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PROCESS DESIGN AND CONTROL

Composition Control in Batch Distillation: Binary and Multicomponent Mixtures

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A strategy is proposed for the control of constant-distillate-composition operations of batch distillation columns. The proposed control law is derived in the framework of Nonlinear Internal Model Control. The distillate composition is estimated from selected tray temperature measurements by an observer. The same observer provides the estimate of the system states that are necessary for the computation of the control algorithm. Results are provided for binary and multicomponent separations of ideal and nonideal systems. Several sources of process/model mismatch are considered in order to provide a realistic test scenario for the proposed procedure. It is shown that the proposed controller ensures fast and smooth transition from the startup phase to the production phase, and tight composition control until the end of the operation. Moreover, the tuning of the controller is performed very easily. Finally, the on-line computation of the control law is straightforward, which is a promising result toward practical implementation of the proposed method.

1. Introduction

A remarkable shift toward batch production technologies has been observed during the past decade. One of the reasons is that the market demand is changing much more frequently than in the past, and product specification requirements are becoming stricter and stricter. The flexibility of a batch process is such that these short-term changes and severe purity requirements can be accommodated. Moreover, because the production amounts in a batch process are usually small, the raw materials inventories can be kept to a minimum, which often results in an economic incentive.

Within a batch process, batch distillation is a ubiquitous way of physical separation of binary and multicomponent mixtures. Many different components and multiple product fractions can be separated in the same batch column, and at a much lower capital cost than would be associated with the relevant train of continuous columns. The streams coming from a batch column may be themselves the desired products; in such a case, they can be directly placed in the market. Alternatively, the distillation may serve as a way of recovering one or more components in order to recycle them to a certain stage of the batch process, or as a method of removing an undesired component from a byproduct stream in order to comply with the regulations on byproduct discharge. However, no matter what the objective of the distillation, the purity requirements are normally strict, and a significant economic penalty exists if the specification requirements are not met. The development of accurate methodologies for tight composition control in batch columns is necessary.

Traditionally, a batch rectifier can be operated in two ways: constant reflux ratio and constant distillate composition. In the first case, the reflux ratio (or the reflux rate) is kept constant throughout the distillation, which results in a continuously changing distillate composition. This is inherently an open-loop strategy. Therefore, unless a proper feedback from the plant is considered, the average product composition is known only at the end of the batch. Therefore, if an off-specification product were obtained, the batch would have to be blended or rerun, with a significant economic loss.

Constant-distillate-composition operation is more difficult. In fact, to keep the distillate composition to the desired value throughout the whole batch, one needs to continuously adjust (increase) the reflux rate. On the other hand, the product composition can be controlled accurately during batch production, i.e., this mode is inherently a feedback operation.

A third operating mode, optimal operation, has gained attention in recent years, and it is determined by maximizing off-line a prespecified profit function. This operating procedure often results in a piecewise constant-reflux-ratio operation, and is normally implemented in an open-loop fashion. Therefore, the drawbacks of optimal operation are somewhat similar to those of constant reflux operation. In fact, the closed-loop implementation of open-loop optimal profiles is still an open issue, at least from a control standpoint.[1] Usually, the optimal profiles cannot be tracked with conventional linear controllers, because of the nonlinear and time-varying nature of the process.[2]

This work provides a framework for obtaining tight distillate composition control in batch rectifiers. It has been recognized[3] that controlling the distillate purity in a batch distillation operation is difficult, even when

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binary mixtures are used. This is due to the inherent dynamic and time-varying nature of batch distillation. However, recent developments of nonlinear control theory[4,5] have made attractive techniques available for the control of a number of nonlinear processes. The nonlinear control strategy sketched by Barolo and Berto[6] is complemented in this paper and extended to both nonideal systems and multicomponent mixtures, thus resulting in a powerful, yet simple to implement, control strategy. The paper is organized as follows. First, an overview of the previous findings in the field of composition control of batch distillation is provided. Then, the process is defined and conventional temperature control is considered. The development of a composition estimator is then illustrated for both binary and ternary mixtures. Nonlinear Internal Model Control[7] (NIMC) is then briefly reviewed, and a control law is derived for binary separations. Results from the NIMC-based control law are compared in the following section to those provided by a conventional composition controller for a binary mixture; relationships with another nonlinear control structure are also clarified. Finally, the NIMC strategy is extended to multicomponent mixtures, and a test case is analyzed on a ternary separation.

2. Summary of Previous Findings

Conventional strategies for constant composition operation of batch distillation columns usually comprise a total-reflux startup phase, which is carried out until the process approaches the steady state. Then, a controller is switched on in order to drive the distillate composition to the desired value and maintain it at the desired level (by manipulating the reflux rate, for example). Finally, the operation is stopped when the reflux ratio reaches a prespecified limiting value.

Early studies on the composition control of batch distillation[8,9] focused on a control strategy similar to that used for one-point composition control in continuous columns. After the startup phase, a proportional–integral (PI) temperature controller (TC) was set in such a way as to maintain the pilot tray temperature at the desired setpoint. This strategy is indeed simple, and can be accomplished with a minimum of measurement and control instrumentation. However, as will be discussed later, a drawback is that the overhead product is usually obtained at a purer level than needed. This is because the reflux ratio is continuously increasing during the batch production, so that if good temperature control is ensured, the fractionating capacity of the column increases as the time progresses.

More recently, a number of studies were published in the field of batch distillation, but they were related mainly to the modeling, simulation, and optimization of batch columns.[10,11] Relatively few papers addressed the issue of feedback control of a conventional (i.e., rectifying) batch column. Quintero-Marmol et al.[12] and Quintero-Marmol and Luyben[13] studied the feedback control of a constant-reflux-ratio-operated batch column. They proposed and compared different methods for estimating on-line the distillate composition, and found that an extended Luenberger observer provided the best overall performance. Bosley and Edgar[14] considered the issue of implementing on-line the optimal operational trajectory determined a priori by optimizing off-line the performance of a batch column; they proved that nonlinear model predictive control is very effective in tracking the optimal performance.

Finefrock et al.[3] studied the problem of composition control in a binary ethanol/water batch column. They indicated that because a batch column is an integrating system, the depletion of light components throughout a run creates a control problem somewhat like a ramp load when composition is being controlled. Moreover, soon after startup, the column is brought from a low-plant-gain composition space (i.e., the steady-state space) to a high-plant-gain composition space. Therefore, if a high controller gain is selected from the beginning of the production phase, oscillations both in the reflux rate and in the distillate composition are observed when the startup phase is switched to the production phase. This is the reason composition control proves to be very challenging even for a binary mixture. They noted that if a PI composition controller is employed, the controller gain should be increased during the operation in order to enable control to be maintained, and the controller integral time constant should be decreased accordingly. The authors suggested using a gain-scheduled PI controller to accomplish this objective (an approach that was recently re-examined by Fileti and Pereira[15]). Assuming that the distillate composition was perfectly known at any time instant, a time-based gain-scheduled PI controller was shown to give good results. However, the scheduling was also heavily dependent upon the composition setpoint and upon the amount and composition of the feed charge. Thus, a gain scheduling based on an on-line analysis of some system states was proposed. This scheduling was obtained by reconstructing the plant dynamics off-line by means of linear models. This approach yields a more flexible controller, which works well only in the range in which the linear models have been fit to the actual plant response. In addition, the off-line computation of the system dynamic characteristics is quite lengthy. Finefrock et al.[3] recognized that nonlinear model predictive control is one of the best approaches for distillate composition control. However, because this strategy requires that an optimization problem be solved on-line, the result may be computationally prohibitive, even with the current availability of computing facilities.

In the field of nonconventional batch distillation, Sørensen et al.[16] proposed to control a reactive batch rectifier by using a conventional PI temperature controller in order to track a prespecified optimal temperature trajectory. Following a strategy devised by Bortolini and Guarise[17] for binary mixtures, Wittgens et al.[18] showed that a TC can be employed for composition control of multivessel batch distillation columns operated at total reflux, if the number of trays is sufficiently large. Farschman and Diwekar[19] considered the dual composition control in a middle vessel batch column. They assumed that the product composition was perfectly known at any instant of time, and showed that the middle vessel provides a decoupling of the two composition control loops. The same decoupling effect of the middle vessel was previously observed by Barolo et al.[20]

3. The Process

In this work, a model is considered as the representation of a batch rectifier; the separation of both binary and ternary mixtures is considered. The process dynamic equations are reported in Appendix 1. Unlike other models used for similar studies,[12,14] this model considers varying molar holdups on each tray. The

Table 1. Model and Systems Characteristics

	binary system	ternary system
vapor boilup, mol/h	5400	6000
tray holdup (steady state), mol	30	30
reflux drum holdup, mol	250	300
total feed charge, mol	8000	30000
nominal feed composition (mole fraction)	0.4/0.6	0.2/0.6/0.2
estimated feed composition (mole fraction)	0.3/0.7	0.3333/0.3333/0.3334
tray hydraulic time constant, h	0.001	0.001
number of ideal trays	8	10
relative volatility	(nonideal)	4/2/1
nominal composition setpoint	0.84	0.95/0.95

Table 2. Vapor-Pressure Coefficients for the Ternary System (Temperature in K and Vapor Pressure in mmHg)

component 1		component 2		component 3	
A_1	B_1	A_2	B_2	A_3	B_3
−4000.44	12.0178	−4000.44	11.3247	−4000.44	10.6316

internal liquid rate L_m on stage m is determined by means of the linearized version of the Francis weir formula:

$$L_m = L_{m,0} + \frac{H_m - H_0}{\tau} \tag{1}$$

where $L_{m,0}$ is the reference value of the internal liquid flow rate, H_m and H_0 are the actual and reference molar holdups on tray m, and τ is the tray hydraulic time constant. The energy balances are not included in the model; therefore, the vapor rate is constant inside the column. Other assumptions are good control of the reflux drum level, ideal trays, well-mixed capacities, boiling feed, total condensation with no subcooling, negligible heat losses, and atmospheric-pressure operation. The model and systems characteristics are illustrated in Table 1 for both of the mixtures considered.

The binary system is a mixture of ethanol and water. This is a highly nonideal mixture, the relative volatility α of which is between 1 (at the azeotropic composition) and ~11 (at large ethanol dilution). The vapor−liquid equilibrium is described through the Nonrandom Two Liquids (NRTL) model, the parameters of which were taken from Gmehling and Onken.[21] The tray temperatures are determined by numerical solution of the vapor-phase constraint equations.

The ternary system exhibits constant relative volatility. The vapor pressure P_j of component j is determined through the Clausius−Clapeyron equation:

$$\ln P_j = \frac{A_j}{T} - B_j \tag{2}$$

where A_j and B_j are listed in Table 2 for each component.

Therefore, as in the approach of Quintero-Marmol et al.,[12] the temperature on stage m is determined by the following equation:

$$T_m = \frac{A_j}{\ln \dfrac{\alpha_j P_{\text{tot}}}{\sum_{k=1}^{3} \alpha_k x_{m,k}} - B_j} \tag{3}$$

where P_{tot} is the total pressure, α_j is the relative volatility of component j, and $x_{m,j}$ is the liquid-phase composition of component j on stage m.

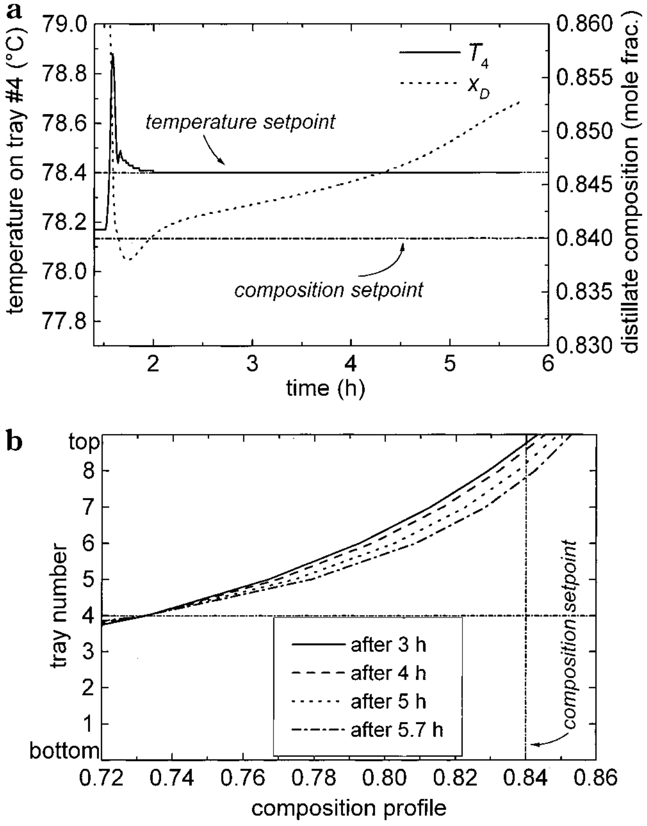

Figure 1. Binary mixture under conventional temperature control: (a) temperature and composition responses; (b) composition profiles in the column at different time instants.

In the following discussion, we refer to this model as "the process" or "the plant".

4. Binary System: Temperature Control

As was noted in the previous Summary section, conventional control strategies consider the implementation of a simple temperature controller in order to keep the distillate composition close to the desired value. The advantage of this procedure is that it is conceptually very simple; plant personnel trust in it, particularly if they have had a previous experience with the one-point composition control of continuous columns. Both Block,[8] and Bortolini and Scali,[9] noted that the measuring element must be placed somewhat lower than the top of the column in order to ensure a sensitive response of temperature to changes in the distillate composition. For a composition setpoint $x_{D,\text{sp}} = 0.84$ (mole fraction), a proper location for the measuring device is on tray no. 4 (the bottom tray is tray no. 1). Figure 1a illustrates the performance of a PI temperature controller with the following tuning: $K_C = -15\,000$ mol/(h×°C) and $\tau_I = 0.1$ h.

Clearly, although the temperature is maintained to the setpoint, the distillate composition drifts away from the specification. This is because the temperature setpoint is chosen in such a way that the composition specification is met from the early steps of the operation; that is, at relatively low values of the reflux ratio. However, as long as the reflux ratio increases (that is, as long as the operation progresses), the fractionating capacity of the column increases (Figure 1b), and therefore the temperature setpoint should be increased in order to achieve the same distillate composition. If the setpoint is not increased, at the end of the batch less product is actually obtained, because the product is purer than required.

Note that although the drift in the distillate composition was significant, the temperature on tray no. 5 changed by only 0.05 °C during the distillation. This did not allow the temperature control tray to move further up in the column.

5. Composition Estimator

A different approach considers controlling directly the distillate composition $x_{D,j}$. It is well-known that an on-line measuring device, such as a gas chromatograph, requires high investment and maintenance costs, and delivers delayed responses. Therefore, practical and economic incentives exist for using available low-cost, fast measurements (such as temperatures) in order to provide an estimate of the product composition. In the field of batch distillation, Quintero-Marmol et al.[12] proved that an extended Luenberger observer can track successfully the product composition profile in a constant reflux ratio operation. The same kind of observer is used here for constant composition operations. The observer structure will be outlined in the following discussion. For an exhaustive description, the reader should refer to the original work of Quintero-Marmol et al.[12]

For a nonlinear system described by the following set of equations:

$$\dot{\mathbf{x}} = \mathbf{f}(\mathbf{x},\mathbf{u}) \tag{4}$$

$$\mathbf{z} = \mathbf{h}(\mathbf{x}) \tag{5}$$

the extended Luenberger observer is the dynamic system defined by

$$\dot{\mathbf{x}} = \mathbf{f}(\mathbf{x},\mathbf{u}) + \mathbf{K}[\mathbf{z} - \mathbf{h}(\hat{\mathbf{x}})] \tag{6}$$

$$\mathbf{z} = \mathbf{h}(\hat{\mathbf{x}}) \tag{7}$$

In the above equations, $\mathbf{x}$ is the vector of the state variables (liquid composition on the trays, reflux drum, and reboiler), $\mathbf{u}$ is the vector of the inputs (including measured disturbances), $\mathbf{z}$ is the vector of output measurements (temperatures), $\mathbf{f}$ and $\mathbf{h}$ are nonlinear vector functions, and the symbol $\wedge$ indicates an estimated property. As explained by Quintero-Marmol et al.,[12] the observer gain matrix $\mathbf{K}$ is chosen in such a way that the estimation error $\mathbf{e} = \mathbf{x} - \hat{\mathbf{x}}$ goes to zero at a desired velocity. It is easy to show that the estimation error dynamics depends on $\mathbf{K}$ through the following dynamic equation:

$$\dot{\mathbf{e}} = (\mathbf{A} - \mathbf{KC})\mathbf{e} \tag{8}$$

where $\mathbf{A}$ and $\mathbf{C}$ are the Jacobian matrixes the elements of which are expressed by

$$a_{i,j} = \partial f_i/\partial \hat{x}_j \tag{9}$$

$$c_{i,j} = \partial h_i/\partial \hat{x}_j \tag{10}$$

To estimate the full state vector $\mathbf{x}$, the system of eqs 4 and 5 must be completely observable. Yu and Luyben[22] proved that a continuous distillation column is observable as long as the number of measurements is at least Nc − 1, where Nc is the number of components; the same argument was used by Quintero-Marmol et al.[12] in order to assess the observability of a batch column. A rigorous test of observability was considered by Berto,[23] and reported by Barolo and Berto.[6]

Quintero-Marmol et al.[12] computed the gain matrix $\mathbf{K}$ with reference to the $\mathbf{A}$ and $\mathbf{C}$ matrixes evaluated at the beginning of the operation. They proved that although $\mathbf{K}$ is kept constant despite the changing system dynamics, an accurate estimation of the state vector is obtained.

Clearly, a process model is needed in order to build the observer. This model is illustrated in the following subsection.

5.1. The Model. The model used as a representation of the process dynamics retains the basic characteristics of the process. However, several sources of process/model mismatch have been introduced in order to provide a realistic test scenario for the proposed procedure. As a major mismatch, the model's tray hydraulics is neglected, thus leading to a constant-molar-holdup model. For the ethanol/water system, we have also considered an estimated representation of the vapor−liquid equilibrium (VLE); in this case, the system VLE has been described by means of an empirical composition-dependent relative volatility, according to the following expression:

$$\alpha(x) = \alpha_0 + p_1 e^{-(x-x_0)/v_1} + p_2 e^{-(x-x_0)/v_2} \tag{11}$$

As for the calculation of the equilibrium temperature at any tray liquid composition x, two different composition-dependent temperature correlations were used:

$$T(x) = T_0 + q_1 e^{-(x-x_0)/w_1} + q_2 e^{-(x-x_0)/w_2} \qquad 0 \le x \le 0.5 \tag{12}$$

and

$$T(x) = \exp(q_1 + q_2 x + w_1 x^2 + w_2 x^3) \qquad 0.5 < x \le 0.89 \tag{13}$$

The parameters for use of eqs 11−13 are listed in Table 3.

6. Nonlinear Controller

The proposed nonlinear controller is the same as that considered by Barolo and Berto.[6] It relies on the NIMC structure proposed by Henson and Seborg.[7] The same dynamic equations used for developing the composition estimator (eq 4) are considered in the development of the controller model. However, it is convenient to recast

Table 3. Parameters for Use of Equations 11−13

	eq 11			eq 12	eq 13
	$0 \le x \le 0.5$	$0.5 < x \le 0.89$		$0 \le x \le 0.5$	$0.5 < x \le 0.89$
α_0	0.63537	0.11098	T_0	76.978	–
p_1	9.76	1.24	q_1	12.02	4.44107
p_2	5.198	0.5264	q_2	10.82	−0.11859
v_1	0.0769	0.4484	w_1	0.03537	−0.04667
v_2	0.3561	0.9804	w_2	0.3844	0.08313
x_0	0.0	0.51475	x_0	0.0	–

the controller model in the following state−space notation:

$$\dot{\mathbf{x}} = \varphi(\mathbf{x}) + \gamma(\mathbf{x})u \tag{14}$$

$$\zeta = \lambda(\mathbf{x}) \tag{15}$$

In the above equations, u is the manipulated input (i.e., the reflux rate R), λ is a scalar function, and ζ is the controlled output, i.e., the overhead composition of the jth component.

NIMC belongs to the class of geometric controllers that linearize the system input−output map exactly. The fundamentals of the method will be illustrated here. Some concepts from the differential geometry theory are recalled.

The Lie derivative $L_\varphi\lambda(\mathbf{x})$ of the smooth scalar function $\lambda(\mathbf{x})$ with respect to the smooth vector field $\varphi(\mathbf{x})$ is the scalar function defined by

$$L_\varphi\lambda(\mathbf{x}) = \sum_{i=1}^{n} \frac{\partial\lambda(\mathbf{x})}{\partial x_i}\varphi_i(\mathbf{x}) \tag{16}$$

where n is the dimension of the state vector $\mathbf{x}$. Moreover, the symbol $L_\varphi^k\lambda(\mathbf{x})$ indicates the k-time repeated iteration of $L_\varphi\lambda(\mathbf{x})$; that is $L_\varphi^k\lambda(\mathbf{x}) = L_\varphi(L_\varphi^{k-1}\lambda(\mathbf{x}))$, with $L_\varphi^0\lambda(\mathbf{x}) = \lambda(\mathbf{x})$. Similarly, $L_\gamma L_\varphi\lambda(\mathbf{x})$ is the Lie derivative of $\lambda(\mathbf{x})$ made first in the direction of $\varphi(\mathbf{x})$, and then in the direction of the smooth vector field $\gamma(\mathbf{x})$. The relative order r of a process model like that in eqs 14 and 15 is defined as the least integer for which the following condition holds true:

$$L_\gamma L_\varphi^{r-1}\lambda(\mathbf{x}) \neq 0 \tag{17}$$

Thus, the relative order of a process is the number of times that the output must be differentiated with respect to time in order to recover the input u explicitly. Roughly speaking, the relative order is a measure of "how directly" the manipulated input affects the controlled output.

Following the above notation, the NIMC control law is the following:[7]

$$u = \frac{\eta_1\delta - \sum_{k=1}^{r+1}\eta_k L_\varphi^{k-1}\lambda(\mathbf{x})}{L_\gamma L_\varphi^{r-1}\lambda(\mathbf{x})} \tag{18}$$

with $\eta_{r+1} = 1$. The error δ provides the feedback signal from the plant, and it is defined as $\delta = \zeta_{\mathrm{sp}} - (\zeta - \hat{\zeta})$, where the suffix sp refers to the setpoint value. The coefficients η_i are the controller design parameters. In the original development, the NIMC technique employs the process model as an open-loop observer, for which observability is not required. It can be proven[7] that for open-loop stable, perfectly modeled processes that operate about a stable equilibrium point with $u(t) = u_0$, the model state converges asymptotically to the plant state.

However, a batch column does not operate about an equilibrium point. Moreover, the control objective is to keep the overhead composition $x_{D,j}$ close to the setpoint, and this is achieved by continuously varying the manipulated input. Because a measure of $x_{D,j}$ is not available, the distillate composition is estimated here by the extended Luenberger observer. The NIMC control law is modified accordingly. In particular, it is assumed that the controller model is a perfect representation of the process ($\zeta = \hat{\zeta}$; $\delta = \zeta_{\mathrm{sp}}$). Note that this assumption is reliable only if the observer estimations are good.

7. Binary System: Nonlinear Control

To keep track of the notation used in eqs 14 and 15, it is useful to note that, for the binary case, the following equalities hold:

$$\mathbf{x} = \begin{bmatrix} x_D \\ x_N \\ \vdots \\ x_m \\ \vdots \\ x_B \end{bmatrix}, \varphi(\mathbf{x}) = \begin{bmatrix} \frac{V}{H_D}(y_N - x_D) \\ \frac{V}{H_m}(y_{N-1} - y_N) \\ \vdots \\ \frac{V}{H_m}(y_{m-1} - y_m) \\ \vdots \\ \frac{V}{H_B}(x_B - y_B) \\ -Vy_B \end{bmatrix}, \gamma(\mathbf{x}) = \begin{bmatrix} 0 \\ \frac{1}{H_m}(x_D - x_N) \\ \vdots \\ \frac{1}{H_m}(x_{m+1} - x_m) \\ \vdots \\ \frac{1}{H_B}(x_1 - x_B) \\ x_1 \end{bmatrix},$$

$$\text{and } \lambda(\mathbf{x}) = x_D \tag{19−22}$$

The relative order of the model in eqs 14 and 15 is $r = 2$ (see Appendix 2). In practice, this is because total condensation of the overhead vapor is assumed ($\gamma_D(\mathbf{x}) = 0$), so that the reflux can affect the distillate composition only through a change in the overhead vapor composition. For a perfect model initially at rest and with $\zeta(0) = \zeta_{\mathrm{sp}}(0)$, the closed-loop transfer function (CLTF) for setpoint changes is[7]

$$\frac{Z(s)}{Z_{\mathrm{sp}}(s)} = \frac{1}{(\epsilon s + 1)^2} \tag{23}$$

where ϵ is the closed-loop time constant, and the controller tuning parameters have been chosen such that $\eta_1 = \epsilon^{-2}$ and $\eta_2 = 2\epsilon^{-1}$. By applying eq 18, the following control law can be derived:

$$R = \frac{\frac{x_{D,\mathrm{sp}} - \hat{x}_D}{\epsilon^2} - \frac{2V(\hat{y}_N - \hat{x}_D)}{\epsilon H_D} - \frac{V}{H_D}\left[\frac{V}{H_m}(\hat{y}_{N-1} - \hat{y}_N)\frac{\partial\hat{y}_N}{\partial\hat{x}_N} - \frac{V}{H_D}(\hat{y}_N - \hat{x}_D)\right]}{\frac{V(\hat{x}_D - \hat{x}_N)}{H_D H_m}\frac{\partial\hat{y}_N}{\partial\hat{x}_N}} \tag{24}$$

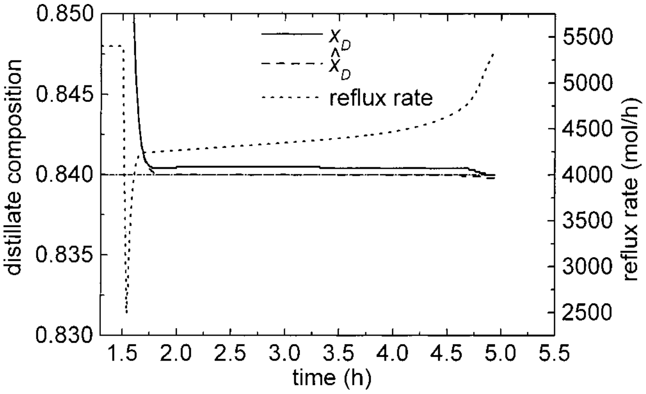

Figure 2. Binary mixture: control of the distillate composition under NIMC ($\epsilon = 0.04$ h).

In eq 24, all of the states appearing in the control law are estimated through the extended Luenberger observer. The effect of the closed-loop time constant ϵ is clear from eqs 23 and 24. A small value of ϵ (i.e., a fast closed-loop response) results in an aggressive controller action. Note that although the off-line derivation of the control law appears to be somewhat difficult to calculate, the calculations that need to be performed on-line are kept to a minimum. This is very important for practical implementation.

A satisfactory tuning of the controller can be achieved by considering the conventional startup procedure.[6] The column is brought to the steady state, and the NIMC controller is then switched on. Therefore, for perfect state estimation, the expected system response is given by eq 23, which allows one to adjust ϵ until a satisfactory response is obtained. Note that immediately after the production phase is started, the control action may be very aggressive, because the distillate composition must be moved from the steady-state value to the setpoint value. This results in severe spiking of the reflux rate. To avoid this occurrence, the reflux rate is passed through a rate-of-change filter.[24] Alternatively, one could consider filtering the setpoint, as indicated by Henson and Seborg.[7]

For the actual composition estimation, only one temperature measurement was made available to the observer; to this purpose, the bottom temperature was chosen, because the reboiler composition dynamics is the slowest in the column. Increasing the number of measurements did not lead to significant improvements in the observer estimations (this evidence is well-known; see, for example, Joseph and Brosilow[25]). The temperature measurement was updated every 0.01 h (36 s). This was also chosen as the controller time interval. Note that because batch processes are run through repeated chains of operations, the actual feed composition may not be known accurately; the composition usually results from the blending of “fresh” feed with recycled (slop) cuts. Therefore, an error in the estimated feed composition was also considered, as indicated in Table 1. This parametric mismatch adds to the structural mismatch that derives from neglecting the tray dynamics.

Figure 2 shows the plant response for a closed-loop time constant of 0.04 h; after 1.5 h the composition profile in the column was expected to be sufficiently close to the steady state composition profile. The PI temperature controller was clearly outperformed by the NIMC controller. In fact, the nonlinear controller drove the distillate composition to the setpoint quickly, and the setpoint was maintained up to the end of the operation; moreover, the reflux rate profile was smooth. Note that by the end of the operation the reflux rate must be increased very rapidly in order to keep the product on specification. This was evidence that the plant gain decreased considerably by the end of the operation.

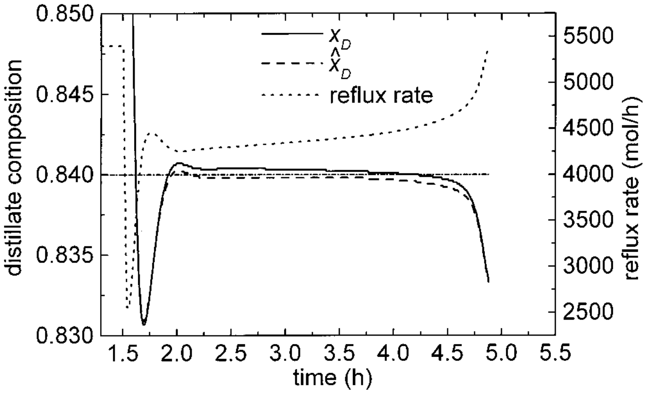

Figure 3. Binary mixture: control of the distillate composition with a PI controller ($K_C = 60\,000$ mol/h; $\tau_I = 0.12$ h).

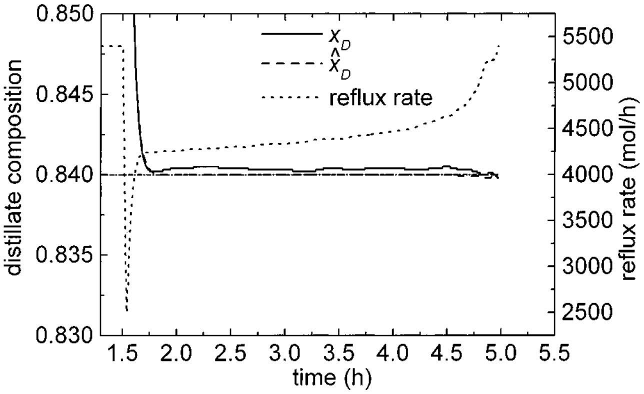

Figure 4. Binary mixture: control of the distillate composition under NIMC and noise in the measured temperature ($\epsilon = 0.04$ h; $\sigma = 0.5$ °C) .

The observer adequately estimates the distillate composition. Only a slight difference between the actual and estimated overhead composition is observed. This estimation error is because the observer employs approximate correlations in order to evaluate the system vapor−liquid equilibria.

As a comparison, the performance of a conventional PI composition controller is reported in Figure 3.

Note that the distillate composition significantly undershoots the setpoint when we switched from the startup phase to the production phase. Moreover, the PI controller cannot cope with the varying plant gain: after $t = 4$ h, the plant response is severely degraded. Increasing the controller gain resulted in an oscillating response at the beginning of the production phase.

The problem in chemical processing is more often the lack of measurements than the low quality of measurements.[26] The Luenberger observer has some degree of built-in robustness, which can often be enough to compensate for noise on the temperature measurements.[27] This was tested in the run represented in Figure 4, in which random numbers with zero mean and standard deviation $\sigma = 0.5$ °C were superimposed onto the temperature measurement in order to mimic the existence of measurement noise.

The performance of the NIMC controller is very good. However, note that if a large amount of noise were expected, the use of a stochastic estimator (like an extended Kalman filter; see, for example, Baratti et al.[28]) would be advisable.

7.1. Relationships with Globally Linearizing Control (GLC). The proposed modification to the NIMC control law makes this control structure resemble the GLC structure proposed by Kravaris and Chung.[29] The GLC control law is described by the following equation:

$$u = \frac{v - \sum_{k=0}^{r} \beta_k L_\varphi^k \lambda(\mathbf{x})}{\beta_r L_\gamma L_\varphi^{r-1} \lambda(\mathbf{x})} \tag{25}$$

where the β_k coefficients are the controller tuning parameters, and v is an external PI controller:

$$v = K_{\mathrm{C,GLC}}\left[(\zeta_{\mathrm{sp}} - \zeta) + \frac{1}{\tau_{\mathrm{I,GLC}}}\int_0^t (\zeta_{\mathrm{sp}} - \zeta)\, \mathrm{d}\theta\right] \tag{26}$$

The state vector can be obtained by either an open-loop or a closed-loop observer.

By applying eq 25 to the model previously described, the following control law is derived:

$$R = \frac{v - \beta_0 \hat{x}_D - \beta_1 \frac{V}{H_D}(\hat{y}_N - \hat{x}_D) - \beta_2 \frac{V}{H_D}\left[\frac{V}{H_m}(\hat{y}_{N-1} - \hat{y}_N)\frac{\partial \hat{y}_N}{\partial \hat{x}_N} - \frac{V}{H_D}(\hat{y}_N - \hat{x}_D)\right]}{\beta_2 \frac{V}{H_D H_m}(\hat{x}_D - \hat{x}_N)\,\frac{\partial \hat{y}_N}{\partial \hat{x}_N}} \tag{27}$$

and the CLTF for setpoint changes results:

$$\frac{Z(s)}{Z_{\mathrm{sp}}(s)} = \frac{K_{\mathrm{C,GLC}}s + \frac{K_{\mathrm{C,GLC}}}{\tau_{\mathrm{I,GLC}}}}{\beta_2 s^3 + \beta_1 s^2 + (\beta_0 + K_{\mathrm{C,GLC}})s + \frac{K_{\mathrm{C,GLC}}}{\tau_{\mathrm{I,GLC}}}} \tag{28}$$

After some cumbersome algebraic computations are performed, which are omitted for conciseness, it can be proved[23] that the GLC closed-loop response (eq 28) is equivalent to the modified NIMC closed-loop response (eq 23) when the GLC tuning parameters are chosen according to

$$\beta_1 = K_{\mathrm{C,GLC}}\epsilon\left(2 + \frac{\beta_0}{2K_{\mathrm{C,GLC}}}\right) \tag{29}$$

$$\beta_2 = K_{\mathrm{C,GLC}}\epsilon^2 \tag{30}$$

$$\tau_{\mathrm{I,GLC}} = \frac{2\epsilon K_{\mathrm{C,GLC}}}{\beta_0} \tag{31}$$

Note that both β_0 and $K_{\mathrm{C,GLC}}$ can be specified arbitrarily; they were set to $\beta_0 = K_{\mathrm{C,GLC}} = 1$ mol/h in this case. Therefore, for $\epsilon = 0.04$ h, the following settings result: $\beta_1 = 0.1$ mol, $\beta_2 = 0.0016$ mol × h, and $\tau_{\mathrm{I,GLC}} = 0.08$ h. With this tuning, a response perfectly equivalent to the one represented in Figure 2 was indeed obtained.

7.2. Modification to the Proposed Approach. Much of the complexity arising in the off-line derivation of control law (eq 24) results because the relative order of the distillate composition with respect to the reflux rate is $r = 2$. If a relative-order-1 system were obtained, the control law would appear to be simpler.

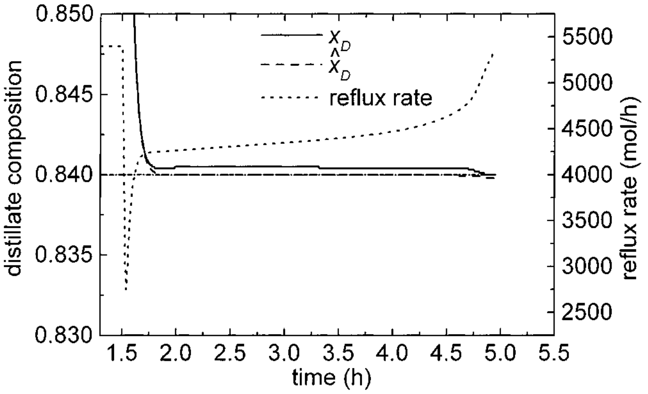

Figure 5. Binary mixture: control of the overhead vapor composition under NIMC ($\epsilon = 0.04$ h).

Note that if the control objective is keeping x_D constant, this is achieved only if the ethanol mole fraction y_N in the overhead vapor is constant and equal to the distillate composition setpoint, too. Therefore, one might choose the (estimated) overhead vapor composition as the controlled variable; a somewhat similar approach was considered by Castro et al.[30] for the composition control of continuous columns. It is intuitive (and can also be proved rigorously) that the relative order of y_N with respect to R is 1. Thus, by using eq 18 with $\eta_1 = \epsilon^{-1}$, one gets the following simpler expression for the manipulated reflux rate:

$$R = \frac{\frac{1}{\epsilon}(x_{D,\mathrm{sp}} - \hat{y}_N) - \frac{V}{H_m}(\hat{y}_{N-1} - \hat{y}_N)\frac{\partial \hat{y}_N}{\partial \hat{x}_N}}{\frac{1}{H_m}(\hat{x}_D - \hat{x}_N)\frac{\partial \hat{y}_N}{\partial \hat{x}_N}} \tag{32}$$

In this case, the CLTF results:

$$\frac{Z(s)}{Z_{\mathrm{sp}}(s)} = \frac{1}{\epsilon s + 1} \tag{33}$$

where $Z(s)$ is now the (Laplace transformed) overhead vapor composition. Figure 5 presents the results for the control of $\hat{y}_N$ with NIMC.

Clearly, the controller performance is as good as that represented in Figure 2.

8. Ternary System: Nonlinear Control

One of the advantages of the proposed procedure lies in the fact that it can be readily extended to multicomponent mixtures. Here, we consider the case of a constant-relative-volatility ternary mixture as specified in Table 1. The objective of the separation is to recover the more volatile component and the intermediate component at a constant purity. In the industrial practice, the column is started up as usual; then, the more volatile component is withdrawn at the required constant purity until the reflux ratio reaches a specified maximum value. At this point, the reflux rate is lowered (for example, following a ramp decrease) in order to allow the intermediate component to reach the

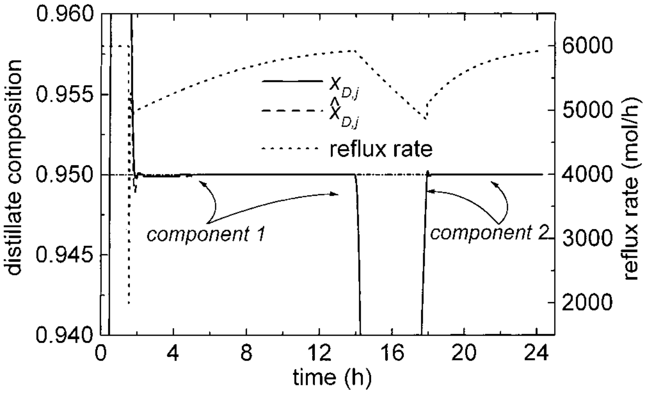

Figure 6. Ternary mixture: control of the distillate composition under NIMC ($\epsilon_1 = 0.06$ h; $\epsilon_2 = 0.06$ h).

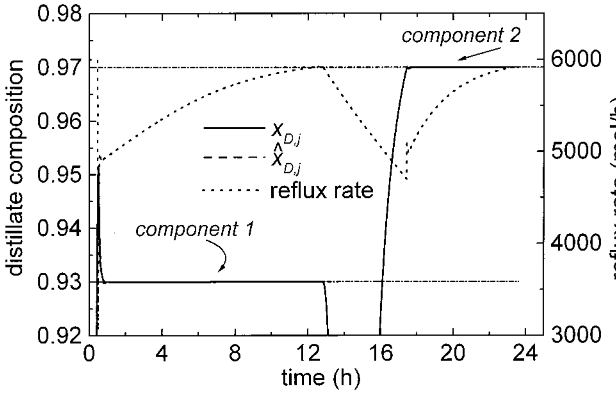

Figure 7. Ternary mixture: control of the distillate composition under NIMC with modified startup procedure and modified product specifications ($\epsilon_1 = 0.06$ h; $\epsilon_2 = 0.06$ h).

top of the column. When this component has reached the desired purity, constant composition control is started again according to the NIMC control law.

To make the derivation and the implementation of the control laws easy, it is assumed that during each of the two production phases a binary mixture is found in the top trays of the column. This approximation holds true for most separations carried out at constant reflux ratio,[13] and it is even more closely met when the column is operated at constant distillate composition. Therefore, the resulting control laws are again expressed by eq 24, where $\hat{x}$ and $\hat{y}$ are now the mole fractions of the component that is being withdrawn at the desired purity. However, note that the composition estimations are still performed with reference to the complete ternary system. Three temperature measurements are employed, and they are located in the reboiler, tray no. 5, and tray no. 10.

The NIMC controller tuning parameter ϵ_1 was tuned as indicated previously, while ϵ_2 was adjusted for satisfactory performance. Figure 6 illustrates that the nonlinear controller performance is very good. Both products are kept at the desired setpoints, and the reflux profile is smooth.

The controller robustness against modifications in the startup policy and product specifications was checked. Because startups and shutdowns of batch columns are very frequent, there is a strong economical incentive to reduce the startup time. For this reason, the more volatile component withdrawal was started as soon as this component met the composition specification, without waiting for the steady state to be attained. Moreover, the composition setpoints were changed to $x_{D,\mathrm{sp}_1} = 0.93$ and $x_{D,\mathrm{sp}_2} = 0.97$, while the same controller tuning was retained.

As is shown in Figure 7, the controller performance is still excellent.

In contrast to the results found by Quintero-Marmol et al.,[12] it was observed that when the number of ideal trays becomes large, the number of available temperature measurements becomes critical. For example, in a 20-ideal-tray column, seven temperature measurements were employed in order to obtain a good performance of the observer; moreover, the sampling interval had to be decreased to 18 s. Although neither the number nor the location of the temperature measurements were optimized in this study, preliminary results showed that ignoring the tray dynamics may result in too rough a simplification when the reflux rate is not constant and the number of trays is large. However, this result needs further verification; we leave thorough study of this point to a future work.

9. Conclusions

A strategy was proposed for the control of constant composition operations of batch distillation. The strategy relies on a modified NIMC[7] structure. State estimation, which is necessary for practical implementation of the proposed control law, was provided by an extended Luenberger observer, as proposed by Quintero-Marmol et al.[12] Structural and parametric mismatches were considered between the process and its model, in such a way as to provide a realistic test scenario for the proposed procedure.

The results for a binary mixture of ethanol and water clearly show the advantage of the proposed approach over conventional PI control. The transition from the startup phase to the production phase is fast and smooth, and the composition control is tight until the end of the operation.

The proposed control strategy can be easily extended to control the distillate purity of multicomponent mixtures. During each production phase, a simple control law can be obtained by assuming that the system is pseudo-binary. A test case for a ternary system indicated that the NIMC controller performance is good, and it is robust in the face of modifications in both the startup policy and the purity specifications.

The analogies with another nonlinear control structure, GLC,[29] were pointed out. In the case considered here, NIMC and GLC can be made equivalent by proper tuning of the GLC controller. In addition, changing the controlled output was shown to result in a simplification of the NIMC control law without loss of performance.

The proposed NIMC controller can be tuned very easily; a single parameter needs to be tuned for each component that is withdrawn as the distillate product. Moreover, the on-line computation of the control law is straightforward, which is a promising result toward practical implementation of this method. The strategy can also be readily extended for the on-line tracking of optimal trajectories determined by off-line optimization studies.

We believe that further research in this field should address the problem of selecting the best temperature measurement locations, especially in the case of multicomponent separations. In fact, it has been found that increasing the number of trays makes the extended Luenberger observer harder to tune.

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Nomenclature

a = element of the matrix $\mathbf{A}$
A = coefficient for the Antoine equation
$\mathbf{A}$ = Jacobian matrix of the function $\mathbf{f}$
B = coefficient for the Antoine equation
c = element of the matrix $\mathbf{C}$
$\mathbf{C}$ = Jacobian matrix of the function $\mathbf{h}$
$\mathbf{e}$ = vector of the estimation errors
f = element of the function $\mathbf{f}$
$\mathbf{f}$ = nonlinear vector function
h = element of the function $\mathbf{h}$
$\mathbf{h}$ = nonlinear vector function
H = holdup (mol)
$\mathbf{K}$ = gain matrix of the Luenberger observer
K_C = proportional gain of the temperature controller (mol/h/°C)
L = reflux rate (mol/h)
Nc = number of components
p = parameter for use of eq 11
P = vapor pressure (mmHg)
q = parameter for use of eqs 12 and 13
r = system relative order
R = reflux rate (mol/h)
s = Laplace operator
T = temperature (K or °C)
t = time (h)
u = manipulated input
$\mathbf{u}$ = vector of inputs
v = parameter for use of eq 11; also external PI controller
V = vapor rate (mol/h)
w = parameter for use of eqs 12 and 13
x = mole fraction in the liquid phase
$\mathbf{x}$ = vector of states
y = mole fraction in the vapor phase
$\mathbf{z}$ = vector of output measurements
Z = Laplace-transformed controlled variable

Subscripts

B = reboiler
D = distillate
j = generic component
m = generic tray
N = total number of trays; also top tray
sp = setpoint
tot = total

Superscripts

$\wedge$ = estimated property
$\bullet$ = time derivative

Greek Letters

α = relative volatility
β = GLC tuning parameter
γ = smooth vector field
δ = error as defined by the NIMC control law (18)
ϵ = closed-loop time constant (h)
φ = smooth vector field
λ = smooth scalar field
η = NIMC tuning parameter
τ = tray hydraulic time constant (h)
τ_I = integral time constant (h)
ζ = controlled output

Acronyms

CLTF = closed-loop transfer function
GLC = Globally Linearizing Control
NIMC = Nonlinear Internal Model Control
NRTL = Nonrandom Two Liquids
PI = proportional + integral
TC = temperature controller

Appendix 1—Process Dynamic Equations

The process (i.e., plant) dynamic equations are detailed here. The internal flow rates of liquid are calculated by means of the Francis weir formula (eq 1). At the beginning of the operation, it is supposed that the reboiler, all the trays, and the reflux drum are filled with the liquid feed.

Reboiler (Subscript *B*).

$$\frac{dH_B}{dt} = L_1 - V \tag{A.1.1}$$

$$\frac{d(H_B x_B)}{dt} = L x_{1,j} - V y_{B,j} \qquad j = 1 \text{ or } j = 1,2 \tag{A.1.2}$$

Bottom Tray (Subscript 1).

$$\frac{dH_1}{dt} = L_2 - L_1 \tag{A.1.3}$$

$$\frac{d(H x_{1,j})}{dt} = L_2 x_{2,j} + V y_{B,j} - L_1 x_{1,j} - V y_{1,j} \qquad j = 1 \text{ or } j = 1,2 \tag{A.1.4}$$

Intermediate Trays (Subscript *m*).

$$\frac{dH_m}{dt} = L_{m+1} - L_m \tag{A.1.5}$$

$$\frac{d(H_m x_{m,j})}{dt} = L_{m+1} x_{m+1,j} + V y_{m-1,j} - L_m x_{m,j} - V y_{m,j} \qquad j = 1 \text{ or } j = 1,2 \tag{A.1.6}$$

Top Tray (Subscript *N*).

$$\frac{dH_N}{dt} = R - L_N \tag{A.1.7}$$

$$\frac{d(H_N x_{N,j})}{dt} = R x_{D,j} + V y_{N-1,j} - L_N x_{N,j} - V y_{N,j} \qquad j = 1 \text{ or } j = 1,2 \tag{A.1.8}$$

Reflux Drum (Subscript *D*).

$$H_D \frac{dx_D}{dt} = V(y_{N,j} - x_{D,j}) \qquad j = 1 \text{ or } j = 1,2 \tag{A.1.9}$$

Appendix 2—Determination of the Relative Order *r* of the Distillate Composition with Respect to the Reflux Rate (Binary Mixture)

Because

$$\frac{\partial x_D}{\partial \mathbf{x}} = [1\ 0\ 0\ \ldots\ 0\ \ldots\ 0] \tag{A3.1}$$

we have

$$L_\gamma \lambda(\mathbf{x}) = L_\gamma x_D = \sum_i \frac{\partial x_D}{\partial x_i} \gamma_i(\mathbf{x}) = 0 \qquad \text{(A3.2)}$$

and therefore the relative order is not 1.

On the other hand

$$L_\varphi \lambda(\mathbf{x}) = L_\varphi x_D = \sum_i \frac{\partial x_D}{\partial x_i} \varphi_i(\mathbf{x}) = \frac{V}{H_D}(y_N - x_D) \neq 0 \qquad \text{(A3.3)}$$

and

$$L_\gamma[L_\varphi \lambda(\mathbf{x})] = L_\gamma\left[\frac{V}{H_D}(y_N - x_D)\right] = \frac{V}{H_D}\sum_i \frac{\partial (y_N - x_D)}{\partial x_i}\gamma_i(\mathbf{x}) = \frac{V}{H_D H_m}\frac{\partial y_N}{\partial x_N}(x_D - x_N) \neq 0 \qquad \text{(A3.4)}$$

Therefore, the relative order is 2.

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