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STATE ESTIMATOR DESIGN FOR MULTICOMPONENT BATCH DISTILLATION COLUMNS

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In the control of batch distillation columns, one of the problems is the difficulty in monitoring the compositions. This problem can be handled by estimating the compositions from readily available online temperature measurements using a state estimator. In this study, a state estimator that infers the product composition in a multicomponent batch distillation column (MBDC) from the temperature measurements is designed and tested using a batch column simulation. An extended Kalman filter (EKF) is designed as the state estimator and is implemented for performance investigation on the case column with eight trays separating the mixture of cyclo-hexane, *n*-heptane and toluene. EKF parameters of the diagonal terms of process noise covariance matrix and those of measurement model noise covariance matrix are tuned in the range where the estimator is stable and selected basing on the least IAE score. Although NC-1 temperature measurements is sufficient considering observability criteria, using NC measurements spread through out the column homogeneously improves the performance of EKF estimator. The designed EKF estimator is successfully used in the composition—feedback inferential control of MBDC operated under variable reflux-ratio policy with an acceptable deviation of 0.5–3% from the desired purity level of the products.

Keywords: batch distillation; simulation; state observer; Kalman filter.

INTRODUCTION

'Batch distillation is generally used as a separation unit in the fine speciality chemicals, pharmaceuticals, biochemical and food industries. The demand and the uncertainty in specifications for these chemicals has increased recently, which increased the popularity of the use of batch distillation' (Barolo and Cengio, 2001; Kim and Ju, 1999). Instead of using many continuous columns in series, multiple products can be obtained from a single batch distillation column during a single batch run. Moreover, batch distillation processes can easily handle variations both in the product specifications and in the feed compositions. This flexibility of batch distillation processes provides the ability to cope with a market characterized by short product life times and strict specification requirements.

In batch distillation, the operation of the column with optimized operation scenario; including reflux ratio policy, switching times, and method of recycling, is required to be realized in a convenient control system. However, in order to employ the operation scenario; the designed controller will require continuous information flow from the column, including the compositions throughout the column or temperatures reflecting the composition knowledge. The reason for this requirement is that, the value of reflux ratio and switching between product and slop cut distillations are optimized which are subject to the composition profile along the column and obtained as a function of it. Therefore, the need for knowledge of current composition in the column becomes obvious.

The composition knowledge can be generated by means of direct composition analysers in the control of a batch distillation column. Although there is a great development in the technology of online composition analysers, such as gas chromatography, they bring large measurement delays and high investment and maintenance costs (Mejdell and Skogestad, 1991; Oisiovici and Cruz, 2000; Venkateswarlu and Avantika, 2001). The most popular alternative to the composition controllers utilizing analysers is standard temperature feedback controllers. Although temperature measurements are inexpensive and have negligible measurement delays, they are not accurate indicators of composition (Mejdell and Skogestad, 1991). Another alternative is inferential control systems incorporating state estimators which use secondary temperature measurements.

State estimation can be defined as the process of extracting information from data which contain valuable

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information about a system and state estimator is the tool responsible for gathering valuable measurements to infer the desired information. Modern estimators also use known relationships in computing the desired information; taking into account the measurement errors, the effects of disturbances and control actions on the system, and prior knowledge about the system and measuring devices. While gathering these elements, they make use of some error criteria and try to minimize errors in some respect. The criteria and the method of minimization characterize the method of estimation and the use of minimization makes the estimate (extracted information) 'optimal'. If this optimality is realized statistically, the estimator type becomes stochastic; if deterministically it becomes deterministic. The estimator used in this work falls in the stochastic category and it is named as Kalman filter.

In this study, the aim is to design a state estimator that infers the component concentrations of the multicomponent batch distillation column from the measured tray temperatures. The designed estimator is further tested using a rigorous column simulation to find its performance. The extended Kalman filter (EKF) is selected as the state estimator. In the literature, EKF has shown to provide good results in the chemical industry that includes model uncertainties, unmeasured process disturbances and noisy measurements. Because it is based on the linear dynamic model of the process, the rigorous model used in the simulation is adapted to the estimator algorithm mainly by simplifying the equilibrium model and by means of linearization. The performance of the developed estimator is tested by using the rigorous column simulation and discrete measurements of the top product compositions.

MULTICOMPONENT BATCH DISTILLATION COLUMN (MBDC) OPERATION

In a batch distillation operation two types of products are handled (Luyben, 1988). The one named as slop-cut which is the byproduct of off-specification material and the other named as product-cut which is the product satisfying the specified purities. The operation of a batch column is divided into a number of stages as in the order of realization; start-up period, distillation at total-reflux, withdrawal of the lightest product, removal of a slop-cut, withdrawal of the next heaviest product, removal of a second slop-cut and, so on.

The operation for the MBDC given in Figure 1 is initiated by charging the feed mixture to the column, from its top, resulting in establishment of initial holdups in the order of condenser, reflux-drum, trays and reboiler. During this initialization period, no distillate is withdrawn from the column but instead the column is operated at total-reflux condition or at high reflux ratios to establish the desired purity level of the lightest compound in the reflux-drum. Then the first product-cut is started to distil by setting the reflux-ratio to a pre-specified value and in the same time the distillate stream is transferred to first product-cut storage tank. Due to the decreasing amount of the lightest compound in the column, after some time its composition level in the first product-cut tank begins to decrease. At this point, the distillate stream is diverted to the first slop-cut tank, if the composition of the next heaviest compound in the reflux-drum is below its specified

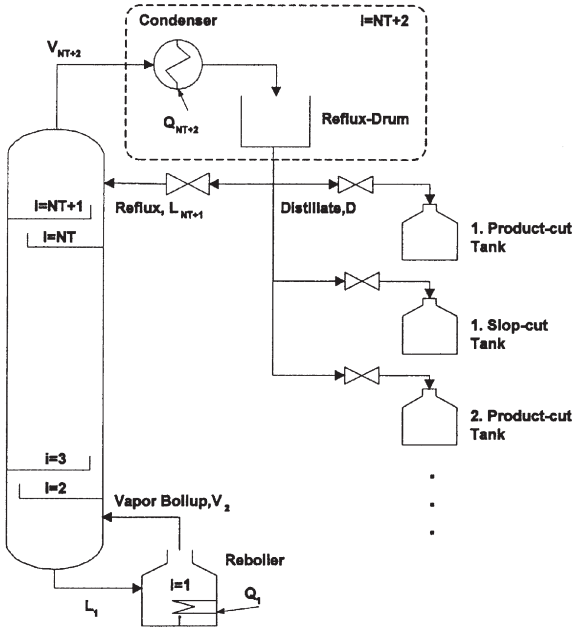

Figure 1. The schematic of a multicomponent batch distillation system.

purity. If however, it is not so low, or, during the slop-cut distillation, it starts to pass the specification level, then again the distillate is diverted to the second product-cut tank and the reflux-ratio is set to its new value. This cyclic operation between the product-cut and the slop-cut distillation continues until all the intermediate compounds is separated. Finally, the content of the reboiler is taken as the final product-cut which is rich in the heaviest compound.

MBDC PROCESS SIMULATION

There are many different rigorous models of batch distillation columns. They use the same basic strategy in the simulation model development which was used initially by the first studies on rigorous modelling of distillation columns. In batch column modelling, this common strategy was initiated by Meadows (1963) and Distefano (1968) which were followed by Stewart *et al.* (1973). In addition, the recent studies of Furlonge *et al.* (1999), Perry *et al.* (1999) and Venkateswarlu and Avantika (2001) can be given as the examples using this common strategy. The rigorous model used in this study is based on the study of Distefano (1968) and its details are given by Yıldız (2002). The assumptions employed in the development of the model can be found in Table 1, besides, Tables 2 and 3 summarize the rigorous model equations.

The assumption of negligible vapour holdup have been discussed by Young and Luyben (1987) and they stated, 'in columns operating at moderate pressures (less than 10 atm), this assumption is usually a good one'. In the study of Distefano (1968) on which the current simulation is based, the assumption of constant volume tray liquid holdup was discussed and it was realized that because of the severe variations in the tray compositions, assumption of constant molar or weight holdup is invalid in batch distillation calculations. Therefore using the constant-volume-holdup assumption will be employed in the model

Table 1. Assumptions made in the model development.

Negligible vapour holdup
Constant volume of tray liquid holdup
Constant liquid molar holdup in the reflux-drum
Total condenser
Negligible fluid dynamic lags
Linear pressure drop profile
Murphree tray efficiency
Approximated enthalpy derivatives
Adiabatic operation

Table 2. Summary of MBDC rigorous model equations.

Compositions and holdups

Reboiler dynamics

$$M_1 = M_f^0 - \sum_{n=2}^{NT+2} M_n - \int_0^i D(\tau)d\tau$$

$$\frac{dx_{1j}}{dt} = [L_2(x_{2j} - x_{1j}) - V_i(y_{1j} - x_{1j})]/M_1, \quad j = 1 \ldots NC$$

Tray dynamics

$$M_i = \frac{\rho_i^{avg}}{Mw_i^{avg}} v_i$$

$$\frac{dx_{ij}}{dt} = [V_{i-1}(y_{i-1,j} - x_{ij}) + L_{i+1}(x_{i+1,j} - x_{ij}) - V_i(y_{ij} - x_{ij})]/M_i$$

$$i = 2 \ldots, NT + 1; j = 1 \ldots NC$$

Reflux-drum dynamics

$$\frac{dx_{NT+2,j}}{dt} = [V_{NT+1}(y_{NT+1,j} - x_{NT+2,j})]/M_{NT+2}, \quad j = 1 \ldots NC$$

Composition sums

$$\sum_{n=1}^{NC} x_n = 1; \quad \sum_{n=1}^{NC} y_n = 1$$

Flowrates for given R and Q_1

Trays

$$V_{i-1} = [V_i(H_i - h_i) + L_{i+1}(h_i - h_{i+1}) + M_i\delta_i(h_i)]/(H_{i-1} - h_i)$$

$$L_i = V_{i-1} + L_{i+1} - V_i - \delta_i(M_i)$$

$$i = NT + 1 \ldots 2$$

Overhead flowrates for total reflux

$$D = 0$$

$$V_{NT+1} = L_{NT+2}$$

$$L_{NT+2} = \frac{-[Q_1 - \delta_i(M_1h_1)] - \sum_{n=2}^{NT+1} \delta_i(M_nh_n)}{(H_{NT+1} - h_{NT+2})}$$

Overhead flowrates for finite reflux ratio

$$D = \frac{Q_1 - \sum_{n=1}^{NT+1} \delta_i(M_nh_n)}{(R+1)H_{NT+1} - Rh_{NT+2}}$$

$$V_{NT+1} = D(R+1)$$

$$L_{NT+2} = RD$$

Table 3. Summary of MBDC rigorous model equations.

Pressure drop profile

$$P_i = P_1 - i \cdot (P_1 - P_{NT+2})/NT$$

Thermodynamic models

VLE calculation

$$[T_i, y_{ij}^*] = f[(x_{ik}, k = 1 \ldots NC), T_i^{guess}, P_i]$$

Murphree tray efficiency

$$y_{ij} = y_{i-1,j} + Eff_{Murphree}(y_{ij}^* - y_{i-1,j})$$

Enthalpy calculations

$$h_i = f[(x_{ik}, k = 1 \ldots NC), T_i, P_i]$$

$$H_i = f[(y_{ik}, k = 1 \ldots NC), T_i, P_i]$$

Physical properties

$$\rho_i^{avg} = f[(x_{ik}, k = 1 \ldots NC), T_i, P_i]$$

$$Mw_i^{avg} = f[(x_{ik}, k = 1 \ldots NC), T_i, P_i]$$

derivation and variations in the molal tray holdup will be provided by variations in the liquid density that is a function of composition, temperature and pressure. Instead of using ideal tray assumption, to represent the non-ideality in the phase equilibrium, Murphree tray efficiency formulation is employed assuming the temperature equilibria between the vapour and the liquid phases. Consequently, the basic assumptions made in the simulation model development are summarized in Table 1.

EXTENDED KALMAN FILTER

The EKF is defined as 'optimal recursive data processing algorithm' (Maybeck, 1979), handling the estimation issues in the nonlinear system theory. EKF uses the nonlinear model of the system given by equation (1)

$$\dot{\underline{x}}(t) = \underline{f}(\underline{x}(t), \underline{u}(t), t) + G(t)\underline{w}(t) \tag{1}$$

where f is the vector of the nonlinear system functions and the noise process, $\underline{w}(t)$ is modelled as white Gaussian noise with statistics

$$E\{\underline{w}(t)\} = 0 \tag{2}$$

$$E\left\{\underline{w}(t)\underline{w}(t')^T\right\} = \begin{cases} \underline{Q}(t), & t = t' \\ 0, & t \neq t' \end{cases} \tag{3}$$

and the nonlinear measurement model written as

$$\underline{z}(t_k) = h[\underline{x}(t_k), t_k] + \underline{v}(t_k) \tag{4}$$

where h is the vector of the nonlinear measurement functions and the noise process, $\underline{v}(t_k)$ is modelled as white Gaussian noise with statistics

$$E\{\underline{v}(t_k)\} = 0 \tag{5}$$

$$E\left\{\underline{v}(t_k)\underline{v}(t_l)^T\right\} = \begin{cases} \underline{R}(t), & t_k = t_l \\ 0, & t_k \neq t_l \end{cases} \tag{6}$$

The EKF has a two-step recursive calculation algorithm. The first named as *the propagation stage* is responsible to calculate the prediction of the state at the current time using the best state estimate at the previous time step. The second is named as *the update stage* and updates the prediction found in the first stage using the measurements taken from the actual process and calculates the best state estimate. The propagation stage integrates the state and error covariance derivatives (see Table 4: Propagation

Table 4. Equations of continuous-discrete extended Kalman filter.

Model	
System	$\dot{\underline{x}}(t) = \underline{f}(\underline{x}(t), \underline{u}(t), t) + G(t)\underline{w}(t);$ $\underline{w}(t){:}N(0, \underline{Q}(t))$
Measurement	$\underline{z}(t_k) = h(\underline{x}(t_k), t_k) + \underline{v}(t_k);\ k = 0, 1, 2, \ldots;$ $\underline{v}(t_k){:}N(0, \underline{R}(t_k))$
Initialization	
State	$\underline{x}(0) = \hat{\underline{x}}(0) = \hat{\underline{x}}_0$
Error covariance	$\underline{P}(0) = \underline{P}_0$
Propagation	
State estimate	$\dot{\hat{\underline{x}}}(t) = f(\hat{\underline{x}}(t), \underline{u}(t), t)$
Error covariance	$\dot{\underline{P}}(t) = \underline{F}(\hat{\underline{x}}(t), \underline{u}(t), t)\underline{P}(t) + \underline{P}(t)\underline{F}^T \times(\hat{\underline{x}}(t), \underline{u}(t), t) + \underline{Q}(t)$
Update	
State estimate	$\hat{\underline{x}}^+(t_k) = \hat{\underline{x}}^-(t_k) + K(t_k)[\underline{z}(t_k) - h(\hat{\underline{x}}^-(t_k), t_k)]$
Error covariance	$\underline{P}^+(t_k) = [\underline{I} - K(t_k)\underline{H}(\hat{\underline{x}}^-(t_k), t_k)]\underline{P}^-(t_k)$
Gain matrix	$K(t_k) = \underline{P}^-(t_k)\underline{H}^T(\hat{\underline{x}}^-(t_k), t_k)\ [\underline{H}(\hat{\underline{x}}^-(t_k), t_k) \times\underline{P}^-(t_k)\underline{H}^T(\hat{\underline{x}}^-(t_k), t_k) + \underline{R}(t_k)]^{-1}$
Definitions	
$N(\mu, \sigma)$: Gaussian distribution with mean, μ and covariance, σ	
$\underline{F}(\hat{\underline{x}}(t), \underline{u}(t), t) = \left.\frac{df(\underline{x}(t), \underline{u}(t), t)}{d\underline{x}(t)}\right\|_{\underline{x}=\hat{\underline{x}}}$	
$\underline{H}(\hat{\underline{x}}^-(t_k), t_k) = \left.\frac{dh(\underline{x}(t_k), t_k)}{d\underline{x}(t_k)}\right\|_{\underline{x}(t_k)=\hat{\underline{x}}^-(t_k)}$	

section) from the previous time step t_{k-1} to the current time t_k and uses the best state estimate $\hat{\underline{x}}^+(t_{k-1})$ and its error covariance $\underline{P}^+(t_{k-1})$ at the previous time step t_{k-1}, in order to calculate the prediction of the state, $\hat{\underline{x}}^-(t_k)$ and its error covariance $\underline{P}^-(t_k)$ at the current time step t_k. The update stage utilizes the equations given in Table 4 (Update section), and updates the prediction of the state $\hat{\underline{x}}^-(t_k)$ and its error covariance $\underline{P}^-(t_k)$ at the current timestep t_k.

In order to initiate the Kalman filter algorithm, the initial conditions incorporating the initial state, $\hat{\underline{x}}(t_0) = \hat{\underline{x}}_0$ and its error covariance, $\underline{P}(t_0) = \underline{P}_0$ are required. At the initialization time step, when the first measurement is taken, requirements of the best state estimate, $\hat{\underline{x}}^+(t_{k-1}) = \hat{\underline{x}}^+(t_{-1})$ and it error covariance, $\underline{P}^+(t_{k-1}) = \underline{P}^+(t_{-1})$ at the time step t_{-1} are supplied by replacing with the initial state $\hat{\underline{x}}_0$ and its error covariance $\underline{P}_0$.

EKF STATE ESTIMATOR FOR BATCH DISTILLATION COLUMNS

The technique of EKF estimation will be applied to MBDC in order to infer the column compositions from the temperature measurements. Firstly, the observability of a multicomponent batch distillation column, which is a must to be able to estimate the system states, is to be analysed. Secondly, due to computational and algorithmic requirements, the simplified models of the system and the measurement devices is to be developed by taking the rigorous simulation model as base. Then, the need for Jacobian matrices both for the system and the measurement models is to be achieved through analytical linearization.

Observability Criteria for MBDC

Most control laws used in the batch distillation are feedback laws and the state-space description of dynamics realizes that the information required for feedback control is the state of the system (Jacobs, 1974). As in the case of batch distillation, in most real controlled processes, the system state (i.e., compositions in the batch distillation) is not identical with the observable outputs (i.e., temperatures in the distillation) but there is a time-varying relationship between the states, X and the outputs, z as given in equation (7).

$$\underline{z}(t_k) = h[\underline{X}(t_k), t_k] \tag{7}$$

Therefore, the question arises whether or not it is possible to evaluate the state form observations of the output (i.e., measurements). The observability criteria is to be satisfied for solving the problem of inferring immeasurable state variables from measurements in the minimum possible length of time. So the system with the measurement model, equation (7) is said to be observable if the output, z embodies sufficient information to infer the state, x in a finite time. Here it should be noted that since the observability criteria is evaluated from the linear dynamic measurement model, although the system is observable in reality, corresponding mathematical model may not possess the property of observability. Employing a degree-of-freedom concept, Yu and Luyben (1987) found that a distillation column is observable if the number of measurements is at least (NC − 1). The study of Quintero-Marmol *et al.* (1991), dealing with the design of an extended Luenberger Observer for MBDC, concluded that even though the linear observer in theory needs only (NC − 1) temperature measurements to be observable, the nonlinear observer needed at least (NC) thermocouples to be effective. In addition, to improve the convergence without affecting the robustness, the use of (NC + 2) measurements is recommended (Quintero-Marmol *et al.*, 1991).

The Design of EKF State Estimator for MBDC

As described previously, EKF algorithm requires the stochastic models of the system (see equation (1)) and the measurement processes (see equation (4)). In equation (1), $G(t)$ is assumed as unity. Moreover, EKF algorithm needs the linearized versions of these two models, specified by the Jacobian matrices and for the system it is given by equation (8) and for the measurement process by equation (9).

$$\underline{F}(\hat{\underline{x}}(t), \underline{u}(t), t) = \left.\frac{df(\underline{x}(t), \underline{u}(t), t)}{d\underline{x}(t)}\right|_{\underline{x}=\hat{\underline{x}}} \tag{8}$$

$$\underline{H}(\hat{\underline{x}}^-(t_k), t_k) = \left.\frac{dh(\underline{x}(t_k), t_k)}{d\underline{x}(t_k)}\right|_{\underline{x}(t_k)=\hat{\underline{x}}^-(t_k)} \tag{9}$$

Lastly, in order to initiate the EKF algorithm, the information of initial conditions is required and stated by $\hat{\underline{x}}_0$ for the states and by $\underline{P}_0$ for the error covariances.

As a result, the nonlinear models for the system and for the temperature measurements are to be developed in the form required for EKF algorithm. However, the model developed for rigorous simulation of the batch column is not suitable for realistic situation in order to be implemented in EKF algorithm. For the reason that it is difficult to obtain the required values of vapor and liquid flowrates and tray holdups with time. In addition, the complexity of the simulation model requires high computational time and memory. Therefore, the rigorous column model for simulation is to be simplified and then the obtained nonlinear model is to be linearized to achieve the Jacobian matrix both for the system and the measurement processes.

Model Simplification and Linearization

Some additional assumptions are needed for the simplification of the rigorous simulation model of MBDC. These assumptions are constant molar holdup on trays, disregard of the energy dynamics in the column, ideal trays, and use of Rault's Law with Antoine's vapour pressure correlation for vapour–liquid equilibrium (VLE) description. As a result, the vapour flowrates throughout the column become equal as well as the liquid flowrates. The simplified model equations for MBDC are given in Table 5.

Next, the nonlinear models in EKF given by equations (1) and (4) are defined in terms of states, inputs and outputs of the column-simplified-model by equations (10) to (16) as

$$\dot{\underline{x}}(t) = \underline{f}(\underline{x}(t), \underline{u}(t), t) + \underline{w}(t) \tag{10}$$

where

$$\underline{x} = [x_{11} \ldots x_{1\mathrm{NC}}, \ldots, x_{\mathrm{NT+2,1}} \ldots x_{\mathrm{NT+2,NC}}]^{\mathrm{T}} \tag{11}$$

$$\underline{f} = [\dot{x}_{11} \ldots \dot{x}_{1\mathrm{NC}}, \ldots, \dot{x}_{\mathrm{NT+2,1}} \ldots \dot{x}_{\mathrm{NT+2,NC}}]^{\mathrm{T}} \tag{12}$$

$$\underline{u} = [R, Q_1]^{\mathrm{T}} \tag{13}$$

$x_{\mathrm{i,j}}$ is the molar fraction of jth component on ith stage, R is the reflux ratio, Q_1 is the reboiler load and

$$\underline{z}(t_{\mathrm{k}}) = h(\underline{x}(t_{\mathrm{k}}), t_{\mathrm{k}}) + \underline{v}(t_{\mathrm{k}}) \tag{14}$$

where

$$\underline{z} = [T_{\mathrm{M(1)}} \ldots T_{\mathrm{M(NM)}}]^{\mathrm{T}} \tag{15}$$

$$\underline{h} = [T_{\mathrm{M(1)}}(x_{\mathrm{M(1),1}} \ldots x_{\mathrm{M(1),NC}}) \ldots T_{\mathrm{M(NM)}} \times (x_{\mathrm{M(NM),1}} \ldots x_{\mathrm{M(NM),NC}})]^{\mathrm{T}} \tag{16}$$

T_{i} is the temperature measurements at ith stage, NM is the number of measurements, $M(n)$ is indexing function returning the tray number of given measurement n. More generally, the elements of $\underline{f}$ and $\underline{x}$ vectors are given by equations (17) and (18) as

$$f_{\mathrm{i}} = f_{(\mathrm{k}-1)\cdot\mathrm{NC}+\mathrm{n}} = \dot{x}_{\mathrm{k,n}} \quad \textit{for} \quad \begin{matrix} k = 1 \ldots NT+2 \\ n = 1 \ldots NC \end{matrix} \tag{17}$$

$$x_{\mathrm{j}} = x_{(\mathrm{p}-1)\cdot\mathrm{NC}+\mathrm{r}} = x_{\mathrm{p,r}} \quad \textit{for} \quad \begin{matrix} p = 1 \ldots NT+2 \\ r = 1 \ldots NC \end{matrix} \tag{18}$$

Lastly, the general forms of the linear system matrix $\underline{F}$ (Table 4)

$$\underline{F}'(\underline{x}(t), \underline{u}(t), t) = \frac{d\underline{f}(\underline{x}(t), \underline{u}(t), t)}{d\underline{x}(t)} \tag{19}$$

and the linear measurement matrix $\underline{H}$ (Table 4)

$$\underline{H}'(\underline{x}(t_{\mathrm{k}}), t_{\mathrm{k}}) = \frac{d\underline{h}(\underline{x}(t_{\mathrm{k}}), t_{\mathrm{k}})}{d\underline{x}(t_{\mathrm{k}})} \tag{20}$$

Table 5. Summary of MBDC simplified model equations.

Compositions and holdups

Reboiler dynamics

$$\frac{dM_1}{dt} = L - V$$

$$\frac{dx_{1j}}{dt} = [L(x_{2\mathrm{j}} - x_{1\mathrm{j}}) - V(y_{1\mathrm{j}} - x_{1\mathrm{j}})]/M_1$$

Tray dynamics

$$\frac{dx_{\mathrm{ij}}}{dt} = [V(y_{\mathrm{i-1,j}} - y_{\mathrm{ij}}) + L(x_{\mathrm{i+1,j}} - x_{\mathrm{ij}})]/M_{\mathrm{i}}$$

Reflux-drum dynamics

$$\frac{dx_{\mathrm{NT+2,j}}}{dt} = [V(y_{\mathrm{NT+1,j}} - x_{\mathrm{NT+2,j}})]/M_{\mathrm{NT+2}}$$

$\mathrm{i} = 2 \ldots NT+1;\ j = 1 \ldots NC$

Flowrates for given R and V_0

Vapour

$$V = V_0$$

Liquid

$$L = RD$$

Composition sums

$$\sum_{n=1}^{NC} x_{\mathrm{n}} = 1;\ \sum_{n=1}^{NC} y_{\mathrm{n}} = 1$$

Pressure drop profile

$$P_{\mathrm{i}} = P_1 - i \cdot (P_1 - P_{\mathrm{NT+2}})/NT$$

Thermodynamic models

Rault's law

$$y_{\mathrm{ij}} = x_{\mathrm{ij}}\left(\frac{P_{\mathrm{j}}^{\mathrm{v}}}{P_{\mathrm{i}}}\right)$$

Antoine's vapour pressure correlation

$$\log(P_{\mathrm{j}}^{\mathrm{v}}) = a_{\mathrm{j}} - \frac{b_{\mathrm{j}}}{c_{\mathrm{j}} - T_{\mathrm{i}}}$$

Equilibrium temperature

$$T_{\mathrm{i}}: \sum_{l=1}^{NC} x_{\mathrm{il}} \cdot 10^{[a_{\mathrm{l}} - (b_{\mathrm{l}}/(c_{\mathrm{l}} - T_{\mathrm{i}}))]} = P$$

$i = 1 \ldots NT+2;\ j = 1 \ldots NC$

are evaluated analytically. Their expanded forms and the details of the derivation are given by Yıldız (2002).

Consequently, all the information required for EKF estimator has been obtained. This information incorporates nonlinear and linearized models for the system of MBDC and the measurement process given respectively by $\underline{f}$, $\underline{h}$, $\underline{F}'$, $\underline{H}'$.

DESCRIPTION OF CASE COLUMN

The case column for simulation is the one which was simulated by Mujtaba and Macchietto (1993) in their study on the subject of optimal operation of MBDC. The column is used to separate the mixture of cyclo-hexane, *n*-heptane and toluene. The sketch of the column can be seen in Figure 1 and the design specifications of the column are listed in Table 6.

The batch distillation column is under the perfect control of reflux-drum level and has two degrees-of-freedom for manipulation which are reboiler heat load, Q_1 and reflux-ratio, R. In this study, the reboiler heat load, Q_1 is kept at its maximum value given by design while the reflux-ratio, R is used as manipulated variable in order to realize the optimal operation policy recommended by Mujtaba and Macchietto (1993) and given in Table 7. This optimal operation policy is used to yield two product-cuts with the desired purity levels of 0.9 and 0.8 from the mixture of cyclo-hexane, *n*-heptane and toluene with the composition of (0.407, 0.394, 0.199). In the simulations, this optimal reflux ratio profile is employed.

RESULTS AND DISCUSSION

The study is performed in three phases. First, a Kalman filter for the estimation of product compositions for a MBDC from temperature measurements is designed. Then the designed EKF is implemented on the case MBDC to check the performance of the EKF. In the third phase the designed EKF is utilized for control purposes in the MBDC.

The Test of the Model Used in EKF

As given in an earlier section, a simplified MBDC model must be utilized in the design of EKF. A simulation run is done to observe the mismatch between the rigorous and simplified models. This run is performed under the reflux ratio policy of Mujtaba and Macchietto (1993) and initialized by the composition of the feed charged to the column. The responses in terms of reflux-drum compositions are given in Figure 2. The trend of the compositions are similar, however, the deviations in compositions increase between the period of 3.5 h and 6 h. This period is the period of slop-cut distillation. In the optimum reflux ratio policy, the reflux-drum concentration values in terms of products are important to determine the change of reflux ratios during the operation. Thus, any deviation will result in wrong timing for the change of reflux ratio and also in the switching between product-cut and slop-cut tanks. The sources of this mismatch are due to the simplification assumptions used in the development of the model for EKF algorithm.

Table 6. Design parameters for the case column.

Number of trays	8
Condenser-reflux-drum Holdup	0.02 kmol
Trays holdup	0.01 kmol
Maximum boil-up rate	2.75 kmol h

Table 7. Parameters for the optimal reflux ratio policy.

Amount of fresh feed	2.93 kmol
Feed composition:	
Cyclo-hexane	0.407
n-heptane	0.394
Toluene	0.199
Desired purity of comp. 1	0.9
Desired purity of comp. 2	0.8
Optimum reflux profile:	
Time interval (h)	*Reflux ratio*
0–2.04	0.875
2.04–3.4	0.911
3.4–6.17	0.933
6.17–6.51	0.831
6.51–8.35	0.876

To realize this argument, one of the major assumptions, specifying the type of VLE relationship is equalized both in the rigorous model representing the process and the simple model for EKF. Therefore, instead of Peng-Robinson equation of state (EOS) for VLE calculations, the same formulation of Rault's Law used in the simple model is utilized to the rigorous model only to see the effect of this simplification. The modified rigorous model is simulated under the same conditions in the previous run. The comparison between the responses of the modified rigorous model and the EKF model are shown in Figure 3.

It is seen that although there are still discrepancies between the modified process model and the EKF model, the mismatch is highly reduced by equalizing the VLE calculations. It is concluded that the major source of the mismatch between the process and the model is due to the selection of VLE formulation utilized in the model for EKF algorithm.

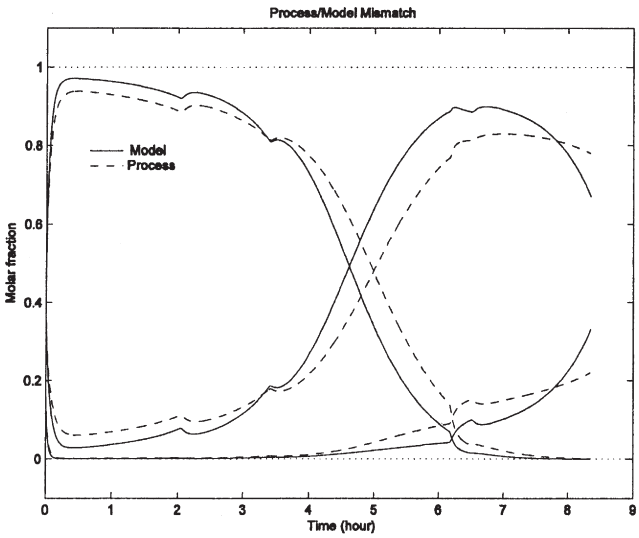

Figure 2. Mismatch between the process and the simplified model used in EKF.

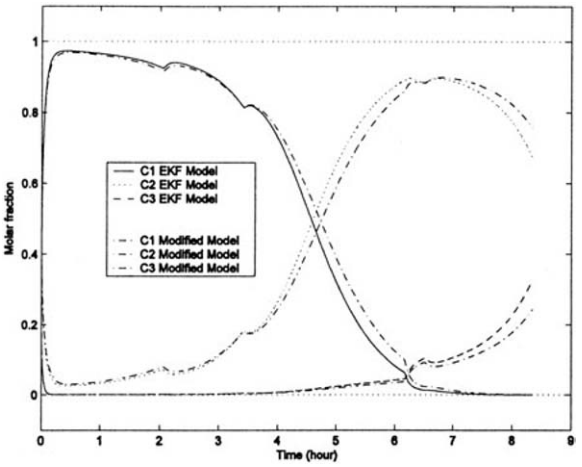

Figure 3. Mismatch between the modified process model and the EKF model.

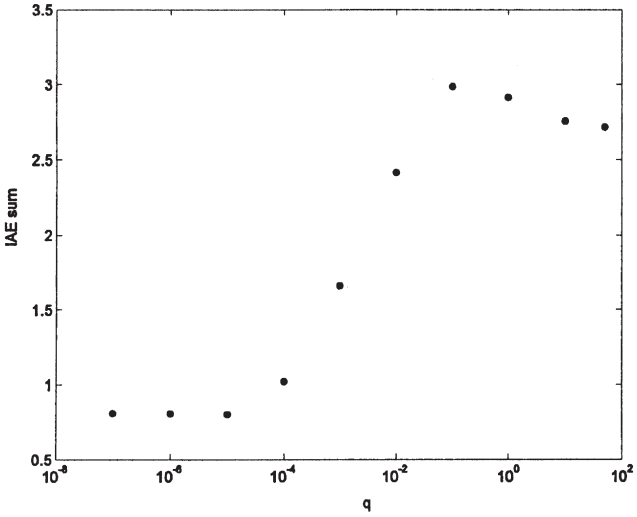

Figure 4. Change of IAE sum with respect to q.

As a result it is decided that in the model development, the most important part is the selection of VLE formulation.

Implementation of EKF

The simulation test runs for tuning the EKF is done without considering any changes in VLE relationship of the EKF model because VLE relationship does not change the effects of tuning parameters on the performance of EKF. Further, it is aimed to obtain the optimum values for these parameters in the worst case (i.e., process/model mismatch). The tuning parameters for EKF are the diagonal terms of process noise covariance matrix, q, and the diagonal terms of measurement model noise covariance matrix, r. Also, the effect of number of measurement points, and measurement period, Δt_m will be illustrated. It is known that, in initialization of the EKF, initial estimates vector, $\underline{x_0}$ and its error covariance vector, $\underline{P_0}$ are also important. These will be discussed also.

In all of the simulation test runs, the integral absolute error (IAE) is chosen as the performance criteria reflecting the fitness of the EKF design parameters. The formulation of IAE between the actual and the estimated fractions of a component is given in equation (21)

$$IAE_i = \int_0^T |X_i(\tau) - x_i(\tau)|\, d\tau \tag{21}$$

where $X_i(\tau)$ is the estimated composition of ith component, $x_i(\tau)$, the actual one and T, the total time of batch. In the performance evaluation, instead of analysing the IAE scores of each component separately, the sum of the IAE scores of the components is selected. Moreover, this total score is calculated both for the reflux-drum and the reboiler composition estimations as given by equations (22) and (23)

$$IAE_{RD} = \sum_{i=1}^{NC} IAE_i \tag{22}$$

$$IAE_{RB} = \sum_{i=1}^{NC} IAE_i \tag{23}$$

where IAE_{RD} and IAE_{RB} are the performance scores in the estimation of the reflux-drum and the reboiler compositions, respectively. As a result, the optimum value of the considered design parameter is obtained from the simulation run giving the lowest sum of IAE_{RD} and IAE_{RB} values. The optimum value of the diagonal terms of process noise covariance matrix, q is searched in the range where the EKF estimator is stable. Performing some trial runs, the stability region of the estimator is found where the value of q is in the range of 50 and 1×10^{-7}. This region is searched by changing the value of q in 10 folds. For $r = 5000$, the change of IAE scores with q is given in Figure 4.

The diagonal terms of measurement model noise covariance matrix, r are changed between 0.5 and 5×10^8 increasing in 10 folds and in each run, the diagonal terms of process noise covariance matrix, q is selected as 0.00001 which was previously determined as optimal. As in the case of q, the searching region for r is also determined by means of the stability concept. Figure 5 presents the relation of IAE sum with respect to r, graphically. The

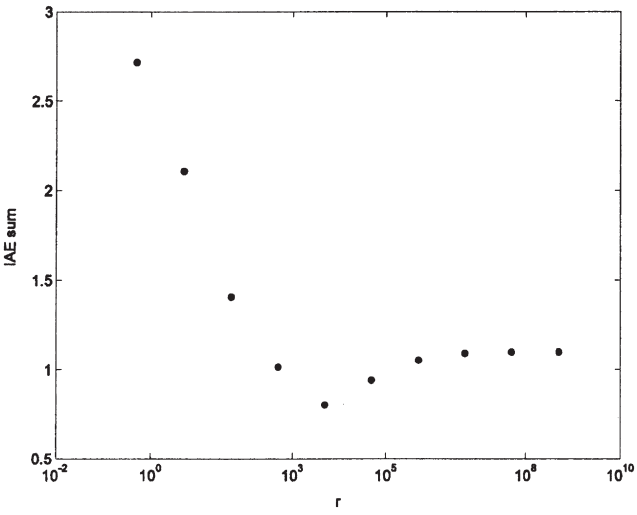

Figure 5. Change of IAE sum with respect to r.

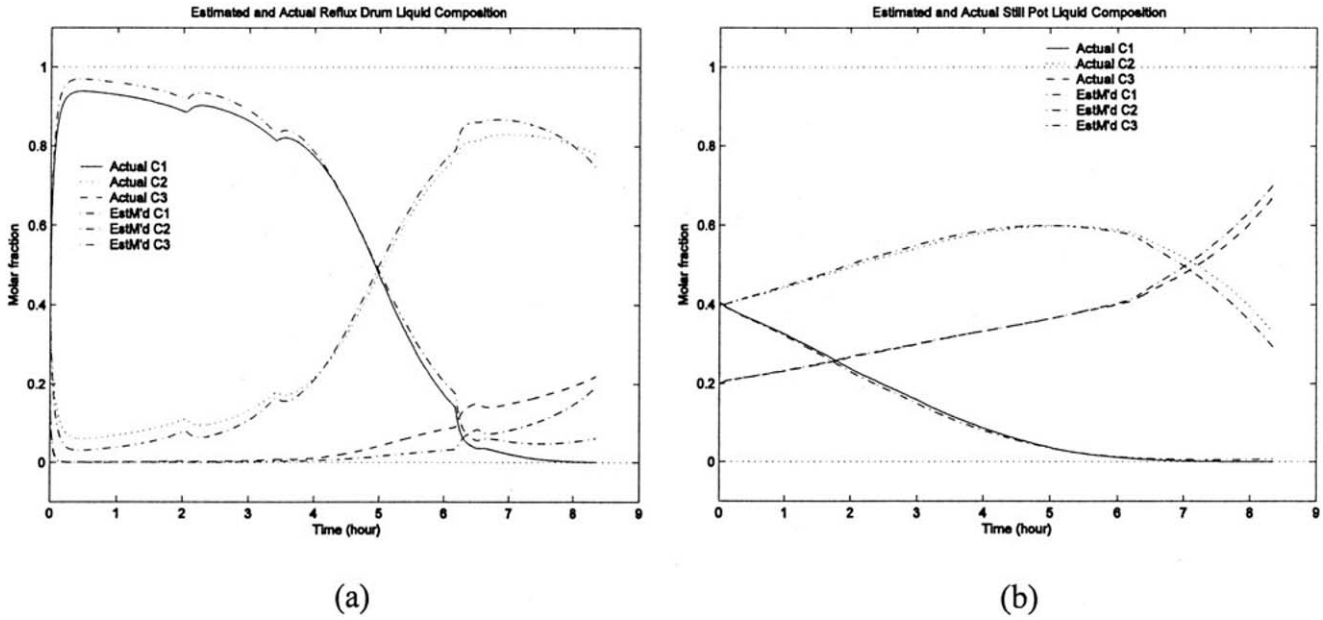

Figure 6. Effect of the diagonal terms of process noise covariance matrix for $q = 0.00001$ and $r = 5000$. (a) Reflux-drum compositions; (b) reboiler compositions.

best result (i.e., one having the lowest IAE sum) is obtained for the diagonal terms of process noise covariance matrix, $q = 5000$ as shown in Figure 6.

The previous runs were done, utilizing three measurement points for temperatures as stated by Quintero-Marmol *et al.* (1991). Several extra runs with one to five measurement points were also conducted in which optimal values for the diagonal terms of process noise covariance matrix, q and the diagonal terms of measurement model noise covariance matrix, r are used, to see the effects of measurement points in EKF performance. Firstly, to decide on the number of measurement points, homogenously spreading the locations of the measurements throughout the column resulted in the IAE sums given in Table 8. The run having the lowest IAE sum is obtained as the one with three measurement points. In addition, the run with two measurements, which is the minimum number of measurements satisfying the observability criteria, has an IAE score, larger than that of the runs with more measurements and it has an IAE less than that of one-measurement run which is the only run violating the observability requirement. Moreover, using NC measurement points was also recommended by Quintero-Marmol *et al.* (1991). Three measurement runs are further simulated using different location alternatives to see the effect of measurement locations. While the locations of two measurements are changed, the reboiler temperature measurement is kept in all of the runs. The IAE scores obtained for different measurement structures are shown in Table 9 additional to the set given in Table 8. All the measurement alternatives given in Table 9 have larger IAE scores than that of the measurement set with reboiler, fourth and top trays (Table 8). Thus this is selected as the temperature measurement points.

The effect of measurement period, Δt_m, on IAE scores is investigated, utilizing the simulation software. The results in terms of IAE scores are given in Figure 7. The responses in terms of reflux-drum and reboiler compositions for the representative values of measurement period of 1 min and 20 min are shown in Figures 8 and 9. As expected, the IAE sum of the estimations increases as the measurement period, Δt_m, increases. Although, the measurement period selected in this simulation study is the minimum possible value $(1 \times \Delta t)$, in real-time estimation problems, the value of Δt_m is to be chosen considering the limits of the computational power. Decreasing the measurement period increases the accuracy of estimation at the expense of the computational burden. The measurement periods of 3 min, even 5 min can satisfactorily be used without much change in IAE scores.

Another simulation run is done to see the effect of initial state estimate, $\underline{x_0}$ and its error covariance vector, $\underline{P_0}$ on the system response. The previous simulations were done using feed composition as the initial estimate throughout the

Table 8. Change of IAE scores with respect to number of measurements.

Measurement locations	IAE_{RD}	IAE_{RB}	IAE_{sum}
Rb, 4, NT	0.6298	0.1706	0.8004
Rb, 3, 5, NT	0.6692	0.1447	0.8139
Rb, 3, 5, 7, NT	0.6756	0.1425	0.8181
Rb, NT	0.6064	0.2219	0.8283
NT	0.6853	0.2744	0.9597

Rb is for reboiler.

Table 9. Change of IAE scores with respect to location of three measurements.

Measurement locations	IAE_{RD}	IAE_{RB}	IAE_{sum}
Rb, 4, 5	0.6598	0.1575	0.8173
Rb, 3, 4	0.6826	0.1413	0.8239
Rb, 2, 3	0.7091	0.1267	0.8358
Rb, 1, 2	0.7803	0.1186	0.8989

Rb is for reboiler.

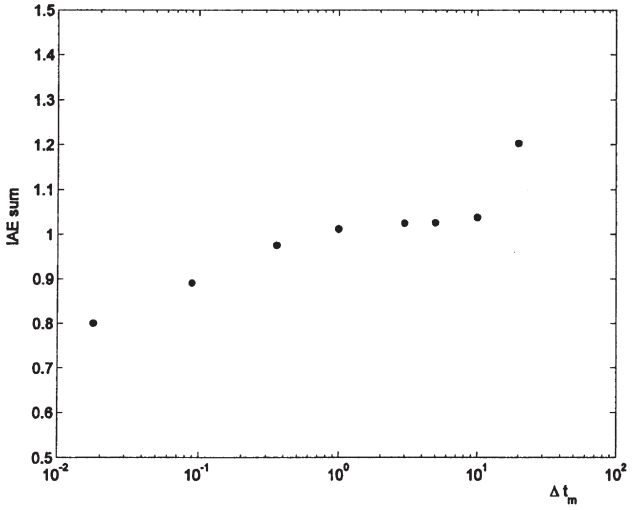

Figure 7. Change of IAE scores with respect to Δt_m.

column. The response for initial state estimate, $\underline{x_0} = [1/3; 1/3; 1/3]$ and the diagonal terms of its error covariance vector, $P_0 = 0.1$ is shown in Figure 10. This is a fictitious composition for feed when the feed composition is not known. Of course, in this run the deviations in estimation are higher than the previous cases, giving IAE sum of 1.8797. However, they can still be considered agreeable in a case where feed composition is not known. Moreover, the estimations can also be improved with trial-and-error using different tuning parameters for the case of unknown feed composition.

Closed-Loop Performance of EKF

In this phase of the study, it is aimed to analyse the performance of the EKF estimator for a MBDC system in a composition-feedback inferential control structure which realizes an actual scheduling policy explained previously in the section entitled MBDC Operation, where reflux-ratio is adjusted to a pre-optimized value with the use of top product composition information. In this control law, the compositions in the reflux-drum, the product-cut tanks and the reboiler are the inputs to the controller and the manipulated variable is the reflux-ratio of the column. The pre-specified reflux-ratio values required for the control algorithm is chosen as the optimized ones used in the previous sections. The tank, to which the distillate stream is diverted, and its timing are decided by monitoring the input compositions to the controller and utilizing the actual reflux-ratio policy. In the simulation of this control structure, the compositions can be obtained directly from the process simulation or from the EKF estimator. Firstly, to create a reference point, a simulation is done, taking the composition knowledge directly from the column as the feedback information to the controller. The desired product purities are the set points of the controller which are taken as 0.9, 0.81, 0.69. The response of this reference run in terms of the liquid compositions, both in the reflux-drum and the reboiler are given in Figure 11.

The capacity factor (CAP) (Luyben, 1988) and batch time (BT) are selected as the performance criteria to compare the closed-loop responses with the reference run. CAP for a batch operation is defined by Luyben (1988) as the ratio between the total amount of specified products and the total batch time plus feeding duration. If the P1, P2 and P3 are the amounts of products obtained and the feeding duration is taken as 0.5 h, the CAP is given by

$$\mathrm{CAP} = \frac{P_1 + P_2 + P_3}{\mathrm{BT} + 0.5} \tag{24}$$

For the previous run, CAP and BT are obtained as 243 mol/h and 8.22 h, respectively. The purities of the

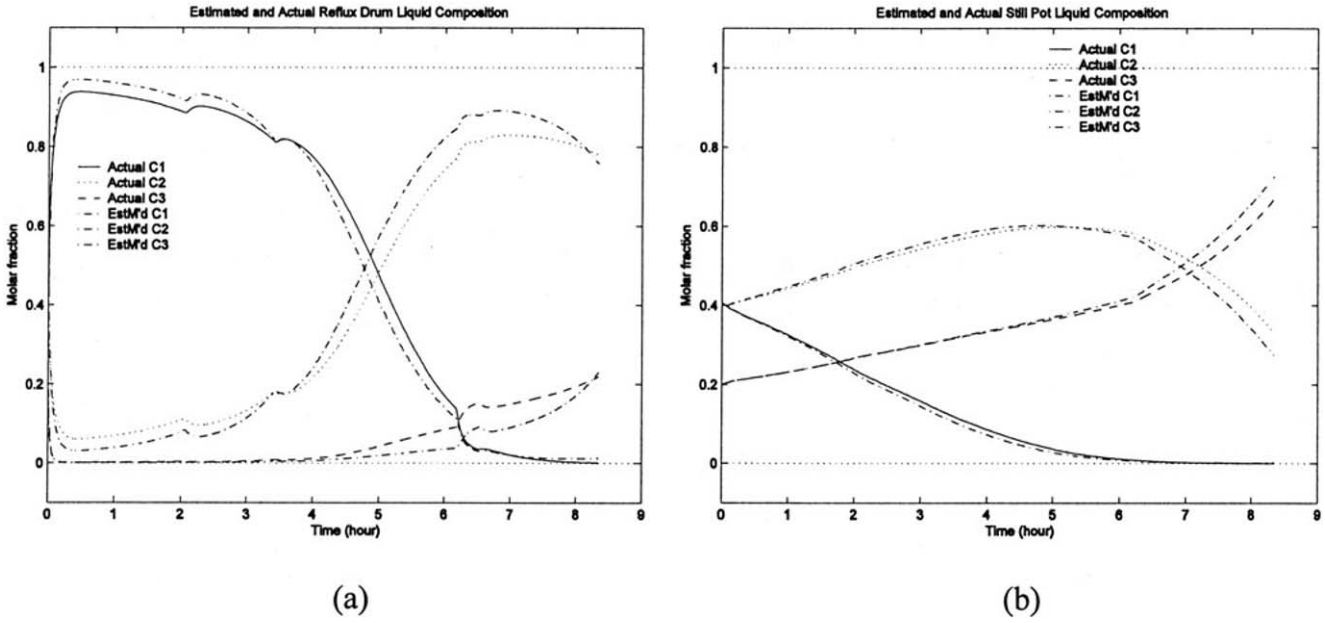

Figure 8. Effect of the period of temperature measurements for $\Delta t_m = 1$ min. (a) Reflux-drum compositions; (b) reboiler compositions.

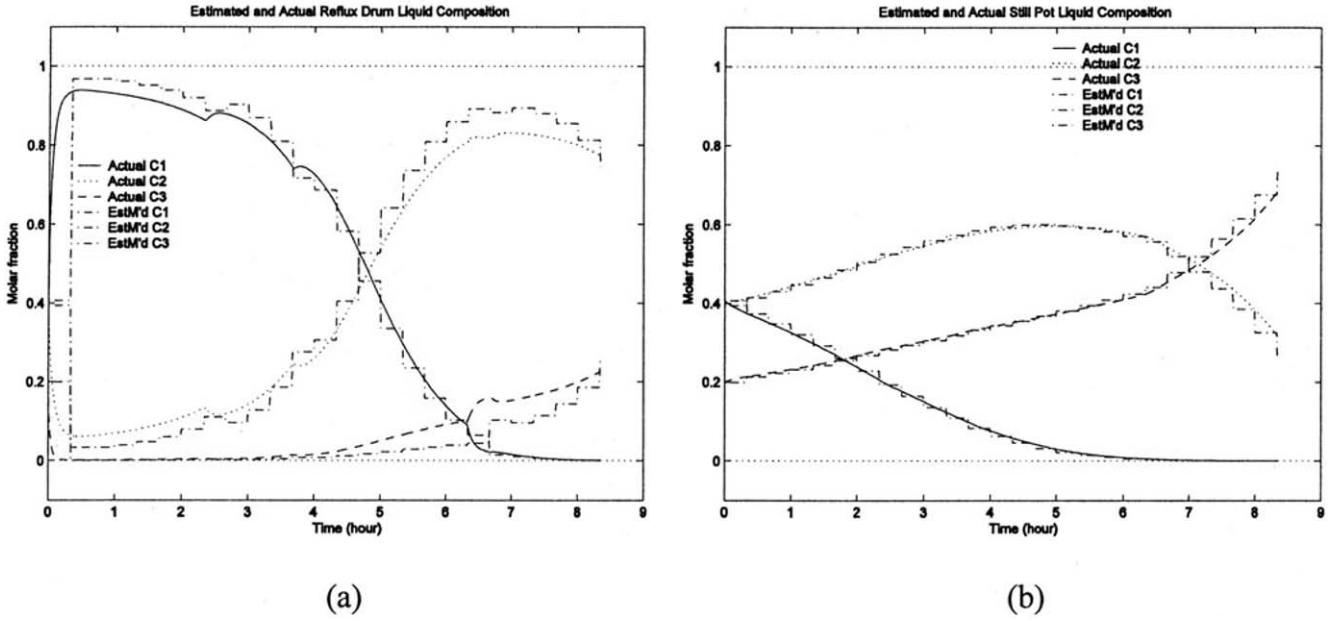

Figure 9. Effect of the period of temperature measurements for $\Delta t_m = 20$ min. (a) Reflux-drum compositions; (b) reboiler compositions.

obtained products are 0.90, 0.81 and 0.69, respectively for cyclo-hexane, *n*-heptane and toluene.

To see the performance of EKF, secondly the same simulation is realized using the estimated compositions as the feedback to the controller. For the design parameters of the EKF estimator, the optimum values obtained in the implementation section (see section 'Implementation of EKF') are used. The responses of the reflux-drum and the reboiler compositions are shown in Figure 12. CAP and BT are found as 308 mol/h and 7.59 h, respectively. The errors in estimation of CAP and BT are 21% and 8%, respectively. The error in CAP is large however; the composition errors are 3%, 2% and 0.5%, respectively for cyclo-hexane, *n*-heptane and toluene. Thus, this batch distillation column can be controlled satisfactorily for variable reflux ratio policy by the use of EKF estimator utilizing a simplified model.

CONCLUSIONS

The study is aimed to estimate the compositions in the multicomponent batch distillation column from temperature measurements using EKF estimator. It is found that, the most important part of the modelling

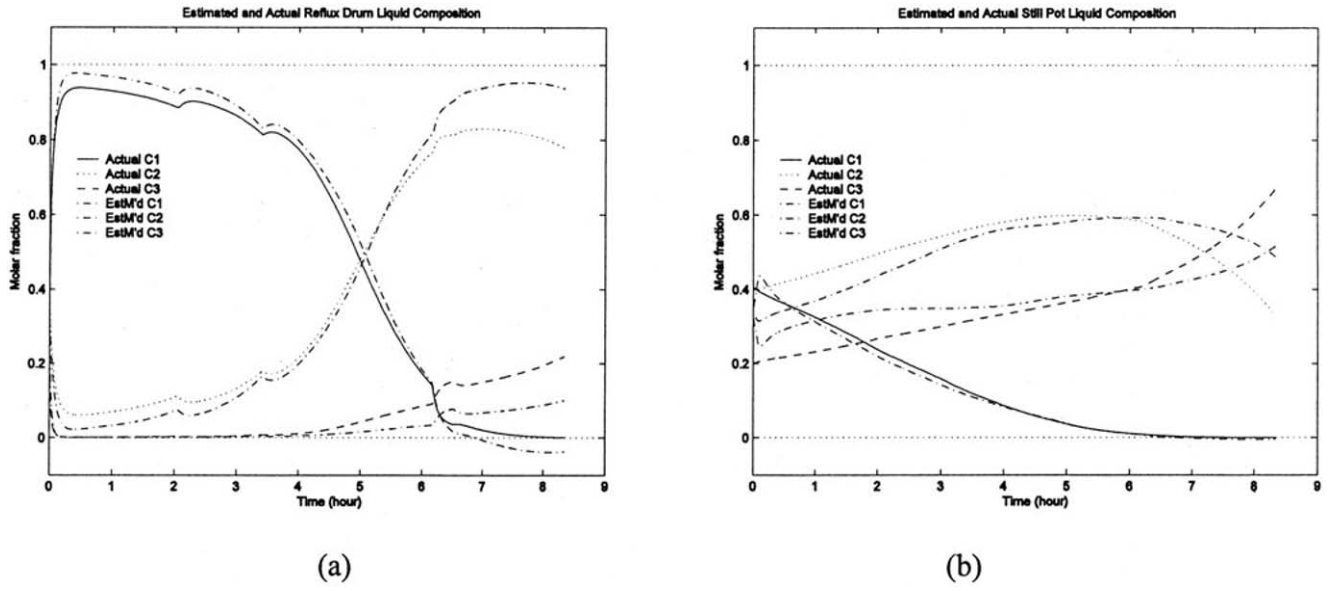

Figure 10. Effect of the initial conditions for $\underline{x_0} = [1/3;\ 1/3;\ 1/3]$ and $P_0 = 0.1$. (a) Reflux-drum compositions; (b) reboiler compositions.

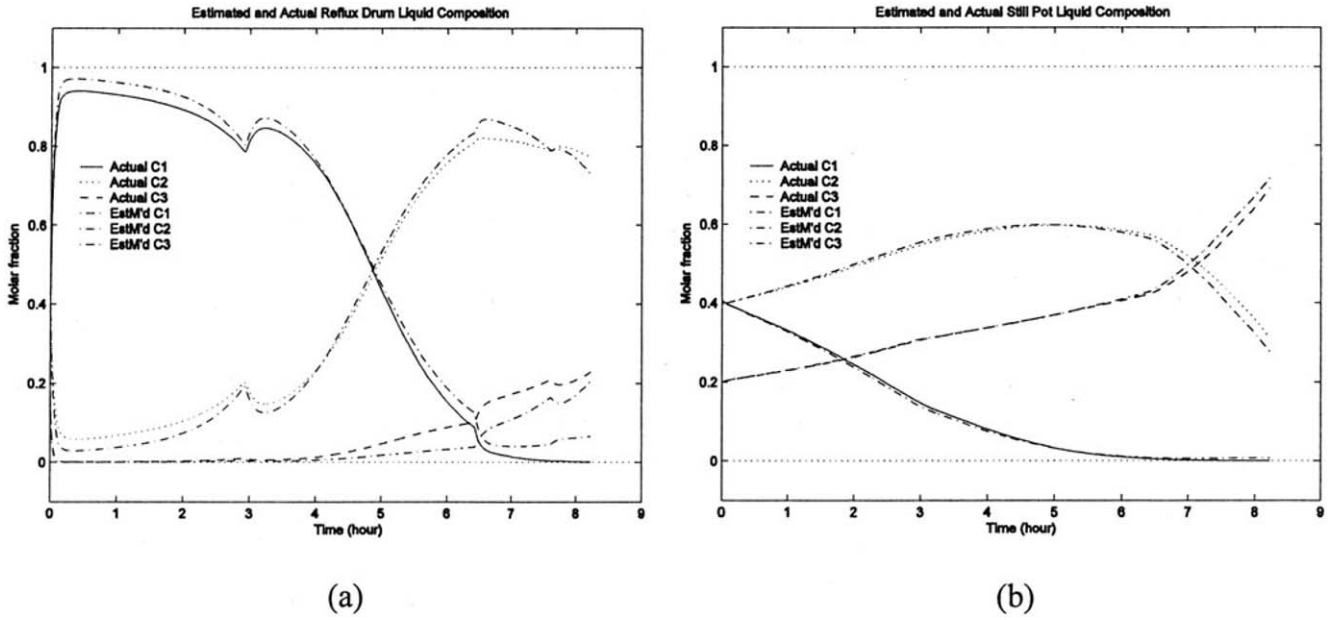

Figure 11. The closed-loop responses of the MBDC under the scheduling controller with actual composition feedback. (a) Reflux-drum compositions; (b) reboiler compositions.

affecting the performance of the EKF estimator is the selection of the VLE formulation. EKF parameters of the diagonal terms of process noise covariance matrix and the diagonal terms of measurement model noise covariance matrix are tuned in the range where the estimator is stable and selected basing on the least sum of individual IAE scores for the reflux-drum and the reboiler composition estimates. It is also found that, increasing the number of temperature measurements above the recommended value of NC does not result in a better performance. Although the observability criterion makes NC − 1 temperature measurements sufficient, using NC measurements improves the performance of EKF estimator. The measurement locations must be spread through out the column homogeneously for a better performance. Decreasing the measurement period value increases the estimator performance, and is limited by the computational power of the digital computer especially in real-time applications. The designed EKF estimator is successfully used in the composition—feedback inferential control of MBDC operated under variable reflux-ratio policy with an acceptable deviation of 0.5–3% from the desired purity level of the

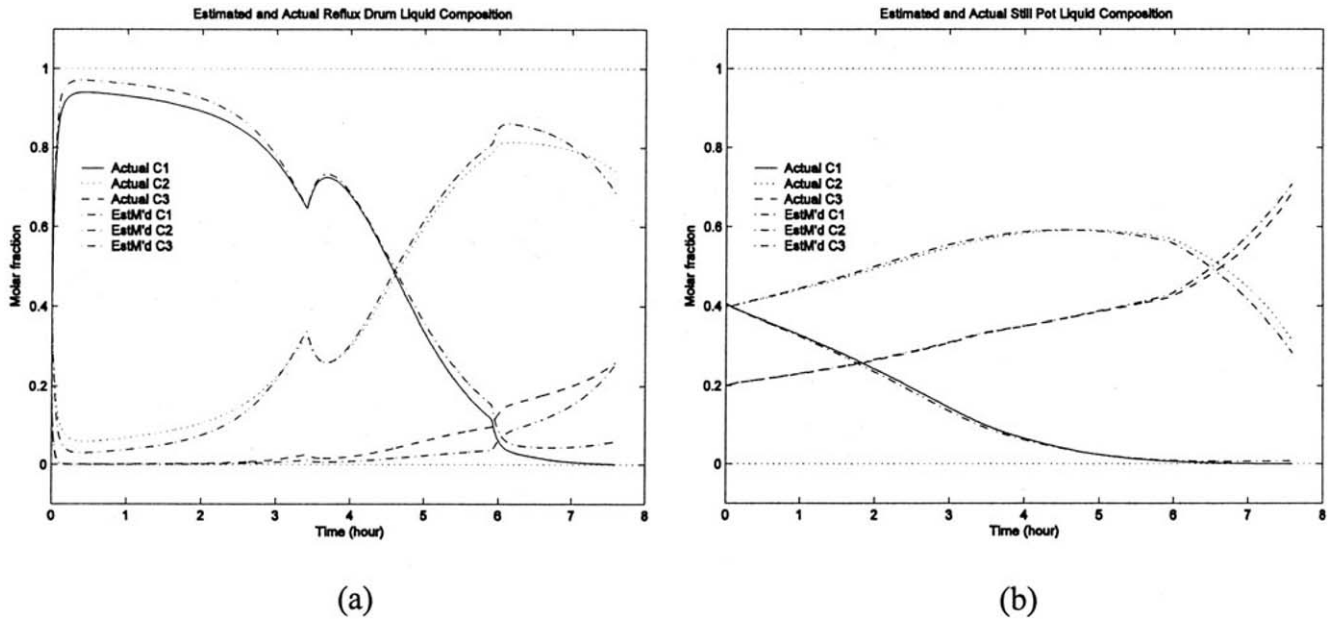

Figure 12. The closed-loop responses of the MBDC under the scheduling controller with estimated composition feedback. (a) Reflux-drum compositions; (b) reboiler compositions.

products. The method proposed in the study utilizes a very simple model for EKF which is tested in a typical batch distillation column for estimation of states and which can also be utilized in continuous distillation columns easily.

NOMENCLATURE

t	time
$\underline{x}$	vector of the nonlinear system states
f	vector of the nonlinear system functions
$\underline{u}$	vector of the nonlinear system inputs
G	process noise coupling matrix
$\underline{w}$	vector of the nonlinear system noise process
$E\{\cdot\}$	expectation
$\underline{z}$	vector of the nonlinear measurements
$\underline{v}$	vector of the nonlinear measurement noise process
$\underline{Q}$	covariance matrix of the nonlinear system noise process
$\underline{R}$	covariance matrix of the nonlinear measurement noise process
$\underline{\hat{x}}$	vector of the state estimates
$\underline{P}$	covariance vector of the estimation error
$\underline{F}$	system matrix of the linearized system model
$\underline{H}$	system matrix of the linearized measurement model
R	reflux ratio
Q_1	reboiler load
T	temperature measurements
q	diagonal terms of process noise covariance matrix
r	diagonal terms of measurement model noise covariance matrix
Δt_m	measurement period
τ	variable for integration
NC	number of components
NT	number of trays
NM	number of measurements
VLE	vapour–liquid equilibrium
Δt	time step for the simulation integration

Subscripts

k	value at kth time index
0	initial time value
_	vector

Superscripts

·	time derivative
T	transpose
∧	estimated values
+	best estimate value
−	prediction value
′	general forms

REFERENCES

Barolo, M. and Cengio, P.D., 2001, Closed-loop optimal operation of batch distillation columns, *Comput Chem Eng*, 25: 561–569.

Distefano, G.P., 1968, Mathematical modelling and numerical integration of multicomponent batch distillation equations, *AIChE J*, 14: 190–199.

Furlonge, H.I., Pantelides, C.C. and Sorensen, E., 1999, Optimal operation of multivessel batch distillation columns, *AIChE J*, 45: 781–801.

Jacobs, O.L.R., 1974, *Introduction to Control Theor*y (Clarendon Press, Oxford, UK).

Kim, J.K. and Ju, D.P., 1999, Shortcurt procedure for multicomponent batch distillation with distillate reciever, *Ind Eng Chem Res*, 38: 1024–1031.

Luyben, W.L., 1988, Multicomponent batch distillation. 1. Ternary systems with slop recycle, *Ind Eng Chem Res*, 27: 642–647.

Maybeck, P.S., 1979, *Stochastic Models, Estimation, and Control* (Academic Press Inc., USA).

Meadows, E.L., 1963, Multicomponent batch distillation calculations on a digital computer, *Chem Eng Progr Symp Ser 46*, 59: 48.

Mejdell, T. and Skogestad, S., 1991, Estimation of distillation compositions from multiple temperature measurements using partial-least-squares regression, *Ind Eng Chem Res*, 30: 2543–2555.

Mujtaba, I.M. and Macchietto, S., 1993, Optimal operation of multicomponent batch distillation—multiperiod formulation and solution, *Comp Chem Eng*, 17: 1191–1207.

Oisiovici, R.M. and Cruz, S.L., 2000, State estimation of batch distillation columns using an extended Kalman filter, *Chem Eng Sci*, 55: 4667–4680.

Perry, R.H., Green, D.W. and Maloney, J.O., 1999, *Perry's Chemical Engineer's Handbook* (McGraw-Hill, New York, USA).

Quintero-Marmol, E., Luyben, W.L. and Georgakis, C., 1991, Application of an extended Luenberger observer to the control of multicomponent batch distillation, *Ind Chem Eng Res*, 30: 1870–1880.

Stewart, R.R., Weisman, E., Goodwin, B.M. and Speight, C.E., 1973, Effect of design parameters in multicomponent batch distillation, *Ind Eng Chem Process Des Develop*, 12: 130–136.

Venkateswarlu, C. and Avantika, S., 2001, Optimal state estimation of multicomponent batch distillation, *Chem Eng Sci*, 56: 5771–5786.

Yıldız, U., 2002, Multicomponent batch distillation column simulation and state observer design, MSc thesis (http://etd.lib.metu.edu.tr/upload/12605551/index.pdf).

Young, S.C. and Luyben, W.L., 1987, Rigorous dynamic models of distillation columns, *Ind Eng Chem Res*, 26: 2158–2161.

Yu, c.C. and Luyben, W.L., 1987, Control of multicomponent distillation columns using rigorous composition estimators, *Distillation and Absorption, Inst Chem Eng Symp Ser*, 104: A29–A69.

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