

## OPTIMAL OPERATING POLICIES OF BATCH DISTILLATION WITH EMPHASIS ON THE CYCLIC OPERATING POLICY

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**Abstract:** Factors that are important for the optimal operation of batch distillation are discussed in this paper. The cyclic operation policy is presented in detail and is compared to the conventional policies (constant distillate, constant reflux ratio and optimal reflux ratio operation) with respect to total batch time. The cyclic policy has been noted earlier because it may be easier to operate and control. Surprisingly, we also find that for some separations, the cyclic policy may significantly reduce the batch time compared to the conventional operating strategies. A simplified cyclic policy is presented which is almost as optimal as the cyclic policy but easier to calculate and implement.

### 1 Introduction

Batch distillation is used in the chemical industry in the production of small amounts of products with high added value and for processes where flexibility is needed, for example, when there are large variations in the feed composition or when production demand is varying. With an increasing demand for product quality and product flexibility, the ability to run the batch distillation columns as efficiently, or as optimally, as possible is of great economic importance.

The optimization of batch distillation has been studied in the past (e.g. [6, 7, 10, 11, 13]), but mostly the optimal reflux ratio, and to a lesser extent the optimal reboiler heat duty, has been considered as optimization variables. However, there are other factors that may be much more important for the optimal operation of a batch column. We will in this paper discuss some of these factors.

Given a batch distillation column with a fixed boilup rate the amount of reflux determines the degree of separation and therefore the product rate. In batch distillation, the reflux rate is normally controlled by one of the following three conventional operating policies ([17]):

- 1) changing the reflux to hold a constant product composition (constant distillate composition policy)
- 2) maintaining a constant ratio of reflux flow to distillate flow (constant reflux ratio policy)
- 3) changing the reflux ratio according to some optimization criterion (optimal reflux ratio policy)

A fourth alternative usually not considered is

- 4) alternating between total reflux and low reflux (cyclic policy)

The first three alternatives all assume constant condenser holdup whereas the fourth uses the condenser holdup as a degree of freedom. A generalization of the cyclic policy is to let the

reflux ratio and the condenser holdup vary freely with time and find their optimal profiles by optimization.

In this paper we focus on the cyclic operating policy. It has several advantages compared to the conventional schemes. It utilizes the maximum attainable separation in the column. There is a minimal need for control and also it is less sensitive to disturbances and therefore safer to operate. For small laboratory columns accurate measurement of flows may be very difficult and the cyclic operation is therefore well suited for these columns since no flow measurements are needed. A disadvantage however, is that the composition of the batch feed must be known quite exactly.

### 2 Factors important for optimal operation

Finding the optimal reflux ratio for batch distillation has received increasing attention in the literature during the last decade. However, there are other factors that may be much more important for the optimal operation of a batch column, such as:

- 1) Active use of varying condenser holdup (e.g., cyclic operation with filling and dumping)
- 2) Starting the batch with product from the previous batch
- 3) Recycling and mixing of off-cuts from different batches
- 4) Concentrating the product in several steps
- 5) Inverting the column, i.e. charging the feed in the condenser
- 6) Operating pressure
- 7) Steam distillation, or generally to continuously add a volatile component to effectively reduce the pressure
- 8) Addition of an entrainer to improve the separation, for example to break an azeotrope

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Most of these items are of course well known, but they have usually not been considered as optimization variables. The two last items are well known also from continuous distillation. The addition of a volatile component to the batch charge, will effectively reduce the pressure and thereby lower the boiling point temperature and make it possible to use a lower-value energy source and to avoid possible thermal decomposition. One special case is where the heat is direct supply of energy in terms of steam as in steam distillation. Another possible strategy, when the top product is sent to waste-water treatment, is to use pure water as reflux similar to an absorption column.

The use of an inverted column, where the feed is charged to the condenser, has been suggested by several authors (e.g. [18]). Intuitively, it seems that this strategy is well suited when the feed mixture is rich in the most volatile component, since conventional batch distillation will be expensive in terms of energy since all of the light component has to be evaporated. However, the inverse column is not truly the inverse of a regular batch column, since this would require the batch, which is stored in the condenser, to be in the vapor phase which is very unlikely. This means that even with an inverted column the light component has to be vaporized at least once, and the main advantage seems to be lost.

Batch distillation is sometimes used as part of a continuous process or to separate a quantity of material much larger than the capacity of the reboiler. This is performed by distilling a sequence of similar batches and any off-specification material from one separation may therefore be returned to the reboiler for reprocessing by mixing it with fresh feed ([5, 14]). The off-cuts may also be collected and processed separately.

When the desired purity is not obtainable in the column, the batch may be processed in several steps with increasing purity of the product. A well known example of this is the separation of ethanol from water by evaporation (Rayleigh distillation).

The operation of a conventional batch distillation column can be divided into three periods: 1) startup period, 2) production period and 3) shutdown period. Normally the production period is the most time consuming, but for difficult separations such as for high purity or azeotropic separations the startup time may also be significant ([9, 15]). Alternative ways of reducing the length of this period such as starting the batch with light product or pure component in the condenser should therefore be considered.

### 3 The cyclic operating policy

In the remaining part of this paper we consider the cyclic operation of a batch column in which the *external* reflux and distillate flows are cycled. This should not be confused with the controlled cycling of continuous distillation columns (e.g. [2, 4]) in which the internal flows are cycled. The *cyclic operation* is characterized by repeating three periods of operation: "filling up", "total reflux" and "dumping". During the first period the condenser is filled up and the filling time is determined by the boilup rate. During the second period the column is run under total reflux and the maximum attainable separation in the given column is achieved. During this period the light component accumulates in the condenser drum until the column reaches equilibrium or steady state. During the last period the condenser is dumped and the product is withdrawn. The dumping time is determined by the maximum distillate flow out of the condenser. This three step procedure is repeated until the specifications are satisfied for either the product in the accumulator or for the remaining material in the reboiler

and column. The number of cycles is largely determined by the ratio between the amount of initial feed to the reboiler and the maximum holdup in the condenser drum but also by the product specifications. The cycle times for this policy will be in the order of minutes and hours.

The *cyclic operating policy* for batch distillation columns has only been mentioned by few authors in the literature ([1, 3, 8, 9, 16]). Gonzalez-Velasco et al. [8] found that the cyclic operating policy (in their paper called *periodic batch distillation*) produces a higher sharpness of separation between components than the conventional constant reflux ratio policy. Their results were based on calculations of the steady state conditions corresponding to the end of the total reflux period for separations with 22 operating cycles and with varying relative volatilities between three components. No comparison was made between the two policies in terms of time consumption. Barb and Holland [1] presented a dynamic and a steady state model for the total reflux period as well as dynamic models for the dumping period of the cyclic operation and for the conventional operation with constant or varying reflux ratio. The results were compared with experiments. However, only the models' ability to describe the course of the distillation in terms of distillate composition as a function of accumulated product was considered. The results by Barb and Holland [1] were also presented in the book by Holland and Liapis [9]. Block [3] discussed control of batch distillation columns. He proposed alternating between total reflux and no reflux (cyclic operation) as a mean for controlling the reflux rate but without giving any details about this operating scheme. However, he reported that one of the main difficulties is how to establish proper intervals of total and zero reflux.

Nowicki and Gorak [16] presented a steady state model for the determination of the end point of the total reflux period for cyclic operation. They presented an example where a high purity separation was performed but used only one cycle to accomplish the separation. They reported that the energy consumption for this one cycle was only slightly higher than for the equivalent constant reflux ratio operation.

The cyclic operating policy has, to the best of our knowledge, not been studied as an alternative to the conventional operating policies with respect to optimal operation. The purpose of this paper is to compare the cyclic operating policy in terms of time consumption with the conventional operating policies mentioned in the introduction. We will present guidelines for

- 1) when the cyclic policy is to be preferred over the conventional operating policies.
- 2) how to determine the end point of the total reflux period and also
- 3) the selection of number of cycles to be used for a given separation

### 4 Comparison of operating policies

In this section we present numerical examples to compare the optimal operation of the various operating policies. By optimal operation we mean the policy that maximizes profit for a given separation in a given column. For many practical cases maximizing the profit will be equivalent to minimizing the total batch time  $t_f$  with the heat input (boilup) at its maximum. This is the problem considered in this paper. We solve the minimum time optimization problem given constraints on the final accumulated light product. The optimization problem can be formulated as:

$$\min t_f \quad (1)$$

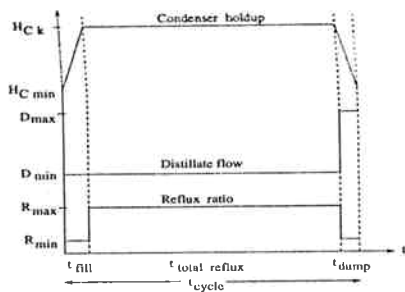


Figure 1: Reflux ratio, distillate flow and condenser holdup for one cycle of a cyclic operation.

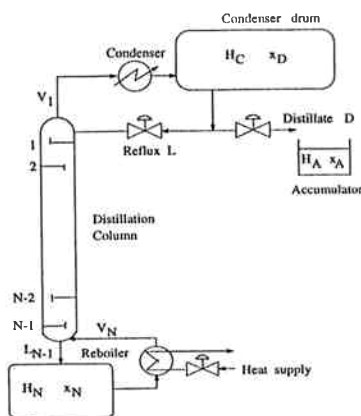


Figure 2: Batch distillation column.

where  $t_f$  is the total batch time and subject to the following end point constraints for the accumulated product:

$$H_A \geq H_{A,spec}, \quad x_A \geq x_{A,spec} \quad (2)$$

In words, the objective is to produce an amount  $H_{A,spec}$  with composition  $x_{A,spec}$  in a minimum duration of time  $t_f$ . In this paper, the optimization problem has been solved in the following way for the four policies:

- 1) *Constant distillate composition.* The distillate composition is kept at a specified set point value  $x_D^j$  using a PI-controller with the reflux ratio  $R$  as manipulated variable. The set point for the composition controller  $x_D^j$  is optimized in order to minimize  $t_f$ . (The use of the distillate composition directly as an optimization variable would result in a high index problem which can not be solved by the given optimization software and which in any case can not be realized in practice).
- 2) *Constant reflux ratio.* The optimal constant reflux ratio  $R$  is found that minimizes the total batch time.
- 3) *Optimal reflux ratio.* The optimal reflux ratio as a function of time  $R(t)$  is found that minimizes the total batch time. (This problem is equivalent to finding the optimal  $x_D(t)$ ). In this paper we use a slightly suboptimal policy where  $R$  is changed linearly at certain intervals.
- 4) *Cyclic policy.* For a given number of cycles,  $N_{cyc}$ , one finds for each cycle  $k$  the optimal condenser holdup  $H_{C,k}$  and the cycle time  $t_{cycle,k}$  that minimizes the total batch time (see Figure 1).

#### 4.1 Numerical example

As an example we consider the separation of a binary mixture with constant relative volatility  $\alpha = 1.5$  in a column consisting of a reboiler, 10 theoretical trays, a condenser and an accumulator. The column is given in Figure 2 and the data used

Number of trays:	10
Initial feed:	$H_F^0 = 10$ kmol
Feed composition:	$x_F^0 = 0.25$
Initial tray holdups, $H_j^0$ :	0.01 kmol
Relative volatility, $\alpha$ :	1.5
Product holdup:	$H_{A,spec} = 1.875$ kmol
Product composition:	$x_{A,spec} = 0.9$
Bounds on variables,	
Condenser holdup:	$[H_{C,min}, H_{C,max}] = [10^{-6}, 1.875]$
Distillate flow (kmol/hr):	$[D_{min}, D_{max}] = [0.0, 25.0]$
Vapor flow (kmol/hr):	$[V_{N,min}, V_{N,max}] = [10^{-6}, 10.0]$
Reflux ratio:	$[R_{min}, R_{max}] = [0.01, 1.0]$
Controller parameters,	
constant distillate policy:	$K_p = 5.0$ and $\tau_I = 0.05$ hr

Table 1: Process data for simulation and optimization.

for the optimizations are given in Table 1. Before the separation starts the feed is added to the reboiler. It is assumed that some of the feed is initially distributed on the trays in the column (i.e.  $H_N^0 = H_F^0 - N \cdot H_j^0$ ) with a composition equal to that of the feed ( $x_j^0 = x_F^0$ ). The initial holdups in the accumulator and in the condenser are negligible. The vapor flow (which is indirectly given by the reboiler heat duty) is in this paper assumed constant at its maximum value except during the dumping period in the cyclic policy where it is reduced. This is done for numerical reasons but the boilup would in practice be constant during the whole cycle. The maximum vapor and distillate flows are given by the column design and are in this paper chosen to be  $V_{N,max} = 10$  kmol/hr and  $D_{max} = 25$  kmol/hr. The minimum reflux ratio is set to a small value for numerical reasons but would in practice be zero. It should be noted that the reflux ratio is given as the internal reflux ratio since this is better solved numerically:

$$R = \frac{L}{L + D} \quad (3)$$

For the cyclic operation, the filling and dumping times are given by

$$t_{fill,k} = \frac{H_{C,k}}{V_{max}} \quad (4)$$

$$t_{dump,k} = \frac{H_{C,k}}{D_{max}} \quad (5)$$

For the conventional policies the condenser holdup is assumed to be negligible ( $H_C = H_{C,min}$ ) since this will normally minimize the batch time [12]. For the constant distillate composition policy, the parameters for the PI-controller are given in Table 1.

The assumptions for the mathematical model of a batch distillation column used in this study are: (1) staged distillation column, (2) perfect mixing and equilibrium between vapor and liquid on all stages; (3) negligible vapor holdup; (4) constant molar flows; (5) linear tray hydraulics; (6) total condensation with no subcooling in the condenser and (7) constant relative volatility. The linear tray hydraulics model has the form  $L_j = L_j^0 + (H_j - H_j^0)/\tau$  where  $L_j^0$ ,  $L_j$ ,  $H_j^0$  and  $H_j$  are the initial and current instant liquid flow rate from and molar holdup on the trays respectively, and  $\tau$  is the hydraulic time constant given by  $\tau = 2/3 \cdot H_j/L_j$ .

#### 4.2 Results

The optimal results for the four policies were obtained using the optimization program DAEOPT developed at Imperial College, London [19]. The optimization accuracy was  $10^{-3}$  for

the cyclic policy and  $10^{-4}$  for the others. It should be noted that for optimizations with more than one interval or cycle, local minima would be expected which makes the problem difficult to solve numerically.

For the optimal reflux ratio both a continuous linear and a piecewise constant policy is presented. The optimal results for the cyclic policy are presented for 1, 2, 3 and 4 cycles. The case with 5 cycles did not converge. The optimization for the cyclic operation is however, very time consuming. The solution for 4 cycles took about 17 CPU hours on a Sparc 10. This is because the number of optimization variables using DAEOPT is  $13N_{cyc}$  where  $N_{cyc}$  is the number of cycles whereas the actual number of optimization variables is only  $2N_{cyc}$  (total reflux period and condenser holdup).  $9N_{cyc}$  variables will be pre-specified (vapor flow, reflux ratio and distillate flow for each period). Of the remaining  $4N_{cyc}$  variables,  $2N_{cyc}$  of these (the values of  $t_{fill,k}$  and  $t_{dump,k}$ ) are found by optimization, even though their optimal values are given by Eq. (4-5). Therefore  $4N_{cyc}$  (periods and condenser holdup) are actually optimized, but the sensitivities for all the  $13N_{cyc}$  variables must be calculated for each intermediate solution of the optimization problem thus making the solution very time consuming.

The optimal results for the four policies are presented in Table 2. The constant distillate composition policy is found to be by far the most time consuming policy for this example with  $t_f = 9.53$  hr. The constant reflux policy (case 2a) yields  $t_f = 5.73$  hr. Normally the constant reflux policy is operated with an initial period with total reflux and the results for case 2b shows that an initial total reflux period of 0.05 hr reduces the total batch time to 5.60 hr.

For the optimal reflux policy we consider two constant intervals (case 3a), two linear intervals (case 3b) and ten constant intervals (case 3c). When comparing these we see as expected that the batch time decreases with increasing number of intervals (cases 2a, 3a and c with  $t_f = 5.73$  hr, 5.53 hr and 5.26 hr).

Note that for the case with two constant intervals (case 3a) letting the reflux ratio vary freely does not yield a total reflux solution at  $t_f = 0$ . However, when the number of degrees of freedom is increased by allowing more intervals, the optimal initial conditions are found to be total reflux (case 3b and c). Case 1 will also have an initial period with total reflux until the specified distillate composition is reached.

As can be seen, using only two linear intervals for the optimal reflux policy (case 3b with  $t_f = 5.32$  hr) is almost as optimal as 10 piecewise constant intervals (case 3c with  $t_f = 5.26$  hr) and the optimization problem is much easier in the first case (5 versus 20 optimization variables). This indicates that our solution is close to the true optimal reflux policy.

Next consider the cyclic policy. With more than one cycle (cases 4b, c and d) it is much less time consuming than any of the other policies. Specifically, the best optimal reflux ratio policy (case 3c with  $t_f = 5.26$  hr) is about 25% more time consuming than the best cyclic policy with 3 cycles (case 4c with  $t_f = 4.03$  hr). Note that for the cyclic policy, the contribution of the filling and dumping time to the total batch time is constant and independent of the number of cycles. For our example,

$$\sum t_{fill} + t_{dump} = \frac{H_{A,spec}}{V_{N,max}} + \frac{H_{A,spec}}{D_{max}} = 0.1875 + 0.075 = 0.2625 \text{ hr}$$

Thus, in the optimal case the filling and dumping time is about 6.5% of the total batch time. Note that we have used only one condenser drum. By implementing two drums in parallel (such that the filling of the second is started at the same time as the

	Operating policy	Optimal $t_f$ [hr]	Optimal variable	$t_f$ [hr]
1	Constant dist. composition 1 interval	9.5268	$x_D^* = 0.90$	9.5268
2a	Constant reflux ratio, 1 interval	5.7345	$R = 0.9673$	5.7345
2b	Case 2a with initial total reflux	0.0529-5.5471	$R = 0.9999^* - 0.9662$	5.6000
3a	Optimal reflux ratio, 2 constant intervals	3.1382-2.3955	$R(t) = 0.9613 - 0.9724$	5.5337
3b	Optimal reflux ratio, 2 linear intervals	0.1420-5.1804	$R(t) = 0.9999^* - 0.9522 - 0.9767$	5.3224
3c	Optimal reflux ratio, 10 constant intervals	0.0626-0.8938-0.9574-0.8925-0.8479-0.3047-0.4996-0.0971-0.7009-0.0025*	$R(t) = 0.9999^* - 0.9530 - 0.9582 - 0.9631 - 0.9675 - 0.9702 - 0.9720 - 0.9728 - 0.9753 - 0.9237$	5.2590
4a	Cyclic policy 1 cycle	7.0896	$H_{c,1} = 1.875^*$	7.0896
4b	Cyclic policy 2 cycles	2.2354-1.8403	$H_{c,1} = 1.1642$ $H_{c,2} = 0.7103$	4.0757
4c	Cyclic policy 3 cycles	2.3560-0.6880-0.9784	$H_{c,1} = 1.2261$ $H_{c,2} = 0.2910$ $H_{c,3} = 0.3574$	4.0224
4d	Cyclic policy 4 cycles	1.2365-0.1155-0.8111-1.8682	$H_{c,1} = 0.6896$ $H_{c,2} = 0.0500^*$ $H_{c,3} = 0.4096$ $H_{c,4} = 0.7255$	4.0313

Table 2: Optimal solutions to the minimum time problem. (\*: on the upper or lower bound). (Data from Table 1).

Cycle	$t_f$ [hr]	$x_A$	$H_A$ [kmol]
1	1.2365	0.9259	0.6896
2	1.3520	0.9244	0.7396
3	2.1631	0.9183	1.1492
4	4.0313	0.9000	1.8750

Table 3: Accumulated product for the cyclic policy with 4 cycles. (Data from Table 1).

dumping of the first one) the dumping time will be zero for all but the last cycle and the total batch time thereby reduced.

The cyclic policy with only one cycle (case 4a with  $t_f = 7.09$  hr) is time consuming because all the specified product has to be accumulated in the condenser in only one cycle. The cyclic policy with 3 cycles (case 4c with  $t_f = 4.02$  hr) seems to be slightly more optimal than with 4 cycles (case 4d with  $t_f = 4.03$  hr). This might be just because of the optimization accuracy. However, it is expected that there is a minimum somewhere since, for an infinite number of cycles, the cyclic policy would approach the optimal reflux policy ( $t_f = 5.26$  hr) but with an additional penalty for filling and dumping (about 0.2625 hr).

The condenser holdup  $H_{C,k}$  in each period varies and from the results no conclusions as to the best condenser holdup profile (e.g. smaller holdups for later cycles) can be made. The accumulated product for the case with 4 cycles is given in Table 3. The composition decreases with time until the specification is reached at the end of the last cycle.

It is interesting to compare the sharpness of separation in the column for the different operating policies. Figure 3 gives the distillate composition as a function of accumulated product for case 2a and 4d and compares it with the total reflux case

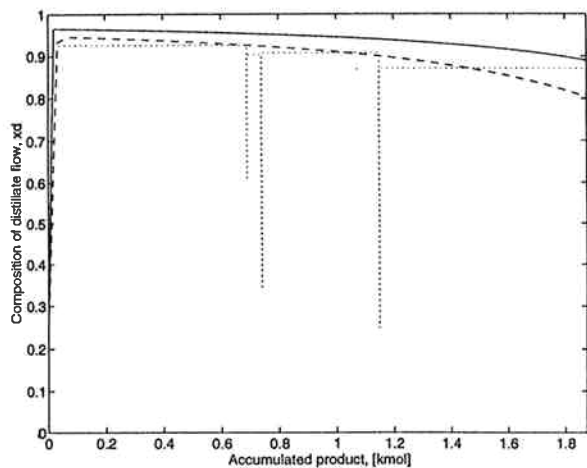


Figure 3: Distillate composition as a function of accumulated product for total reflux (solid line), constant reflux ratio (case 2a, dashed line) and cyclic policy with 4 cycles (case 4d, dotted staircase). (Data from Table 1).

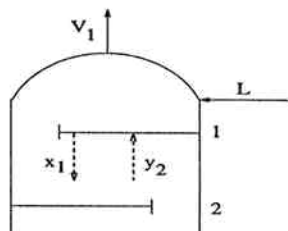


Figure 4: Steady state in the top of the column when  $x_1 = y_2$ .

(maximum separation). It can be seen that, for the cyclic policy (dotted staircase), the distillate composition drops at the end of each cycle. This is because the vapor flow is reduced for numerical reasons and this would not occur in practice. The compositions for the cyclic policy (dotted staircase) and the reflux policy (dashed line) are very close. The difference between the two cases is surprisingly small when one knows that the difference in batch time is 40% (5.73 hr and 4.03 hr). The results thus show that the cyclic policy is able to achieve the desired separation in a shorter time.

## 5 Simplified cyclic policy

It was found above that the cyclic policy is less time consuming and therefore more optimal than the conventional policies. The optimization of this policy is however, very time consuming. To simplify the calculations for the cyclic policy we therefore propose the following approach:

1. The same condenser holdup is used for each cycle

$$H_{C,k} = H_{A,spec} / N_{cyc} \quad (6)$$

2. A cycle is stopped when a given approach to equilibrium has been reached in the column. At equilibrium with total reflux, the liquid and vapor between two trays have the same composition,  $x_j = y_{j+1}$ . In this paper the approach to equilibrium is defined in terms of the difference  $\epsilon$  in liquid and vapor composition between tray 1 and 2 (see Figure 4):

$$|x_1 - y_2| \leq \epsilon \quad (7)$$

The total reflux period is thus stopped when Eq. 7 is satisfied. The same value for  $\epsilon$  is used for each cycle  $k$ .

3. The parameter  $\epsilon$  is adjusted to meet the product composition specification. (The specification on the amount is

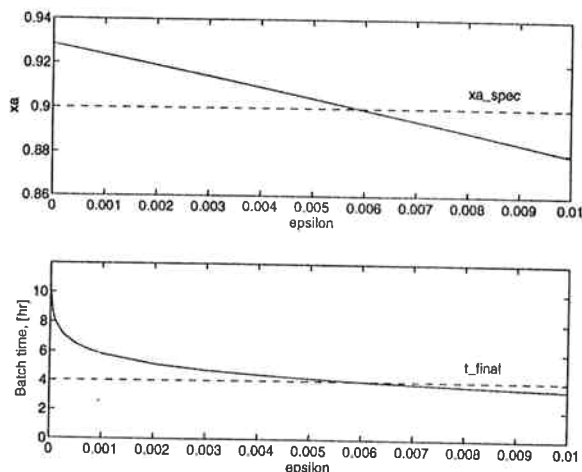


Figure 5: Accumulated product composition and batch time for the cyclic policy with 3 equal cycles for different values of  $\epsilon$ . (Data from Table 1.)

always satisfied due to the way the condenser holdup is defined).

Instead of optimizing the condenser holdup and the total reflux time ( $2N_{cyc}$  optimization variables) we now have only one variable  $\epsilon$  which is selected such that the composition specification is met. The procedure is demonstrated in Figure 5 where the final batch time and the composition of the accumulated product is presented for different  $\epsilon$  assuming 3 equal cycles with the same  $\epsilon$  for all cycles. The relationship between  $\epsilon$  and  $x_A$  is almost linear and only a few trial values for  $\epsilon$  are needed to find the value where  $x_A = x_{A,spec}$ . A simple batch distillation simulation program can therefore be used to find the right  $\epsilon$  in the simplified policy instead of a large and complicated optimization program which is needed for the optimal cyclic policy.

This policy is fast and has few convergence problems. Importantly, we have found for the examples presented above that the optimal total batch time found using this simplified approach is only marginally larger than the batch time found by the very time consuming optimization procedure (for 3 cycles 4.05 hr with one selected parameter  $\epsilon$  versus 4.02 hr for the optimal solution). This also shows that varying the condenser holdup for each cycle as given by the optimal results is not very important.

## 6 Comparison of operating policies for varying conditions

We have shown above for a given example that the cyclic policy is less time consuming than any of the other operating policies. An obvious question is therefore: Is this always the case and if not *when is the cyclic policy to be preferred?* In this section we will try to answer this question with reference to a measure for the degree of difficulty of separation  $q$ . We will compare the simplified cyclic operating policy with the constant reflux ratio policy for varying values of  $q$ , that is for varying operating conditions. We will also give guidelines for how to select the number of cycles  $N_{cyc}$  in the simplified cyclic policy.

### 6.1 A measure for the degree of difficulty of separation

A measure for the difficulty of a given separation in a distillation column is the ratio between the minimum number of trays  $N_{min}$  required to make a specified separation at total re-

case	$\alpha$	$N_t$	$x_{F0}$	$x_{A,spec}$	$x_{R,spec}$	$H_{A,spec}$	$q$
1	1.50	15	0.15	0.975	0.05	1.0811	0.91
2	1.25	15	0.45	0.900	0.10	4.3750	0.91
3	2.00	5	0.55	0.950	0.10	5.2941	0.90
4	1.50	10	0.80	0.975	0.10	8.0000	0.88
5	1.50	10	0.60	0.950	0.05	6.1111	0.85
6	1.25	20	0.45	0.950	0.10	4.1176	0.85
7	1.50	10	0.25	0.900	0.10	1.8750	0.85
8	1.50	10	0.35	0.900	0.05	3.5294	0.83
9	1.25	15	0.65	0.900	0.05	7.0588	0.83
10	1.50	15	0.30	0.975	0.05	2.7027	0.82
11	2.00	5	0.40	0.900	0.10	3.7500	0.81
12	1.25	20	0.55	0.950	0.10	5.2941	0.80
13	1.25	15	0.65	0.900	0.10	6.8750	0.78
14	2.00	5	0.85	0.950	0.05	8.8889	0.78
15	1.25	20	0.40	0.900	0.05	4.1176	0.76
16	2.00	10	0.20	0.975	0.05	1.6216	0.74
17	2.00	10	0.15	0.975	0.10	0.5714	0.74
18	1.50	15	0.30	0.950	0.05	2.7778	0.71
19	1.50	10	0.50	0.900	0.10	5.0000	0.70
20	2.00	10	0.35	0.975	0.05	3.2432	0.68
21	1.50	15	0.30	0.950	0.10	2.3529	0.67
22	2.00	5	0.75	0.900	0.05	8.2353	0.65
23	1.50	15	0.45	0.950	0.05	4.4444	0.64
24	2.00	10	0.40	0.975	0.10	3.4286	0.63
25	2.00	10	0.25	0.950	0.05	2.2222	0.62
26	2.00	10	0.55	0.975	0.05	5.4054	0.61
27	2.00	10	0.30	0.950	0.10	2.3529	0.57
28	2.00	5	0.85	0.900	0.10	9.3750	0.56
29	1.50	15	0.65	0.950	0.10	6.4706	0.54
30	1.50	15	0.50	0.900	0.05	5.2941	0.51
31	2.00	15	0.35	0.975	0.10	2.8571	0.45
32	2.00	15	0.25	0.950	0.10	1.7647	0.41

Table 4: Operating conditions for examples used in comparing the simplified cyclic policy and the constant reflux ratio policy. flux (i.e. the minimum value of  $N$ ) and the actual number of trays  $N_T$  in the given column.

$$q = \frac{N_{min}}{N_T} \quad (8)$$

If  $N_T \gg N_{min}$  the separation is considered easy but if  $N_T \approx N_{min}$  it will be difficult. Fenske's equation can be used to calculate  $N_{min}$  for a binary separation in a continuous column:

$$N_{min} = \frac{\log \left( \frac{x_D}{1-x_D} \right) \left( \frac{1-x_B}{x_B} \right)}{\log \alpha} \quad (9)$$

For continuous distillation  $x_D$  and  $x_B$  will be constant while as for batch distillation one or both compositions will vary with time according to the operating policy chosen. For the constant overhead policy  $x_D$  will be constant and only  $x_B$  will vary with time. For the other operating policies both  $x_D$  and  $x_B$  will vary.

One alternative is therefore to use an average number of  $N_{min}$  for batch distillation where  $N_{min}$  is averaged over time:

$$\overline{N_{min}} = \frac{\int_{t_0}^{t_f} N_{min}(t) dt}{(t_f - t_0)} \quad (10)$$

where

$$N_{min}(t) = \frac{\log \left( \frac{x_D(t)}{1-x_D(t)} \right) \left( \frac{1-x_B(t)}{x_B(t)} \right)}{\log \alpha} \quad (11)$$

By assuming that  $x_D$  is constant as for the constant overhead policy and  $x_B$  is a linear function of time eq. 10 can be rewritten to an integral which can be solved numerically a priori (Christensen and Jørgensen [5]).

$$\overline{N_{min}} = \frac{\int_{x_{B0}}^{x_{Bf}} N_{min}(x_B) dx_B}{x_{B0} - x_{Bf}} \quad (12)$$

case	$q$	Simplified cyclic policy			Const. reflux policy	
		$N_{cyc}$	$\epsilon \cdot 10^{-3}$	$t_f$ , hr	$R_{opt}$	$t_f$ , hr
1	0.91	4	0.929	4.6603	0.9912	12.3027
2	0.91	18	1.873	16.5059	0.9866	32.6746
3	0.90	22	5.814	5.7257	0.9391	8.6926
4	0.88	50	3.530	10.5270	0.9378	12.8583
5	0.85	40	4.290	10.7480	0.9662	18.0554
6	0.85	9	2.259	14.2470	0.9811	21.7462
7	0.85	4	6.538	4.0447	0.9673	5.7345
8	0.83	11	5.945	7.7058	0.9701	11.8134
9	0.83	50	3.783	17.2487	0.9715	24.7788
10	0.82	4	2.480	6.8609	0.9747	10.6619
11	0.81	8	15.740	3.4761	0.9037	3.8951
12	0.80	8	3.085	14.7089	0.9718	18.7778
13	0.78	50	5.878	13.3382	0.9552	15.3404
14	0.78	50	14.900	4.6369	0.8037	4.5291
15	0.76	25	4.054	13.8704	0.9782	18.8505
16	0.74	1	5.662	2.2721	0.9422	2.8077
17	0.74	1	9.167	-0.7706	0.9365	0.9001
18	0.71	2	5.778	5.5731	0.9611	7.1477
19	0.70	5	13.286	5.9458	0.9186	6.1403
20	0.68	1	5.543	3.4312	0.9242	4.2789
21	0.67	1	8.629	3.6180	0.9395	3.8888
22	0.65	50	30.050	3.9657	0.7745	3.6528
23	0.64	2	5.915	6.7323	0.9477	8.4907
24	0.63	1	9.245	3.0103	0.8750	2.7435
25	0.62	1	14.385	2.2837	0.9200	2.7763
26	0.61	50	10.411	4.7857	0.9000	5.4036
27	0.57	1	20.135	1.9192	0.6611	0.6943
28	0.56	50	37.297	2.2012	0.5440	2.0560
29	0.54	50	13.347	6.1717	0.8917	5.9747
30	0.51	50	15.60	6.1937	0.9188	6.5177
31	0.45	1	24.11	1.4403	0.8607	1.2669
32	0.41	1	12.25	2.5579	0.8596	2.0346

Table 5: Results for the simplified cyclic policy and the constant reflux ratio policy for varying operating conditions or  $q$  values.

where

$$N_{min}(x_B) = \frac{\log \left( \frac{x_D}{1-x_D} \right) \left( \frac{1-x_B}{x_B} \right)}{\log \alpha} \quad (13)$$

and  $q$  is given by

$$q = \frac{N_{min}}{N_T} \quad (14)$$

Equation (12-14) can then be solved numerically and thereby provide a measure of the degree of difficulty of separation a priori. Christensen and Jørgensen [5] used this measure in the study of waste cut recycling.

## 6.2 Numerical examples

In the following we compare the cyclic policy with the constant reflux policy. We use the simplified cyclic policy with the same condenser holdup  $H_{C,k}$  and approach to equilibrium  $\epsilon$  in each cycle. For the constant reflux policy the batch is started directly without an initial total reflux period. The cases studied are given in Table 4. We have varied the number of theoretical stages in the column  $N$ , the relative volatility  $\alpha$ , the feed composition  $x_F$  and the product specifications for the distillate and the residual in the column,  $x_{A,spec}$  and  $x_{R,spec}$ . The initial feed was equal to 10 moles in all cases. The specification for the accumulated distillate holdup  $H_{A,spec}$  can then be calculate from a total mass balance. The same assumptions for the dynamic model and the bounds on the flows as given in section 4.1 were used.

The results are presented in Table 5 and the time saved by using the simplified cyclic policy compared to the constant reflux policy is given in Figure 6. The time saving is defined as

$$\text{time saved} = \frac{t_{cyclic} - t_{reflux}}{t_{reflux}} \cdot 100\% \quad (15)$$

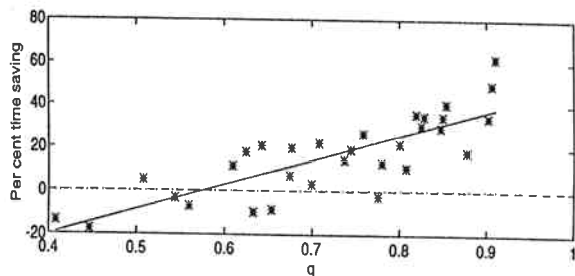


Figure 6: Per cent time saved by using the simplified cyclic policy compared to the constant reflux policy as a function of the measure  $q$ . (Solid line: results fitted to a linear function).

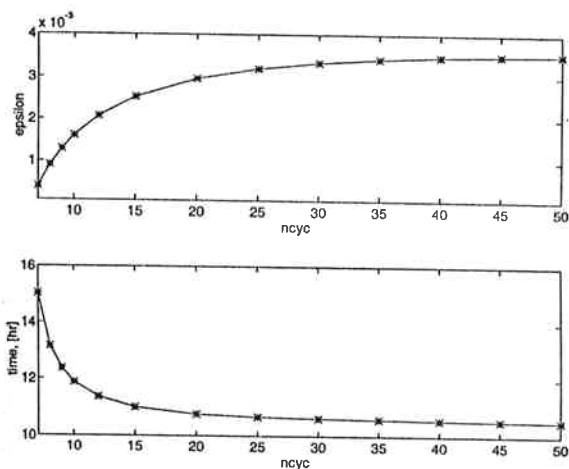


Figure 7:  $\epsilon$  and total batch time as a function of number of cycles for the simplified cyclic policy (case 4:  $q=0.8776$ ).

From the plot it can be seen that the results vary considerably. Still, it can be concluded that the time saved increases with increasing  $q$  value, that is, when the separation becomes more difficult. This is as expected because of the improved separation attainable under total reflux.

From the plot it can be concluded that the cyclic policy is to be preferred when the measure  $q$  is larger than 0.7. Even though there are few results for  $q$  values less than 0.5 it is still reasonable to assume that the constant reflux policy will be less time consuming than the cyclic policy for  $q$ -values below 0.5 and therefore to be preferred in this range. In the range 0.5-0.7 either one of the policies may be preferred depending on the given separation.

### 6.3 Number of cycles in the cyclic policy

The number of cycles  $N_{cyc}$  for each separation in Table 5 is found by running the simplified cyclic policy for different number of cycles until the minimum in terms of time consumption is found. The upper bound was in this study 50 cycles. An example of values for  $\epsilon$  and total batch time as a function of number of cycles is given in Figure 7. For this example, the total batch time decreases continuously when the number of cycles is increased.

Other separations may show the opposite effect, that is, 1 cycle is the optimum. Others again may have an interior optimum.  $\epsilon$  usually increase with  $N_{cyc}$ . This is because as the number of cycles is increased, the need for maximum separation in each cycle is decreased and the total reflux period can be stopped earlier or at higher  $\epsilon$ -values.

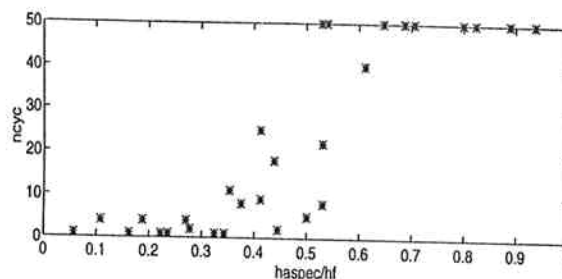


Figure 8: Number of cycles as a function of the ratio between  $H_{A,spec}$  and  $H_F$  for the simplified cyclic policy.

From Table 5 it can be seen that  $N_{cyc}$  varies considerably from only 1 cycle for some separations up to the maximum of 50 for others. Figure 8 gives the optimal number of cycles as a function of the ratio between the accumulated product  $H_{A,spec}$  and the feed  $H_F$  for all the cases in Table 4. It can be seen that for high ratios between accumulated light product and initial feed,  $H_{A,spec}/H_F > 0.5$ , it is optimal to use many cycles whereas when only small amounts of light product are to be separated,  $H_{A,spec}/H_F < 0.5$ , it is optimal to use few cycles.

## 7 Discussion

We have in this study found that the simplified cyclic policy with the same condenser holdup in each cycle, is almost as optimal as the cyclic policy were the condenser holdup is varied. Also the length of the total reflux period can be found easily in terms of the steady state parameter  $\epsilon$  and no time consuming and complicated optimizations are needed.

We have found that the cyclic policy can be recommended for difficult separations ( $q > 0.7$ ) and where the amount to be separated is less than 50% of the initial feed ( $H_{A,spec}/H_F < 0.5$ ). Using a large number of cycles (typically larger than 5-10) as found to be optimal for separations where  $H_{A,spec}/H_F > 0.5$ , will not be practical in real operation. We have in this paper compared the results from the cyclic policy with the constant reflux ratio policy. When using the cyclic policy with a high number of cycles one approaches the optimal reflux policy and this policy will probably be the best in these cases.

### 7.1 The cyclic policy in practice

No design changes are necessary for the column in order to run it with the cyclic policy except that a larger condenser drum might be needed. One alternative is also to use two condenser drums as mentioned in section 4.2 (such that the filling of the second is started at the same time as the dumping of the first one). To implement the cyclic policy several alternatives may be suggested:

1. "Open-loop" operation where the times for each cycle  $t_{cycle,k}$  (obtained from the optimization, eg. Table 3) is specified. This mode of operation is however, sensitive to changes and disturbances.
2. "Feedback" operation where the cycle is stopped when a given composition or temperature (obtained from the optimization, eg. Table 3) is achieved.
3. "Feedback" operation where the cycle is stopped when a given approach to equilibrium is achieved. Since it may be difficult to measure  $\epsilon = x_1 - y_2$  as we have defined it in this paper it is probably better to stop when  $dT_i/dt < \epsilon T$ . In words, when a temperature change in the column is

less than a given bound. In this case, it is important to select a sensitive temperature (eg. a temperature in the middle of the column).

One problem with the cyclic procedure is that the feed composition has to be known quite exactly since  $H_{A,spec}$  is calculated based on the value of this composition. If  $H_{A,spec}$  is set too large, the required separation might not be achievable. It is therefore better to select  $H_{A,spec}$  too low, and operate the column with the constant reflux policy after the last cycle until the specifications are met.

## 8 Conclusion

In this paper we have presented some factors that are important for the optimal operation of batch distillation other than the optimal reflux ratio and reboiler heat duty normally studied. We have presented the cyclic operating policy in detail and compared it with the conventional operating policies for some separations and found it to be favorable for difficult separations with  $q$  values larger than 0.7. Normally, batch columns are design with  $N_T \approx 2N_{min}$  ( $q \approx 0.5$ ) and the constant reflux policy will be the most favorable. Batch columns are however, often used for separations they are not designed for ( $q > 0.5$ ) and the cyclic policy might be favorable for these separations. The number of cycles for the cyclic policy is important for the attainable separation in the column and also for the batch time.

## NOTATION

$D$	distillate flow, $kmol/hr$
$H_A$	liquid holdup in the accumulator, $kmol$
$H_C$	liquid holdup in the condenser, $kmol$
$H_F$	feed, $kmol$
$H_j^0$	initial liquid holdup on tray $i$ , $kmol$
$K_P$	gain for PI-controller
$N$	number of trays in the column section
$L$	reflux flow, $kmol/hr$
$N_{cyc}$	number of cycles in the cyclic policy
$q$	a measure for the degree of difficulty of separation
$R$	internal reflux ratio, $L/(L + D)$
$t$	time, $hr$
$t_{fill}$	filling period for cycle $k$ , $hr$
$t_{totalreflux}$	total reflux period for cycle $k$ , $hr$
$t_{dump}$	dumping period for cycle $k$ , $hr$
$t_{cycle}$	cycle time, $hr$
$t_f$	total batch time, $hr$
$V_N$	vapor flow from the reboiler, $kmol/hr$
$x_A$	mole fraction in accumulator
$x_B$	mole fraction in reboiler
$x_F$	initial mole fraction in feed
$x_R$	mole fraction in residual in column/reboiler
$x_D^d$	desired mole fraction in distillate
$\alpha$	relative volatility
$\tau$	hydraulic time constant, $hr^{-1}$
$\tau_I$	integral time for PI-controller, $hr$

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