Heteroazeotropic batch distillation in a new double-column system

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We investigate a new double-column batch heteroazeotropic distillation system (DCS) by rigorous simulation. Its performance is compared with that of the conventional batch rectifier (BR). For the new configuration the minimal operational time is determined. The separation of mixture isopropanol – water + cyclohexane is studied by using a professional dynamic flowsheet simulator. In the case studied we obtained better results (eg. lower specific energy consumption) with the DCS than with the BR.

1. Introduction

The batch heteroazeotropic distillation (BHD) is frequently applied in pharmaceutical and fine chemical industries for the recovery of organic solvents. If the components of a mixture form a heteroazeotrope or by the addition of an entrainer a heteroazeotrope can be formed, the azeotropic composition (boundary) can be crossed by decantation. To our best knowledge the process is exclusively applied in the industry in batch rectifiers (BR) in open operation mode (with continuous distillate withdrawal). The BR was investigated with variable decanter holdup by Rodriguez-Donis et al. (2002) and with continuous entrainer feeding by Modla et al. (2001, 2003) and Rodriguez-Donis et al. (2003), respectively. The heterogeneous batch distillation in batch rectifiers (BR) and also in multivessel columns was extensively studied by Skouras et al. (2005a,b). Pommier et al. (2008) developed a specific software architecture based on the BatchColumn® simulator and on both SQP and GA algorithms for the optimisation of sequential batch columns and heterogeneous batch distillation. Lang et al. (2008) suggested a new double-column system (DCS) for BHD. The two column system is operated in closed circuit without any continuous product withdrawal (*Fig. 1*). They made feasibility studies based on a very simplified model (e.g. assumption of maximal separation, neglecting of plate and decanter hold-ups etc.). The above authors (Denes et al. (2008)) made also rigorous simulation calculations for two test mixtures: for a binary heteroazeotropic mixture (n-butanol - water) and for the separation of the minimum azeotrope isopropanol – water by applying the classical entrainer benzene (which is very efficient but nowadays not permitted for the practical application). The new configuration was found competitive with the BR and worthy of further investigations. For the latter, ternary mixture it gave even better results than the BR.
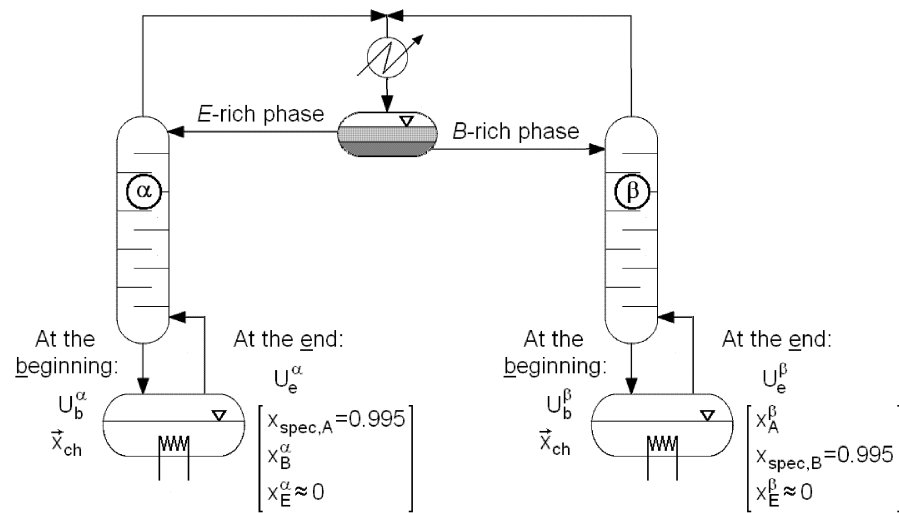
Figure 1. The scheme of the double-column system

In this paper
-we study the separation of the (homo)azeotropic mixture isopropanol (*A*) – water (*B*) in the new (DCS) and the traditional configurations (BR) applying cyclohexane as entrainer (*E*),
-the performances of the two configurations are compared by rigorous simulation,
-for the calculations the dynamic simulator CC-DCOLUMN (of the professional flowsheet simulator ChemCad 6.0) is applied.

2. Simulation method

The following simplifying assumptions are applied:
- theoretical trays,
- constant volumetric liquid holdup on the trays and in the decanter,
- negligible vapour holdup,
- negligible duration of pumping between the two steps (BR).
The model equations to be solved are well-known:
a. Ordinary non-linear differential equations (material and heat balances)
b. Algebraic equations (VLE, LLE relationships, summation equations, hold-up and physical property models).
For describing phase equilibria the UNIQUAC model is applied. For the solution of the above equations the dynamic simulator CC-DCOLUMN is applied. Column sections are modelled by the module DYNAMIC COLUMN, reboilers and the (vertical cylindrical flat bottom) decanter by the DYNAMIC VESSEL, respectively (*Fig. 2*). Besides these units the flow-sheet still contains HEAT EXCHANGERs (condensers), MIXERs and DIVIDERs.
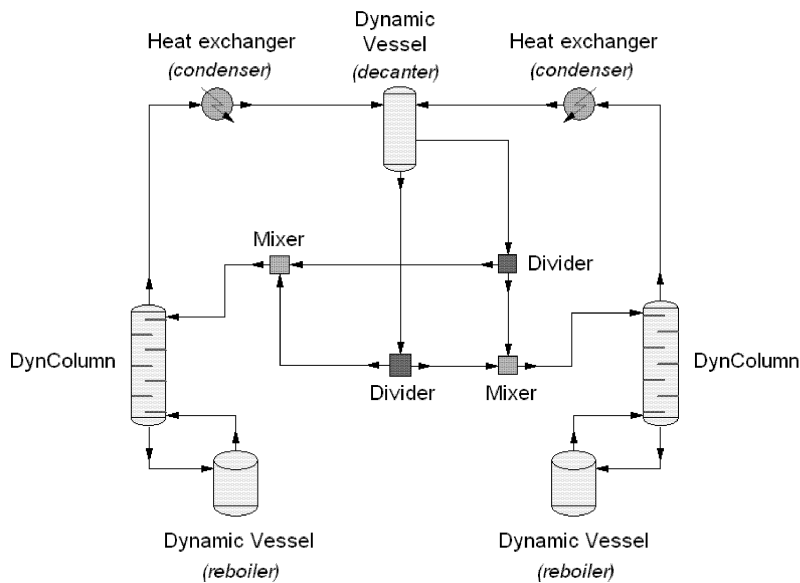
Figure 2. ChemCAD model of the new double-column system

3. Results

In each case the total number of theoretical stages (N) without the condenser, decanter and reboiler is 10 for the BR. For the DCS two different cases are studied: either the sum of the numbers of stages of the two columns is 10 or both columns contain 10 stages. The separation is performed at atmospheric pressure.

Both reflux and distillate (BR) are homogeneous. In the decanter (D=0.5 m) the volume of liquid phases are prescribed constant (after the start-up). The ratio of the volumes of the two phases is equal to that of the ternary heteroazeotrope. The holdup of the plates is taken ~ 1 % of the initial liquid volume in the reboiler of the given column. The (total) amount of charge (U_{ch}) is 100 kmol (5.69 m^3), its composition is that of the binary homoazeotrope isopropanol (A) - water (B): $x_{ch,A}$=67.4 mol%. The prescribed purity of both products is 99.5 mol%. The total heat duty is 12 MJ/min in each case. The prescribed levels of the decanter: B-rich phase: 0.134 m, E-rich phase: 0.6 m. The calculations are started with dry plates and decanter.

For both configurations there is an optimal amount of E for which the (total) duration of the production cycle is minimal. For the DCS the duration of the distillation can be also influenced by the division of the total number of plates (N^{α}/N), total heat duty (Q^{α}/Q), and the amount of charge (U_b^{α}/U_{ch}) between the two columns (α and β). In this paper for each configuration we publish the results only for the optimal case with *minimal duration*. For the DCS this means that the prescribed purity is reached in both columns at the same time.

For the BR the optimal amount of E by which the duration of Step 1 (determining primarily the duration of the production cycle) is minimal: 3.8 kmol (0.408 m^3). In Step 1 A is produced (*Table 1*). The duration of this step is much longer than that of Step 2.

Table 1. Optimal parameters and results

		BR			DCS (1)		DCS (2)	
		Step 1	Step 2	Total	Col. α	Col. β	Col. α	Col. β
Number of stages	-	10	10	10	7	3	10	10
Division of charge	kmol	-	-	-	57	43	55	45
Heat duty	kW	200	200	-	169.0	31.0	168.3	31.7
Plate holdup	dm³/plate	60	10	-	35	25	35	25
Entrainer	kmol	3.8	0.0	3.8	4.0	0.4	4.0	0.4
Duration	hour	36.83	1.07	37.9	28.5		28.8	
Product A	kmol	50.75	0.00	50.75	59.33	0.00	58.78	0.00
Product B	kmol	0.00	21.30	21.30	0.00	25.46	0.00	26.16
Recovery of A	%	74.89	-	74.89	87.53	-	86.74	-
Recovery of B	%	-	65.10	65.10	-	77.89	-	79.97
Distillate	kmol	37.90	3.35	3.35*	0.00		0.00	
Distillate composition	mol% A	17.07	53.44	-	-		-	
	mol% B	82.51	43.09	-	-		-	
	mol% E	0.42	3.47	-	-		-	
Column holdup	kmol	13.28	10.49	23.77	17.68		17.50	
Column holdup composition	mol% A	75.24	34.41	-	44.12		47.41	
	mol% B	1.50	65.21	-	34.00		30.38	
	mol% E	23.16	0.38	-	21.88		22.21	
Decanter holdup	kmol	1.87	2.76	4.63	1.94		1.96	
Decanter holdup composition	mol% A	25.24	35.13	-	26.34		26.80	
	mol% B	44.61	64.76	-	46.45		46.95	
	mol% E	30.15	0.11	-	27.21		26.25	
Byproducts	kmol	15.15	16.60	31.76	19.61		19.46	

* The distillate of Step 1 is processed in Step 2 hence it is not byproduct.

Though the amount of distillate (*B*-rich phase) in Step 1 is not too low (37.9 mol% of the charge) but the *E*-content of this distillate, which must be removed (together with *A*) in Step 2, is very low (0.42 mol %). At the beginning of Step 2 the concentration of *E* decreases quickly in the reboiler (*Fig. 3*), and later on *E* disappears from top vapour as well (*Fig. 4*). The concentration of *A* continuously decreases in the reboiler during the whole step.

For the DCS the optimal division of the total number of plates is unequal: $N^{\alpha}=7$ $N^{\beta}=3$.

In the case where the 10 stages are optimally divided between the two columns the optimal total amount of *E*: 4.4 kmol. The 90.9% of the amount *E* is filled in reboiler α. The heat duty in column α (84.5 %) is much higher than in the other column. In this case 57% of the charge must be filled in the reboiler α.

The increase of the number of stages of both columns to that of the BR (DCS (2)) does not improve considerably the performance of the system. This can be partially explained by the increase of column holdups.
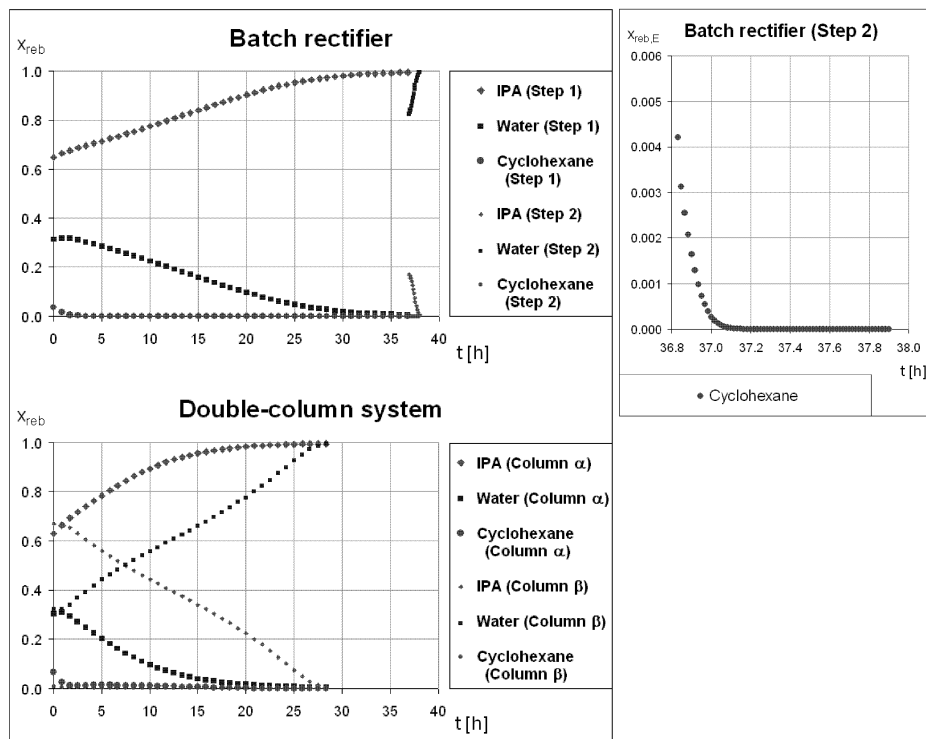
Fig. 3. The evolution of liquid compositions in the reboiler(s) (for the DCS $N^{\alpha}=7$, $N^{\beta}=3$)

Comparing the two different configurations it can be concluded that
-The duration of the process is shorter for the DCS.
-The recovery of both components is higher for the DCS.
The evolution of liquid compositions in the reboilers for both configurations is shown in *Fig.* 3, that of top vapour compositions in *Fig. 4.*, respectively.

Conclusion

We investigated a new double-column batch heteroazeotropic distillation configuration by rigorous simulation using a professional dynamic simulator (CC-DColumn). The performance of the new configuration was compared with that of the conventional batch rectifier. The simulations were performed for the separation of the minimum homoazeotropic mixture isopropanol – water by applying cyclohexane as an entrainer. For both configurations we determined the optimal quantity of entrainer to be applied and for the new double-column system (DCS) the optimal divison of the entrainer, the charge and the total heat duty providing the minimal operational time for the different configurations. The new double column system provided better performance than the batch rectifier in the case studied. By the results obtained so far DCS appears to have high potential and deserves further investigations (closed loop control etc.).
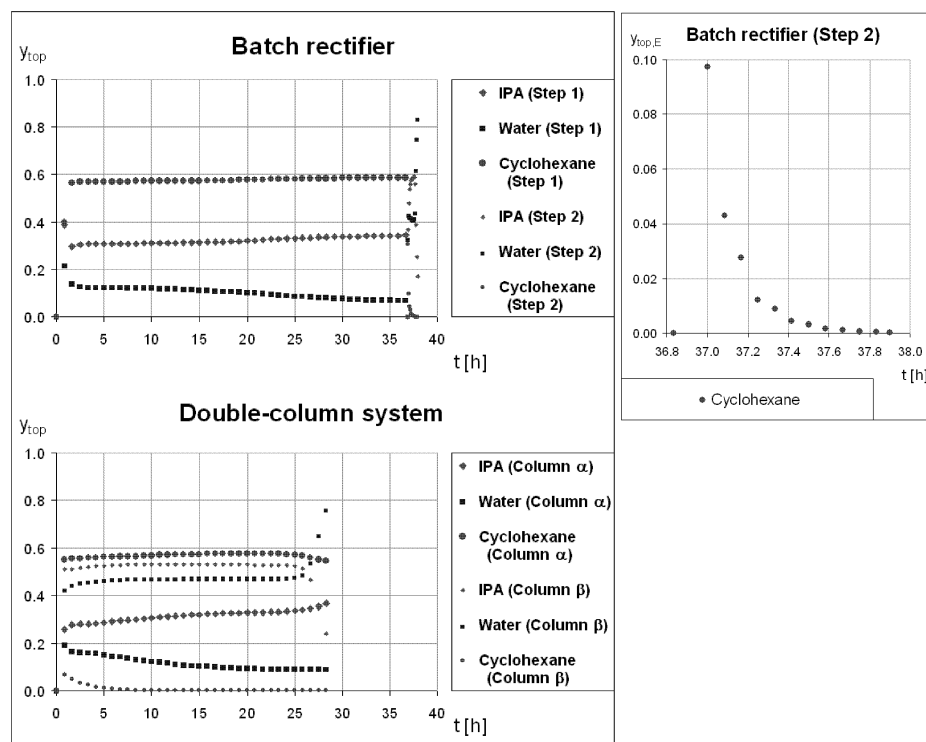
Figure 4. The evolution of the top vapour composition(s) (for the DCS N^α=7, N^β=3)

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