


Figure A2: Realization structure of referential control.

Hence, under this control structure, the controller $C(s)$ should see only the residual dynamics G all along the progress of the batch process.