Development of a software sensor for compositions in continuous reactive distillation

C Sumana and C Venkateswarlu*
Process Dynamics and Control Group, Chemical Engineering Sciences, Indian Institute of Chemical Technology,
Hyderabad 500 007

Received 29 January 2007; revised 13 June 2007; accepted 16 July 2007

A model based soft sensor is presented for composition estimation in continuous multicomponent reactive distillation. Composition soft sensor is supported by a simplified dynamic model of the process that includes component balance equations together with thermodynamic relations and reaction kinetics. Temperature measurements used as inputs to the soft sensor are suitably selected in order to obtain optimal estimates for compositions. Design and performance of the composition soft sensor is evaluated by applying to a metathesis reactive distillation column. Results demonstrate effectiveness of the soft sensor for inferential estimation of compositions in reactive distillation column.

Keywords: Composition estimation, Dynamic model, Reactive distillation

IPC Code: B01J8/00

Introduction

In chemical processes, software sensors or inferential estimators are effective alternative for estimating primary process variables using existing process model and measurements of secondary variables. In multicomponent distillation under disturbances, composition control is more effective to achieve better performance, though analyzers to measure online compositions are expensive and introduce significant measurement delays into the control loop thus affecting desired continuous column operation. This difficulty in distillation can be overcome through inferential estimation of product compositions, either by a rigorous mathematical model1,2 or an empirical model3,4 determined from process data in distillation. Reactive distillation is an integrated process operation involving combined operation of reaction and separation in a single unit, causing increased conversion and high selectivity, especially useful for systems involving reversible reactions. Performance of conventional distillation is governed by separation of mixture components, and chemical reaction rates. Integrated process, therefore, requires a precise matching of different counteracting affects such as reaction kinetics and mass transfer to achieve efficient operation.

Present study focuses on development of a software sensor for compositions in continuous reactive distillation using a simplified dynamic model of the process. Performance of the proposed inferential control scheme is evaluated by applying to a metathesis reactive distillation column that uses 2-pentene as reactant and produces 2-butene and 2-hexene as products.

Inferential Estimation Algorithm

Stochastic estimators like Kalman filters estimate process states in presence of random process disturbances and measurement errors. Dynamic inferential state of reactive distillation, which is an intrinsically complex dynamic process involving interactive influence of reaction kinetics, mass transfer and thermodynamics, can be effectively realized by designing a model based estimator such as extended Kalman filter (EKF), also known as a composition soft sensor. General process representation for composition estimation by EKF is described as follows:

Time varying model of nonlinear process is represented by
\[ x(t) = f(x(t), t) + w(t), \quad x(0) = x_0 \]  

(1)

where \( x(t) \) is \( n \) dimensional state vector; \( f \), nonlinear function; \( w(t) \), Gaussian noise with zero mean.

Nonlinear measurement model with observation noise can be expressed as

\[ y(t) = h(x(t)) + v(t) \]  

(2)

where \( h \) is a nonlinear function of state \( x(t) \). Expected values of noise covariance matrices for the initial state \( x(0) \), process noise \( w(t) \) and observation noise \( v(t) \) are given by the following relations,

\[ E \left[ \{x(0) - x(0)\}^T \{x(0) - x(0)\} \right] = P_0 \]
\[ E \left[ w(t) w^T(t) \right] = Q(t) \]  

(3)
\[ E \left[ v(t) v^T(t) \right] = R(t) \]

(4)

where \( P_0 \), initial state covariance matrix; \( Q(t) \), process noise covariance matrix; and \( R(t) \), observation noise covariance matrix. EKF is computed in following two steps:

**Prediction Equations**

With an initial estimate \( \hat{x} \) and its covariance \( P_0 \) at time zero and measurements between \( t_{k-1} \) and \( t_k \), propagating expression for the state estimate and its covariance from \( t_{k-1} \) to \( t_k \) are as follows:

\[ \hat{x}(t_{k-1}) = f \left( \hat{x}(t_{k-1}), t \right) \]  

\[ \hat{P}(t_{k-1}) = \frac{d}{d\hat{x}_{ij}(t)} \left( f(\hat{x}(t_{k-1}), t) \right) \bigg|_{x(t) = \hat{x}(t_{k-1})} \]  

(5)

(6)

where \( F(x(t), t) \) is the state transition matrix whose \( (ij) \)th element is given by

\[ F(\hat{x}(t_{k-1}), t) = \frac{d}{d\hat{x}_{ij}(t)} \left( f(\hat{x}(t_{k-1}), t) \right) \bigg|_{x(t) = \hat{x}(t_{k-1})} \]

Solution of propagated estimate \( \hat{x}(t_{k+1}) \) and its covariance \( P(t_{k+1}) \) at time \( t_k \) are \( \hat{x}(t_{k+1}) \) and \( P(t_{k+1}) \). Using measurements at time \( t_k \), update estimate \( \hat{x}(t_k) \) and its covariance \( P(t_k) \) are computed.

**Correction Equations**

Estimates are corrected by following equations:

\[ \hat{x}(t_k / t_{k-1}) = \hat{x}(t_{k+1} / t_{k-1}) + K(t_k) \left[ y(t_k) - h(\hat{x}(t_k / t_{k-1})) \right] \]  

\[ P(t_k / t_{k-1}) = (I - K(t_k) H(x(t_k))) P(t_{k+1} / t_{k-1}) \]  

(7)

(8)

where,

\[ H(x(t_k)) = \frac{\partial h(x(t_k))}{\partial x(t_k)} \bigg|_{x(t_k) = \hat{x}(t_k / t_{k-1})} \]

(9)

The recursive initial conditions for state and covariance are,

\[ \hat{x}(t / t_{k-1}) = \hat{x}(t_k / t_k) \]
\[ P(t_k / t_{k-1}) = P(t_k / t_k) \]

(10)

More details of algorithm can be referred elsewhere\(^2\).

**Process and Model**

In Olefin metathesis, two moles of 2-pentene yield one mole each of 2-butene and 2-hexene

\[ 2C_5H_{10} \leftrightarrow C_4H_8 + C_6H_{12} \]

pentene butene hexene

In this reaction, boiling points of the products straddle the boiling point of reactant. Normal boiling points of components allow an easy separation between the reactant 2-pentene (310 K), top product 2-butene (277 K) and bottom product 3-hexene (340 K). Since there is no formation of azeotropes, products of this reaction can be easily removed by minimizing side reactions or additional metathesis of products. In steady state design of metathesis reactive distillation, reaction and separation simultaneously reduces byproduct formation and overcomes the equilibrium limitations. This results in increased selectivity and higher yield over conventional series processing\(^3\).

Alejski & Duprat\(^6\) reported model equations (Table 1) for continuous reactive distillation. Boublík\(^7\) referred Antoine coefficients for all components of the system. Tray hydraulic computations involve Francis weir formula. Coefficients of heat capacity equations are reported for gases\(^8\) and liquids\(^9\). Multicomponent reactive distillation dynamic simulator of present study has major computation functions like vapor flow, liquid flow and tray holdup, enthalpy, average molecular weight & density, vapor-liquid equilibrium and reaction rate calculations.
Table 1—Equations for mathematical model of the process

Total Mass Balance

Total condenser, \[ \frac{dM_n}{dt} = V_n - (D + R) + \Delta R_n \] (11)

Feed plate \( n \), \[ \frac{dM_n}{dt} = FL + V_{n-1} + L_{n+1} - (V_n + L_n) + \Delta R_n \] (12)

Other plate \( m \), \[ \frac{dM_m}{dt} = V_{m-1} + L_{m+1} - (V_m + L_m) + \Delta R_m \] (13)

Reboiler, \[ \frac{dM_b}{dt} = L_1 - (V_b + B) + \Delta R_b \] (14)

Component Balance

Total condenser, \[ \frac{d(M_n x_{n,j})}{dt} = V_n x_{n,j} - (D x_{d,j} + R x_{d,j}) + \Delta R_{d,j} \] (15)

Feed plate \( n \), \[ \frac{d(M_n x_{n,j})}{dt} = FL x_{f,j} + V_{n-1} y_{n-1,j} + L_{n+1} x_{n+1,j} - (V_n y_{n,j} + L_n x_{n,j}) + \Delta R_{n,j} \] (16)

Other plate \( m \), \[ \frac{d(M_m x_{m,j})}{dt} = V_{m-1} y_{m-1,j} + L_{m+1} x_{m+1,j} - (V_m y_{m,j} + L_m x_{m,j}) + \Delta R_{m,j} \] (17)

Reboiler, \[ \frac{d(M_b x_{b,j})}{dt} = L_1 x_{1,j} - (V_b y_{b,j} + B x_{b,j}) + \Delta R_{b,j} \] (18)

Energy Balance

Condenser, \[ \frac{dE_c}{dt} = V_n H v_n - (D H_l + R H_l) + \Delta H r_d \] (19)

Feed plate \( n \), \[ \frac{dE_n}{dt} = FL H l + V_{n-1} H v_{n-1} + L_{n+1} H l_{n+1} - (V_n H v_n + L_n H l_n) + \Delta H r_n \] (20)

Other plate \( m \), \[ \frac{dE_m}{dt} = V_{m-1} H v_{m-1} + L_{m+1} H l_{m+1} - (V_m H v_m + L_m H l_m) + \Delta H r_m \] (21)

Reboiler, \[ \frac{dE_b}{dt} = L_1 H l_1 - (V_b H v_b + B H l_b) + \Delta H r_b \] (22)

Tray Hydraulics

Francis weir formula, \( h_l = \left( h_l + h_{eq} \right) = \left( M v_n / A_n \right) \) (23)

Flow of liquid over weir, \( M v_n = \left( (h_l - h_w) / 1.33 \right)^{1/2} l_w \) (24)

Mole fraction normalization, \[ \sum_{i=1}^{N_c} x_i = \sum_{j=1}^{N_c} y_j = 1 \] (25)

Antoine Equations

\[ \log P_{n,j}^{sat} = a_j - \frac{b_j}{c_j + T_n} \] (26)

\[ y_{n,j} P_n = x_{n,j} P_{n,j}^{sat} \] (27)

Physical Properties

\[ H_l = f (P_n, T_n, x_{n,j}) \] (28)

\[ H v_n = f (P_n, T_n, y_{n,j}) \] (29)

\[ \rho_{liq} = f (P, T, y_j) \] (30)
Soft Sensor Design

Initial process conditions of metathesis reactive distillation column are as follows: operating pressure, 1 atm; operating temperature, K (reflux drum, 281.1; reboiler, 333.5; feed, 308.2); total trays, 12; feed tray, 7; condensing, total; reboiling, partial; distillate composition (pentene, 0.064854; butene, 0.934597; hexane, 0.00583); bottom product composition (pentene, 0.063969; butene, 0.000484; hexane, 0.935512); feed composition, liquid pentene with unit mole fraction (feed, 100 Kmoles/h; top product, 50 Kmoles/h; bottom product, 50 Kmoles/h; reflux, 200 Kmoles/h; vapor boilup, 225 Kmoles/h; vapor hold up 24.8 Kmoles; and reflux drum holdup 3.1 Kmoles).

Composition soft sensor design using a rigorous mathematical model of multicomponent reactive distillation is not suitable for realistic situation, since it is difficult to obtain measured values of liquid and vapor flow rates and tray holdups with time. Therefore, a simplified model, which assumes constant tray, reflux drum holdups, constant vapor and liquid flow rates, is considered for designing the composition estimator. P, Q and R matrices involving in the estimation algorithm are heuristically considered as design parameters. State variables are component concentrations in reboiler, in all trays and in reflux drum. State vector for a ternary reactive distillation system is expressed in terms of the first and second components. Third component composition can be found by subtracting summation of the two component compositions from unity. State vector for the metathesis reactive distillation column (Fig. 1) is given by

\[ x = [x_{b,1} \ x_{b,2} \ x_{b,3} \ x_{i,1} \ x_{i,2} \ .......... \ x_{Nt,1} \ x_{Nt,2} \ x_{d,1} \ x_{d,2}]^T \]

Elements \( f_{ij} \) of \( F \) matrix in Eq. (5) of the estimation algorithm are obtained by taking partial derivatives of the component balance equations with respect to the state vector. Temperature measurement of the process model is given by:

\[ T_n = \frac{b_j}{a_j - \log\left(\frac{P_{n,j} \ P_n}{\sum_{i=1}^{Nc} P_{n,i} \ x_{n,j}}\right)} - c_j \]

...(31)

where \( a \), \( b \) and \( c \) are coefficients in vapor liquid equilibrium relation. Elements of \( \tilde{H} \) matrix in Eqs (8) and (9) of estimation algorithm are obtained by taking partial derivatives of temperature measurement expression with respect to state vector. Inferential estimator is thus designed and implemented in conjunction with dynamic model of process for composition estimation in reactive distillation column.

Results and Discussion

Temperature measurements as inputs to state estimator are obtained through bubble point calculation procedure by solving the rigorous model equations (step size, 0.001 h). In order to reflect real situation, temperature data of every sampling instant is mixed with a random Gaussian noise of zero mean and a standard deviation of 0.26°C. All column temperatures cannot be

Fig. 1 — Schematic of inferential composition estimator for metathesis reactive distillation column