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Comparison of Extractive and Pressure-Swing Batch Distillation for Acetone-Methanol Separation

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The performance of extractive (BED) and pressure swing distillation (PSBD) for the separation of the mixture acetone(A)-methanol(B) forming a minimum azeotrope are compared. For the BED, where the solvent(S) is water, three different operational policies (basic, modified, controlled) are studied. For the PSBD the double column batch stripper (DCBS) is applied with and without heat integration (HI). The rigorous simulation calculations are made with a professional dynamic simulator.
From the same equimolar binary charge A and B are produced in the same purity with all methods. The specific overall energy consumptions and recoveries are compared.

1. Introduction

For the continuous separation of binary homogeneous azeotropes widespread methods are pressure swing (PSD) and extractive distillation (ED). PSD is effective if the composition of the azeotrope varies significantly with pressure. Its main advantage is that it does not need the application of a separating agent contrary to the ED requiring an efficient solvent (S). Luyben (2008) compared the two methods for the separation of the mixture acetone-methanol which is a frequent waste in pharmaceutical industry forming a pressure sensitive minimum azeotrope. The column producing acetone was operated at 1.013 bar while the methanol column at 10 bar for PSD and 5 bar for ED, respectively. Applying heat integration (HI) for both methods he stated that the controllability of continuous PSD and ED is quite similar and total annual cost of ED is somewhat lower.
The batch distillation is more advantageous than the continuous one in many cases (eg. when the amount and composition of the mixture frequently changes). Moreover in a single batch column an arbitrary number of products can be produced. However neither PSD nor ED is well-known in the industry in batch.
The basic operational policy (BOP) of the batch extractive distillation (BED, Fig. 1a.) in a single column was suggested by Yatim et al. (1993) for the mixture acetone (A) – methanol (B) + water (S). The main particularity of the BED is continuous feeding of S ($F>0$) in two steps of the batch process (purification under total reflux ($R=\infty$) and production of A). Several BED operational policies were studied by Lelkes et al. (1998). Based on industrial experiences Lang et al. (2006) modified the BOP of the BED. By

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the modified policy (MOP) the first two steps (heating-up and purification) of the BOP were combined. The solvent feeding was already started during heating-up of the column under R=∞ when the vapour reached the solvent feed plate.

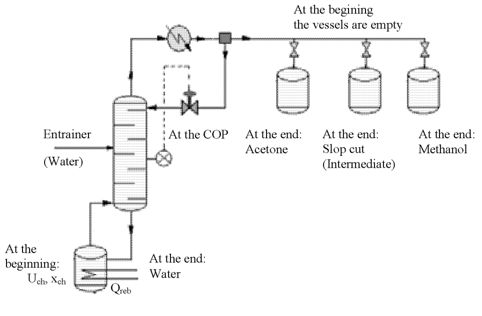

Figure 1 a. Scheme of the BED

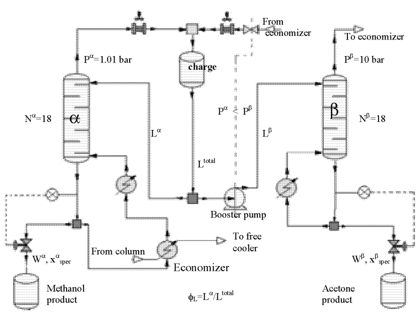

Figure 1b. Scheme of the DCBS

The BED was studied by Luyben and Chien (2010) with simulation for the above mixture (under fixed R and F) and the mixture isopropanol (A)-water (B)+DMSO (S). For the second mixture, where S is much heavier than A and B, during the step of production of A both R (on the basis of temperature of the reflux drum) and F (on the basis of temperature of the S feeding stage) were varied. The aim of varying feeding of S is to reduce its quantity applied.

For the pressure swing batch distillation (PSBD) new double column configurations (for min. azeotropes double column batch stripper (DCBS, Fig. 1b)) were suggested by Modla and Lang (2008). Kopasz et al. (2009) presented a simple scheme for the control of product compositions for DCBS. DCBS provides several advantages, among others the possibility of HI (Modla and Lang 2010).

The purpose of this paper is to compare BED and PSBD (with and without heat integration) in terms of specific overall energy consumption. For the rigorous simulation we used the dynamic simulator of CHEMCAD 6.0 (CC-DCOLUMN, Chemstations, 2007).

2. VLE data of mixture acetone-methanol

For the two different pressures (1.01 and 10 bar) the azeotropic data of the mixture acetone-methanol are shown in Table 1. The large shift in the azeotropic composition from 78 to 37 mol % acetone indicates that a pressure swing separation should be feasible.

Table 1: Calculated data of the azeotropes

Mixture	P [bar]	x_{AZ} [%]	T_{AZ} [°C]	$T_{BP,A}$ [°C]	$T_{BP,B}$ [°C]
Acetone (A)-	1.01	78	55.2	56.0	64.4
Methanol (B)	10	37	142.9	142.9	136.7

3. Rigorous Simulation Results

3.1 Input data

The initial charge contains 50 mol% acetone ($x_{ch,A}=0.5$). The specified purities are 94 mol% for Acetone and Methanol and 99 mol% for Water. The liquid hold-up is 4 dm^3/plate. The number of theoretical stages (N) for each column is 18. (The total condenser and total reboiler do not provide a theoretical stage.) The S feeding stage is the 6-th for the BED (from the top).

In each case the quantity of the charge is 0.9 m3 ($U_{ch}=15.96$ kmol). At the start the columns are filled with boiling point liquid (at the pressure of given the column).

For the BED the heat duty of reboiler is 400 MJ/h, the pressure of the column is 1.01 bar and the temperature of the water feeding is 80 °C.

The DCBS columns are operated at 1.01 bar (producing methanol) and 10 bar (producing acetone).

3.2 Batch extractive distillation

Besides the basic and modified operational policy a new one is also studied.

Controlled operational policy (COP)

The controlled operational policy (COP) consists of the following steps:

1. Heating-up and purification: Operation under total reflux with solvent feeding ($R1=\infty$, $F1>0$), Step 1 is ended when acetone concentration in the distillate ($x_{d,A}$) reaches 0.95.
2. Acetone production (P1): Operation under finite varying reflux ratio ($x_{d,A}$ is kept constant at its prescribed value: $x_{d,A,spec}=0.94$) with solvent feeding ($R_2<\infty$, $F_2>0$), Step 2 is ended when the acetone recovery takes its prescribed value (94%).
3. Collection of a slop cut (S1): Operation under finite fixed reflux ratio without solvent feeding ($R_3<\infty$, $F_3=0$), Step 3 is ended when the methanol concentration in the distillate ($x_{d,M}$) reaches the specified purity of methanol product (0.94)
4. Methanol production (P2): Operation under two different fixed reflux ratios without solvent feeding ($R_4<\infty$, $F_4=0$): a. The reflux ratio at the beginning of this step equals with that of Step 3 (R_3). b. When $x_{d,M}$ is higher than 0.98 the reflux ratio is changed. Step 4 is ended when the mole fraction of Methanol in the product tank ($x_{d,M,av}$) falls to its prescribed value (0.94).
5. Production of water (P3) as bottoms (optional): If at the end of Step 4 the water product has not reached yet its purity specified another slop-cut (S2) must be collected. (In our case at the end of Step 4 the bottom product has already reached its purity specification therefore Step 5 was omitted.)

We investigate which parameter is suitable for controlled parameter. The criteria for selection of controlled parameter are the following ones:

- unambiguous relation with product quality (acetone concentration in the distillate),
- sensibility,
- it must be easy to measure.

The evolution of the stage temperatures are investigated. The location of temperature control stage is selected by the above criteria.

The dynamic responses of $x_{d,A}$ and several stage temperatures in Step 2 are studied.

First the distillate composition is stable (Figs. 2, between 40-170 min). After that we make oscillate $x_{d,A}$ by applying a PI controller (Figs. 3). On most of the stages the temperature is not stable in spite of the constancy of the acetone concentration (Figs. 2). However the temperature of stage 9 (T_9) only slightly varies (Fig. 2b). The top temperature (stage 1) can not be used as controlled parameter for ensuring $x_{d,A,spec}$, because it is not sensitive enough (Fig. 3a). However T_9 (Fig. 3b) varies sensitively and simultaneously with $x_{d,A}$. Hence R is varied by T_9 (set point: 62.7 °C) with the PI controller.

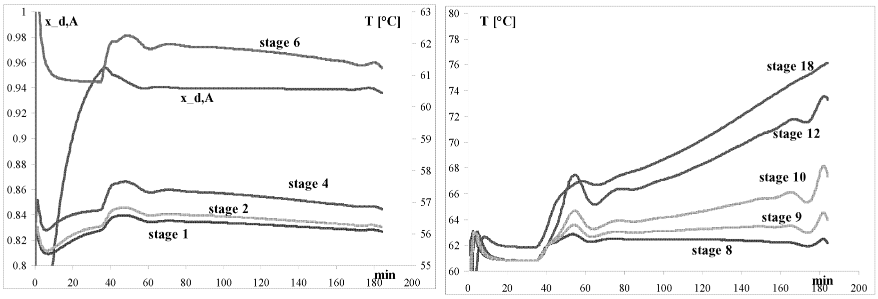

Figure 2a. *Figure 2b.*

Figure 2. Evolution of the stage temperatures (stable $x_{d,A}$)

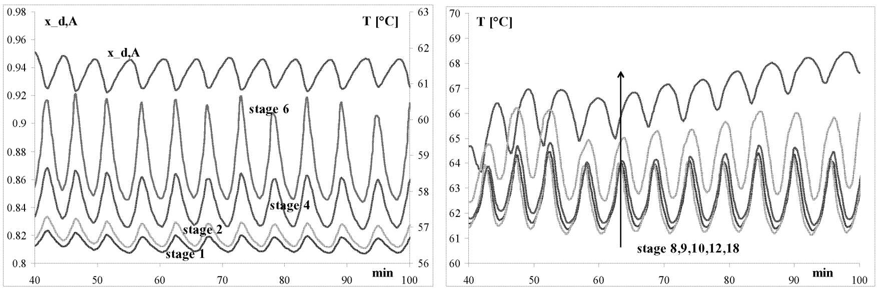

Figure 3a. *Figure 3b.*

Figure 3. Evolution of the stage temperatures (oscillating $x_{d,A}$)

We investigate the effects of different disturbances (e.g. water flow rate and temperature, reboiler heat duty) on the product quality and recovery of acetone. We stated that the suggested control policy is efficient and robust.

The influence of the most operational parameters (e.g. R, F) on the overall specific energy consumption (SQ/SPr) was studied for all operational policies (BOP, MOP, COP) in order to determine the minimum of SQ/SPr. The results are summarized in Table 2.

Table 2: Results of the different BED and PSBD methods

	BED			**DCBS**	
	BOP	**MOP**	**COP**	**Without HI**	**With HI**
Start-up&Purification [min]	122	33	33	-	-
Acetone production [min]	206.5	202	172.5	-	-
Slop-cut withdrawal [min]	64	62.5	38.0	-	-
Methanol production [min]	183	190	241.5	-	-
Duration of the process [min]	575.5	487.5	485	213	213
Acetone product [kmol]	8.73	8.68	8.61	8.23	8.23
Methanol product [kmol]	6.67	6.94	7.32	8.44	8.44
Acetone recovery [%]	95	94	94	84	84
Methanol recovery [%]	73	76	80	86	86
Water consumption [dm^3]	1095	785	685	-	-
Overall spec. energy consumption [MJ/kmol]	269	223	205	245	145
Slop cut Acetone [%]	22.8	24.2	31.1	-	-
Slop cut Methanol [%]	77.1	75.7	68.0	-	-
Slop cut Water [%]	0.1	0.1	0.7	-	-
Slop cut product [kmol]	2.37	2.34	2.36	-	-

3.3 Pressure swing batch distillation (PSBD)

For the separation of the minimum azeotrope the DCBS is applied. The main operational parameter is the liquid division ratio ($\phi_L = L^\alpha / L^{total}$). First the optimum value yielding the minimal overall specific energy consumption (SQ/SPr) is determined by varying ϕ_L in the region 0.1-0.7 without applying the economizer. The best result (245 MJ/kmol) is obtained at $\phi_L=0.45$. The boiling point of azeotrope (top stream temperature of the high-pressure Column β (133.9 °C at 10 bar)) is higher than the boiling point of methanol product (bottom stream temperature of the low-pressure Column α (near to 64 °C at 1.01 bar), so heat integration could be attractive in terms of energy consumption. The vapour leaving the top of the high-pressure column is fed into an economizer. The economizer is used to recover energy from the top vapour of Column β by (partial) vaporisation of the reboil stream of Column α. The operational parameter of the economizer is that the temperature of the hot stream outlet (the recycled top vapour from Column β) is higher by 11 °C than that of the cold stream outlet. The overall specific energy consumption is much lower than it is without HI (Table 2).

4. Comparison of the BED and PSBD

On the basis of the results obtained for the BED and DCBS (Table 1) we can state that
-The specific energy consumption is the lowest for the DCBS with HI. However the energy demand of both of the MOP and COP of the BED was lower than that of the DCBS without HI.

-By all BED policies higher recovery of acetone (A) was achieved than by the PSBD. However the recovery of methanol (B), which is produced between A and S by the BED, remained below that of the PSBD. By the BED the products A and B are polluted mainly with S contrary to the PSBD where they are polluted with the other original component obviously decreasing its recovery.

-By the BED there is a slop-cut contrary to the PSBD, which must be recycled to the next charge and/or processed with another charge (of different composition).

- The BED needs much more operation steps than the PSBD.
- The control of the DCBS is easier than that of the BED since the columns are operating practically in steady state.
- The capital costs of the DCBS (requiring two columns) are obviously higher than that of the BED.

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