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Reactive Distillation: Control Structure and Process Design for Robustness

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1. Introduction

Reactive Distillation (RD) is the combination of reaction and distillation in a single vessel (Backhaus, 1921). Over the past two decades, it has emerged as a promising alternative to conventional "reaction followed by separation" processes (Towler & Frey, 2002). The technology is attractive when the reactant-product component relative volatilities allow recycle of reactants into the reactive zone via rectification/stripping and sufficiently high reaction rates can be achieved at tray bubble temperature. For equilibrium limited reactions, the continuous removal of products drives the reaction to near completion (Taylor & Krishna, 2000). The reaction can also significantly simplify the separation task by reacting away azeotropes (Huss et al., 2003). The Eastman methyl acetate RD process that replaced a reactor plus nine column conventional process with a single column is a classic commercial success story (Agreda et al., 1990). The capital and energy costs of the RD process are reported to be a fifth of the conventional process (Siirola, 1995).

Not withstanding the potentially significant economic advantages of RD technology, the process integration results in reduced number of valves for regulating both reaction and separation with high non-linearity due to the reaction-separation interaction (Engell & Fernholtz, 2003). Multiple steady states have been reported for several RD systems (Jacobs & Krishna, 1993; Ciric & Miao 1994; Mohl et al., 1999). The existence of multiple steady states in an RD column can significantly compromise column controllability and the design of a robust control system that effectively rejects large disturbances is a principal consideration in the successful implementation of the technology (Sneesby et al., 1997).

In this Chapter, through case studies on a generic double feed two-reactant two-product ideal RD system (Luyben, 2000) and the methyl acetate RD system (Al-Arfaj & Luyben, 2002), the implications of the non-linear effects, specifically input and output multiplicity, on open and closed loop column operation is studied. Specifically, steady state transitions under open and closed loop operation are demonstrated for the two example systems. Input multiplicity, in particular, is shown to significantly compromise control system robustness with the possibility of "wrong" control action or a steady state transition under closed loop operation for sufficiently large disturbances.

Temperature inferential control system design is considered here due to its practicality in an industrial setting. The design of an effective (robust) temperature inferential control system requires that the input-output pairings be carefully chosen to avoid multiplicity in the vicinity of the nominal steady state. A quantitative measure is developed to quantify the severity of the multiplicity in the steady-state input output relations. In cases where an appropriate tray temperature location with mild non-linearity cannot be found, it may be possible to "design" a measurement that combines different tray temperatures for a well-behaved input-output relation and consequently robust closed loop control performance. Sometimes temperature inferential control (including temperature combinations) may not be effective and one or more composition measurements may be necessary for acceptable closed loop control performance. In extreme cases, the RD column design itself may require alteration for a controllable column. RD column design modification, specifically the balance between fractionation and reaction capacity, for reduced non-linearity and better controllability is demonstrated for the ideal RD system. The Chapter comprehensively treats the role of non-linear effects in RD control and its mitigation via appropriate selection/design of the measurement and appropriate process design.

2. Steady state multiplicity and its control implications

Proper regulation of an RD column requires a control system that maintains the product purities and reaction conversion in the presence of large disturbances such as a throughput change or changes in the feed composition etc. This is usually accomplished by adjusting the column inputs (e.g. boil-up or reflux or a column feed) to maintain appropriate output variables (e.g. a tray temperature or composition) so that the purities and reaction conversion are maintained close to their nominal values regardless of disturbances. The steady state variation in an output variable to a change in the control input is referred to as its open loop steady state input-output (IO) relation. Due to high non-linearity in RD systems, the IO relation may not be well behaved exhibiting gain sign reversal with consequent steady state multiplicity.

From the control point of view, the multiplicity can be classified into two types, namely, input multiplicity and output multiplicity as shown in Figure 1. In case of output multiplicity, multiple output values are possible at a given input value (Figure 1(a)). Input multiplicity is implied when multiple input values result in the same output value (Figure 1(b)).

To understand the implications of input/output multiplicity on control, let us consider a SISO system. Let the open loop IO relation exhibit output multiplicity with the nominal operating point denoted by '*'(Figure 1(a)). Under open loop operation, a large step decrease in the control input from u_0 to u_1 would cause the output to decrease from y_0 to y_1. Upon increasing the input back to u_0, the output would reach a different value y_0' on the lower solution branch. For large changes in the control input (or alternatively large disturbances), the SISO system may exhibit a steady state transition under open loop operation. For RD systems, this transition may correspond to a transition from the high conversion steady state to a low conversion steady state. The transition can be easily prevented by installing a feedback controller with its setpoint as y_0. Since the output values at the three possible steady states corresponding to u_0 are distinct, it is theoretically possible to drive the system to the desired steady state with the appropriate setpoint (Kienle & Marquardt, 2003). Note

that for the IO relation in Figure 1(a), the feedback controller would be reverse acting for y_0/y_0' and direct acting for y_0'' as the nominal steady state.

The implications of input multiplicity in an IO relation are much more severe. To understand the same, consider a SISO system with the IO relation in Figure 1(b) and the point marked '*' as the nominal steady state. Assume a feedback PI controller that manipulates u to maintain y at y_0. Around the nominal steady state, the controller is direct acting. Let us consider three initial steady states marked a, b and c on the IO relation, from where the controller must drive the output to its nominal steady state. At a, the initial error (y^{SP}-y) is positive and the controller would decrease u to bring y to the desired steady state. At b, the error is again positive and the system gets driven to the desired steady state with the controller reducing u. At c, due to the y^{SP} crossover in the IO relation, the error signal is negative and the direct acting controller would increase u, which is the *wrong* control action. Since the IO relation turns back, the system would settle down at the steady state marked '**'. For large disturbances, a SISO system with input multiplicity can succumb to wrong control action with the control input saturating or a steady state transition if the IO relation exhibits another branch with the same slope sign as the nominal steady state. Input multiplicity or more specifically, multiple crossovers of y^{SP} in the IO relationship thus severely compromise control system robustness.

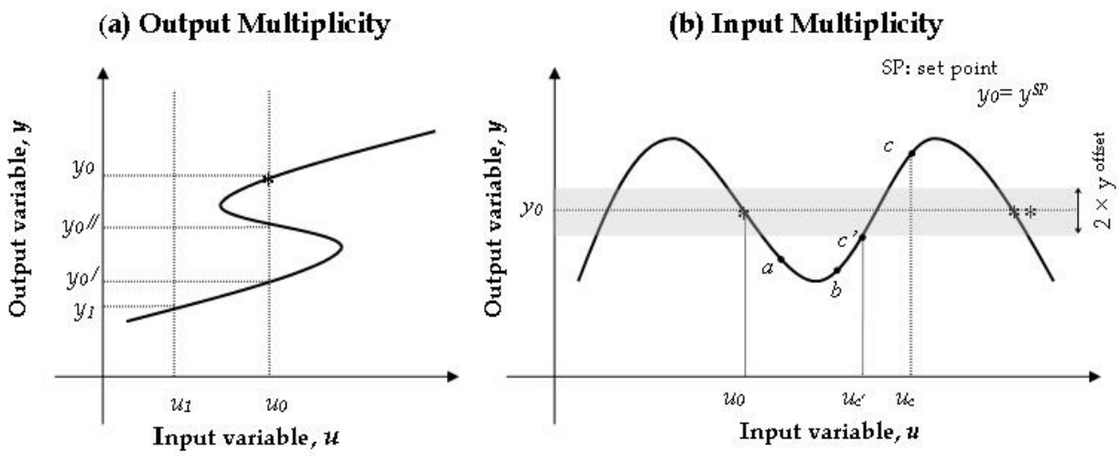


Fig. 1. Steady state multiplicity, (a) Output multiplicity, (b) Input multiplicity

The suitability of an input-output (IO) pairing for RD column regulation can be assessed by the steady state IO relation. Candidate output variables should exhibit good sensitivity (local slope in IO relation at nominal operating point) for adequate muscle to the control system where a small change in the input drives the deviating output back to its setpoint. Of these candidate sensitive (high open loop gain) outputs, those exhibiting output multiplicity may be acceptable for control while those exhibiting input multiplicity may compromise control system robustness due to the possibility of wrong control action. The design of a robust control system for an RD column then requires further evaluation of the IO relations of the sensitive (high gain) output variables to select the one(s) that are monotonic for large changes in the input around the nominal steady state and avoid multiple y^{SP} crossovers. If

such a variable is not found, the variable with a y^{SP} crossover point (input multiplicity), that is the furthest from the nominal operating point should be selected. It may also be possible to combine different outputs to design one that avoids crossover (input multiplicity). The magnitude $|u_0\text{-}u_c|$, where u_c is the input value at the nearest y^{SP} crossover can be used as a criterion to screen out candidate outputs. For robustness, Kumar & Kaistha (2008) define the rangeability, r, of an IO relation as

$$r = |u_0 - u_c'|$$

where u_c' is obtained for $y = y^{SP} - y^{offset}$ as shown in Figure 1(b). The offset from the actual crossover point ensures robustness to disturbances such as a bias in the measurement. In extreme cases, where a suitable output variable is not found that can effectively reject large disturbances, the RD column design may require alteration for improving controllability. Each of these aspects is demonstrated in the following example case studies on a hypothetical two-reactant two-product ideal RD column and an industrial scale methyl acetate RD column.

3. RD control case studies

To demonstrate the impact of steady state multiplicity on RD control, two double feed two-reactant two-product RD columns with stoichiometric feeds (neat operation) are considered in this work. The first one is an ideal RD column with the equilibrium reaction $A + B \leftrightarrow C + D$. The component relative volatilities are in the order $\alpha_C > \alpha_A > \alpha_B > \alpha_D$ so that the reactants are intermediate boiling. The RD column consists of a reactive section with rectifying and stripping trays respectively above and below it. Light fresh A is fed immediately below and heavy fresh B is fed immediately above the reactive zone. Product C is recovered as the distillate while product D is recovered as the bottoms. The rectifying and stripping trays recycle the reactants escaping the reactive zone and prevent their exit in the product streams. This hypothetical ideal RD column was originally proposed by Luyben (2000) as a test-bed for studying various control structures (Al-Arfaj & Luyben, 2000).

In terms of its design configuration, the methyl acetate column is similar to the ideal RD column with light methanol being fed immediately below and heavy acetic acid being fed immediately above the reactive section. The esterification reaction $CH_3COOH + CH_3OH \leftrightarrow CH_3COOCH_3 + H_2O$ occurs in the reactive zone with nearly pure methyl acetate recovered as the distillate and nearly pure water recovered as the bottoms.

Figure 2 shows a schematic of the two RD columns. The ideal RD column is designed to process 12.6 mol s^{-1} of stoichiometric fresh feeds to produce 95% pure C as the distillate product and 95% pure D as the bottoms product. Alternative column designs with 7 rectifying, 6 reactive and 7 stripping trays or 5 rectifying, 10 reactive and 5 stripping trays are considered in this work. For brevity, these designs are referred to as 7/6/7 and 5/10/5 respectively. The methyl acetate RD column is designed to produce 95% pure methyl acetate distillate. The 7/18/10 design configuration reported by Singh et al. (2005) is studied here. Both the columns are operated neat with stoichiometric feeds. The reaction and vapor liquid equilibrium model parameters for the two systems are provided in Table 1.

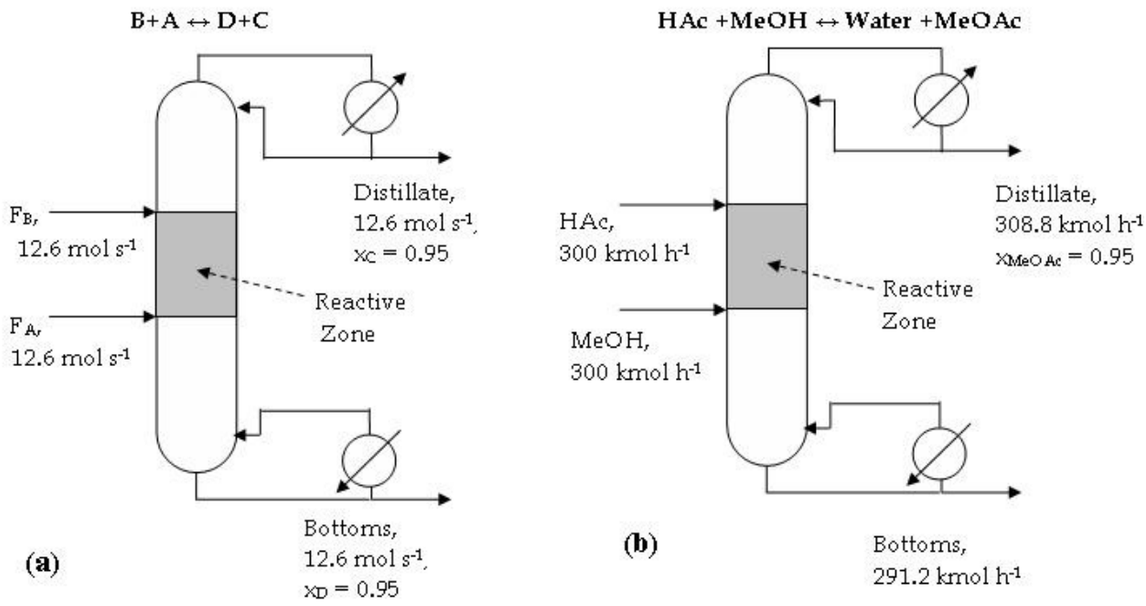


Fig. 2. Schematics of example RD columns. (a) Ideal, (b) Methyl acetate

	Ideal RD column	**Methyl acetate RD column**
Reaction	B+A ↔ D+C	Acetic acid + Methanol ↔ Water + Methyl Acetate
Relative volatility	$\alpha_C : \alpha_A : \alpha_B : \alpha_D = 8:4:2:1$	Extended Antonie Equations are used for the estimation of saturation vapour pressure, temperature dependent
Liquid phase activity	Ideal	Wilson
Vapour phase	Ideal	Ideal with Marek Method (Marek, 1995) (Vapour dimerization of Acetic acid)
Reaction kinetics	$r_C(mol/mol\ s^{-1}) = k_f x_A x_B - k_b x_C x_D$	$r_{MeOAc} = \frac{M_C k_f \left[a_{HAc} a_{MeOH} - a_{MeOAc} a_{H_2O} / K_{eq} \right]}{\left[1 + K_{HAc} a_{HAc} + K_{MeOH} a_{MeOH} + K_{MeOAC} a_{MeOAC} + K_{H_2O} a_{H_2O} \right]^2}$
	$k_f = 2.4260 \times 10^{16} e^{15098.1/T}$ $k_b = 2.11768 \times 10^{-6} e^{5032.47/T}$	$K_{HAc} = 3.18; K_{MeOH} = 4.95; K_{H_2O} = 10.5$ $K_{MeOAc} = 0.82$ $k_f(kmol/kg_{cat}/h) = 69.42 \times 10^9 e^{-52275.93/(RT)}$ $K_{eq} = 2.32 e^{782.98/T}$
Heat of reaction	- 41840 kJ/kmol Temperature independent	- 33566.80 kJ/kmol at 330 K Temperature dependent

Table 1. VLE and reaction parameters of the example RD systems

3.1 Output multiplicity effects

To demonstrate the impact of output multiplicity on column operation, the 7/6/7 design with 1 kmol reaction holdup per reactive tray is considered for the ideal RD system. For 95% pure distillate and 95% pure bottoms, the reflux ratio and vapor boilup is found to be 2.6149 and 28.32 mol s^{-1}, respectively. For the methyl acetate RD column, the 7/18/10 design is considered. At the nominal design, the reflux ratio and reboiler duty is 1.875 and 4.6021 MW respectively for 95% methyl acetate distillate and 96.33% water bottoms.

3.1.1 Ideal RD column

The variation in the bottoms *D* purity with respect to the vapor boilup at constant reflux rate in the 7/6/7 ideal RD column design is shown in Figure 3(a). Both input and output multiplicity are present in the relation with respect to the nominal steady state. Output multiplicity is observed with three distinct purities for the product *D* other than the basecase

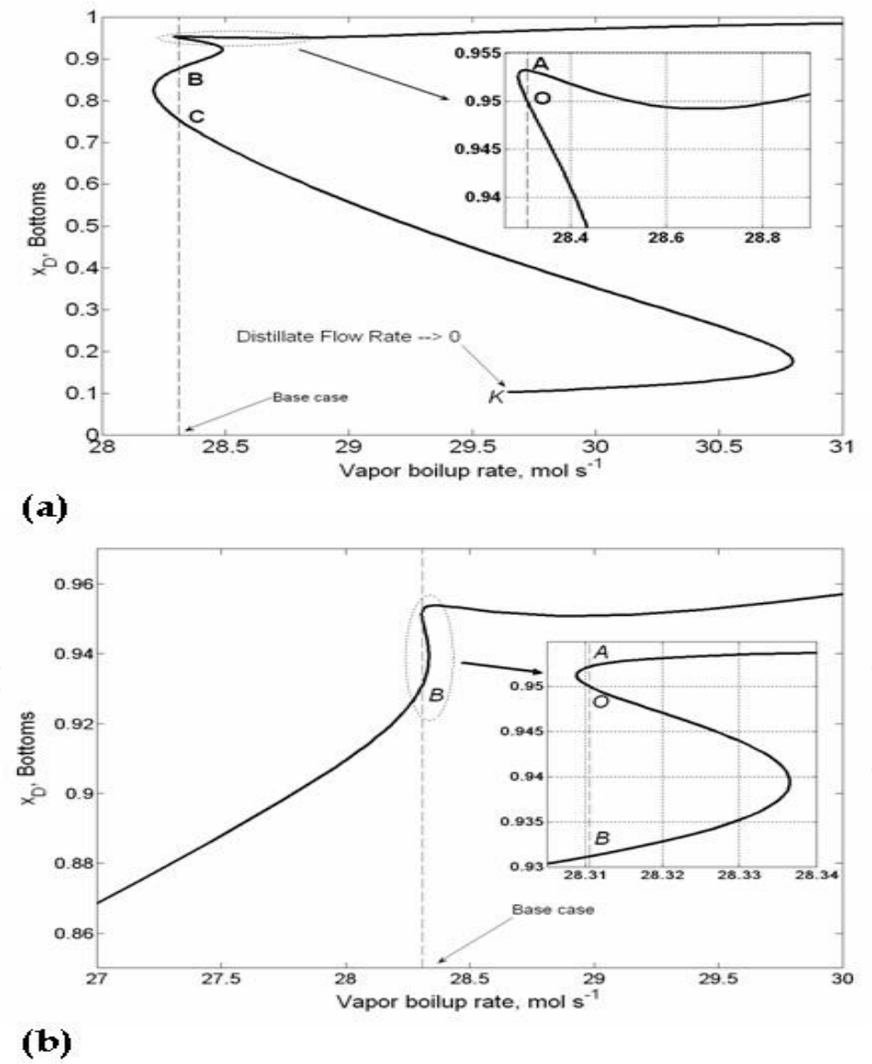


Fig. 3. Variation of ideal RD column bottom product purity with boilup at (a) fixed reflux rate, (b) fixed reflux ratio

design purity of 95%. At point *K* on the solution diagram, the distillate flow rate almost reaches 0 beyond which a steady solution is not found.

Figure 3(b) shows that IO relation of bottoms purity with vapor boilup at constant reflux to distillate ratio, a common operating policy implemented on distillation columns. Output multiplicity at the nominal steady state is evident in the Figure. Notice that a feasible steady state solution now exists for boilups below its nominal value, unlike for column operation at fixed reflux rate. From the column operation standpoint, maintaining reflux in ratio with the distillate is therefore a more pragmatic option as a feasible steady state exists for large changes in the vapor boilup in either direction.

To understand the implication of the observed steady state solution diagrams on column operation, the dynamic column response to a ±5% pulse change of one hour duration in the vapor boilup is obtained at a fixed reflux rate or at a fixed reflux ratio. The reflux drum and bottom sump levels are maintained using respectively the distillate and the bottoms flow (P controller with gain 2). The dynamic response is plotted in Figure 4. At constant reflux rate (Figure 4(a)), for the -5% boilup step change, the distillate rate quickly goes down to zero corresponding to no feasible solution in the solution diagram. For the +5% pulse change, the distillate rate settles at a slightly higher value of 12.623 mol s^{-1} (nominal value: 12.6 mol s^{-1}) implying an open loop steady state transition. This new steady state corresponds to Point *B* in the bifurcation diagram in Figure 3(a). For the -5% pulse, the distillate valve shuts down due to the absence of a feasible steady state solution for a large reduction in the boilup.

At fixed reflux ratio, a stable response is obtained for the ±5% pulse in boilup (Figure 4(b)). The column however ends up transitioning to different steady states for a +5% and a -5% pulse change, respectively. This is in line with the bifurcation diagram in Figure 3(b) with the column transitioning to a high conversion steady state (*A*) or a low conversion steady state (*B*) solution under open loop column operation.

Given the possibility of an open loop steady state transition due to output multiplicity, a PI controller is implemented that adjusts the reflux rate/reflux ratio to hold the distillate purity at 95%. The loop is tuned using the ATV method (Astrom & Hagglund, 1984) with Tyreus-Luyben settings (Tyreus & Luyben, 1992). At constant reflux rate, a boilup pulse change of -5% is handled with the column returning to its nominal steady state. In addition, a -5% step change is also handled with a stable response implying the existence of a steady state solution (feasibility) at low boilups with the distillate purity held constant. This is in contrast to the no feasible solution at reduced boilups for column operation at constant reflux rate. With the composition control loop on automatic, an unstable response is however observed for a large -20% step change which is likely due to the absence of a feasible steady state for low boilups at constant distillate composition. With the composition control loop, a +5% pulse change in the vapor boilup does not result in a steady state transition unlike for column operation at constant reflux and the column returns to its nominal steady state.

The implementation of a feedback loop controlling distillate purity by adjusting the reflux ratio results in the column returning to its nominal steady state for a ±5% pulse change in the boilup. The open loop steady state transition observed for the same pulse disturbance at constant reflux ratio is thus prevented. In addition, a -20% step change in the boilup results in a stable response with the column settling at a new steady state implying feasibility. These dynamic results serve to highlight that the implementation of feedback control serves to mitigate the non-linear effects of output multiplicity so that an open loop steady transition is prevented (Dorn et al., 1998). Feedback control also ensures feasible operation over a larger disturbance range.

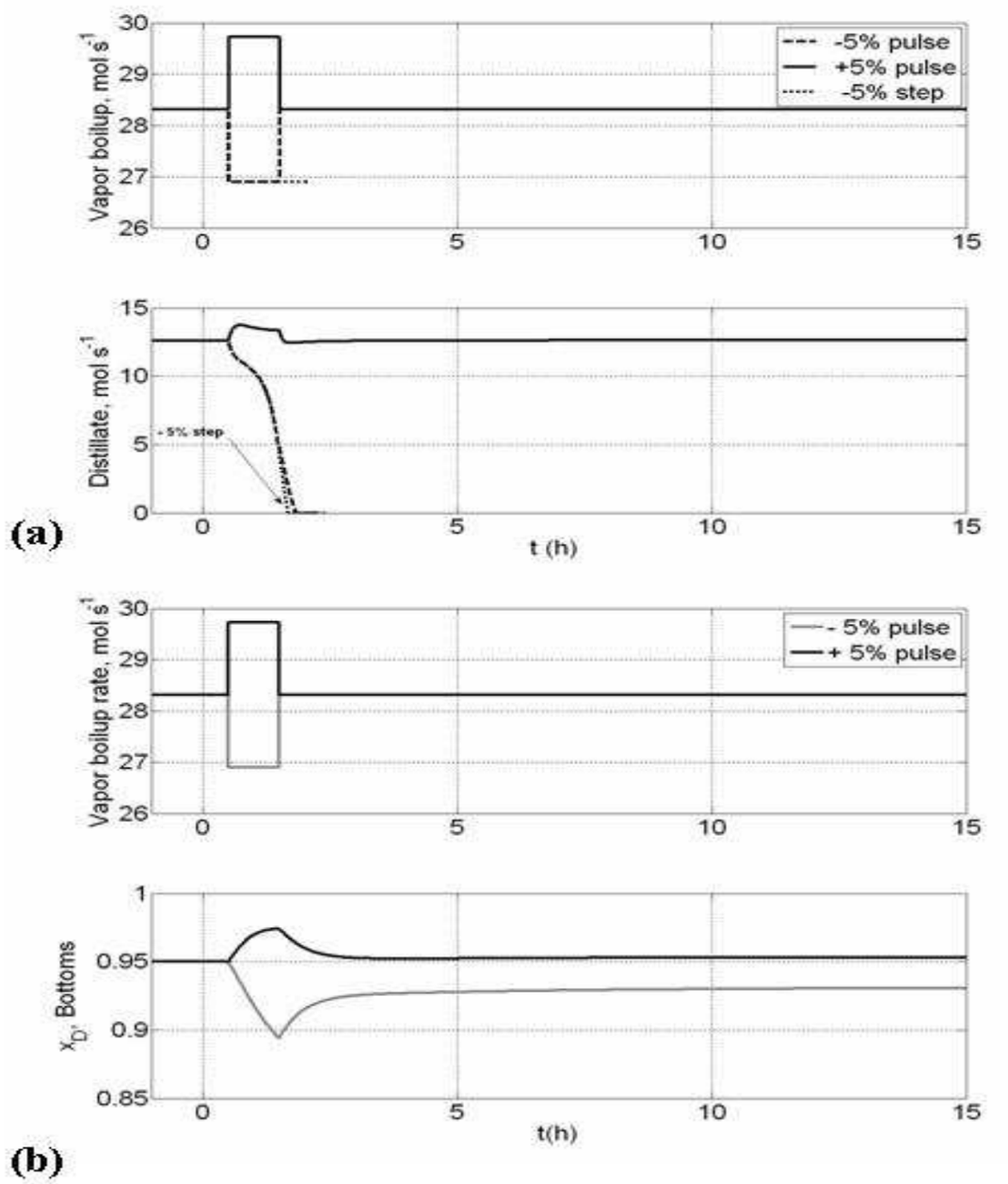


Fig. 4. Open loop dynamics of ideal RD column (7/6/7 design), (a) fixed reflux rate, (b) fixed reflux ratio

3.1.2 Methyl acetate RD column

The 7/18/10 methyl acetate RD column design is studied (Singh et al., 2005). The steady state variation of reaction conversion with respect to reboiler duty at a fixed reflux ratio and a fixed reflux rate is shown in Figure 5. At fixed reflux ratio, the nominal steady state is unique with a 97.77% conversion while two additional low conversion steady states (conversion: 72.95% and 59.66%) are observed at fixed reflux rate. The column dynamic response to a 5 hour duration -3% pulse in the reboiler duty at alternatively, a fixed reflux

rate, a fixed reflux ratio or controlling a reactive tray temperature using reflux rate is shown in Figure 6. The liquid levels in the reflux and reboiler drums are controlled using the distillate and bottoms, respectively (P controller with gain 2). Whereas the column returns to its nominal steady state for a fixed reflux ratio or for reactive tray temperature control using reflux, a steady state transition to a low conversion steady state is observed at a fixed reflux rate. This transition is attributed to the output multiplicity at constant reflux rate in Figure 5. Maintaining the reflux in ratio with the distillate is thus a simple means of avoiding output multiplicity and the associated open loop column operation issues (Kumar & Kaistha, 2008).

3.2 Input multiplicity and its implications on controlled variable selection

As discussed, the existence of input multiplicity in an IO pairing can severely compromise control system robustness due to the possibility of wrong control action. In this section, we demonstrate wrong control action in the ideal and methyl acetate RD systems. We also demonstrate the systematic use of steady state IO relations to choose CVs (controlled variables) that are better behaved (more robust) in terms of their multiplicity behavior and the consequent improvement in control system robustness for the two example RD systems.

3.2.1 Ideal RD column

The 5/10/5 design with 1 kmol reaction holdup per reactive tray is considered here. For 95% distillate and bottoms purities, the reflux ratio and vapor boilup are respectively 2.6915 and 29.27 mol s^{-1} respectively. As with the 7/6/7 design, maintaining reflux in ratio with the distillate mitigates nonlinear effects and is therefore implemented. The simplest policy of operating the column at fixed reflux ratio is first considered.

At a fixed reflux ratio, there are three available inputs for control, namely the fresh A feed (F_A), the fresh B feed (F_B) and the vapor boilup (V_S). Of these, one of the inputs must be used

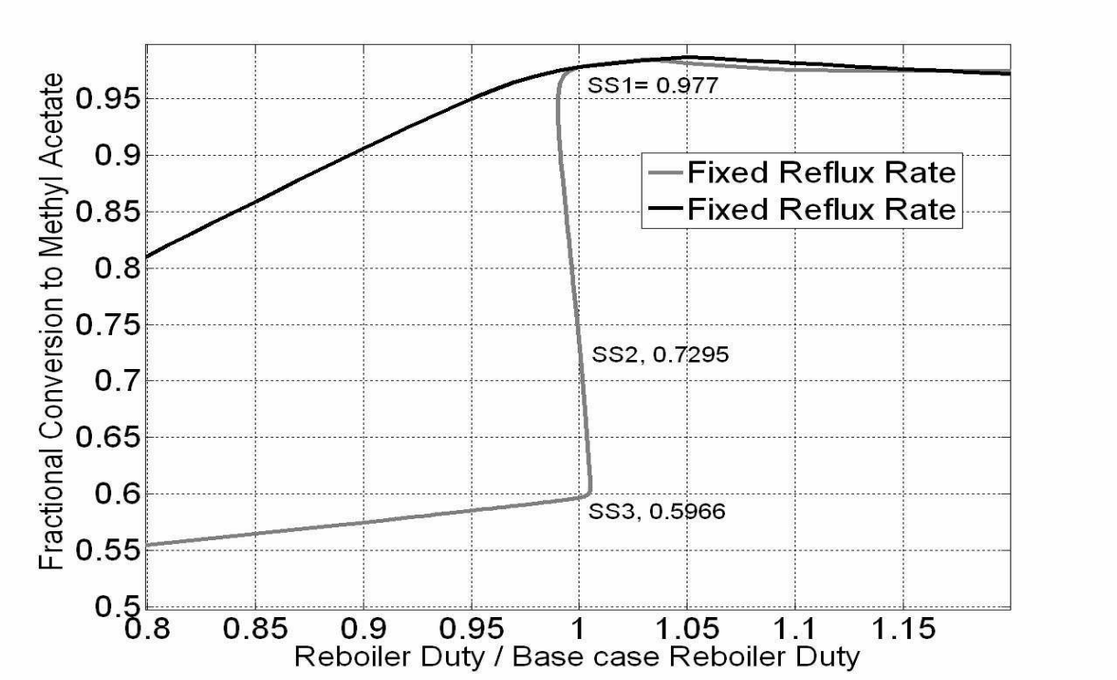


Fig. 5. Steady state conversion to methyl acetate with respect to reboiler duty

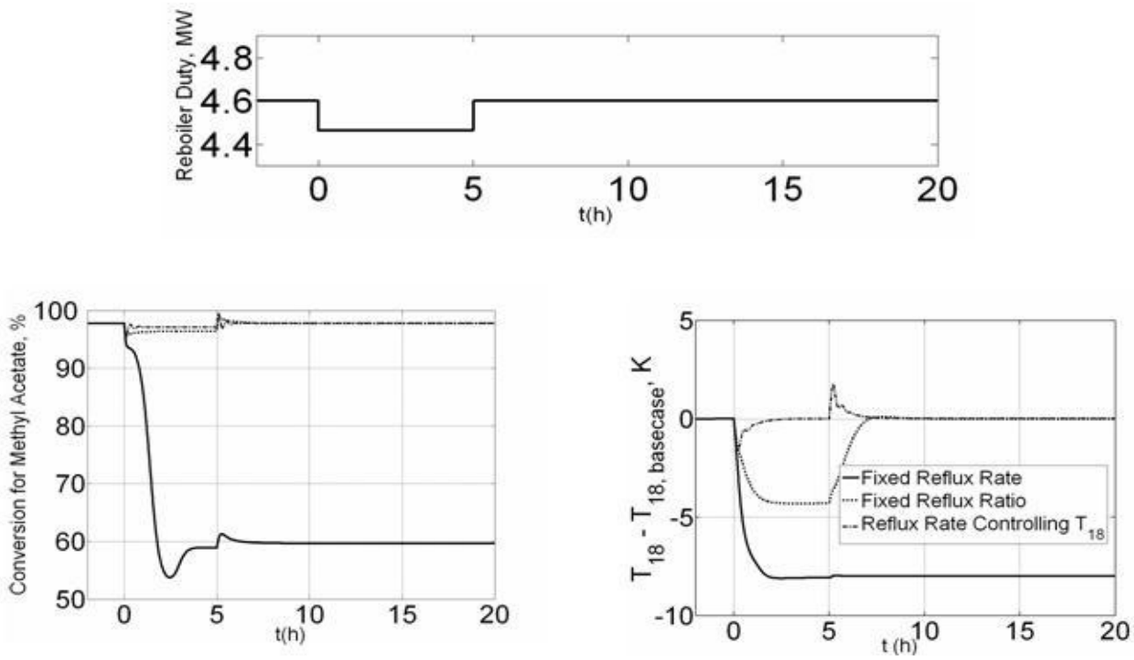
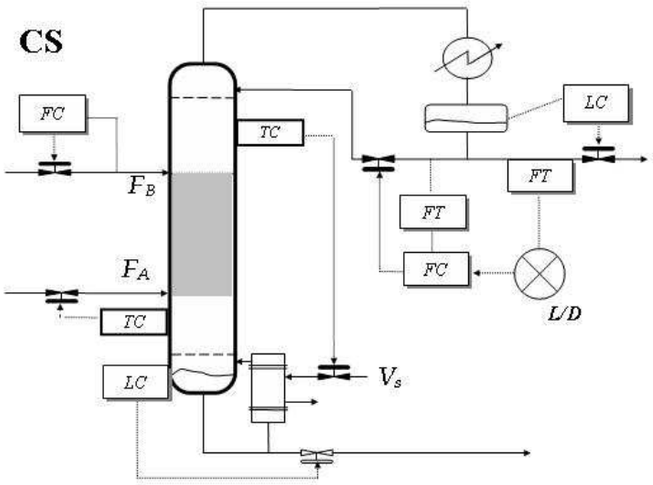


Fig. 6. Dynamic response of methyl acetate RD column for a pulse change in reboiler duty

to set the production rate (throughput) with the remaining two inputs available for column regulation. F_B is chosen as the throughput manipulator as the dynamic response of the tray temperatures (potential controlled outputs) to F_B is sluggish compared to V_S or F_A due to the associated large liquid hydraulic lags. V_S and F_A would thus be more effective manipulation handles for column regulation. From sensitivity analysis, a stripping tray temperature is the most sensitive to a change in F_A. Accordingly, F_A is paired with the sensitive stripping tray temperature (T_2, bottom-up tray numbering). V_S is then used as the manipulation handle for controlling a non-stripping (reactive or rectifying) tray temperature. Sensitivity analysis shows T_{18} to be the most sensitive rectifying tray temperature with T_{12} being the most sensitive reactive tray temperature, which is however lower than T_{18}. We therefore consider two alternative pairings namely T_{18}-V_S or T_{12}-V_S. A schematic of the two-temperature control structure is shown in Figure 7. The Niederlinski Index and Relative Gain Array of the two alternative control loop pairings are also given in the Figure and are found to be acceptable. These local metrics suggest T_{18} to be the better controlled variable.

The steady state input-output relations between the manipulated and controlled variables are now evaluated for multiplicity. The variation of three tray temperatures (T_2, T_{18} and T_{12}) with respect to all three inputs (F_B, F_A and V_S) is plotted in Figure 8. For easy comparison, the difference in the temperature from its nominal value is plotted with respect to percentage change in the inputs around the nominal steady state. Input-output relations are nearly monotonic with respect to V_S with an increase in V_S causing the tray temperature to increase. Although gain sign reversal is seen in T_{12} and T_2 for large negative change in V_S, the IO relations remain away from a crossover. On the other hand, crossover is seen with respect to F_B. In the T_{18}–F_B IO relation, crossover is observed at -22.5% and -30.8% and +22.7% change in F_B. With respect to F_A, directionality in response is observed with no change in T_{12} or T_{18} for an increase in F_A but a visible change for a decrease in F_A. The

response of T_2 (controlled using F_A) is better behaved with gain sign reversal for a decrease in F_A. But the IO relation remains away from crossover for a ±35% input change.



CS pairing	NI	RGA
$V_S - T_{18}$; $F_A - T_2$	0.8862	$\Lambda = \begin{matrix} \\ T_{18} \\ T_2 \end{matrix} \begin{matrix} V_s \\ \begin{bmatrix} 1.128 & -0.128 \\ -0.128 & 1.128 \end{bmatrix} \end{matrix} \begin{matrix} \\ F_A \end{matrix}$
$V_S - T_{12}$; $F_A - T_2$	0.7345	$\Lambda = \begin{matrix} \\ T_{12} \\ T_2 \end{matrix} \begin{matrix} V_s \\ \begin{bmatrix} 1.361 & -0.361 \\ -0.361 & 1.361 \end{bmatrix} \end{matrix} \begin{matrix} \\ F_A \end{matrix}$

Fig. 7. Two temperature control structure with *Niederlinski Index (NI)* and *Relative Gain Array (RGA)* of control loop pairings

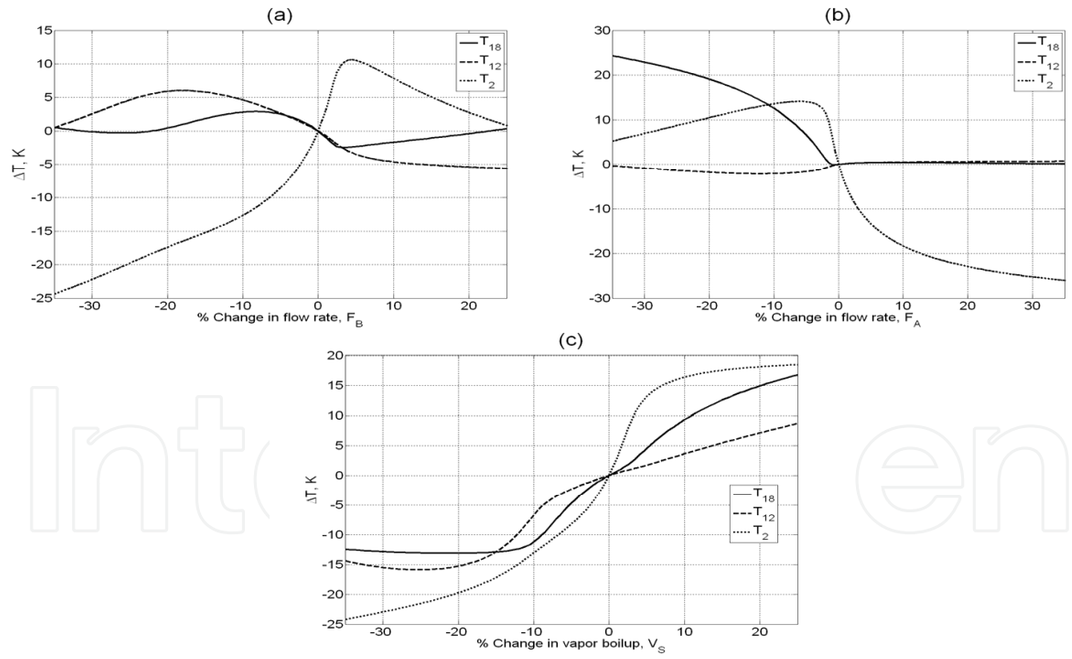


Fig. 8. Open loop variation of ideal RD column tray temperatures with inputs (F_B, F_A and V_S)

The open loop IO relation that a control loop 'sees' can be significantly different depending on whether the other loop is on manual (its input is fixed) or automatic (its output is fixed). To evaluate the same, open loop IO relations for the T_{18}-V_S pairing and T_2-F_A pairing are obtained with the output for the other loop (T_2 or T_{18}) maintained at its setpoint (nominal value). Similarly the T_{12}-V_S (T_2 fixed) and T_2-F_A (T_{12} fixed) IO relations are also obtained.

These are shown in Figure 9. The nominal steady state is marked *O* and the corresponding crossover points are marked *A*, *B* etc. A non-nominal steady state on a solution branch is stable if the local slope in the IO relation has the same sign as for the nominal steady state O, else it is unstable. Accordingly, the stable solution branch is shown as a continuous curve while the unstable solution branch is shown as a dashed curve.

For the T_{18}-V_S and T_2-F_A pairing, the input multiplicity steady states *A* and *B* are unstable with respect to controller action (reverse or direct) as the local slope sign of at least one of the IO relations is opposite the nominal slope sign. Steady state *C* on the other hand is stable. Disturbances that push the column towards *A* i.e., cause a large decrease in F_A/V_S, can result in wrong control action with saturation of a control input. On the other hand, disturbances that cause large increases in F_A/V_S can result in a closed loop steady state transition to steady state solution *C*. For the T_{12}-V_S and T_2-F_A pairing, both the input multiplicity steady states *A'* and *B'* are unstable with respect to controller action so that wrong control action with consequent valve saturation is expected for large changes in F_A/V_S in either direction (increase or decrease).

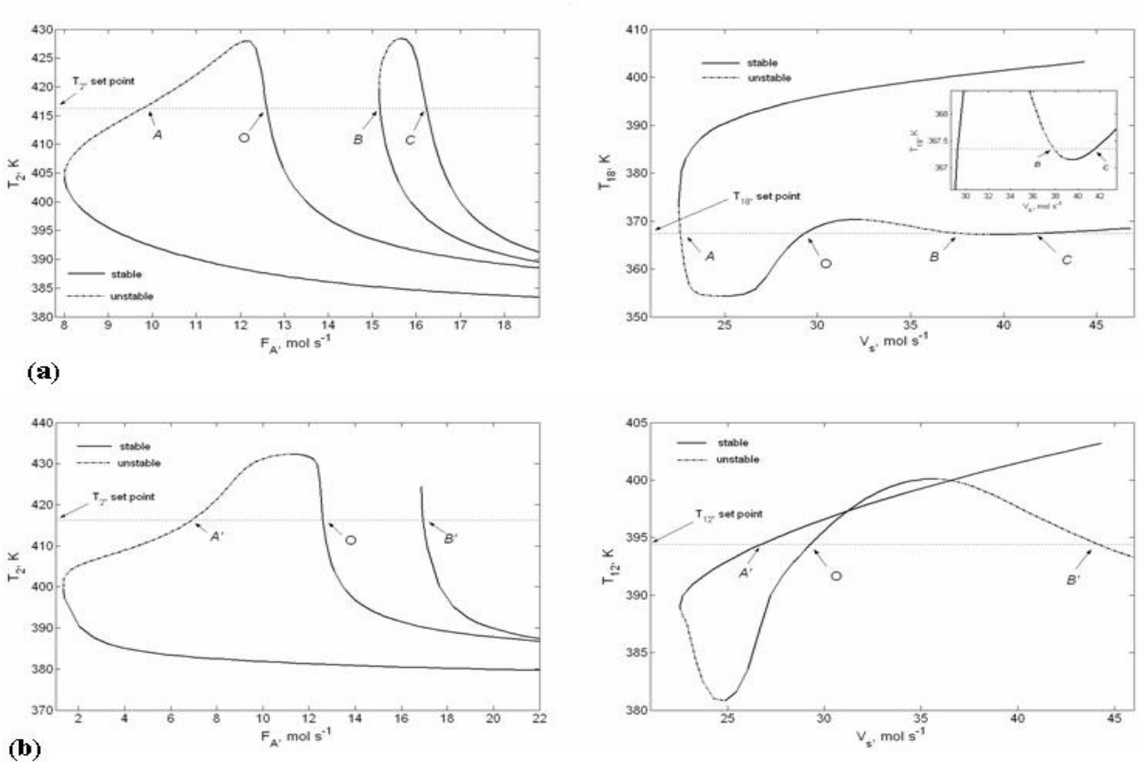


Fig. 9. Ideal RD column IO relations,
(a) T_2-F_A (fixed T_{18}) & T_{18}-V_S (fixed T_2) (b) T_2-F_A (fixed T_{12}) & T_{12}-V_S (fixed T_2)

Which pairing (T_{18}-V_S/T_2-F_A versus T_{12}-V_S/T_2-F_A) would handle larger disturbances without succumbing to wrong control action depends on the degree of tightness of control of the outputs. Usually tightest tray temperature control is usually possible with boilup as the manipulation handle. T_{18}/T_{12} is therefore likely to be controlled tightly without significant deviations from its nominal setpoint. Larger deviations in T_2 (controlled using F_A) can result in wrong control action due to input multiplicity corresponding to higher F_A feed into the

column (Figure 8 and Figure 9). In the T_2-F_A IO relation (Figure 9), notice that a crossover in T_2 occurs earlier when T_{18} is held constant compared to when T_{12} is held constant. Accordingly, one would expect controlling T_{12} to handle larger disturbances without wrong control action.

Using T_{18}/T_{12} and T_2 as controlled variables to manipulate V_S and F_A respectively, two different series of step changes are given to the throughput manipulator F_B to demonstrate the impact of input multiplicity under closed loop operation. The temperature controllers are tuned individually using the relay feedback test. The T_{18}-V_S loop must be detuned by a factor of 5 from its Tyreus Luyben settings to avoid a highly oscillatory response while not detuning is necessary when the T_{12}-V_S loop is implemented. In the first (second) series of step changes, the F_B flow rate value is decreased (increased) to 15% (20%) and then 30% (40%) below its basecase value at time 0 and 15 hr respectively, and then restored back to its nominal value of 12.6 mol s^{-1} at 30 hour. The closed dynamic results for these step changes when T_{18} is controlled are shown in Figure 10(a).

For the first series of step changes, stable closed loop responses are obtained for the changes made at 0 and 15 hr (Figure 10(a)). Tight control of the product purities with less than 1% deviations is achieved suggesting that two-point temperature inferential control provides effective column regulation holding the reaction and separation close to the nominal steady state. Upon restoration of the F_B flow rate to its nominal value at 30 hrs with a large 30% step increase, the F_A and V_S valves are completely closed. A sudden large increment of F_B

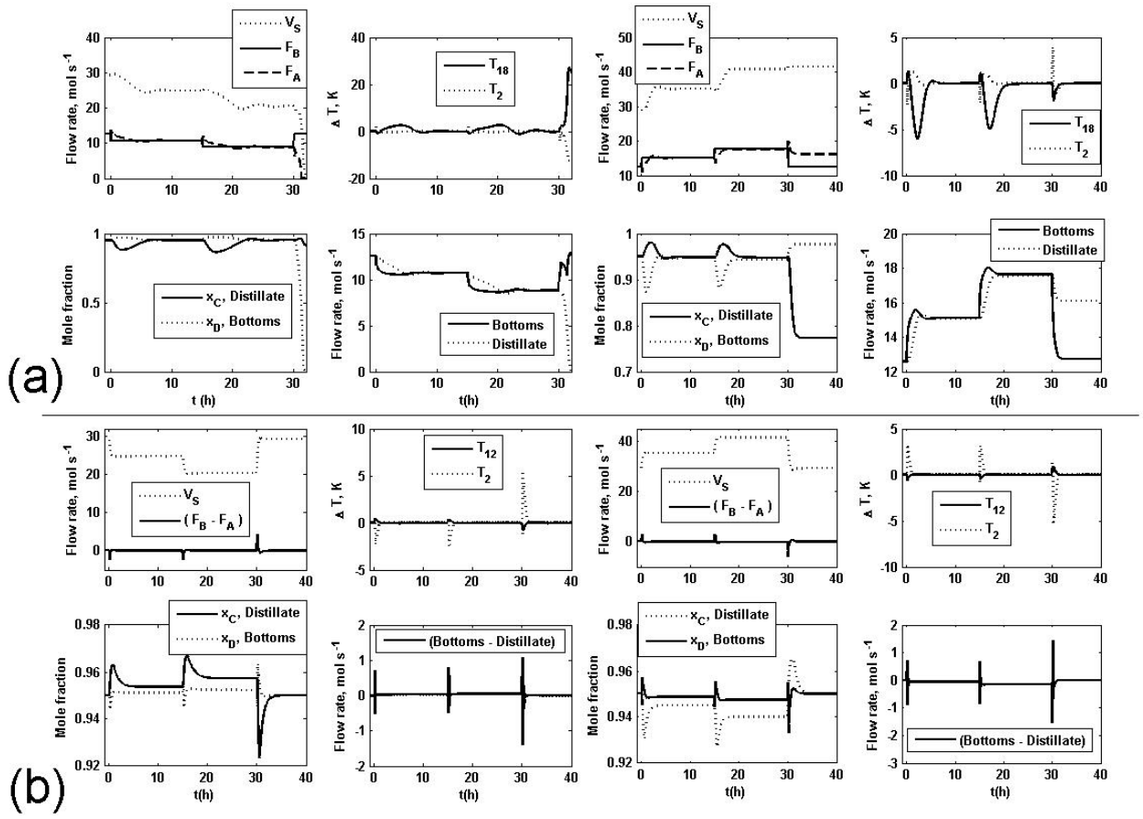


Fig. 10. The closed loop dynamics of ideal RD column for the two different series of step changes in F_B when (a) T_{18} (b) T_{12} is controlled variable

flow rate from 8.82 to 12.6 mol s^{-1} brings the column operation in the vicinity of point *A* in Figure 9 (relatively low F_A) with the consequent wrong control action causing a valve shutdown.

For the second series of step changes (+20%, +20% and -40%), a stable and well behaved response is observed for the two +20% step changes with acceptably small product purity deviations. However, for the -40% step change to bring F_B back to its nominal value, the column drifts to new steady state, i.e., settles at steady state *C* in Figure 9. The large F_B flow value decrease 17.64 to 12.6 mol s^{-1} at 30 hr, results in excess *A* input which causes a steady state transition to the stable steady state *C* in Figure 9. The same series of step changes in F_B (-15%, -15%, +30% and +20%, +20%, -40%) is effectively handled with no valve saturation or steady state transition due to wrong control action when T_{12} is used as the controlled variable manipulating V_S instead of T_{18}. The closed loop dynamic response is shown in Figure 10(b). The small steady state product purity deviations for the large throughput changes again highlight two-point temperature inferential control as an effective means of column regulation.

These results clearly demonstrate that proper choice of the controlled output variable can significantly improve the robustness of the control system in rejecting large disturbances. The results also highlight that the conventional wisdom of choosing controlled variables using local steady state metrics such as open loop gain or Niederlinski Index/relative gain may lead to the wrong conclusions. In the current example, the open loop sensitivity and relative gain for the T_{18}-V_S pairing are better than for the T_{12}-V_S pairing. A more comprehensive bifurcation analysis however reveals T_{12} to be the more robust CV. Such a comprehensive steady state analysis is strongly recommended for designing robust control systems for highly non-linear RD systems.

3.2.2 Methyl acetate RD column

In this RD column, column trays are numbered from top to bottom with the condenser as tray 0. As seen earlier, column operation at fixed reflux ratio avoids output multiplicity. Accordingly, the simple constant reflux ratio policy is implemented leaving the remaining three inputs, namely acetic acid feed (F_{HAc}), methanol feed (F_{MeOH}) and reboiler duty (Q_r) for column regulation. Sensitivity analysis shows that the temperature of tray 18 in the reactive section is very sensitive with respect to F_{HAc} and Q_r. In the stripping section, temperature of tray 34 is sensitive to all three inputs. Based on these sensitivities, two decentralized temperature inferential control structures, labelled CS1 and CS2, are synthesized, which are schematically depicted in Figure 11. In CS1, Q_r is the throughput manipulator, F_{HAc} controls a reactive tray and F_{MeOH} controls a stripping tray. This control structure was originally proposed by Roat et al. (1986). In CS2, F_{HAc} is the throughput manipulator with a reactive tray temperaure controlled using Q_r and a stripping tray temperature controlled using F_{MeOH}.

Further analysis is now conducted to check for multiplicity in the IO relations. As shown in Figure 12(a), all reactive tray temperatures (including the most sensitive T_{18}) exhibit input multiplicity with respect to changes in F_{HAc} and Q_r. To quantify the severity of input multiplicity, the rangeability (with a 3K offset) of the reactive tray temperatures with respect to Q_r and F_{HAc} are reported in Table 2. Even as reactive tray temperature, T_{18}, is the most sensitive to F_{HAc} and Q_r as evidenced from the slope at the nominal steady state in Figure 12(a), its rangeability is lower compared to reactive tray temperature T_{20}. To eliminate a

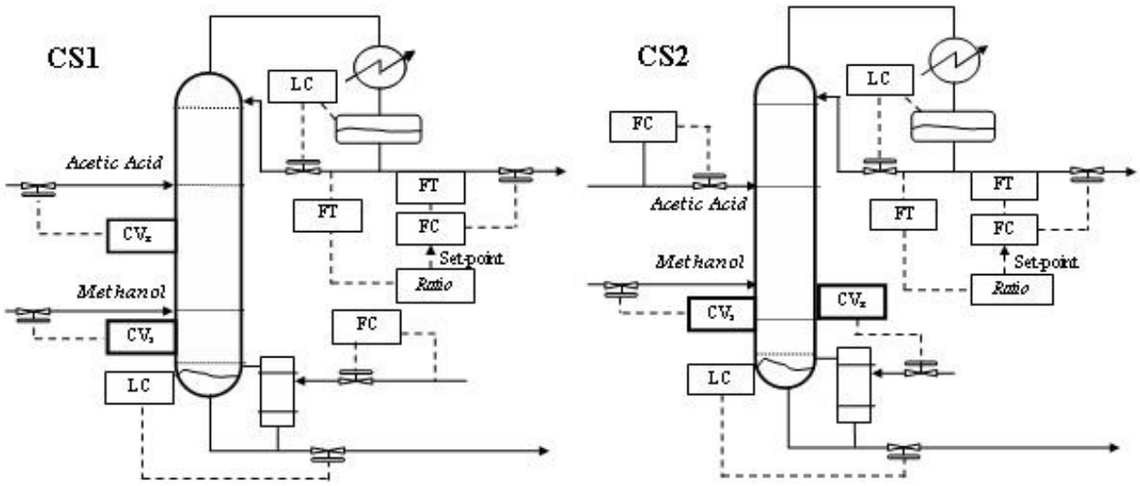


Fig. 11. Schemetics of two temperature control structures used for the methyl acetate RD column

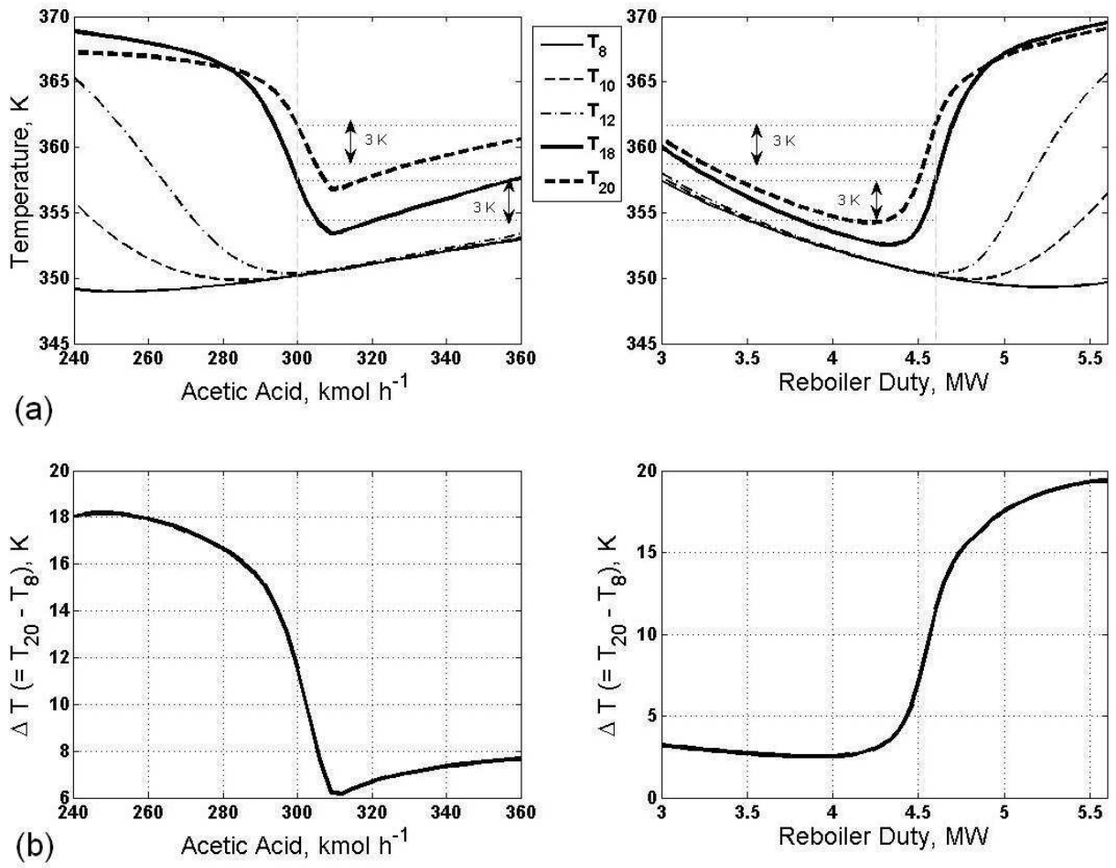


Fig. 12. Variation of (a) reactive tray temperatures and (b) $\Delta T = T_{20} - T_8$ with F_{HAc} and Q_r

crossover in the IO relations for high rangeability, we also consider a combination of tray temperatures. The difference between two reactive tray temperatures ($\Delta T = T_{20} - T_8$) was found to avoid input multiplicity with respect to F_{HAc} and Q_r with the corresponding IO relations in Figure 12(b).

In the T_{34}-F_{MeOH} IO relation, a crossover does not occur (data not shown) so that this pairing is fixed in both CS1 and CS2. For the reactive tray temperature control loop, there are three candidate controlled outputs in both CS1 and CS2, namely, T_{18}, T_{20} and ΔT ($T_{20} - T_8$). Superscripts '*a*', '*b*' and '*c*' are appended to the control structure label (CS1 or CS2) corresponding to T_{18}, T_{20} and ΔT, respectively, as the controlled reactive zone measurement. Note that T_{18} exhibits the highest sensitivity but low rangeability, T_{20} exhibits reasonable sensitivity with higher rangeability while ΔT exhibits the best rangeability with reasonable sensitivity. The three variants of each control structure are tested using rigorous dynamic simulations for the maximum throughput change handled in the worst-case direction. From the IO relations in Figure 12(a), for CS1, a step decrease in Q_r is the worst-case direction due to input multiplicity at reduced Q_r while for CS2, a step increase in F_{HAc} is the worst-case direction due to input multiplicity at increased F_{HAc}.

Tray Number	**HAc**		**Reboiler Duty**		**MeOH**	
	Decrease	Increase	Decrease	Increase	Decrease	Increase
16	>20	0	0	>20	>20	0
17	>20	0	6.9	>20	>20	0
18	>20	8.8	17.32	>20	>20	0
19	>20	10.9	24.63	>20	>20	0
20	>20	11.1	25.19	>20	>20	0
21	19.2	7	31.14	>20	>20	0

Values are in % change about their basecase values
A 3K offset is used in calculating rangeability

Table 2. Rangeability of reactive tray temperatures

The PI temperature loops are systematically tuned (Kumar & Kaistha, 2008). The two level controllers are P only with a gain of 2. The column pressure is assumed fixed, which is reasonable as in practice tight pressure control is achieved by manipulating the condenser duty. Also instantaneous flow control is assumed which is again reasonable in that tray temperature dynamics are significantly slower than flow dynamics.

Table 3 reports the maximum throughput step change handled by the different variants of the two control structures. CS1^a and CS1^b fail for a 20% and 30% throughput decrease respectively while CS1^c effectively handles 40% (larger changes not tested). The throughput increase for which CS2^a and CS2^b fail are respectively 25% and 40% while CS2^c works even for a 50% throughput increase (larger increase not tested). The trend in both CS1 and CS2 is in direct agreement with the increasing rangeability of the controlled outputs T_{18} (CS1/2^a), T_{20} (CS1/2^b) and ΔT (CS1/2^c). The result confirms the direct relationship between control system robustness and input multiplicity with rangeability being a useful metric for selecting 'robust' controlled variables. The result also shows that a well designed controlled variable such as ΔT with high rangeability and acceptable sensitivity results in a robust control system that effectively rejects large disturbances.

CS	CS1[a]		CS1[b]		CS1[c]		CS2[a]		CS2[b]		CS2[c]	
	-15%	40%	-25%	40%	-40%	40%	-40%	20%	-40%	35%	-40%	40%

Table 3. Maximum throughput change in either direction handled by the control structures

For the sake of brevity, the dynamic response to throughput change for CS1 and CS2 is not shown and may be found in Kumar & Kaistha (2008). These dynamic results show that controlling ΔT better prevents the breakthrough of heavy acetic acid from the reactive zone. In fact, the cause of input multiplicity in the IO relations is heavy acetic acid moving down and breaking through the reactive zone. This breakthrough would occur if the F_{HAc} is sufficiently increased above F_{MeOH} or if Q_r is sufficiently reduced, which results in the input multiplicity in the IO relations in Figure 12(a). For successful regulation of the RD column, such accumulation or breakthrough of acetic acid must be prevented and the same is effectively achieved by controlling ΔT.

In this example, an appropriate temperature based measurement could be designed that does not exhibit output multiplicity for robust column control. If such a temperature-based measurement is not evident for an RD system, controlling an appropriate tray composition may be considered. Even as online composition measurements are expensive, the additional expense would be justified in order to make the practical implementation of RD technology feasible.

4. RD design for controllability

The two case studies on control of RD columns clearly demonstrate that the existence of steady state multiplicity can result in hard-to-fathom nonlinear dynamic phenomena such as an open loop or a closed loop steady state transition, which can be particularly confusing for operators. In extreme cases where the non-linear effects cannot be sufficiently mitigated by appropriate choice/design of the controlled variable (including composition control), it may be necessary to alter the design of the column to mitigate the non-linearity for better controllability.

How to alter the column design to mitigate the non-linear effects? Several researchers have attempted to address this question for the ideal RD system with often contradictory claims (Huang et al., 2006; Kumar & Kaistha, 2008a, 2008b). To us, it appears that design modifications that help prevent escape of reactants from the reactive zone improve the controllability. To that end, for RD systems with exothermic reactions, the extension of the reactive zone into the stripping section with catalyst redistribution helps prevent the breakthrough of the heavy reactant from the reactive zone. Alternatively, the lower feed tray location may be moved up into the reactive zone. Reduced energy consumption has been demonstrated using a catalyst redistribution and lower feed tray location alteration. With respect to the original 5/10/5 ideal RD column design, controllability improves with catalyst redistribution only but deteriorates significantly when the lower feed tray location is moved up. A combination of the two provides acceptable controllability with significant energy savings. The extension of reactive zone into the rectifying section or upper feed tray alteration does not help improve controllability or energy consumption as the exothermic reaction causes the light reactant to escape up the top. For an endothermic reaction however, such a strategy may have merit (Huang et al., 2006).

For the methyl acetate column studied earlier, input multiplicity caused the control system to succumb to wrong control action for large throughput changes. Redistributing the catalyst onto the adjacent eight stripping trays results in significantly improved

controllability and energy savings (Kumar & Kaistha, 2008b). Figure 13 plots the variation in the methyl acetate purity with reboiler duty at a fixed reflux rate for this alternative design. Notice that unlike the original 7/18/10 design with conventional feed tray locations, the revised design does not exhibit output multiplicity with respect to the nominal steady state (compare with Figure 5). The non-linearity is thus mitigated in this alternative design with expectedly improved control performance. Thus for example, where CS1 for the original design with the most sensitive reactive and stripping tray temperatures as the controlled outputs succumbs to wrong control action for a -20% step change in the reboiler duty, the corresponding change is easily handled in the revised design (Kumar & Kaistha, 2008b).

The IO relation of product purity (top or bottom) with respect to a column input can be a useful tool to screen out poor designs exhibiting output/input multiplicity with respect to the nominal steady state. To demonstrate this for the ideal RD system, we consider the 7/6/7 design which is the most difficult to control using temperature inferential control (Luyben, 2000). The catalyst hold up on each tray is kept fixed at 1 kmol. Keeping the distillate rate equal to the fresh feed rate, the reflux ratio can be adjusted for reaction conversions of 90%, 95% or 98.5% with corresponding product purities of 90%, 95% and 98.5%. As shown in Figure 14, for a column pressure of 9 bars, the distillate and bottoms purity IO relations exhibit input and output multiplicity with respect to the nominal steady state for high conversions (and purities) of 95% and 98.5%. The multiplicity disappears for 90% conversion suggesting that high conversion RD columns are likely to exhibit multiplicity and therefore susceptible to consequent non-linear dynamic phenomena.

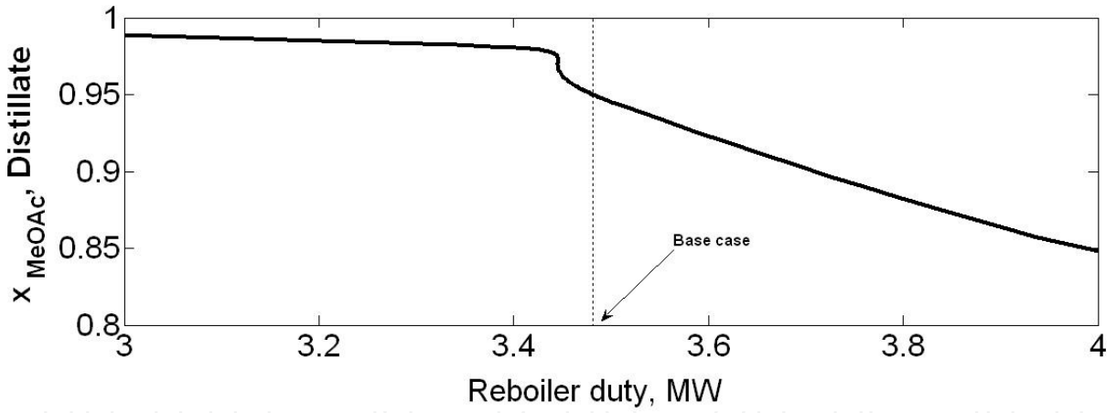


Fig. 13. Steady state variation of methyl acetate purity with respect to reboiler duty

We now consider column re-design for the highest considered conversion (and purity) of 98.5%. Holding the number of stripping trays equal to the number of rectifying trays, the number of reactive trays is increased and the IO relation of the distillate purity with respect to vapor boilup at constant reflux ratio is obtained. Similarly, holding the number of reactive trays constant, the number of stripping trays (equal to rectifying trays) is altered and the distillate purity-boilup IO relation is generated. Table 4 reports whether input or output multiplicity is observed in the different designs. From the Table, observe that simply reducing the number of rectifying (and stripping) trays from 7 to 4 causes the IO relation to be well behaved with no input/output multiplicity. The boilup is however too high and the design is uneconomical. No multiplicity is also observed for column designs with higher

number of reactive trays and not too many fractionation trays, specifically, in the 4/9/4 and 7/12/7 designs. Of these, the latter consumes much less energy with a 30.17% lower boilup than the former. This design thus appears to be a good one both from the process economics and controllability perspectives.

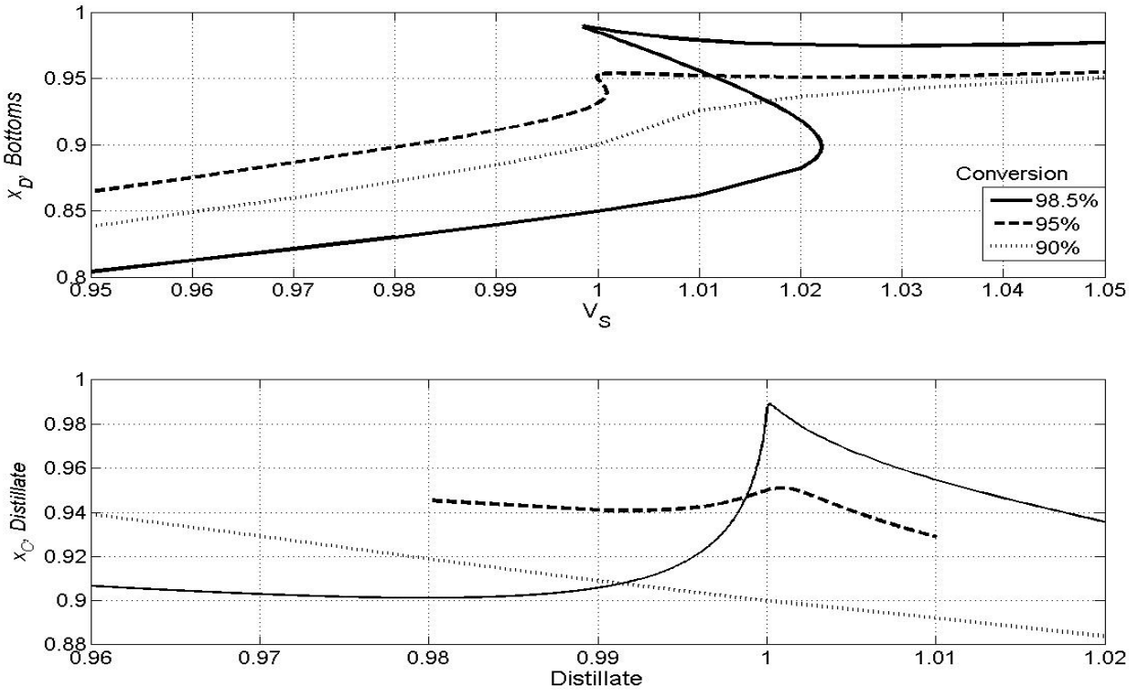


Fig. 14. Variation of $x_C{}^D$ & $x_D{}^B$ with vapour boilup and distillate for ideal RD 7/6/7 design

Design	Input multiplicity	Output multiplicity	Reflux ratio	Vapor boilup, mol s^{-1}
4/6/4	No	No	12.4400	151.4698
7/6/7	Yes	Yes	3.2841	36.1073
10/6/10	Yes	Yes	2.8155	30.2030
13/6/13	Yes	Yes	2.7311	29.1397
4/9/4	No	No	4.0037	45.1734
7/9/7	Yes	Yes	2.8312	30.4013
10/9/10	Yes	Yes	2.7868	29.8415
13/9/13	Yes	Yes	2.7774	29.7223
4/12/4	No	No	3.0407	33.0401
7/12/7	No	No	2.9055	31.3368
10/12/10	No	Yes	2.9007	31.2766
13/12/13	No	Yes	2.8996	31.2621
16/12/16	No	Yes	2.8989	31.2543

Table 4. Nature of the IO relation of bottom product purity versus vapour boilup (Ideal RD)

The multiplicity trends in the Table also suggest that excess fractionation capacity causes output multiplicity to 'appear' in the IO relations (compare e.g. 4/9/4 design with 7/9/7 design). The process design must therefore seek the appropriate balance between reaction capacity and fractionation capacity for well behaved IO relations (Bisowarno et al., 2004). For an economical design, sufficient reaction capacity must be provided.

We have dynamically tested both the 4/9/4 and 7/12/7 designs of 95% conversion ($x_{C,\ Distillate} = x_{D,\ Bottoms} = 0.95$) using two-point temperature inferential control structures similar to the ones studied earlier. Large throughput changes (up to 40%) in either direction are handled without wrong control action suggesting that these designs are inherently more controllable. This simple example demonstrates the power of steady state bifurcation analysis in arriving at economical RD column designs with good controllability.

5. Conclusions

To conclude, we hope that this Chapter has convinced the reader that a systematic evaluation of steady state multiplicity in RD systems is fundamental for designing robust control systems that effectively reject large disturbances. Specifically, the possibility of an open loop steady state transition due to output multiplicity and wrong control action under closed loop operation due to input multiplicity has been demonstrated for the example systems studied here. To improve the robustness of the control system, the controlled variables should be selected with care for a larger operating window around the nominal steady state without a crossover in the IO relation. In conjunction with local linear tools such as open loop gain, Niederlinski Index and relative gain, the proposed rangeability metric is a useful tool for selecting 'robust' controlled variables and rejecting poor choices that may potentially succumb to non-linear dynamic phenomena. The steady state IO relations (bifurcation analysis) can also help in arriving at an inherently more controllable and economical RD process design.

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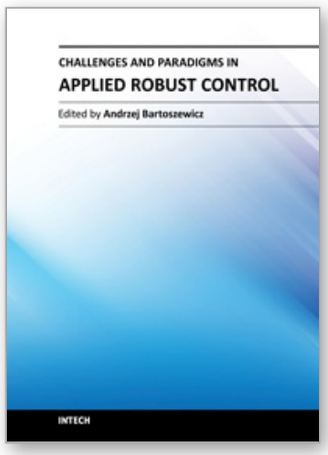

Challenges and Paradigms in Applied Robust Control

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The main objective of this book is to present important challenges and paradigms in the field of applied robust control design and implementation. Book contains a broad range of well worked out, recent application studies which include but are not limited to H-infinity, sliding mode, robust PID and fault tolerant based control systems. The contributions enrich the current state of the art, and encourage new applications of robust control techniques in various engineering and non-engineering systems.

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