OPTIMAL STARTUP PROCEDURES FOR BATCH DISTILLATION

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Abstract - Operation of a conventional batch distillation column can be conveniently described in three parts: 1) startup period, 2) production period and 3) shutdown period. For standard separation processes, the production period is the most time consuming. However, for difficult separations, such as for high purity or azeotropic separations, the startup time may also be significant. In this paper, we present results for optimal operation of a number of different separations taking the startup time into consideration. It is generally found that the startup time is significant compared to the total operating time for difficult separations (high purities and recoveries) and when the combined column and condenser holdup is large. It is also found that the exact value of the startup time is of limited significance unless it is very different from the optimal value. Alternative ways of reducing the duration of the startup period are discussed.

INTRODUCTION

Operation of a conventional batch distillation column can be conveniently described in three parts: 1) startup period (normally under total reflux), 2) production period and 3) shutdown period. For standard separation processes the production period is the most time consuming. However, for difficult separations, such as for high purity or azeotropic separations, the startup time may also be significant as shown in this paper. The importance of the startup period relative to the total separation has only been studied by few authors. Nad and Spiegel (1987) presented experimental results for the separation of 3 components into two main cuts, two off-cuts and a residue where the startup period was more than 1/4 of the total operating time. Holland and Liapis (1983) presented simulation results for the separation of a five component mixture where the startup time was more than 50% of the total operating time. Luyben (1971) presented simulation results for a number of cases where the startup time varied from negligible up to 50% of the total operating time.

The purpose of this paper is to discuss various aspects of startup procedures for batch distillation. We will discuss *under which conditions* the optimal startup time is significant compared to the optimal total operating time. Also, the influence of a correct startup time on the optimal operating time is considered. To the best of our knowledge, no studies of optimal startup procedures have been presented in the literature so far. In the second part of the paper we discuss alternative ways of reducing the startup time by either using partial backmixing equipment or by adding light material to the condenser drum initially.

DYNAMIC MODEL

The dynamic model used in this paper for a binary mixture is valid under the following assumptions: 1) Staged batch distillation column, 2) perfect mixing and equilibrium on all trays, 3) negligible vapour holdup, 4) constant stage pressures and tray efficiencies, 5) constant vapour flows, 6) total condensation with no subcooling in the condenser, 7) constant relative volatility, 8) constant molar condenser drum holdup and 9) constant molar liquid holdup on all trays. It is assumed that the vapour flow V can be manipulated directly. Note that the reflux ratio R used here is the internal reflux ratio $R = L/V$.

SIGNIFICANCE OF STARTUP TIME FOR OPTIMAL OPERATION

In this section we will discuss under which conditions the optimal *startup time* is significant compared to the optimal *total operating* time. We will consider a binary mixture separated into two or three fractions (see Figure 1):

Two fractions. After the initial startup period under total reflux ($t_{startup}$), there is a production period (time t_1) where a light product with mole fraction x_A and amount H_A is accumulated.

Three fractions. After the first production period (t_1), there is a second production period (time t_2) where an intermediate off-cut is produced (with composition x_{off} and amount H_{off}) and collected in a second accumulator tank.

In both cases, the heavy product is the residual material in the reboiler with mole fraction x_R and amount H_R. Note that this residual also includes the holdup in the condenser and the column section which is

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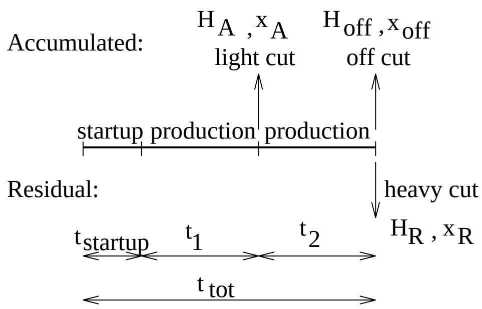

Figure 1: Binary separation into two main cuts and one off-cut.

assumed to be drained to the reboiler at the end of the batch. (If the condenser holdup was large, one would probably want to drain this to the accumulator instead).

In the following we will consider an operating policy with constant reflux ratios R_1 and R_2 in each of the two production periods. For a given separation the optimal startup time $t^{opt}_{startup}$, the times to reach the specifications for the product cuts, t_1 and t_2, and the corresponding constant reflux ratios R_1 and R_2 can be found by minimising the total operating time t_{tot}. In addition, there are constraints on the composition and recovery percentage of light product at t_1 and heavy product at t_2:

$$\min_{t^{opt}_{startup}, t_1, R_1, t_2, R_2} \quad t_{tot} = t^{opt}_{startup} + t_1 + t_2 \tag{1}$$

$$\begin{aligned} & x_A(t_1) \geq x_A^{spec} && x_R(t_2) \leq x_R^{spec} \\ & 100\% \cdot \tfrac{x_A H_A}{x_F H_F}(t_1) \geq \ \% \text{ light recovery} && 100\% \cdot \frac{(1-x_R)H_R}{(1-x_F)H_F}(t_2) \geq \ \% \text{ heavy recovery} \end{aligned} \tag{2}$$

Also, there may be upper and lower bounds on times and reflux ratios. However, all the constraints are not necessarily active at the optimal solution (inequalities instead of equalities). The initial compositions are assumed to be equal to the feed composition. The column is run under total reflux during startup ($R_{startup} = 1.0$). The optimisation program DAEOPT (Vassiliadis, 1993) is used with an optimisation accuracy of 10^{-4}. The purpose of this study is to investigate the relative importance of the startup time for a given separation. We do therefore not consider what is done with the off-cut after the separation, e.g. recycling or separate reprocessing.

Optimal results

Optimal results for several cases are given in Table 1. Cases C1 and C2 contain 0.5% of light component, cases C3 and C4 contain 5% of light component and cases C5, C6 and C7 are equimolar mixtures. The specifications are chosen so as to illustrate the influence of startup time for different types of separations (varying purity and recovery specifications). For all cases the total holdup in the column section $\sum^N H_j$ is 1% of the initial charge. The condenser drum holdup H_C is assumed to be 0.25% of the initial charge for cases C1 and C2 and 1% for the others. A time t_2 equal to zero means the constraints are met without the production of an off-cut (C2, C3 and C6). Note that for some cases (C2, C3, C6 and C7), not all the constraints are active (for example C2 where $x_R = 0.0025 < 0.01 = x_R^{spec}$). This will in general be the case when an off-cut is not produced (C2, C3 and C6) since then only two constraints can be independently specified. The approach to equilibrium given in Table 1 is defined as:

$$\% \text{ approach to equilibrium} = [x_D(t^{opt}_{tot}) - x_F]/[x_D^{ss} - x_F] \cdot 100\% \tag{3}$$

Let us now consider the results in more detail. For case C1, the startup time is almost 70% of the total operating time. In this case only a very small amount of light component (impurity) is to be recovered from the feed charge leaving a heavy product of high purity. (It should be noted that the startup time is significantly reduced if there is no purity constraint on the light product). For case C2, the startup time is 99.5% of the total operating time. The optimal solution is in fact a cyclic operating policy with one cycle where a condenser drum holdup equal to the desired amount of light product is used and the column is run under total reflux until the desired product is obtained (see Sørensen and Skogestad, 1994).

For case C5, which is an easy equimolar separation with lower product purities, the startup time is only 1 % of the total operating time. The results depend of course on the specifications for the product compositions and the percentage of recovery of the components. If a high percentage of recovery with a high purity is desired, the startup time will be longer than if the specifications are less strict. This is illustrated by case C3 and C4 which only differ in the specifications for the products.

Table 1: Optimal results (minimum operating time) for different separations for a piecewise constant reflux policy with one startup period and one or two production periods ($H_F = 10\ kmol$ and $V = 10.0\ kmol/hr$ for all cases) (*: on the lower bound, −: not specified).

	C1	C2	C3	C4	C5	C6	C7
Cases:							
N	20	20	10	10	10	10	10
α	4.0	4.0	3.0	3.0	2.0	2.0	2.0
H_j, $kmol$	0.005	0.005	0.01	0.01	0.01	0.01	0.01
H_C, $kmol$	0.025	0.025	0.1	0.1	0.1	0.1	0.1
x_F	0.005	0.005	0.05	0.05	0.5	0.5	0.5
Specifications:							
x_A^{spec}	0.5	0.9	0.99	0.95	0.95	0.99	0.9
% light recovery	90	50	75	-	75	90	-
x_R^{spec}	0.0001	0.01	0.05	0.01	0.05	0.1	0.01
% heavy recovery	90	-	-	90	75	-	90
Results:							
x_A	0.500	0.900	0.990	0.950	0.950	0.990	0.955
% light recovery	90.00	50.00	75.00	9.88	75.00	90.00	83.04
x_R	0.00010	0.0025	0.013	0.010	0.050	0.092	0.010
% heavy recovery	90.00	99.97	99.96	90.00	75.00	99.09	90.00
x_{off}	0.0043	-	-	0.277	0.500	-	0.725
$t_{startup}^{opt}$, hr	0.886	0.556	0.183	0.162	0.031	0.077	0.035
t_1, hr	0.009	0.003	2.285	0.0008	1.652	4.674	2.178
R_1, hr	0.01*	0.01*	0.983	0.342	0.761	0.903	0.800
t_2, hr	0.387	0	0	0.699	0.860	0	1.963
R_2, hr	0.754	-	-	0.806	0.755	-	0.944
t_{tot}^{opt}, hr	1.282	0.559	2.468	0.862	2.543	4.751	4.175
$t_{startup}^{opt}/t_{tot}^{opt}\cdot 100\%$	**69.1 %**	**99.5 %**	**7.4 %**	**18.8 %**	**1.2 %**	**1.6 %**	**0.8 %**
$x_D(t_{tot}^{opt})$	0.9984	0.9927	0.9841	0.9609	0.8246	0.9725	0.8450
x_D^{ss}	0.9995	0.9995	0.9998	0.9998	0.9995	0.9995	0.9995
approach to eq.	**99.9 %**	**99.3 %**	**98.3 %**	**95.9 %**	**65.0 %**	**68.7 %**	**69.0 %**

We see from Table 1 that the approach to equilibrium at the end of the startup time as defined by Eq. 3, varies considerable between the cases (99.9 % for case C1 to 65.0 % for case C5). Thus, the approach to equilibrium during startup will, as the startup time, depend on the mixture and the specifications.

In summary, the startup time will be significant compared to the total operating time for difficult separations (high purities and recoveries). It will also be significant when the combined column and condenser holdup is large. The last conclusion regarding holdup follows from the fact that the equilibrium time increases proportionally with holdup whereas the total operating time increases much less than proportionally.

Optimal results with pre-specified startup time

In the previous section we found that for difficult separations the column is run closer to steady state than for easier separations. The question is now *how sensitive* the results are to the exact value of the startup time. To this effect, we preselect the startup time equal to an equilibrium time $t_{eq}^{98\%}$ defined as the time when the distillate composition has reached 98% of its steady state value:

$$[x_D(\mathbf{t}_{\mathbf{eq}}^{\gamma}) - x_F]/[x_D^{ss} - x_F] = \gamma \tag{4}$$

where $\gamma = 0.98$ (see Sørensen, 1994) and compute the corresponding optimal total operating time $t_{tot}^{98\%}$. (The results are found by first running a simulation under total reflux to find the steady state distillate composition x_D^{ss} and thereby the startup time $t_{eq}^{98\%}$. In the new optimisations, the startup time is pre-specified to this value and $t_{tot}^{98\%}$ is found.) These values are compared with the previous optimal values in Table 2, $t_{startup}^{opt}$ and t_{tot}^{opt}. Interestingly, although the startup time is quite different ($t_{eq}^{98\%}/t_{startup}^{opt} \neq 1$), the increase in total operating time as expressed by $t_{tot}^{98\%}/t_{tot}^{opt}$ is very small for all the cases studied (except for case C2 where a cyclic operating policy is better than the reflux policy studied here).

Total operating time as a function of startup time

For a given prespecified startup time one may compute the optimal total operating time. This relationship is shown graphically in Figure 2 for cases C3, C5 and C7. We see that for all cases, the total operating time increases moderately when the operating time is longer than minimum. However, the optimum is very flat for cases C5 and C7 but not for case C3. For case C3 there is a very large penalty in terms of longer operating time when the startup time is less than the optimal value. Two factors contribute to this: 1) The

Table 2: Optimal results ($t_{tot}^{98\%}$) when the startup time is pre-specified, $t_{startup} = t_{eq}^{98\%}$.

	C1	C2	C3	C4	C5	C6	C7
$t_{eq}^{98\%}$, hr	0.429	0.429	0.179	0.179	0.100	0.100	0.100
$t_{tot}^{98\%}$, hr	1.287	0.909	2.469	0.863	2.589	4.760	4.218
$t_{eq}^{98\%}/t_{startup}^{opt}$	0.51	0.81	0.98	1.10	3.23	1.30	2.86
$t_{tot}^{98\%}/t_{tot}^{opt}$	**1.004**	**1.626**	**1.000**	**1.001**	**1.018**	**1.002**	**1.010**

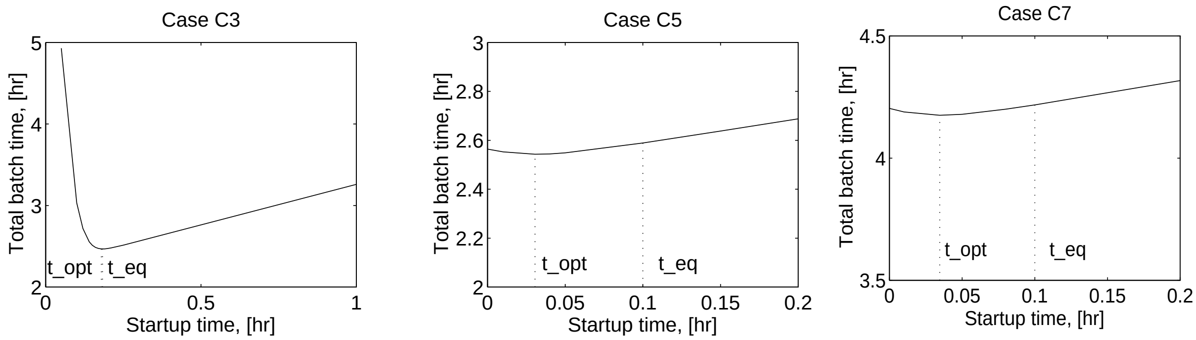

Figure 2: Total operating time t_{tot} as a function of startup time $t_{startup}$ for cases C3, C5 and C7 (Note that the scaling is different).

startup time and approach to equilibrium is more important for cases where the tightest specification is for the light product (case C3) and 2) the startup time and approach to equilibrium is less important when an off-cut is produced since then there are more degrees of freedom (cases C5 and C7).

IMPROVEMENTS IN THE STARTUP PROCEDURE

Obviously a quick composition change has to take place during the startup period in order to reach steady state or a prescribed reflux composition as soon as possible. Changes in the startup procedure and/or the equipment characteristics may be necessary in order to reduce the duration of this period. The following suggested improvements will be considered in the last part of this paper: 1) Partial backmixing equipment and 2) light material in the condenser drum initially.

Partial backmixing equipment

González-Velasco *et al.* (1987) proposed to modify the overhead equipment of the batch distillation unit in order to more closely approach a plug flow behaviour within the condenser. They demonstrated that this behaviour, instead of the normal complete mixing, reduces the inertia to composition changes and permits a more rapid startup of the unit. One of the modifications, *partial backmixing equipment* is illustrated in Figure 3. When the lowest drum has initially filled up during startup, the valve between the drums is closed and total reflux operation begun. The reflux is provided from the lower drum but the condensed vapour stored in the upper drum. As soon as the lower drum is empty, the upper drum is completely full; at this time the valve is opened and liquid transferred from the upper to the lower drum during a period of time which is considered to be negligible. This cycle is repeated until steady state or a prescribed reflux concentration is reached. The authors reported time savings in the startup time of up to 25%.

In this section we will compare the equilibrium startup time, defined as the time for 98% approach to equilibrium (Eq. 4), for a number of cases using one condenser drum (conventional equipment) and two condenser drums (partial backmixing equipment). The bottom condenser drum is assumed initially filled ($H_{C1}^o = H_C$) whereas the top drum is empty ($H_{C2}^o = 0$). The results are given in Table 3 for different feed compositions and condenser holdups. The condenser drum holdup H_C is 0.5, 5 and 10% of the initial charge respectively. Note that the total drum holdup for the case with two condenser drums is equal to the holdup in the single drum case ($H_{C1} + H_{C2} = H_C$). The difference in equilibrium time is small when $x_F = 0.1$, or when there is a low amount of light component in the feed. Using two drums will actually slightly increase the equilibrium time for low x_F and large H_C. The largest time saving (12%) is for the case with the largest feed composition and the largest condenser drum. González-Velasco *et al.* (1987) reported time savings from 2 to 25% using two drums instead of one. However, they only studied cases where the condenser holdup was 5 or 10% of the initial charge and the feed composition $x_F = 0.5$ to 0.6. It can therefore be concluded that *the partial backmixing equipment* is advantageous only if the feed composition x_F is large and the condenser holdup H_C has to be large too for some reason.

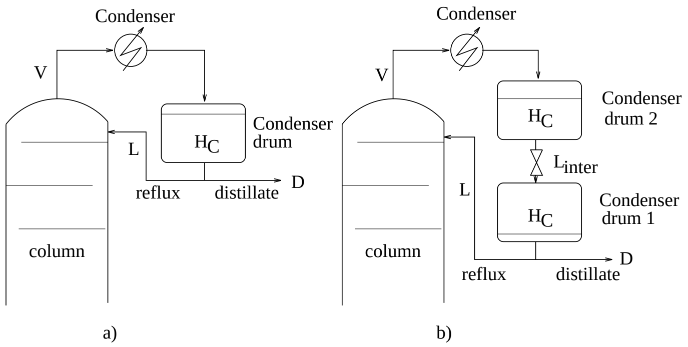

Figure 3: a) Usual equipment with backmixing in the condenser; and b) partial backmixing equipment with two condenser drums.

Table 3: Steady state distillate composition x_D^{ss}, equilibrium time $t_{eq}^{98\%}$ and % time saving for one and two condenser drums for different feed composition x_F and condenser drum holdup H_C. (Parameter values: $N = 10$, $\alpha = 2.0$, $V = 10.0\ kmol/hr$, $H_j = 0.05\ kmol$, $H_F = 10\ kmol$).

x_F	H_C $kmol$	composition x_D^{ss}	one drum $t_{eq}^{98\%}$, hr	two drums $t_{eq}^{98\%}$, hr	% time saving
	0.05	0.9931	0.421	0.418	0.7%
0.1	0.5	0.9827	1.429	1.428	0.07%
	1.0	0.8566	3.230	3.263	-1.0%
	0.05	0.9995	0.144	0.143	0.7%
0.5	0.5	0.9994	0.502	0.458	8.8%
	1.0	0.9993	0.927	0.814	12.2%

Light material in the condenser drum initially

A second possible improvement in the startup procedure is to start the batch with either light product from the previous batch or pure light component in the condenser initially. This way, the reflux will already be at its prescribed value and steady state will be reached quicker. This mode of operation is particularly easy to implement if the light component is water. Luyben (1988) proposed to use the first off-cut from a batch to fill up the condenser drum prior to startup of the next batch. However, no results were given. One drawback with this method is that light component which has already been separated is returned to the column and re-processed. The time saved during startup must therefore compensate for a longer production time if this procedure is to be beneficial.

We here consider the following alternative startup procedures: **P1**) Normal startup where the condenser is initially empty, **P2**) The condenser is initially filled with light product from the previous batch ($x_D^o = x_A^{spec}$) and **P3**) The condenser is initially filled with pure light component, $x_D^o = 1.0$. (This is reasonable if the light component is water.) The feed is initially charged to the reboiler and the tray holdups are assumed negligible. The optimal separation of a given mixture according to the three procedures can be found in terms of minimum operating time. Here, we consider a piecewise constant reflux ratio policy with one startup and one production period subject to constraints on the composition and amount of accumulated product:

$$\min_{t_{startup}^i, t_{prod}^i, R_{prod}^i} \quad t_{tot}^i = t_{startup}^i + t_{prod}^i \quad , \quad H_A(t_{tot}^i) \geq H_A^{spec} + H_C^o \quad , \quad x_A(t_{tot}^i) \geq \frac{H_A^{spec} x_A^{spec} + H_C^o x_D^o}{H_A^{spec} + H_C^o} \tag{5}$$

where i is the given procedure $P1$, $P2$ or $P3$ and H_C^o is the amount of added material for procedures $P2$ and $P3$. ($H_C^o = 0$ for procedure $P1$.) In words, the problem is to find the optimal time periods $t_{startup}$ and t_{prod} and the optimal constant reflux ratio R_{prod} which minimises the total operating time t_{tot} subject to constraints on the composition and amount of accumulated product, x_A and H_A. (Equivalently we could have specified the compositions x_A and x_R.) Note that the startup time is also optimised. For procedures $P2$ and $P3$, the amount of light product added initially must also be removed during the course of operation if these procedures are to be beneficial. For procedure $P3$ this means that the purity specification is increased. This will ensure the same purity and recovery of the heavy product (but will give a more difficult separation). During startup the column is run under total reflux ($R_{startup} = 1.0$).

The optimal results for some examples are given in Table 4. It is found that filling the condenser drum with light product from the previous batch ($P2$), may be beneficial for mixtures with a very low contents of light component. However, the time saved with procedure $P2$ relative to the normal procedure $P1$ is small. Filling the condenser with pure light component ($P3$) yields in all cases a longer operating time than the normal procedure ($P1$). However, note that the reflux ratio was assumed constant during the production

Table 4: Optimal results for a constant reflux ratio policy for procedures P1, P2 and P3. (Parameter values: $N = 10$, $\alpha = 2.0$, $V\ kmol/hr$, $H_F = 10\ kmol$, $H_j = 1e-6\ kmol$).

x_F	H_C $kmol$	x_A^{spec}	H_A^{spec} $kmol$	% light rec.	t_{tot}^{P1} hr	t_{tot}^{P2} hr	t_{tot}^{P3} hr
0.2	0.1	0.95	1.895	90	**4.85**	4.88	5.00
0.1	0.1	0.95	0.789	75	**2.85**	2.92	3.09
0.08	0.1	0.95	0.632	75	**3.35**	3.43	3.76
0.07	0.1	0.95	0.553	75	**3.83**	3.90	4.43
0.06	0.1	0.95	0.474	75	4.77	**4.75**	5.79
0.05	0.1	0.95	0.395	75	7.36	**6.74**	10.03
0.01	0.01	0.90	0.056	50	2.83	**2.76**	4.48

period and a varying reflux ratio might yield different results. Also the effect of holdup was not considered. The startup times for procedures $P2$ and $P3$ are negligible for all the examples, i.e. product removal can be started immediately. Thus although the startup time is reduced in procedures $P2$ and $P3$, this advantage is lost when the total operating time is considered.

CONCLUSIONS

In this paper we have discussed various aspects of startup of a batch distillation column. We have compared the optimal startup time with the optimal total operating time for several cases. It was found that the startup time is a considerable part of the total operating time for difficult separations with high purity and recovery specifications. This is as expected. However, the actual duration of the startup time is of limited significance unless it is very different from the optimal value. The correct startup time is most important for separations with high purity constraints for the lightest product since this is the component taken off first. Alternative ways of reducing the duration of the startup period where discussed and it was found that using two condenser drums in series can reduce the startup time for some separations.

NOTATION

D	distillate flow, $kmol/hr$	$t_{eq}^{98\%}$	time to reach 98% of steady state, hr
H_A	accumulator holdup, $kmol$	V	vapour flow, $kmol/hr$
H_C	condenser drum holdup, $kmol$	x_D	mole fraction in distillate
H_F	amount of initial feed, $kmol$	$x_D^{98\%}$	mole fraction in distillate, 98% appr. to equilibrium
H_j	liquid holdup on tray j, $kmol$	x_F	mole fraction of feed
H_{off}	amount of off-cut, $kmol$	x_{off}	mole fraction in off-cut
H_R	amount of residual, $kmol$	x_R	mole fraction in residual
L	reflux flow, $kmol/hr$	*Greek letters*	
N	number of trays in column section	α	relative volatility
R	reflux ratio $=L/V$	*Scripts*	
t	time, hr	opt	optimal value
t_{tot}	total operating time, hr	$spec$	specified value
$t_{startup}$	startup time, hr	ss	steady state

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