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Integrated Process Design and Control of Cyclic Distillation Columns

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Abstract: Integrated process and control design approach for cyclic distillation columns is proposed. The design methodology is based on application of simple graphical design approaches, known from simpler conventional distillation columns. Here, a driving force approach and McCabe-Thiele type analysis is combined. It is demonstrated, through closed-loop and open-loop analysis, that operating the column at the largest available driving force results in an optimal design in terms of controllability and operability. The performance of a cyclic distillation column designed to operate at the maximum driving force is compared to alternative sub-optimal designs. The results suggest that operation at the largest driving force is less sensitive to disturbances in the feed and inherently has the ability to efficiently reject disturbances.

Keywords: Cyclic distillation, Process control, Process design, Process intensification, Driving force

1. INTRODUCTION

Distillation has been the dominant separation process over several decades in the chemical industry worldwide. However, the operating costs associated with separation by distillation account for substantial fractions of the total operational cost of the industry due to the significant energy demands (Kiss *et al.* 2012, Kraller *et al*. 2016, Sholl *et al.* 2016). Therefore, it is desirable to improve the energy and economic efficiency of distillation processes. A substantial body of the literature attempts to address this need for separations by introducing intensified and highly integrated process design alternatives such as reactive distillation, diabatic distillation, heat-integrated distillation, divided wall columns and cyclic distillation (Kiss *et al.* 2014). Here we focus on cyclic distillation, which has shown promising results by lowering the operational cost by 30-50% relative to conventional distillation due to the lower energy requirement (Bîldea *et al.* 2016).

Cyclic distillation is a highly efficient method of separation, with tray efficiencies substantially greater than those of classical trays. The underlying theoretical concepts and the engineering models of periodic cycling were developed sometime between 30's and 50's. However, practical results leading towards realistic applications have been much slower. A significant body of both experimental and theoretical studies have been made on cyclic distillation. Despite the significant benefits over conventional distillation methods, its large-scale implementation has not been as extensive as one might expect (Bîldea *et al.* 2016). Further investigations in relation to process control are needed to uncover the potential and reap fully the benefits of the technique.

Traditionally, process design and control have been considered separate sequential tasks, with process design coming before control system design. In intensified separation systems, this sequential approach can lead to controllability limitations and require unduly complex process control structures, even in distillation columns with side draws (Udugama *et al.* 2017a, 2017b). In more complex intensified distillation systems, such as reactive distillation, the sequential approach can be limiting due to the lack of degrees of freedom as process design decisions might influence the process control and operation (Mansouri *et al.* 2015, 2016a, 2016b, 2016c). Additionally, the sequential approach does not guarantee a robust performance due to its limitations related to dynamic constraint violations such as operating points, process over-design, or under-performance (Seferlis and Georgiadis, 2004). One way to overcome these limitations is to tackle design and control issues in an integrated fashion. Therefore, to assure that design decisions give the optimum operational and economic performance, operability and controllability issues are preferably considered simultaneously with the process design issues.

In this work, a methodology for integrated process and control structure design for reactive distillation in conventional columns, proposed by Mansouri *et al.* (2016a) is used to demonstrate the integrated process and control structure design of cyclic distillation columns, also known as periodic distillation. The applicability of the proposed methodology has been highlighted in various cases involving binary reacting mixtures (Mansouri *et al.* 2016a) and ternary reacting mixtures with one inert component (Mansouri *et al.* 2016b). Here, first cyclic distillation column design at the maximum driving force is obtained using the method of Nielsen *et al*. (2017). Next, we demonstrate that the same concepts that are valid at maximum driving force (for design and controllability) for conventional non-reactive and reactive distillation columns are also valid in case of non-reactive cyclic distillation columns.

2. CYCLIC DISTILLATION COLUMN DESIGN

Design of cyclic distillation columns, due to the separate vapor and liquid flow periods during a cycle, is a more complex task than design of conventional distillation columns, using graphical tools such as the McCabe-Thiele method. However,

a similar analysis using operating lines and the corresponding McCabe-Thiele constructions can be used for design of a cyclic column. To draw the operating lines of a cyclic column, the time-averaged vapor composition that enters tray n ($\overline{y_{n+1}}$) can be plotted against the liquid composition at the tray at the end of the vapor flow period ($x_n^{(V)}$). The McCabe-Thiele diagram for conventional distillation columns assumes continuous internal and external flows. However, for the cyclic column, the internal and external flows are also constant when expressed in terms of amounts per cycle during steady operation. Therefore, the McCabe-Thiele steps for the cyclic system are different from the classical McCabe-Thiele steps, as the tray efficiency, E_T, of a cyclic tray,

$$E_T = \frac{\overline{y_n} - \overline{y_{n+1}}}{y_n^{(V)} - \overline{y_{n+1}}} \quad (1)$$

This efficiency is substantially greater than that of a classical tray. To calculate the ideal number of stages for cyclic operation, a backwards integration method, like the one of Toftegård and Jørgensen (1987) and extended by Pătruț et al. (2014), can be utilized. The design algorithm requires a specified bottoms composition and knowledge about all the internal and external flows for the column. The algorithm integrates hereafter the mass-balances, for each stage, backwards in time, stage-by-stage. With this procedure, an approximate feed location is found together with the number of required stages for obtaining the specified separation. This design algorithm is however limited to only model saturated liquid feeds, which restricts the possibilities of operation. With an extended mass balance model, as suggested by Nielsen et al. (2017), the design algorithm can be used for mixed phase feeds ($0 < q < 1$). This makes it possible to obtain a driving force design for the cyclic distillation. The driving force, FD_i, is defined as the difference in composition of a component i between the vapor phase and the liquid phase:

$$FD_i = |y_i - x_i| \quad (2)$$

The driving force concept, based on identification of the largest driving force (see Figure 1), is used to find the optimal design target values of the process variables for separation systems. The algorithm for this combines the method of Pătruț et al. (2014) with parts of the driving force procedure by Bek-Pedersen and Gani (2004). The procedure is as follows:

Step 1: Find the maximum driving force composition (D_x) for the mixture,

Step 2: Specify product and feed compositions, all external flows, and the number of stages (NT),

Step 3: Specify the internal vapor-flow rate ($V \cdot t_{vap}$) and calculate the rest of the internal flow rates,

Step 4: Adjust q, so the operating lines intersect at $x = D_x$, and calculate the corresponding molar fractions x_F and y_F,

Step 5: Run the design algorithm for NT stages and place the feed stage NF where $x_{NF} \approx D_x$,

Step 6: If the distillate composition at the start of the vapor flow period matches the specified composition, the design has been obtained. If not, go to *Step 3* and adjust $V \cdot t_{vap}$ accordingly.

In this work, the design method of a cyclic distillation column is considered for a binary mixture of ethanol and water. In Table 1, the feed and product target compositions are given.

Table 1. The feed and product molar fraction specifications.

Component	z_F	x_B	x_D
Ethanol	0.1500	0.0001	0.8300
Water	0.8500	0.9999	0.2700

The Wilson thermodynamic model was employed to predict liquid phase activity coefficients, and the vapor phase is assumed to behave ideally. Figure 1 shows the driving force diagram to perform the separation task by a cyclic distillation column together with the operating lines (*SOL: stripper operating line, ROL: rectifying operating line*). The corresponding operating lines and analogous McCabe-Thiele constructions are shown in Figure 2.

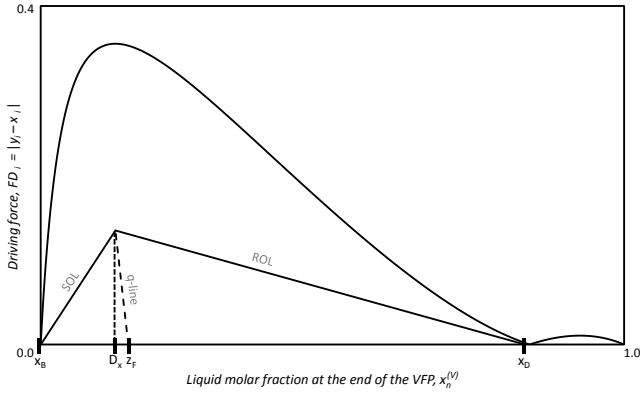

Fig. 1. Driving force diagram for the separation of ethanol-water mixture.

Table 2 lists the operating parameters for the separation (outputs of the design approach). The optimal feed location is two trays above the reboiler, which is tray 12. Figure 2 shows that cyclic distillation requires far less trays, compared to a conventional distillation column with the same internal flows, due to the enhanced tray efficiency.

Table 2. The operating parameters for the driving force based design of cyclic distillation column (*RR* is the reflux ratio, and *RB* is the boil-up ratio).

$V \cdot t_{vap}/F$	q	x_F	y_F	RR	RB
0.778	0.837	0.093	0.441	4.212	0.950

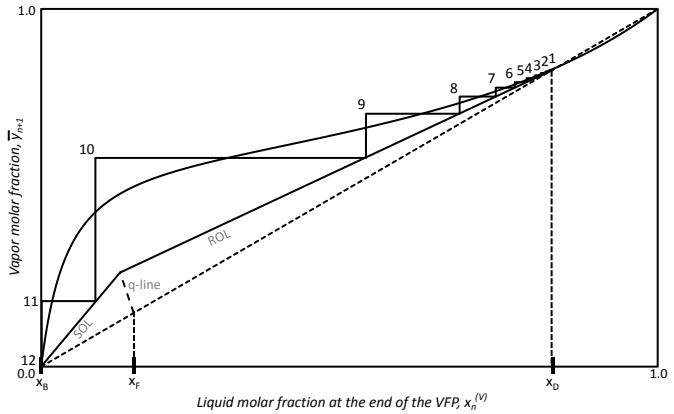

Fig. 2. McCabe-Thiele constructions for the cyclic distillation (stage 12 is the reboiler).

3. DYNAMIC PROCESS MODEL

Here we employ the dynamic process model of Andersen (2016), based on the model of Lita *et al.* (2014). The purpose of this model is to describe the internal and external vapor and liquid flows, the temperature, the composition profiles and the energy requirements in a cyclic distillation column. The process model is developed under the following assumptions: (a) vapor-liquid equilibrium is reached instantaneously, (b) perfect mixing on each stage, (c) negligible vapor hold-up, (d) negligible pressure drop throughout the column, (e) negligible heat exchange with surroundings, (f) complete condensation of entering vapor, and (g) any boiling liquid will remain boiling throughout the VFP. Energy and mass balances are shown for the liquid and vapor flow periods. The notation uses capital *H* for vapor enthalpies and lower case *h* for liquid enthalpies, where *M* is the liquid hold up. Superscript (*V*) and (*L*) respectively denotes values at the end of the vapor and liquid flow period, where subscript *j* indicates the specific component. Q is the energy input for the condenser and reboiler and *B*, *D* and *L* are respectively the bottoms, distillate and reflux streams. The equations are as follows:

Vapor flow period (VFP):

a. Mass balances for VFP:

Condenser:
$$\frac{d}{dt}M_{1,j} = V_2 y_{2,j} \tag{3}$$

Trays ($n = [2 \dots NF-2; NF \dots NT-1]$):
$$\frac{d}{dt}M_{n,j} = V_{n+1}y_{n+1,j} - V_n y_{n,j} \tag{4}$$

Tray above feed tray:
$$M_{NF-1,j} = V_{NF}y_{NF,j} + F_V y_{F,j} - V_{NF-1}y_{NF-1,j} \tag{5}$$

Reboiler:
$$\frac{d}{dt}M_{NT,j} = -V_{NT}y_{NT,j} \tag{6}$$

b. Energy balances for VFP:

Condenser:
$$\frac{d}{dt}h_1 = H_2 - Q_C \tag{7}$$

Trays ($n = [2 \dots NF-2; NF \dots NT-1]$):
$$\frac{d}{dt}h_n = H_{n+1} - H_n \tag{8}$$

Tray above feed tray:
$$\frac{d}{dt}h_{NF-1} = H_{NF} + H_F - H_{NF-1} \tag{9}$$

Reboiler:
$$\frac{d}{dt}h_{NT} = Q_B - H_{NT} \tag{10}$$

Liquid flow period (LFP):

a. Mass balances for LFP:

Condenser:
$$M_{1,j}^{(L)} = M_{1,j}^{(V)} - (D+L)x_{1,j}^{(V)} \tag{11}$$

Tray below condenser:
$$M_{2,j}^{(L)} = Lx_{1,j}^{(V)} \tag{12}$$

Trays ($n = [3 \dots NF-1; NF+1 \dots NT-1]$):
$$M_{n,j}^{(L)} = M_{n-1,j}^{(V)} \tag{13}$$

Feed tray:
$$M_{NF,j}^{(L)} = M_{NF-1,j}^{(V)} + F_L x_{F,j} \tag{14}$$

Reboiler:
$$M_{NT,j}^{(L)} = M_{NT,j}^{(V)} + M_{NT-1,j}^{(V)} - Bx_{NT,j}^{(V)} \tag{15}$$

b. Energy balances for VFP:

Condenser:
$$h_1^{(L)} = h_1^{(V)}\left(1 - \frac{D+L}{M_1^{(V)}}\right) \tag{16}$$

Tray below condenser:
$$h_2^{(L)} = \frac{L}{M_1^{(V)}}h_1^{(V)} \tag{17}$$

Trays ($n = [3 \dots NF-1; NF+1 \dots NT-1]$):
$$h_n^{(L)} = h_{n-1}^{(V)} \tag{18}$$

Feed tray:
$$h_{NF}^{(L)} = h_{NF-1}^{(V)} + h_F \tag{19}$$

Reboiler:
$$h_{NT}^{(L)} = h_{NT}^{(V)} + h_{NT-1}^{(V)} - \frac{B}{M_{NT}^{(V)}}h_{NT}^{(V)} \tag{20}$$

The liquid is assumed boiling at all times once it has reached its boiling point (see assumption (g) above). Therefore, during the VFP time derivatives of the tray temperatures can be

determined from chain-rule algebra when $df = 0$; thus, $f = 1 - \sum_j x_j K_j$.

Note also that the LFP is non-dynamic because all liquid hold-up on each tray dumps down to the stage below with no back mixing or any other interaction with the rest of the process. This is justified by the assumption of each tray having a sluice chamber, allowing for plug flow. The model is applicable for cases of multiphase feed, though the parameter q does not explicitly appear in the model equations. Its effect is incorporated by flashing the feed prior to introducing it to the column, thereby separating the feed flow F in a liquid and vapor fraction, respectively F_L and F_V. Thereby the vapor feed fraction is continuously supplied during the VFP to stage $NF - 1$ as shown in equation 5. The liquid fraction is transferred to stage NF during the LFP, as shown by equation 14. A pressure-enthalpy flash calculation evaluates the separation of the feed mixture. In order to implement this feature, the previous definition of the feed tray was altered, where the liquid feed would previously drop to stage $NF + 1$, meaning the designated feed stage differs by a single stage with this definition. Similarly, the mass and energy balances were modified, by splitting the feed into two individual stages. Inclusion of energy balances means that a non-constant vapor flow profile is obtained for the column. This means that one of the controlled variables (*RR* or *BR*) will deviate slightly from the pseudo-steady-state design based on models not employing energy balances.

4. OPTIMAL DESIGN-CONTROL SOLUTION

The development of an integrated approach can be achieved by taking into consideration key process variables and their target values that influence process-controller design. The solution to this optimization problem must balance the trade-offs between opposing process design and control requirements. As such, a systematic analysis needs to be performed to identify optimal design together with design-manipulated variables u, process-controlled variables y, and their target set points. It is important to note that their pairing significantly contributes to the integration of process design, operation and control. A systematic analysis in this context may provide additional or innovative options to address the conflicting trade-offs between process design, control and operation of an intensified distillation process such as cyclic distillation.

From a process design point of view, a set of process design objectives (specifications) needs to be determined at the maximum driving force that also satisfy the specified inputs, u, and disturbances, d, values for states, x, and outputs, y. In this case x and y also represent some of the operational conditions for the process. From a controller design point of view values of u need to be determined that are able to recover the process to its optimal designed condition at the maximum driving force, for any changes in d and/or set point values in y. It is also important to note that x and y are directly influenced by θ (the constitutive variables such as reaction rate or equilibrium constant). This concept is illustrated through representation of a dynamic process system in Figure 3. The optimal solution for x (states) and y (outputs) is obtained at the maximum point of the driving force; see diagram in Figure 3, which is based on θ (the constitutive variables). By model analysis, the corresponding derivative information (with respect to x, y, u, d and θ) which satisfies controller design objectives, can be obtained.

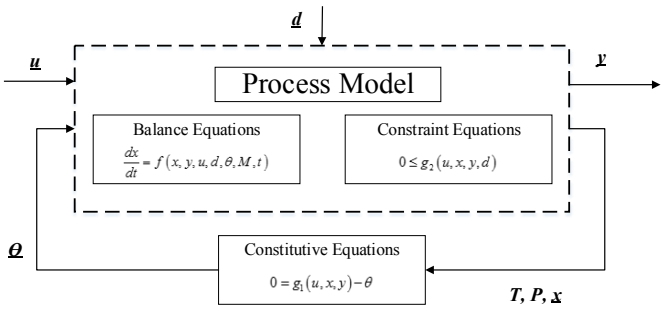

Fig. 3. Dynamic process system representation (Mansouri et al., 2016a)

As shown in section 2, selecting the design targets at the maximum driving force when designing the cyclic distillation column, the optimal design objectives are obtained. Furthermore, these design targets achieve the best controllability and operability of the process from a controller point of view. This means that, the derivative of the controlled variables y with respect to disturbances in the feed, d (*dy/dd*) and manipulated variables, u (*dy/du*) will determine the process sensitivity and influence of the control structure selection. Accordingly, *dy/dd* and *dy/du* are defined as (Russel et al., 2002):

$$\frac{dy}{dd} = \left(\frac{dy}{d\theta}\right)\left(\frac{d\theta}{dx}\right)\left(\frac{dx}{dd}\right) \tag{21}$$

$$\frac{dy}{du} = \left(\frac{dy}{d\theta}\right)\left(\frac{d\theta}{dx}\right)\left(\frac{dx}{du}\right) \tag{22}$$

The values for $d\theta/dx$ can be obtained from the process (dynamic and/or steady state) constraints:

$$\frac{dx}{dt} = f(x, y, u, d, \theta, Y, t) \tag{23}$$

and values for $dy/d\theta$, dx/dd and dx/du can be obtained from constitutive (thermodynamic) constraints:

$$0 = g(u, x, y) - \theta \tag{24}$$

It must be noted that at the maximum driving force, the sensitivity of controlled variables, y, to disturbances, d, is minimum while the sensitivity of y variables to manipulated variables, u, is maximum. This has been demonstrated in detail by Mansouri et al. (2016a, 2016b). Interested readers are referred to that work for further details and analytical analyses. Dynamic simulations were conducted in MATLAB on a system defined by the design parameters through the method by Nielsen (2017) – see section 2. The reboiler duty was approximated from the overall vapor flow for a total cycle, as the dynamic model simulates a dynamic and altering vapor

flow. The system was allowed to reach pseudo-steady state under these conditions, where the combined mass and energy model's response to the design decisions was obtained. The reboiler duty was gradually altered as to obtain the exact averaged vapor flow, where the reflux ratio differs. Perturbations of ±10% were introduced in the feed flow rate as to evaluate the open loop response of this. This was applied to different systems, where all parameters were unchanged except the feed stage.

The condenser's liquid hold-up, the reflux flow and the distillate flow are constrained, leaving only a single control variable as stated by Matsubara (1982). This may be the vapor flow duration, reboiler duty or changes in the feed. Easy controllability was provided by the reboiler duty, which was altered to achieve the desired purities of the products, meaning the reflux ratio changed as well as to retain a constant condenser hold-up. This clearly illustrated that the smallest disturbance in both top and bottom composition was obtained while operating at a feed stage corresponding to the optimal driving force, as expected. A simple discrete PI controller was introduced as stated by Matsubara (1982), of the following form

$$(\theta_v)_{i+1} = (\theta_v)_i - K_P(e_i - e_{i-1}) - K_I e_i. \quad (25)$$

The manipulated value θ_v was chosen as the reboiler duty, Q_B, and e is the control error of the bottom product concentration. It was chosen to monitor the offset in the ethanol concentration. In this case, the controller was tuned relatively aggressively as the inherent cyclic nature of the process coupled with relatively long column time constants required aggressive control actions to keep the column within specification.

The fastest response time for the closed loop simulations was obtained while operating as close to the maximum driving force as possible. Not only is the fastest response time observed, but generally the magnitude of the fluctuations is smaller, though there are exceptions. To test the ability of the proposed optimal design to reject disturbances, the optimal cyclic distillation column design was compared with two suboptimal designs. Critical process parameters of these three designs are recorded in the table below, Where B = 245.8 (bottom flow rate), D = 54.2 (distillate flow rate) and F = 300 (feed flow rate) are recorded in moles per cycle. The design alternatives are considered by altering the feed location.

Table 3. Operating parameters for optimal design and alternative sub-optimal designs

Design	$\mathbf{N_F}$	$\mathbf{N_T}$	$\mathbf{t_{VFP}}$ [s]	$\mathbf{Q_B}$ [kW]
Optimal (FD Design)	12	13	9.41	1019
Suboptimal 1	10	13	9.41	1136
Suboptimal 2	9	13	9.41	1460

Based on the results in Table 1, it can be expected that the optimal distillation column design should have a lower reboiler duty on average. In Figure 4, the response of the optimal cyclic distillation column design along with two suboptimal designs for a ±10% disturbance in the feed flow rate is observed. All these designs use the same process control strategy and process tuning parameters and the results shown are these closed-loop responses. Analyzing the all-important reboiler duty variable, which is the manipulated variable in this control structure, illustrates that the optimal design has a noticeably lower reboiler duty usage for both feed flow perturbations.

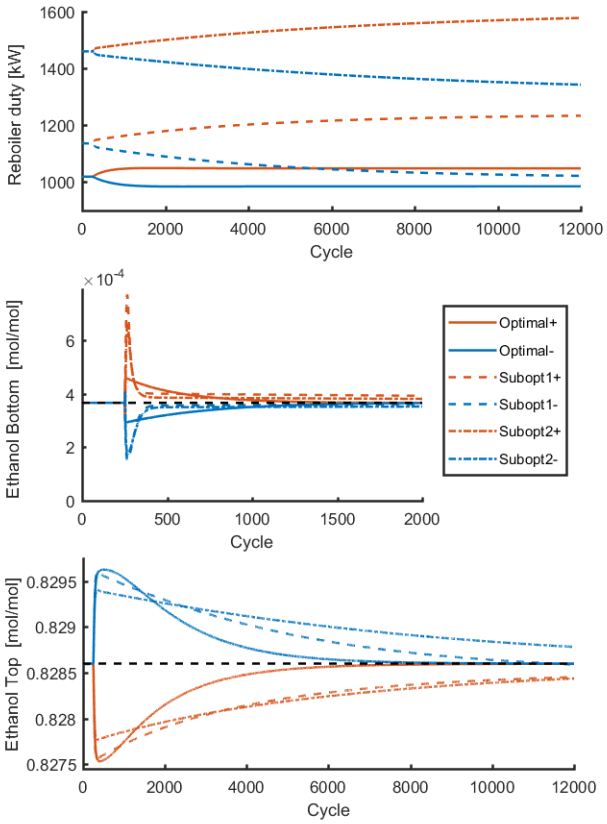

Fig. 4. The closed loop process response of the optimal process design and suboptimal process designs to ±10% change in feed flow rate.

Closer inspection reveals that the two suboptimal controllers continue to have a process offset for a relatively long duration. Thus, it can be concluded that the optimal process design has better disturbance rejection characteristics as the integral absolute error (IAE) is two times smaller than for the competing suboptimal designs. If a design is able to quickly return the controlled process variable to its steady state value it illustrates the design's ability to stabilize the column during process disturbances. In this instance, the superior controllability is associated with the optimal process design, which swiftly is able to bring the bottom ethanol concentration back to its set point with a smaller absolute offset than the sub-optimal designs. The variation in the controller variable is also smaller, illustrating that operating at the optimal driving force ensures economic optimization as well. It should be stressed that the objective was not to optimize the controller, but to exemplify the principle of optimized controllability, where further tuning of the controller would increase the efficiency vastly. Long settling times are observed in the top, however

due to the control structure this response is the system's natural response, on which the controller performance cannot be evaluated where the relative offset is furthermore low.

Overall, based on this evidence it can be concluded that the optimal cyclic distillation column design based on the maximum driving force has the best controllability. In terms of process controllability and potential operational optimization, this means that designing a cyclic distillation column at its maximum driving force would allow for relatively tight and very responsive process control. As such, in comparison to the two suboptimal designs presented, the optimal design would possess much better disturbance rejection characteristics. This in turn would allow industrial operators to operate an optimally designed column much closer to hard product specifications.

5. CONCLUSIONS

In this work, for the first time an integrated process design and control approach based on the driving force has been explored for cyclic distillation columns. The approach has been applied to design a cyclic distillation column for separation of a binary mixture of water and ethanol. For comparison, two suboptimal process designs (that are not operating at the maximum driving force) were also considered. The controller performance of the design at the maximum driving force (optimal design) and suboptimal design alternatives were then fitted with a standard process control scheme proposed by Matsubara (1982) with identical process tuning parameters. These design alternatives were then tested against feed flow disturbances where it was clearly demonstrated that the optimal process design at the maximum driving force provides better process controllability, column stability as well as energy efficiency.

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