Influence of the Size of Prefractionator on Processing Capacity for Two-Column Batch Distillation Processes

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Abstract. Depending on the feed composition, the processing capacity (Cap) of a batch distillation column may be increased by applying another, smaller column (Column1), which can serve as prefractionator or can operate in parallel, independently of the large column (Column2). The two-column processes are investigated with varying the number of trays of Column1 (N_1) by rigorous simulation for the separation of acetone-water and water-acetic acid mixtures. The purity of the organic product is specified. Three operational policies are studied: only the large column is operated (Policy I), Column1 is operated either sequentially as prefractionator for Column2 (Policy IIa) or in parallel with it (Policy IIb). All policies are optimised by maximising Cap. The goal is to find the range of N_1 for different charge compositions where Cap of the single-column process (Cap^I) can be increased by applying the smaller column by reaching at least its recovery (η^I).

Keywords: Optimisation, Processing capacity, Batch distillation, Two-column process.

Introduction
Batch distillation is frequently applied in the fine chemical and pharmaceutical industry since it can be used for treating mixtures of variable quantity and composition (Mujtaba, 2004). Typically, the separation is performed in a single batch column. However, the processing capacity (Cap) can be increased by operating an additional, even smaller batch column simultaneously, which can be operated parallel or sequentially (Nemeth et al., 2020) to the larger column.
Several authors investigated the possibilities of increasing the capacity (or annual profit) of batch columns. However, their main focus is usually the optimisation of the operation of a single existing column. Fidkowski (2009) increased the capacity of an existing, industrial multicomponent batch distillation process by maximising the production rate.
When the capacity of an existing column is already optimised for its single operation (Policy I), the simplest way of increasing the capacity is to install a similar column (or to use another existing one) and to operate the columns in parallel (Policy IIb). However, installing a new column of similar size requires considerable capital investment. Hence it is more favourable to use an existing, smaller column. The increase of Cap by using two batch distillation column is rarely studied in the literature. The main focus of this work is the determination of the minimal column size (expressed by the number of theoretical plates) necessary for reaching higher Cap for the two-column process (Policy II) than for Policy I. Another option is to operate the two columns sequentially (Policy IIa) by using the smaller one (Column1) for preliminary separation. Pre-fractionation is commonly used in continuous distillation processes, which also gave later the idea of the dividing-wall columns. Adi and Chang (2010) studied the scheduling of a two-column batch heteroazeotropic distillation system. The process was not modelled rigorously but by using only material balance equations. For several case studies, scheduling optimisation was performed to maximise the overall profit of a production campaign. The authors concluded that equipment sharing in the case of distillation tasks causes a significant decrease in the overall profit.
In our previous work (Nemeth et al., 2020), Policy IIa was already investigated for the mixture dichloromethane-acetone-water, but the main objective was to minimise the specific energy demand of the process. The processing capacity significantly increased compared to the single-column process. However, the recovery of acetone from the ternary mixture was not kept constant contrary to another work of the authors (Nemeth et al. 2022), where the recovery was equal to that of the single-column process for the two-column process, and the goal was to investigate by simulation and optimisation whether higher Cap can be reached with the two-column process than with the single-column one. The separation of two mixtures was studied: acetone-water (Mixture 1) and water-acetic acid (Mixture 2). The two batch distillation columns had different sizes, but constant numbers of trays, contrary to the present work, where the effect of varying the number of trays is studied. The effects of varying charge composition and operation

parameters on Cap were studied. It was found for both mixtures that there is a region of charge composition, where the two-column process has higher Cap than the single-column one.

In this work, the influence of varying the number of trays of the smaller column (N_1) is studied for the first time. Different feed compositions (x_F= 20 – 50 – 80 mass% organic component, referred to as Cases 1-3, respectively) of the mixtures studied by Nemeth et al. (2022): acetone(A)-water(B) and water(A)-acetic acid(B) are considered. The feed compositions with average (50 %), lower (20 %) and higher (80 %) organic component concentrations were selected in order to map the space of composition for the influence of N_1 on Cap. Three operating policies are studied: single-column process (Policy I); two-column process, where the two columns are operated either sequentially (Policy IIa) with the product of the first column processed further in the second one, or independently (Policy IIb): both columns produce on-specification organic product. The goal is to find the range of N_1 for all charge compositions and operating policies, where Cap of the given policy is the highest. The processing capacity of both the two- and single-column processes are maximised either by using a genetic algorithm (GA) coupled to the professional flow-sheet simulator ChemCAD or by sensitivity studies. For all processes, the purity of the organic product is specified (x_{sp}=99.7 mass%) in addition to a minimum recovery constraint, which is equal to the calculated recovery of the single-column process. The results of the optimised two-column processes are compared to those of the single-column one.

Separation process and calculation method

Water forms a tangent azeotrope with acetone at high acetone concentrations and with acetic acid at high water concentrations. The UNIQUAC model was used for describing the vapour-liquid equilibrium for Mixture 1, while the NRTL model was used for Mixture 2. A more detailed description of the equilibrium is found in Nemeth et al. (2022). By Policy I, the separation is performed in a single column providing N_2=22 theoretical stages (including the reboiler and the condenser). The volume of the charge is 10 m^3 (25 °C), and the dead time is 1.6 h. The reboiler duty is 720 MJ/h, and it is assumed that the capacity of the condenser and that of the column are always sufficient. The pressure drop of the column is 0.4 kPa. Hold-ups: 5 dm^3 (condenser) and 3 dm^3/plate. One distillation step is performed in which the product is obtained as distillate (acetone) for Mixture 1 or as still residue (acetic acid) for Mixture 2. The effect of the reflux ratio (R^I) is studied on the recovery (η^I) and Cap^I. Based on the results of Policy I, minimum recoveries are specified for the different feed compositions of both mixtures.

By Policy IIb, the separation is performed in two independent (parallelly operated) columns. The larger column (Column2) is the same as the one used for Policy I, while Column1 is the smaller one (with dead time of 3.5 h). The effect of N_1 is investigated on the total recovery (Eq. 1) and capacity (Eq. 2):

$$\eta^{IIb} = \frac{SPF_1 + SPF_2}{Cap_1 + Cap_2} \cdot \frac{1}{x_F} \qquad (1) \qquad\qquad Cap^{IIb} = Cap_1 + Cap_2 \qquad (2)$$

where SPF is the specific product flow rate defined as the amount of the organic component in the distillate (Mixture 1) or in the residue (Mixture 2) divided by the distillation time t (including the dead time) (Eqs. 3 and 4), and Cap is calculated as the mass of feed processed divided by the distillation time.

$$SPF = \frac{P \cdot x_{sp}}{t} \qquad (3) \qquad\qquad Cap = \frac{F}{t} \qquad (4)$$

where P is the mass of the product, x_{sp} is the mass fraction of the organic component in the product.

The lowest N_1 considered is 5, with a volume of 5 m^3 feed and 360 MJ/h reboiler duty. By increasing N_1, the feed volume and the reboiler duty are also increased proportionally until the size of the larger column (Column2) is reached (22 stages, 10 m^3 feed, 720 MJ/h reboiler duty). (The dead time of 3.5 h is constant.) The design parameters of the columns, including the volume of the charge, are based on an existing industrial separation process. The goal is to find for all N_1 those R_1 (reflux ratio in Column1) and R_2 values for which η^{IIb} is at least equal to that of Policy I, while Cap^{IIb} is maximal. η^{IIb} and Cap^{IIb} were calculated with sensitivity studies by varying N_1, R_1 and varying R_2.

By Policy IIa, the distillate or residue of Column1 is transferred to Column2 for further processing. This two-column system is optimised by minimising N_1 with the following constraints: (1) η^{IIa} and Cap^{IIa} must be higher than those of Policy I (Table 1) for the same feed composition, (2) the mass of residue in Column2 must be higher than 800 kg.

For all optimisations performed, the optimisation variables are N_1, R_1, R_2 and the stopping criterion for Column1, which is the mass fraction of acetone in the distillate ($\bar{x}_{D1,A}$) for Mixture 1, while it is that of acetic acid in the still residue ($x_{W,B}$) for Mixture 2. As the volume of charge of Column1 is smaller than that of Column2, several batches can be processed in Column1 prior to fully filling the still pot of Column2. The number of batches (n) processed in Column1 is determined so that the material transferred fill the still pot of Column2 to the greatest extent possible.

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Minimising N_1 was not performed for Mixture 1 based on the results of the previous works because of the very low value R_1 (<0.01), which practically means that the capacity is independent of N_1.

After the minimal N_1 values are obtained for Mixture 2 for each charge composition, a new optimisation is performed by maximising Cap at given N_1 values. The goal is to find those N_1 values for each charge composition, where Policy IIa has higher Cap than Policy IIb. If no solution is found fulfilling the constraints at the given N_1 value, N_1 is increased, and the optimisation is performed again. With the above procedure, different N_1 regions can be found as the function of x_F, where Policy I, IIa or IIb has the highest Cap.

In all the above cases, the optimization method used is a genetic algorithm (GA) running under Excel coupled to ChemCAD. The parameters of the optimization are: number of individuals: 30; mutation probability: 5%; crossover probability: 70% and the number of maximum generations: 100.

Results

The results are shown first for Policy I (Table 1) chosen as references, then for Policies IIa and IIb.

Table 1. Results for Policy I (Nemeth et al., 2022).

	Mixture 1			Mixture 2		
	Case 1	Case 2	Case 3	Case 1	Case 2	Case 3
Feed composition, mass%	20	50	80	20	50	80
R^I	5	5	5	10	12	13
η^I, %	94.50	99.08	99.92	93.00	93.73	94.17
Cap^I, t/day	21.989	9.917	6.494	0.861	1.156	2.606

Independent operation of the two columns (Policy IIb)

Table 2 summarizes the results for Mixture 1. Below N_1=14, the separation is not feasible. In all cases above N_1=14, the total capacity of Policy IIb is higher than that of Policy I. On the increase of N_1, R_1 decreases, while Cap^{IIb} increases for all compositions studied (the distillation time of Column1 is the dominating one through Cap_1 and SPF_1 in Eqs. 1 and 2. On the increase of x_F, R_1 decreases at lower N_1 values, while it remains almost unchanged at higher N_1 values, similarly to R_2 for all N_1. R_1 values are higher compared to those of Policy I (because of the lower N_1 values), but R_2 values are lower due to the lower η required in Column2. At given N_1 and R_1 values, there is a maximum in η^{IIb} as the function of R_2 (Fig. 1a). The maximum η^{IIb} for each R_1 value is marked by red dots in Fig. 1a.

Table 2. Results for Policy IIb for Mixture 1.

	Case 1				Case 2				Case 3			
N_1	R_1	R_2	Cap^{IIb}, t/day	η^{IIb}, %	R_1	R_2	Cap^{IIb}, t/day	η^{IIb}, %	R_1	R_2	Cap^{IIb}, t/day	η^{IIb}, %
14	28.0	4.81	27.11	94.50	19.5	4.78	12.76	99.08	19.0	4.98	8.172	99.92
15	-	-	-	-	11.7	4.81	14.19	"	10.6	5.30	8.895	"
16	9.80	4.83	33.12	94.50	8.90	4.75	15.32	"	9.00	4.98	9.814	"
18	6.90	4.82	36.63	"	6.50	4.79	17.08	"	7.00	4.95	10.82	"
20	5.60	4.94	38.91	"	6.00	4.67	17.98	"	6.00	4.91	11.67	"
22	5.00	5.00	43.98	"	5.00	5.00	19.83	"	5.00	5.00	12.99	"

Table 3 summarizes the results for Mixture 2. The separation is practically not feasible below N_1=9 for Case 1, and below N_1=8, for Cases 2 and 3 (although for Case 3, at N_1=7 Cap^{IIb} is higher than Cap^I, but only with extremely high R_1, resulting in a distillation time higher than 500 h). For all compositions, Cap^{IIb} is higher than Cap^I. On the increase of N_1, both R_1 and R_2 show a slightly decreasing tendency. R_1 has a maximum as the function of x_F at 50% acetic acid concentration at lower N_1 values (N_1<12). Above N_1=12, the maximum disappears, and R_1 increases on the increase of x_F. R_2 values are slightly higher than R^I for all feed compositions. Similarly to the results of Mixture 1, at given N_1 and R_1 values, there is a maximum in η^{IIb} as the function of R_2 (Fig. 1b). However, the change in η^{IIb} is not as sharp as in the case of Mixture 1. The maximum η^{IIb} for each R_1 value is marked by red dots in Fig. 1b.

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Sequential operation of the two columns (Policy IIa)

The minimal value N_1 is determined where Cap^{IIa} is still higher than Cap^I for Mixture 2 (Table 4). On the increase of x_F, the minimum number of trays decreases. However, it must be noted that according to Nemeth et al. (2022), for Cases 1 and 2, there are lower N_1 values, where Cap^{IIa} is higher than Cap^I. The reason for this is that the optimum is close to the constraints, and thus, it is found with a lower probability (more generations would be required). Since η and Cap are included in the optimisation problem as constraints only, their values are higher than the specified ones.
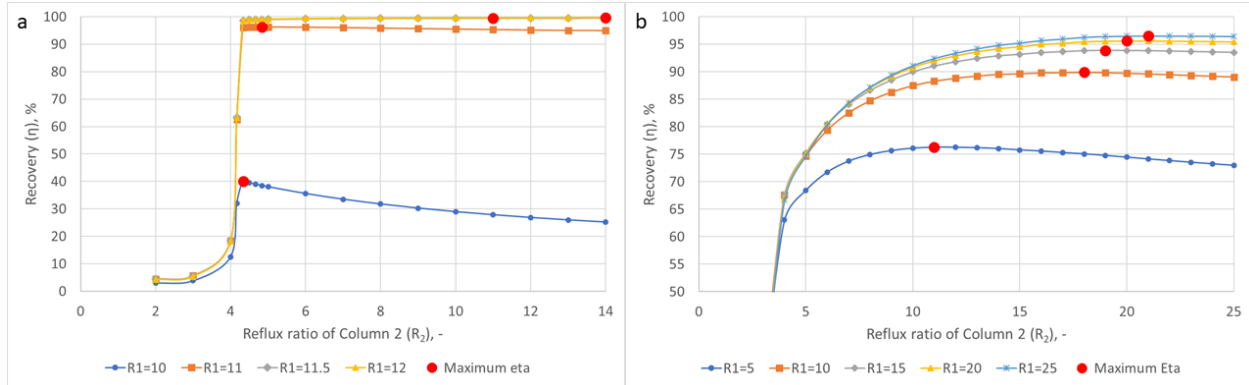

Fig. 1 Recovery of Policy IIb for Case 2 of Mixture 1 as a function of R_2 at $N_1=15$ (a) and Mixture 2 at $N_1=10$ (b)

Table 3. Results for Policy IIb for Mixture 2.

	Case 1				Case 2				Case 3			
N_1	R_1	R_2	Cap^{IIb}, t/day	η^{IIb}, %	R_1	R_2	Cap^{IIb}, t/day	η^{IIb}, %	R_1	R_2	Cap^{IIb}, t/day	η^{IIa}, %
7	-	-	-	-	-	-	-	-	67.0	14.3	2.688	94.17
8	-	-	-	-	39.0	13.7	1.243	93.73	31.0	15.1	2.947	"
9	23.0	11.3	1.014	93.00	25.0	13.7	1.384	"	22.0	15.0	3.264	"
10	17.0	11.3	1.111	"	20.0	13.2	1.525	"	18.0	14.7	3.561	"
12	13.0	10.9	1.271	"	15.0	12.8	1.751	"	15.0	14.0	4.023	"
14	12.0	10.5	1.378	"	14.0	12.4	1.886	"	15.0	13.0	4.329	"
16	12.0	9.88	1.468	"	13.0	12.3	2.012	"	14.0	13.1	4.566	"
18	10.0	10.8	1.556	"	12.0	12.7	2.111	"	14.0	12.7	4.768	"
20	11.0	9.67	1.628	"	12.0	12.3	2.212	"	13.0	13.2	4.969	"
22	10.0	10.0	1.722	"	12.0	12.0	2.313	"	13.0	13.0	5.212	"

Table 4. Results of minimising N_1 for Mixture 2.

	N_1	R_1	R_2	$x_{W,B}$	η^{IIa}, %	Cap^{IIa}, t/day
Case 1	12	6.60	15.1	0.8578	93.54	0.908
Case 2	9	9.37	18.4	0.7561	95.01	1.282
Case 3	4	10.6	13.0	0.7074	95.14	2.623

For given N_1 values ($N_1=5$ and $N_1=22$), Cap^{IIa} was maximised for both mixtures and all feed compositions. The results of this optimisation for Case 2 are shown in Fig. 2, along with the maximal Cap^{IIb} at different N_1 values for Case 2 for both mixtures. (Cap^I is shown as a horizontal line.)

In all cases, Cap^{IIb} was higher than Cap^I because more feed is processed by Policy IIb. However, Cap^{IIb} is still lower than Cap^{IIa} (Fig. 2b) for Mixture 2. Although the amount of feed processed is higher by Policy IIb than by Policy IIa, as two charges are processed in parallel, the total number of trays is higher in the latter case for processing only one

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charge in the two columns. This results in a lower reflux ratio, thus lower distillation time and higher Cap^{IIa}. By using broader intervals of the optimisation variables, higher Cap^{IIa} values were reached for Cases 2 and 3 than by Nemeth et al. (2022). Optimal results for $N_1=5$ are shown in Table 5. (Results of Policy I were considered as 100%).
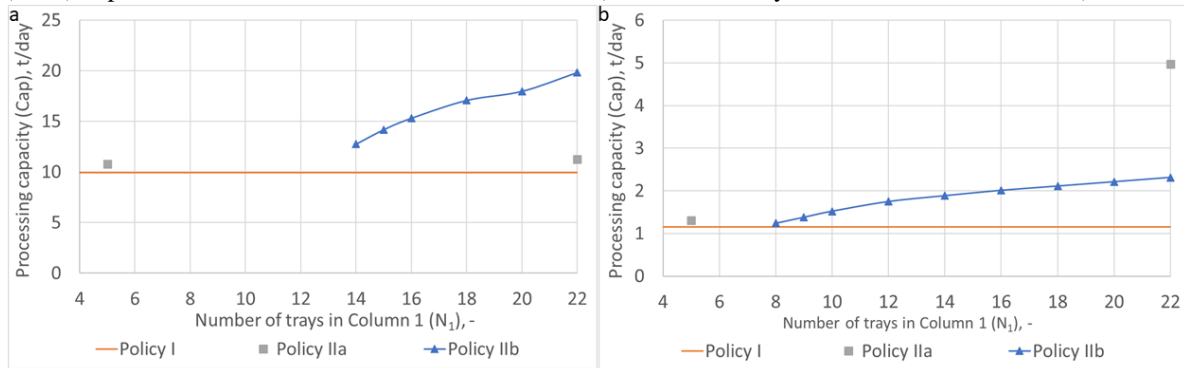

Fig. 2 Cap of the different operational policies for Case 2 of Mixture 1 (a) and Mixture 2 (b)

Table 5. Optimal results of Policy IIa for Mixture 2 ($N_1=5$).

	Case 1	Case 2	Case 3
Operation parameters	$R_1=10.9$ $\bar{x}^1_{D,B}=0.029$ $R_2=12.2$	$R_1=12.2$ $x^1_{W,B}=0.526$ $R_2=12.0$	$R_1=14.9$ $x^1_{W,B}=0.816$ $R_2=13.6$
$n \cdot t_1 / t_2$, h	187.50 / 236.71	28.10 / 94.40	16.30 / 44.30
η^{IIa}, %	93.01 (100 %)	93.82 (100 %)	94.18 (100 %)
Cap^{IIa}, t/day	1.03 (119.4 %)	1.3 (113 %)	2.8 (108 %)

For Mixture 1 (Fig. 2a), Cap^{IIb} is the highest, at the N_1 values higher than the minimum feasible ($N_1=14$). For Case 2 at $N_1=22$, Cap^{IIa} is by 43% lower than Cap^{IIb}. However, by Policy IIa, higher Cap can be achieved than by Policy I at lower N_1 values, where Policy IIb is infeasible. Case 1 is an exception, where Policy IIa is better than Policy I above $N_1=14$, only (Fig. 3). This is caused by the high dead time of Column1 and the high number of charges that must be processed in Column1 (a higher N_1 is required to achieve lower distillation time, which can compensate the high dead time). Consequently, N_1 has a higher effect on Cap^{IIa} for Case 1 than for Cases 2 and 3 due to the higher R_1.

The capacity results can be summarized in a map of x_F-N_1 (Fig. 4) for both mixtures. The maximisation of Cap^{IIa} was not performed for Case 3 of Mixture 1 (Fig. 4a) as Cap^{IIa} was independent of N_1 above $x_F=0.5$ due to the very low R_1.
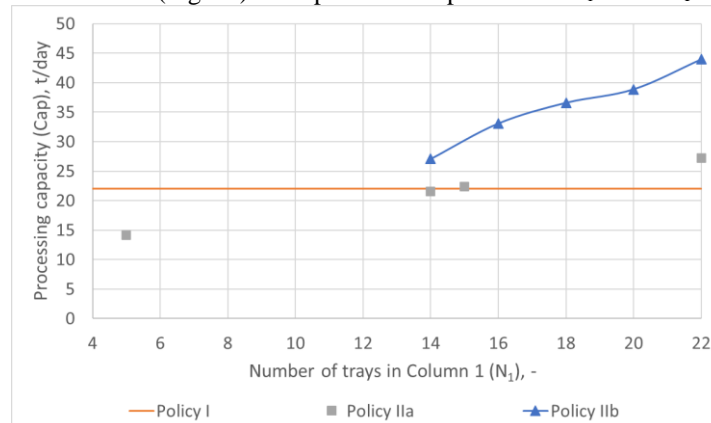

Fig. 3 Cap of the different policies for Case 1 of Mixture 1

For both mixtures, Policy I is the best at low N_1 because Policy IIb is not feasible and Cap^{IIa} is lower than Cap^I (Zone A). In Zone B, Cap^{IIa} is the highest: N_1 is high enough for prefractionation for Policy IIa but lower than its feasible minimum for Policy IIb. In Zone C, N_1 is already higher than the minimum for Policy IIb. For Mixture 2, Cap^{IIa} is still

the highest in this zone, although Cap^{IIb} is also higher than Cap^I. However, Cap^{IIb} is the highest in Zone C for Mixture 1. For Mixture 1, in Zone D, Cap^{IIb} is the highest, similarly to Zone C (for Mixture 1), while Cap^{IIa} is lower than Cap^I.
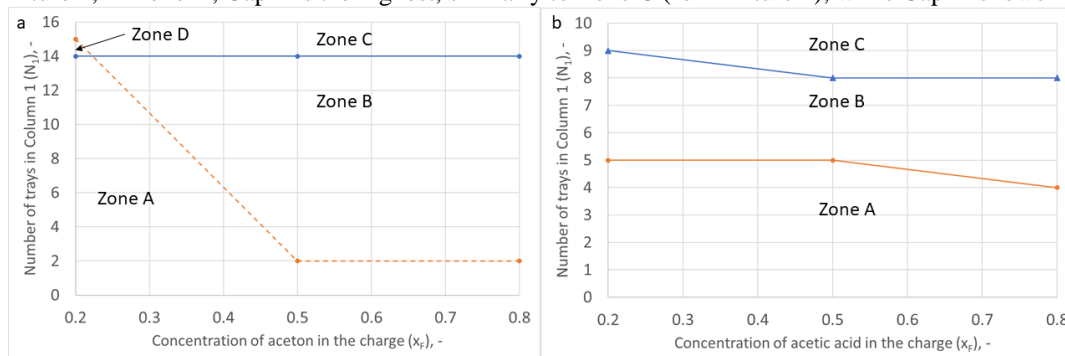

Fig. 4 Map of x_F-N_1 for all policies for Mixture 1 (a) and Mixture 2 (b)

Conclusions

The effect of the size of the smaller column (Column1) in a two-column system on processing capacity (Cap) was studied for separating acetone-water (Mixture 1) and water-acetic acid (Mixture 2) for three different feed compositions: 20 (Case 1), 50 (Case 2) and 80 mass% (Case 3) of organic component. The purity requirement is 99.7 % for acetone (as distillate) and acetic acid (as residue). Three operational policies were studied: a single-column process (Policy I); two two-column processes, where the two columns are operated sequentially (Policy IIa) or in parallel independently (Policy IIb). The two-column policies were optimised by maximising Cap. The optimisation variables were the reflux ratios of both columns (R_1 and R_2) and for Policy IIa the stopping criterion of Column1: the concentration of the organic component in the accumulated distillate (for Mixture 1) or in the residue (for Mixture 2). It was shown for Policy IIb that at given N_1 and R_1 values, there is a maximum in η^{IIb} as the function of R_2 for both mixtures. The optimised Cap^{IIb} was higher than Cap^I for both mixtures and for all feed compositions studied above a minimum N_1 (number of trays in Column1), which was 14 for Mixture 1 (for all three cases), while 8 (Cases 2 and 3) or 9 (Case 1) for Mixture 2. The difference between Cap^{IIb} and Cap^I increased on the increase of N_1. However, Cap^{IIa} was even higher than Cap^{IIb} for Mixture 2: it was by 40-110 % higher at N_1=22. For Mixture 1, Cap^{IIa} was lower (by 43 % at N_1=22) than Cap^{IIb}. The minimum N_1 was higher for Policy IIa than for Policy IIb where only partial separation is required in Column1. A map of N_1 was drawn as the function of charge composition (x_F), where different zones were distinguished based on the order of Cap values.

The highest increase in Cap (compared to Policy I) can be achieved by using Policy IIb for Mixture 1, while Policy IIa for Mixture 2. In general, Policy IIb is not feasible below a minimum N_1, contrary to Policy IIa, where only prefractionation is performed in Column1.

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