



Short Communication

Pressure-Driven Batch Distillation Optimal Control for Ethanol-Water Separation

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Abstract: Ethanol-water separation by pressure-swing batch distillation in a double-batch rectifier system is a process under investigation. In this work, a complete global solution to the optimal control problem in the form of a sequential synthesis of controlled trajectories is derived. During this study, the optimal reflux strategy through cyclic operation was extended to the separation of a second non-ideal minimum boiling azeotrope of industrial importance. The optimal cyclic reflux ratio operating policy, brought significant reductions in time duration, going up to even 50%. Consequently even more significant reductions in energy expenditure, since the derived discontinuous energy function tends to show cyclic “behavior” as well. Lastly, the proposed strategy provides the possibility to highly recover the desired product even within a single period, and finally maybe even the most importantly, flexibility in operating different units within the time frame.

Keywords: pressure-swing, batch distillation, ethanol-water, optimal control, direct method, Pontryagin’s Maximum Principle

1. Introduction

Dussel and Stichlmair,¹ investigated the separation of different binary mixtures by extractive distillation: among them, ethanol-water separation by ethylene glycol, whereas, a hybrid process proposed replacing one distillation step by the absorption operation, at the top of the column high boiler is fed in the first step in order to absorb the water from the vapor arising from the water-rich mixture place in the decanter, enriched in glycol, whereas, in the second step, water and ethylene glycol are rectified, presented the course of distillate concentrations and boundaries on ternary diagrams. In the same year, Watson et al.,² detailed the industrial operating cyclic method to separate a quaternary mixture of methanol-cyclohexane-ethanol-water: providing the possibility to concentrate binary heteroazeotrope of methanol-cyclohexane at very high reflux ratios: two periods of “drainage” interconnected by the total reflux period were necessary to concentrate the maximum of the heteroazeotrope, which is to disappear in the next step of finite reflux. This is possible only due to the fact that the initial mixture is poor in methanol, afterwards, even four infinite reflux periods intertwined with periods of drainage, were necessary to concentrate the binary homoazeotrope of ethanol-water, finally to produce ethanol, started after 10.3 h, and last till 15 h, ie. 4.7 h in total. It is worth noting, that “periods of drainage”

were gradually increased, but one should note that the second drainage period's total duration is longer than the first by 46.3088%, consecutively, the third drainage period is longer than the second by 31.5468%, and, the fourth longer than the third for 24.0698%. A particular step duration decrease (%), (third-second) vs (second-first) is 14.7620%, but, (forth-third) vs (third-second) only 7.4770%. Furlonge,³ detailed the comparison of breaking the binary homoazeotrope of ethanol-water, in regular extractive batch column vs non-conventional configurations (middle vessel), according to the author unlike the regular column the operating policy involves a relatively long initial total reflux period, whereas ethanol composition in distillate vs time remained well above the azeotropic point for more than 30% of the total time, thanks to the high solvent feed rate. Tavan and Hosseini,⁴ investigated the continuous reactive distillation process for the system containing even 4 azeotropes, one of them heterogeneous, two feeds are introduced into the column, ethanol/water, and ethane: interesting to note is that the total duty (en.) via stages, for the reflux ratio ranged [3-4], keeps fluctuating in the interval (13,000-17,000 [kW]), also, these functions are different with the respect to the azeotropic feed inlet stage - reflux ratio interval ranged [0.5-2.5], the total duty is ranged in the even broader interval (2,000-14,000 [kW]), in particular reboiler duty up to 10,000 [kW]. Esteban-Decloux et al.,⁵ tempted to propose the best strategy for the separation of the particular complex industrial mixture issued from spirit plants: important was to "track" four components present initially in traces, to observe that D-limonene reached the maximum during the production of approx. 0.2 or up to 20 times increase, but, an even greater jump could be stated for linalool oxide, as approx for a hundred times. Iqbal and Ahmad,⁶ detailed the professional software designed (HYSYS) optimal operation results, whereas, defined the optimal reflux ratio for HP (column) as greater than for LP (column) for approx. 13.5%, whereas, the ratio of distillate to feed flowrate (like "liquid ratio for batch distillation"), greater for even 43.75%. Heras-Heras-Cervantes et al.,⁷ a special case of fuzzy-logic control is introduced, ie. Takagi-Sugeno controller, significantly, among the three premise variables the reflux valve opening percentage is placed as recognized to influence all the states of the system together with light component compositions in condenser/reboiler. Zhu et al.⁸ studied the control of the pressure swing distillation process of benzene/isopropanol/water separation, developed four improved control structures, and achieved good control results, providing a reference for industrial development. Aqar et al.⁹ proposed a semi-batch reactive distillation column in which additional methanol would be fed in a continuous fashion at the bottom of the column, allowing the synthesis of methyl palmitate to continue, shown, in terms of product quality, the gap between reflux ratios on double-period intervals drops with the product specification constraint imposed. Diwekar et al.¹⁰ analyzed the separation wall column of batch distillation in detail and established the operating procedure and mathematical model for the separation wall stripping column. The performance of this new configuration is compared with that of the conventional intermittent rectifiers and intermediate container columns. The results show that the performance of the new intermittent separator column is significantly better than that of the rectifier column or intermediate cylinder column. Treating the mixture of hydrocarbons, they showed in two study cases for a high concentration of the first component, (>95%), for double period reflux ratios to a difference of 1.54% between, it seems that the gap between the first and the second-period reflux ratio, increase for more than 2.5 times. Most recently, Putri et al.,¹¹ showed from normalized product concentration output from a particular neural network approach, observed the "quasi zero-bang" sequence in the very beginning, fluctuating further around a mean value (of 0.8), subsequently dropped for "quasi-zero" almost instantaneously to switch to the instantaneous increase in the next moment, however, the combined control made up from neural network and Kalman filter, ("smoothened") resulted in lowering the "quasi-bang-zero" sequence (from previous maximum 1 or 0.99) to less than 0.95. Mahida et al.¹² studied a pressure swing distillation process for separating ternary azeotropic mixtures of acidic aqueous solutions, having examined three compositions of feed mixture: the first component in excess (ie. formic acid, 70%) showed the greatest total energy consumed, ie. almost twice more compared to the second case (ie. water, 60%), and even more than 30% compared to the third case (acetic acid, 60%). Desikan et al.¹³ designed a fast mid-reactor batch rectification column for the three-way separation of ethanol/propanol/butanol mixtures using the steam compression method. Also, compared cases for the configuration of middle vessel batch distillation column, whereas, the column functions in a "classical way" and/or "decoupled" in terms of pressure, to conclude: in the first case, starting from the atmospheric pressure, rectifier/stripper, resp., arrives to almost 3.8/2.75 ([bar]), resp., whereas, for the latter case, starting pressures are different for rectifier/stripper, resp., ie 1 and 4.2 ([bar]), respectively, and higher pressures set at, 4.2 and 5.1 ([bar]), respectively. Zhang et al.,¹⁴ studied the enhanced configuration energy saving of benzene/isobutanol binary azeotropic pressure swing distillation, and, came up with the conclusion for the discharge pressure difference gap ([atm]), to coincide with the gap

in the phase transition temperature ([K]), however, the gap in pre-treatment temperature was reduced compared to the later (halved for the examined values of higher/lower pressures (8.2/7.2)). Zhang et al.¹⁵ studied the pressure swing distillation separation of butanone/isopropanol/n-heptane mixed system, implemented the full/partial/non-thermal integration process, resp., whereas, the first/second/third column set at 1.013/3.546/0.334 ([Mpa]), resp., to combine the integral of squared value error of the invented control structure with the dynamic response, finally to compare the performance of the control structure: one can perceive that difference between partial and full heat integration case, has never been found lower than the value of 0.02. Yu et al.¹⁶ designed Three-column Pressure-Swing Distillation (TPSD) and a novel Azeotropic combination Pressure-Swing Distillation (APSD) for the separation of n-propanol/water/tetrahydrofuran ternary mixtures. Interestingly, the authors found for the novel configuration, that after the use of heat integration and steam recompression heat pump technology, the order of the pressures being for the first/second column, resp., 7 and 1 ([atm]), resp., the pressure of the third column brings (the most) benefits if set at 2, but rises again if pressure increase from 5 to 6 (whereas for the gap between it drops).

In this study, the optimal control for the operation of pressure-swing batch distillation for ethanol production in an open-mode double rectifier column configuration is researched. Having considered the safety constraints, ie. avoidance of overpressuring and/or overheating at any moment of the operation, the cyclic reflux ratio strategy is implemented, and show significant reductions in time, since only within a single reflux period, both high desired purities and recoveries achieved. From here, the discontinuous energy function is partially derived, showing cycling tendency, which gives the reason to believe that the part of the process can already be controlled by the “energy variable”. However, it is a must to upgrade the optimal controller to the “combined” one so as to give the best approximation of the latter function on the entire process. Last but not least, the proposed strategy; permits flexibility in the employability of different operating units in time, which only goes in favor of its future applications in the industrial environment, especially food, and beverages, due to the fact that it would permit “the ease” in both starting/restarting production (serial) batches.

2. Materials and methods

In this short communication, the author aims to expand the optimal control strategies to the separation of the other mixtures with minimum boiling azeotrope, by pressure-swing batch distillation. In particular, a case of ethanol-water is taken into examination, due to its significance in the beverage and food industry, as well as the possibility to expand further the study to the more complex mixtures of alcohols. The optimal control problem can be described as follows: For a given pressure-swing batch distillation configuration (N -total number of trays, P -working pressure), batch composition, the distillation task, and overall time horizon (t_f), determine the optimal reflux ratio so as to maximize the distillate, subject to any constraints (model equations, bounds on the optimization variables). The dynamic model equations to be solved are already known from the work of Stojkovic,¹⁷ and handling the thermodynamics constraints lying behind the effects risen from elevated pressure conditions, from the work of Stojkovic.¹⁸

The pressure-swing process scheme presented in Figure 1, assumes “one-pass” through the column at the time, in the other words it is assumed that columns work consecutively (“two-step” process), in double batch rectifier configuration. But, the maximum achievable distillate concentration at lower pressure $P_1 = 1$ atm is 95.5% of ethanol, but, at $P_2 = 10$ atm only 82% of water. Moreover, in Tables 1-3, the parameters corresponding to the thermodynamics model Wilson, and, originated from Gmehling et al.,¹⁹ are tabulated, and the choice of the thermodynamic model coincides with the work of the authors Binous et al.²⁰

In Figure 2, the vapor-liquid equilibria is presented for a mixture of ethanol-water at different pressures, whereas the Wilson model applied is verified by ChemCad professional simulator.

Last but not least, the thermodynamics model of Wilson, incorporated in optimal control solver, is compared against, the more complex thermodynamics model named The Nonrandom Two Liquid (NRTL) (Table 1). As a consequence, very close optimal solutions are obtained, both in terms of the gain and control pattern, therefore, the choice is made for the previously described thermodynamics model of Wilson, due to the savings in computational time.

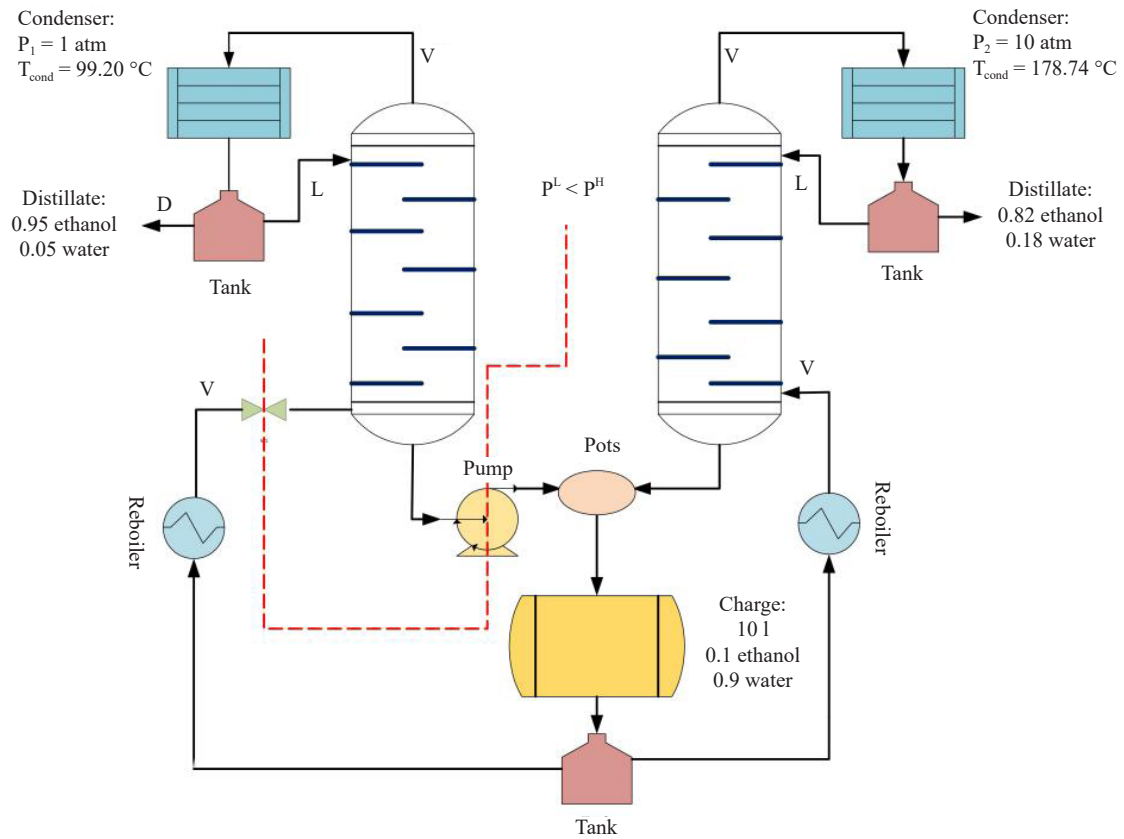


Figure 1. A regular double-rectifier scheme for pressure-swing batch distillation

Table 1. Binary interaction parameters for Wilson model

Component <i>i</i>	Component <i>j</i>	Wilson			NRTL		
		A_{ij} [cal/mol]	A_{ji} [cal/mol]	A_{ij} [cal/mol]	A_{ji} [cal/mol]	B_{ij} [cal/mol]	B_{ji} [cal/mol]
Ethanol	Water	-2.5035	-0.0503	-0.80	3.46	246.18	-568.08

Table 2. Antoine parameters for ethanol from Dechema

<i>n</i> °	A	B	C	T_{min} , [C°]	T_{max} , [C°]
1.	8.20417	1642.89	230.3	-57	80
2.	7.68117	1332.04	199.2	77	243

Table 3. Antoine parameters for water from Dechema

<i>n</i> °	A	B	C	T_{min} , [C°]	T_{max} , [C°]
1.	8.07131	1730.63	233.426	1	100
2.	8.14019	1810.94	244.485	99	374

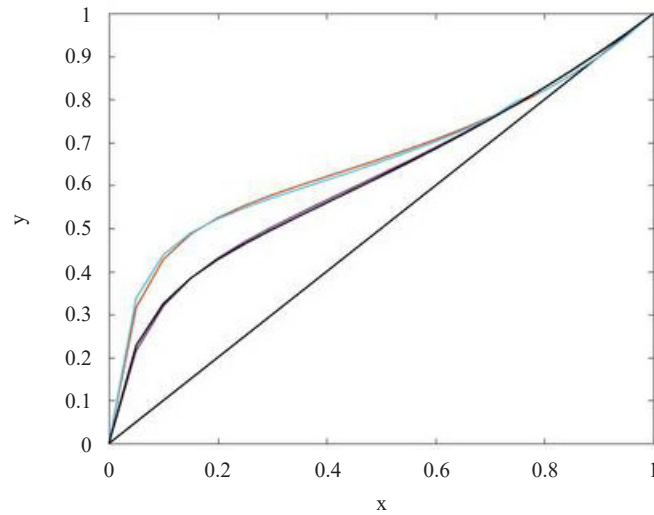


Figure 2. Vapor-liquid equilibrium curves for ethanol-water mixture at different pressures: light colors - $P_1 = 1$ atm, dark colors: $P_2 = 10$ atm

3. Results and discussion

The working conditions and predefined parameters are tabulated in Table 4, whereas concentrations are expressed from acetone, and LP/HP refers to low/high-pressure column, respectively. In the next table, Table 5, the optimal results obtained by control solver BOCOP, are tabulated, for the total number of stages, N , for LP/HP, rectifier column, respectively.

Table 4. Working conditions

Parameter	P [atm]	U_N^0 [l]	U_i^0 [l]	V [mol/h]	t_f [h]	x_N^0	y^*
Predefined/initial value	LP:1	10	0.1	4	1.6	0.1	LP: 1.0.85 or 2.0.95
	HP:10						HP: 0.82

** 1. purity set at 95%, 2. purity set at 85%

Table 5. The optimal results by BOCOP solver

Product recovered	N	$U_0(t_f)$	Recovery rate	Discr. scheme, nb. of points
Ethanol	21	1. 1.8340 2. 1.1138	1. 66.26% (single period) against 93.11% (quadruple), 2. 47.19% (single period) against 90.29% (double period)	Gauss, 2200
Water	10	0.748	1. 72.20% (quadruple) 76.46% (5 periods)	Gauss, 2200

(*) 1. 95%, 2. 85%

Discussion for the interesting findings for the desired product purity: first strategy seeking 94%, yielded more than 90% only after a quadruple period of time, 4 consecutive runs, ie. final time of 3.2 [h], whereas the second proposed

strategy where only 85% of purity seeking, yielded its maximal only after a double period (1.6 [h]).

The HP run, for the first strategy proposed, could last for effectively 0.2 [h], whereas all the control trajectory is constituted from two consecutive bang arcs, concatenated by a very short singular arc, lasting not more than $3E-3$ [h]: it seems that for this short interval, the instantaneous switching to the at least two extremely short piques reaching their minimums in the period of 1.8-1.4 and maximums at approx 2.2-2.4. The second bang arc is of about double more duration compared to the very first one, whereas the final zero arcs appears for not more than 5% of the total batch time.

Moreover, then the HP step should be repeated for even four times, ie. “quadruple period” in order to achieve more than 70% of the recovery, and even five periods necessary to achieve more than 75%, for the desired purity of 82%, ie. slightly more than an azeotropic point.

Otherwise if again, the relaxation on the purity constraint is introduced, 95% for the first case, but, 85% for the second case, then it is suggested for the minimum batch time, to be 1 h, (LP + HP), whereas for the satisfactory recovery, It is suggested for the minimum batch time of the two-step process, (LP + HP), 1) in the case of the first proposed strategy: $(3.2 + 0.2 \times 4 = 4.0$ [h]) for the second proposed strategy: $(1.6 + 0.2 \times 4 = 2.4$ [h]). It is to examine, which strategy would bring more in terms of overall performance, (ie. yield, productivity, desired purity, and most importantly energy requirement). As longer running the operation, the cost of power utilities will be higher heating, and electric energy, assuming they are constant with time. Otherwise, a cyclic strategy should be additionally proposed for a particular case.

The optimal control policies for the azeotropic mixture of ethanol-water are depicted in Figure 3, as the LP and HP step, respectively.

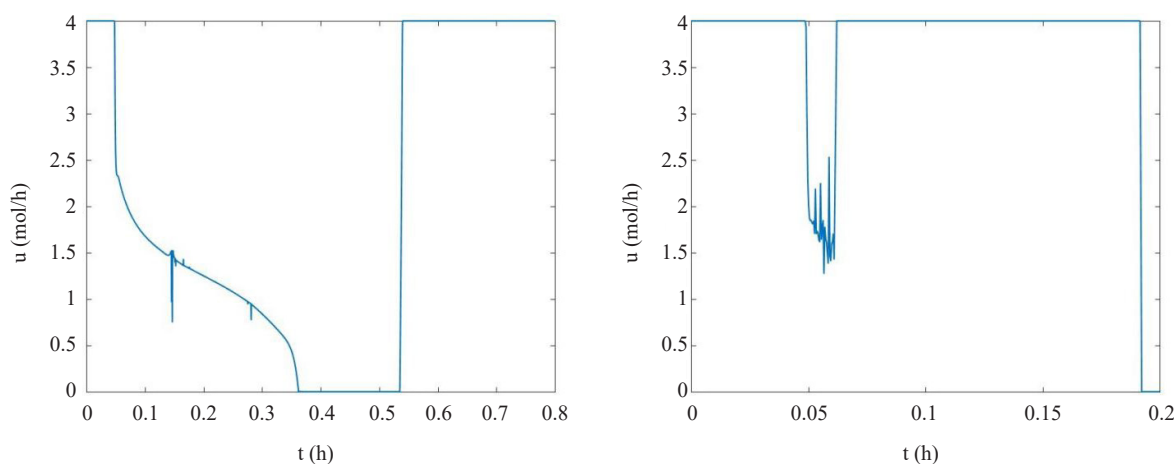


Figure 3. The optimal distillate rate for $u(t)$ for the: 1) left: LP step, 2) right: HP step

Moreover, it is noted, during the elevated pressure part of the process: the purified water (91.60%) is obtained in the reboiler and withdrawn, for the quadruple period duration. Next, the recovery rate is 88.47% of water calculated per single period. This means, at this point, there is a choice to make between ethanol/water, resp., purification, in accumulator/reboiler.

Despite, the fact, that the property of distilling under the pressure change, is exploited at maximum, the recovery rate for the ethanol after the LP step is 72.20%, and, still 24.90% more than the best case study reported by Alvarez et al.²¹ for the particular mixture separation in the batch rectifier. And, consequently the recovery rate for ethanol from the more complex mixtures, such as the example of bitter orange authored by Esteban-Decloux et al.,⁵ as expected, is even less than 20%. Lara-Montaña et al.,²² grace to the concentration of the other components (ie. methanol, ethyl-acetate, 1-propanol, isobutanol), the process started to work without fluctuations and disturbances, after 360 mins. Additionally, Esteban-Decloux et al.,²³ performed malt whiskey distillation with a stupfler column, in order to separate a very complex aromatic mixture of 30 compounds: the simulation resulted in the same percentage of ethanol in the residue, however,

only at the expense of the time horizon longitude - more than 340 min of process running, with the clear difference with the LP step proposed in this work, where the first/second strategy proposed (Table 5), resulted in not more than 320/160 (min), resp. In conclusion, the proposed operation is recommended as a two-step ethanol production and purification.

Moreover, in Figure 4, the output for the temperature evolutions along with the optimal solution is presented. Above, the temperatures on all the stages of the column for the rectifier (LP step) are depicted. From here, the interval of temperature increases along with the first rectifier, going from the bottom to the top (batch to accumulator), following the optimal trend from the minimal temperature of 78.32 to the maximal one at 100.09 °C, at the specified working pressure of $P_1 = 1$ atm. As previously stated, verify that the ethanol is recovered in the product tank. Below, the optimal temperature evolutions are presented for the HP step of the process, ie. the second rectifier, at the specified working pressure of $P_2 = 10$ atm, temperature rise, in the same sense, following the trend within the temperature interval from the minimal temperature of 151.55 °C to the maximal temperature of 179.78 °C. Hereby, previously written goes in favor of the fact that ethanol is recovered (again) in the first rectifier column product tank. As a consequence, it is verified, that the ultimate goal of the designed two-step process, ie. consecutive production of ethanol and water, is achieved.

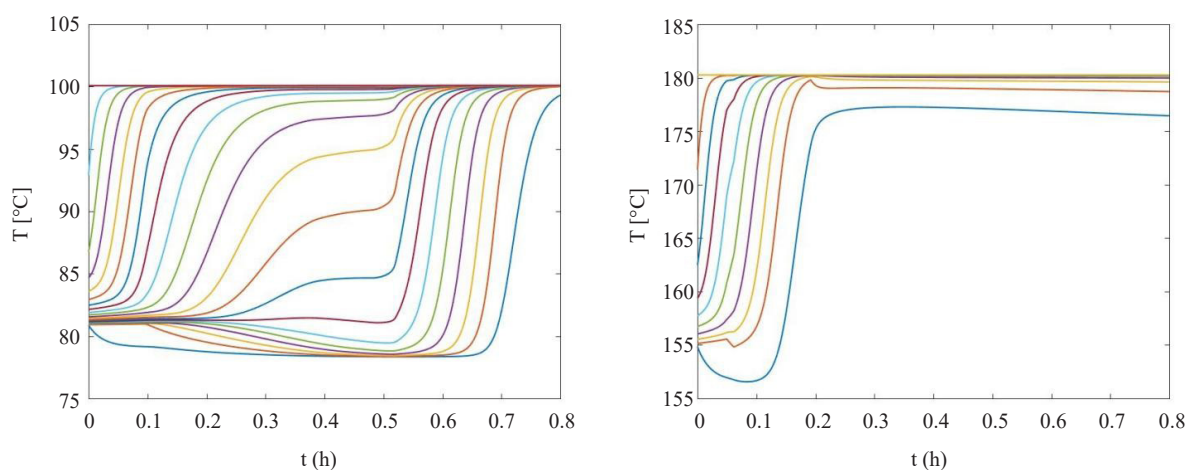


Figure 4. The optimal temperature evolution for the: 1) left: LP step, 2) right: HP step

3.1 The influence of vapor boil-up

The influence of vapor boil-up on the structure of the optimal control pattern is observed, as well. In Figure 5, on the left/right, respectively, the optimal control patterns for purity constraint set at 85%/95%, respectively, are plotted for varied values of vapor boil-up. One can observe that, with the increase of the vapor boil-up parameter, the structure becomes more complex: firstly, the singular arcs are to appear more and more frequently, ie. from a total number of 1 to 3, for both the desired purities; moreover, the unique zero arcs tends to last more, as it triples for an increase of only 25% and becomes even 5 times longer for an increase of 2.5 times in vapor boil-up; the last mentioned change, invokes also the introduction of the additional bang arc for all desired purities.

In Figure 6, finally, the elevated pressure part of the process, (ie. $P_2 = 10$ atm), could only be approximately simulated by the professional simulator, ChemCad, but it gives a clear idea of the “discontinuous energy function”, almost ie. quasi-zero-bang policy, since it is recommended to lower the heat at the minimal values close to zero for almost 25% of the total duration of the process. Moreover, at the subsequent shortage interval the heating should be even double lowered, before the final period of “quasi-bang” where recommended instantaneously to rise the heating to its maximum and keep it to approximately 95% before the end of the process, since here a very low stepwise increase proposed or less than 3% of the maximum previously achieved. The LP step, however, due to the complexity of its optimal control structure, should be assessed by the “combined control” approach, ie. the classical control tools should be designed in order to give “the best” approximation on its “discontinuous energy function”. The cyclic reflux ratio

optimal control strategy also, provides with temperature difference control, which implies that it “copes” with the feed disturbances keeping the variations in product compositions within the satisfactory limits.

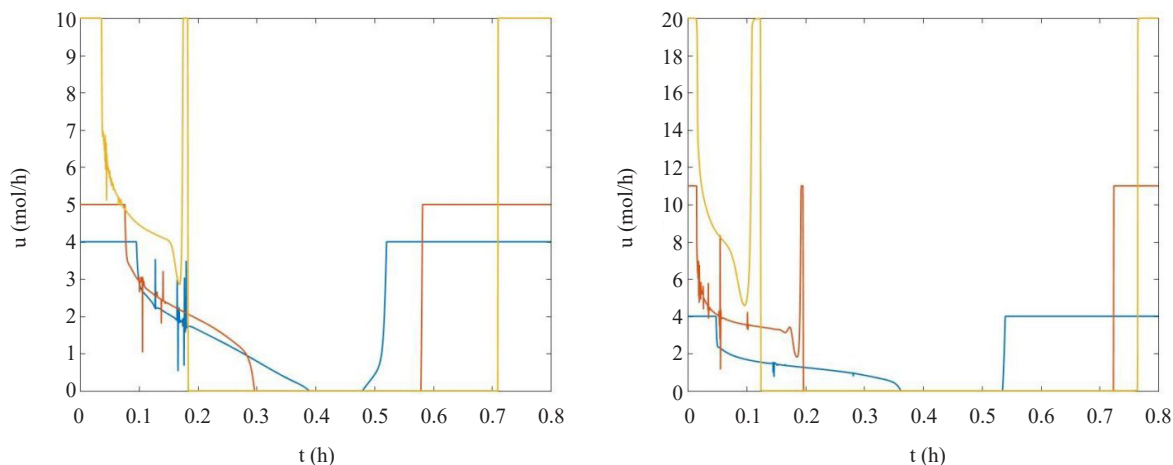


Figure 5. The influence of vapor boil-up variation on the control structure: 1) left: purity set at 85%, 2) right: purity set at 95%

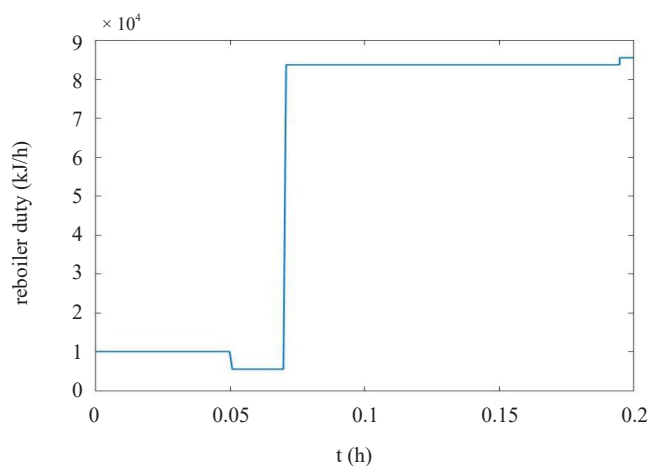


Figure 6. Discontinuous reboiler energy function

4. Conclusions

The use of optimal cyclic reflux policy has solved the problem of optimizing a multi-cut pressure-swing batch distillation campaign using variable reflux ratios with the ‘recycling’ of cuts at predetermined times, whereas, the gap between compositions in reboiler and recycled cuts are minimized. The additional analysis of process constraints their possible influence on the duration of the process. Last but not least, the author reported the details on productivity, as well as significant achievements in time savings depending on process conditions and constraints and ranging from [6-50 (%)], and the possibility to achieve a high recovery rate even within a single time period of the process. Also, the possibility to redesign the process according to the needs of the industrial environment. The cyclic reflux ratio optimal control strategy also provides temperature difference control, which implies that it “copes” with the feed disturbances keeping the variations in product compositions within satisfactory limits. Even more, the scheme could be accompanied by the means of the classical control tools, so as to give the unique optimal discontinuous function for reboiler energy

for the entire process, which can be done by implementing the specially designed composition-temperature cascade structure. Grace to this, future studies should focus on more complex mixtures originating from industrial waste, especially those treated after the effluents were trashed from the beverage and food industry, containing complex compounds following the alcohols such as esters, aldehydes, carboxylic acids, etc. Consequently, the primary goal should be direct implementation into the industrial environment after the pilot-plant checking for predesigned control platform structures.

Conflict of interest

I declare there is no conflict of interest, and the entire responsibility for the published results and data is up to the author.

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