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Different Feeding Policies for Semi-Batch Distillation

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Semi-batch distillation (SBD) differs from the batch distillation (BD) in that one part of the feed is continuously fed. The liquid volume in the reboiler has usually a minimum value because of different reasons. The application of semi-batch distillation might be advantageous in cases where the prescribed purity of the less (B) or the loss permitted of the more volatile (A) component cannot be reached because of the above constraint. In the first case study morpholine (B) must be produced from a dilute aqueous solution in high purity. In the second case study acetone (A) is produced from a mixture with low-water content. Different BD and SBD operational policies are studied by dynamic simulation with a professional flow-sheet simulator, and compared on the basis of the specific energy demand of production of the valuable component. The SBD policies differ from each other in the feeding location. The possibility of varying feed location is also studied.

1. Introduction

By batch distillation (BD) the feed is processed in several batches. The volume of the liquid (hold-up) in the reboiler is continuously decreasing but usually there is a minimal volume of residue at the end of processing of each batch because of several practical reasons. If the reboiler is heated with a tube bundle or heating spiral at the bottom of the reboiler their surface must be always covered by liquid. For BD the heating jacket provides a relatively low, continuously decreasing active heating surface area. A minimum volume might be also necessary for other reasons, such as if a dissolved solid considerably increases the viscosity of the residue or if it precipitates on concentration. If a binary mixture is separated, the product is more frequently the more volatile component (A), obtained as distillate, but in some cases the less volatile component (B) obtained as residue. In certain cases, B must be produced from a dilute solution in high purity or A from its concentrated solution with low loss that cannot be solved in one batch because of the above constraint on the reboiler hold-up. The process where one part of the feed is continuously fed (usually) into the reboiler during the distillation is called rather semi-batch distillation (SBD) than semi-continuous distillation even if the majority of the feed is introduced continuously and not in batch. By Mujtaba (2004), SBD is also suitable for extractive and reactive distillation (e.g. Bashah et al., 2015).

There are the following possibilities (operational policies) to produce B in the purity prescribed:

1. To leave the residue in the reboiler and to fill the next amount of fresh feed onto it until the purity prescribed of B is reached (BD1 policy).
2. At the end of the batches the reboiler is drained, the residues are collected in a tank. After processing several batches when the necessary amount of residue (containing enough B) has been collected, the residues collected are redistilled (BD2 policy).
3. Semi-batch operation of the BD column (SBD policy) when one part of the feed is introduced continuously, usually into the reboiler (SBD1). However, the possibility of feeding into the column (SBD2) is worthy of studying. SBD was studied with fixed feeding location into the still (SBD1) by Lang et al. (2017). As the composition profile of the column is varying in time, the optimal feeding location will also vary. By shaping multiple feeding location in the column (Figure 1), a varying location policy (SBD3) can be also realized, where the mixture is fed into the column higher and higher to follow the change of liquid composition (x) profile in the column.

The aim of the paper is (1) to study different BD operational policies by dynamic simulation, (2) to suggest and investigate new SBD policies and (3) to compare the specific energy demands (SED) and recoveries (η) of the

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different policies. Two case studies are presented with different mixtures to be separated in the same BD column. Mixture 1 is dilute aqueous solution (water: A) of morpholine (B) from which B must be produced in high purity. Mixture 2 is acetone (A) with low water (B) content, where A is the valuable component. The simulation calculations are performed with the ChemCad professional flow-sheet simulator (‘CCBATCH’, ‘CCDCOLUMN’).

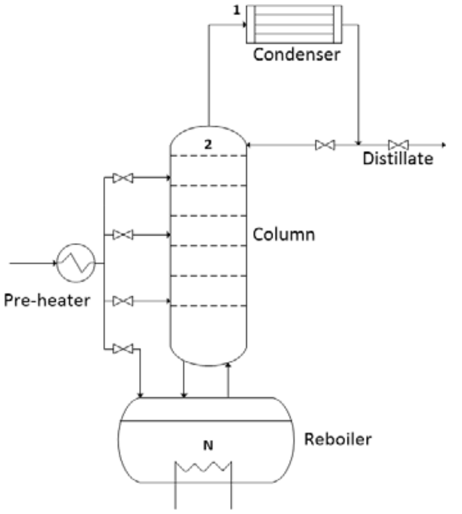

Figure 1: Scheme of a SBD column with variable feeding location

2. Phase equilibrium conditions

For the VLE calculations, the Margules model (a_{12}=0.4816, a_{21}=0.0085) was applied for Mixture 1 and UNIQUAC (U_{12}-U_{22}=601.61 cal/mol, U_{21}-U_{11}=-52.302) for Mixture 2. Figure 1 shows the relative volatility (α) profile for CD (for optimal feed locations). For Mixture 1, α is much higher at the lowest stages than at the highest ones. For Mixture 2, α decreases on the increase of the acetone concentration in a monotonous way. At the highest stages, α is quite low. (Mixture 2 forms a tangent azeotrope at high x_A values.)

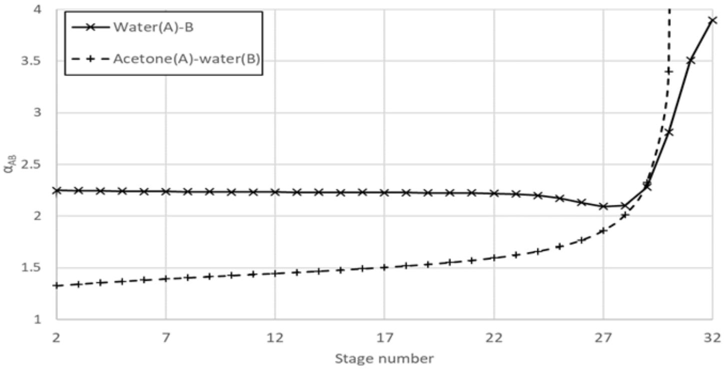

Figure 2: Relative volatility profiles for continuous distillation with optimal feed plates (f=19 and 30)

3. Results

The data of the column (operating at atmospheric pressure) are the same for both case studies. The volume of the reboiler: 10 m^3, the maximum and minimum liquid volumes in it are 8 and 2 m^3, respectively. Number of theoretical plates (in the CC model): N=32 (including the total condenser and reboiler). The hold-ups: condenser: 0.08 m^3, column: 0.08 m^3. The volumetric flow rate of distillate: 1 m^3/h. The hold-up remaining in the condenser and column are separately collected. At the start the reboiler is filled up to its maximal volume. The feed is always liquid at its boiling point. For the SBD policies the reboiler volumetric hold-up (U_N) is kept constant (which can be made at different levels between the maximum and minimum volumes) so the flow rate of the continuous feeding equals to that of the distillate (1 m^3/h). At the end of the process (last batch for BD, last step for SCD policies) both products must satisfy their purity requirements.

3.1 Case study 1

The feed contains 90 mass% water (x_F=0.9). The prescribed product compositions: $x_{D,av}$=0.99 and x_W=0.005. The reflux ratio (R) is so determined that at the end of each step the average composition of the distillate ($x_{D,av}$) collected in this step equals to 0.99.

For the sake of comparison, the results obtained previously (Lang et al., 2017) are presented briefly here. By CD with the given N, the optimal location of the feed plate (f_{opt}=19) was determined, where the energy demand of the separation is minimal (R=0.683). The SED: 37.83 MJ/kg B and the recoveries: η_B=0.9091, η_A=0.9994.

3.1.1 Batch distillation policies

Several BD policies were studied that differ from each other in what happens with the off-spec residues whose A content (x_W) is still higher than 0.005. The initial volume of the charge was always 8 m^3. By the BD1 policy, the distillation was finished when U_N reached the minimum (2 m^3) or x_W decreased to 0.005 (in the last, 4th batch). The off-spec residues remain in the reboiler and the next amount (6 m^3) of fresh feed is filled onto it. The SED was 55.5 MJ/kg B, η_B=0.823. By the BD2 policy, the distillation of each batch was finished when the minimum volume (2 m^3) was reached. The residues collected were then redistilled. A minimal amount of B is necessary for redistillation that was reached after 3 batches (6 m^3 residue; BD2b policy). The residue of a 4th batch can also be collected to use the full volume of the reboiler (BD2a policy). The SED was 48.2 MJ/kg B for BD2a and 47.7 MJ/kg B for BD2b. η_B was 0.849 (BD1a) and 0.842 (BD2b), respectively.

Without reboiler hold-up constraint (BD0 policy) both products of prescribed purity could be produced at the same time in one batch, but with highest SED (60.0 MJ/kg B) and lowest recovery (0.767). By the BD policies R varied between 0.78 (first batch) and 2.25 (last batch) depending on the policy and the batch processed.

3.1.2 Semi-batch policies

First, a brief summary of the results of Lang et al. (2017) on the SBD1 policy is given. In the present work, feeding into the column with fixed location (SBD2) and with varying location (SBD3) is then studied.

After the start-up continuous feeding is applied (usually from the start of distillate withdrawal). There is a minimum amount (ca. 14,500 kg) that must be continuously introduced in order to have at least the minimum amount of B in the system (>2,000 kg). Hence, the minimal duration of continuous feeding is ca. 14.5 h.

a. SBD1 policies

Lang et al. (2017) studied two versions of the SBD1 policy. By SBD1a, U_N is kept all the time near to its maximal value (7.84 m^3). By SBD1b, U_N is kept constant at a lower value (5 m^3). By SBD1a, even if a higher reflux ratio (R_1=4) was applied x_W could not be reduced in this step onto 0.005 even if the distillate was too pure. In this work, we found that x_W can be reduced onto 0.005, but only with a very high, uneconomical reflux ratio (R_1=12.5). When the residue becomes pure enough, the top product is still too pure ($x_{D,av}$=1.0000). For lower reflux ratios x_W cannot be reduced onto 0.005 even if $x_{D,av}$>0.99 (e.g at R=12 $x_{W,min}$=0.0051 $x_{D,av}$=0.9999). The process must be stopped when the residue reaches the prescribed purity because at that moment we have product B in the maximum amount and prescribed quality.

Hence, an additional BD step without continuous feeding (Step 2) is needed (with $R_2>R_1$), at the end of which both purity requirements are satisfied (Lang et al., 2017). Both steps are finished when $x_{D,av}$ falls onto 0.99.

The (minimal) reflux ratio for Step 2 (R_2) was determined for two different appropriate R_1 values. For R_1=1.0, R_2 was 2.8, the SED 50.0 MJ/kg B and the recovery of B 0.885. For R_1=0.9, R_2 was 2.4 and the SED was slightly lower than for R_1=1.0 (49.6 MJ/kg B). The recovery of B was 0.876.

In the case of SBD1b, a first BD step is added to reduce reboiler hold-up to 5 m^3 before the continuous feeding starts. The reflux ratios of the steps were: 0.67, 1.0 and 2.4. The SED was 49.1 MJ/kg B, slightly lower than that of SBD1a for R_1=1.0. The recovery of B was also slightly lower (0.873). However, the SBD1b policy is more complicated and the surface area of the heating mantle which can be covered by liquid is smaller.

Lang et al. (2017) concluded that SBD1 policies gave better results than BD1, but their SED were higher than those of BD2 policies. However, SBD1 policies gave higher η_B than all BD policies. SBD1b is more complicated than SBD1a. The main advantage of SBD1a is that U_N can be kept at the maximum and the interruption of the production (filling up, heating up and draining of the reboiler), which causes dead time, is much more seldom.

b. SBD2 policy

The main shortcoming of SBD1 policies is that the column has no stripping section. By SBD2 policy the feed is introduced not into the reboiler but into the column e.g. onto the plate found optimal for the CD (f=19). In this case very favorable results were obtained (Table 1). An additional BD step was not necessary to reach the prescribed purities.

Table 1: Results of SBD2 policy (Case study 1)

Step	total feed		R	Δt	SQ	distillate		Residue	
	kg A	kg B		h	MJ	kg A	kg B	kg A	kg B
1	76,721	8,525	0.70	77.24	295,354	76,563	692	39	7,813

SED is only 37.8 MJ/kg B, very near to the CD value. η_B=7813/(800+77.24x100)=0.9165; which is slightly higher than that of the CD. (η_A=0.9979, slightly lower than that of CD). One step is enough and the mass of product B is the maximum possible (7.85 t). However, this policy requires shaping of a feed location in the BD column.

Figure 3 shows the x-profile at different moments, as well as the profile of CD for comparison. At end of start-up (t=0), the profile shows that the feeding location is not optimal (local minimum on plate 19). At halftime ($t=t_{total}/2$), however, the profile crosses the x=0.9 line at f=19, as that of CD does. Above this stage, the two profiles are essentially identical. At the end of the operation ($t=t_{total}$), f_{opt} would be 18 instead of 19. This profile lies even closer to that of CD above stage 18, while below this stage, x of SBD2 is lower than that of CD. This means that at a certain point of time the profile of SBD2 crosses the CD one.

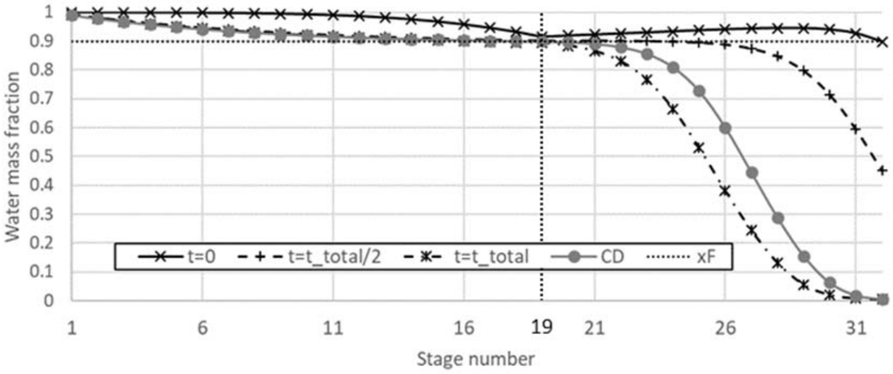

Figure 3: Evolution of the x-profile for SBD2 and the CD profile (Case study 1, f=19)

f_{opt} for SBD2 is 21,very close to that of CD (19). By increasing f from 19 to 21, SED decreased by 0.13%, only.

c. SBD3 policy

For the BD the x-profile of the column is varying and the plate whose liquid composition is close to that of the feed is moving upwards. Therefore, we studied also a new SBD policy for a column with variable feed locations (Figure 1). The actual feed location used is moved upwards during the process. First the feed enters the still, and when $x_{25}<x_F$ it is introduced to plate 25, when x_{17} becomes smaller than x_F to plate 17 and so on (Table 2).

Table 2: Results of SBD3 policy (Case study 1)

Step	feed plate	R	Δt	SQ	distillate		residue	
			h	MJ	kg A	kg B	kg A	kg B
1	32	0.70	0.7	2,499	698.3	1.4	6,944.2	858.2
2	25	0.70	46.05	176,158	45,657.6	401.7	2,743.3	5,058.7
3	17	0.70	29.95	114,796	29,676.9	279.1	38.4	7,764.6
4	9	-	-	-	-	-	-	-

There was no need for Step 4 since at the end of Step 3 the purity of residue (99.51 % B) reached the prescribed one and that of $x_{D,av}$ was slightly above it (99.11 % A). The SED is 37.75 MJ/kg B, and the recovery of B: 7,764.6/(800+76.65x100)=0.9172; both are slightly more favourable than those of SBD2. It must be noted that SBD3 policy have not been optimized yet.

3.1.3 Summary of the results

The most important results of the operational policies are compared in Table 3, including the number of start-ups and discharges that increase the dead time, the amount of fresh feed placed in the reboiler as charge (SF_{batch}) and fed continuously (SF_{conti}), and the amount of B product (SW). The lowest SED is reached by SBD3, which also gives the highest η_B. The worst results are given by BD1 (except for the infeasible BD0).

BD2 policies might be preferred for heat sensitive materials, since their maximal residence time in the reboiler (τ_{max}) are lower than those of BD1 and all SBD policies. All BD policies gave better results than BD0 except for residence time.

Table 3: Summary of results for Case study 1

Step	BD1	BD2a	BD2b	SBD1a (R_1=0.9)	SBD2 (f=19)	SBD3	BD0
No. of start-ups	4	5	4	1	1	1	1
No. of discharges	1	4	3	1	1	1	1
No. of steps	4	5	4	2	1	3	1
SF_{batch}, t	26	32	24	8	8	8	8
SF_{conti}, t	-	-	-	26.7	77.2	76.65	-
SW, t	2.15	2.73	2.03	3.06	7.85	7.8	0.612$^+$
Δt_{total}, h	23.3	28.5	21.4	31.5	77.2	76.65	7.28
SED_B,MJ/kgB	55.5	48.2	47.7	49.6	37.80	37.75	60.0
Recovery of B	0.823	0.849	0.842	0.885	0.9165	0.9172	0.767
$\tau_{max}/\Delta t_{total}$	1.0	2/5=0.4	2/4=0.5	1.0	1.0	1.0	1.0

$^+$ below the minimum

3.2 Case study 2

The feed contains 90 mass% acetone (x_F=0.9). The prescribed product compositions: $x_{D,av}$=0.995 and x_W=0.01. For CD, f_{opt}=30. The reflux ratio is 1.7, the SED is 1.433 MJ/kg A, and the recovery of A 0.9988.

3.2.1 Batch distillation policies

For the BD1 policy, the reflux ratio of the steps (R_1=R_2=1.65, R_3=R_4=1.66) are so determined that when the volume of the residue reaches the minimum, $x_{D,av}$ be 99.5 %. In this case, at the end of Step 4 the loss of A is still too high, therefore a 5th step is necessary under considerably higher R (2.2). Steps 1-4 last ca. 5.85 h each; Step 5 is 5.50 h long. SED is only by 3% higher than that of CD. η_A=22,987/(5867.8+4x0.75x5867.8)=0.9794.
By BD2, the necessary amount of B can already be collected in 4 batches (R=1.65), and then processed in a fifth one (R=2.2). The total duration of the first four batches is 23.44 h, the last one is 5.38 h long. The SED is by 2 % lower than that of BD1, however the recovery of A is by 0.27 % lower: η_A=22,925/(4x5,867.8)=0.9767.
If the minimum volume of the residue is not taken into account (BD0 policy), lower SED can be obtained (1.41 MJ/kg A) than with CD, although the η_A is significantly lower (0.9868). Process duration is 7.28 h; R is 1.65.

3.2.2 Semi-batch policies

a. SBD1 policies
The SBD1 policy was studied both at maximal U_N (SBD1a) and at a lower U_N value (5 m^3, SBD1b) (Table 4). By SBD1a, both purity requirements can be reached in one step only with uneconomically high R (> 5). If R of CD is applied, an additional BD step is necessary (with increased R) since when $x_{D,av}$ falls on the prescribed one, the loss of A is still too high. At the end of Step 2, both products have their prescribed purity. The SED is close to that of BD1: 57,937/39,292=1.475 MJ/kg B, but the recovery of B (39,292/(5,867.8+45.68x733.48)=0.9979) is much higher, than those of the BD policies, it is close to that of CD.By SBD1b, U_N is first reduced in a BD step with the same R as in CD, then the continuous feeding is started. The last, short BD step needs a higher R than by SBD1a. With reduced U_N, the SED is lower: 62,899/43,555.3=1.444 MJ/kg A, although η_A decreased slightly (43,555.3/(5,867.8+51.68x733.48)=0.9950). The SBD1b policy has a lower SED than the BD1 and BD2 policies, and higher η_A.

b. SBD2 policy
The continuous feeding arrives at the plate found optimal for CD (f=30). The process is shorter (Table 4) than by SBD1, meaning that less feed can be processed in one production. Step 2 requires a lower reflux ratio. The SED, however, is slightly higher: 30,803/20,715=1.487 MJ/kg A, while η_A is slightly lower: 20,715/(5,867.8+20.34x733.48)=0.9965, therefore the more favorable and simpler SBD1 must be applied. The SBD3 policy is not worthy of studying in this case.

3.2.3 Summary of the results

The most important results of the operational policies are compared in Table 5. The variation in both the SED and recovery of A is low. BD2 has again lower SED than BD1. Except for BD0 SBD1b has the lowest SED. The recoveries of the SBD policies are significantly higher than those of the feasible BD policies.

Table 4: Results of SBD policies (Case study 2)

Policy	Step	R	Δt	SQ	distillate		residue	
			h	MJ	kg A	kg B	kg A	kg B
SBD1a	1	1.7	45.68	51,424	36,336	179	2,920	4,194
	2	2.8	3.72	6,513	2,956	18.0	35.1	4,084
SBD1b	1	1.7	2.86	3218	2276	10.4	3474	640
	2	1.7	51.68	58595	40993	205.6	322	4585
	3	4.8	0.36	1086	286.3	1.4	42.7	4575
SBD2	1	1.7	20.34	22894	16179	79.7	4490	2228
	2	2.2	5.7	7909	4536	19.6	20.3	2123

Table 5: Summary of results for Case study 2

Step	BD1	BD2	SBD1a	SBD1b	SBD2	BD0
No. of start-ups	5	5	1	1	1	1
No. of discharges	1	4	1	1	1	1
No. of steps	5	5	2	3	2	1
SF_{batch}, t	26.08	26.08	6.52	6.52	6.52	6.52
SF_{conti}, t	-	-	45.7	51.7	20.3	-
SW, t	2.34	2.42	4.12	4.62	2.14	0.568^{+}
Δt_{total}, h	28.9	28.8	49.4	54.9	26.0	7.28
SED_A,MJ/kgA	1.475	1.458	1.475	1.444	1.487	1.41
Recovery of A	0.9794	0.9767	0.9979	0.9950	0.9965	0.9868
$\tau_{max}/\Delta t_{total}$	1.0	2/5=0.4	1.0	1.0	1.0	1

4. Conclusions

Different batch (BD) and semi-batch distillation (SBD) operational policies were studied by dynamic simulation with the ChemCad professional flow-sheet simulator for two case studies. Semi-batch policies were studied by keeping reboiler volumetric hold-up constant during the continuous feeding. The policies were compared on the basis of the specific energy demand (SED) and the recovery (η) of the valuable component. In Case 1, the less volatile component (morpholine, B) must be produced from a dilute aqueous solution in high purity. In Case 2, acetone (A) is produced from its aqueous solution of low water (B) content. In both cases, the prescribed purity of B cannot be reached in one batch, because of the minimum volume of the residue at the end of the batch. For the BD polices we stated that it is better to empty the reboiler at the end of the batches, and redistill the residues collected in a tank (BD2) than filling the next amount of fresh feed on the residue of the previous batch (BD1), which is a common practice in the industry. For Case 1 the best results (lowest SED, highest η_B) were obtained with SBD3 policy where the continuous feeding is performed into the column with varying locations. SBD2 (fix feeding into the column) gave slightly worse results. SED of SBD1 policy (continuous feeding into the reboiler) was near to that of BD2 with lower dead times. For Case 2, the differences in SED and η_A of the different policies were low. The optimal feeding location was very close to the reboiler. SBD1 policy with reduced reboiler hold-up (SBD1b) gave slightly better SED than all the other feasible policies. However SBD1b is more complicated, as it contains one step more than SBD1a where the reboiler hold-up is kept on the maximum. With continuous feeding of one part of the mixture to be separated, the production capacity of existing batch distillation columns can be increased. The dead times can be reduced and the liquid volume in the reboiler can be kept constant even on the maximum whose advantage is that the heating surface can be completely covered by liquid during the distillation. The proper selection of the feed plate(s) has great influence on the effectiveness of the process. For this the optimal feed plate of the continuous process provides useful information.

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