Batch distillation of binary mixtures: preliminary analysis of optimal control

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Abstract: The present work concerns the distillation of a binary non-ideal zeotropic mixture in an N-stage batch column under total condensation and constant vapour flow rate assumptions. The aim is to maximize the final product of desired purity using the reflux as a control parameter. We discuss certain methodological aspects of the numerical resolution, and present the first results obtained by the application of the direct method based on a full discretization of the optimal control problem.

1. INTRODUCTION

Distillation is the technique of separation of the components of a liquid mixture using the principle of relative volatility and different boiling temperatures of the components. It is the most widely used separation technique in chemical industry. In simple distillation, the liquid mixture is kept boiling in a still under thermodynamic equilibrium conditions. The vapour produced along the process is richer in light components than the liquid, it is evacuated from the still by condensation allowing to obtain the *distillate* rich in desired light component. The process can be intensified by reversing the part of the distillate back to the still, i.e., by using the *reflux*, which makes the transfer of lighter components to the vapour phase more efficient.

In the industrial context the process is carried out in continuous, batch, or semi-batch distillation mode in distillation columns. The batch distillation is used when a small amount of product of high quality is required, or the flexibility of the production is needed. The most commonly used control parameters are the reflux policy and the reboiler heat duty.

The present paper focuses on the distillation of homogeneous non-ideal binary mixtures. Different strategies were proposed to optimize this process by choosing the best step-wise constant reflux ratio (Farhat et al., 1991), by selecting an optimized repeating sequence of zero and total reflux periods (cyclic operation, Sorensen (1999)), etc. Optimal control of batch distillation of binary mixtures was analysed in minimum time, maximum distillate or maximum economical profit formulations (Mujtaba, 2004). In particular in Converse, Gross (1963) and then in Diwekar (1992), Kim et al. (2001) Potryagin's maximum principle

(PMP) was applied to solve the maximum distillate problem. The authors considered the dynamics on the internal part of the column in almost steady-state approximation. The singular type control policy found by the authors is now considered as the reference optimal solution of the problem. In our opinion certain optimal solutions were neglected in this studies.

We consider in this paper the problem of maximisation of production of the distillate of prescribed purity over a fixed interval of time by controlling the distillate rate. In Section 2 we analyse the mathematical model of the problem. We show that the purity constraint can be transformed into an additional state of the system. Then the maximum distillate problem can be formulated as a standard affine control problem with bounded control and simple state constraints. According to PMP, optimal controls can be both of bang and singular types. In Section 3 we present the results of the numerical resolution for three binary mixtures in batch columns with different number of plates. The presented solutions were obtained by the direct method implemented in the BOCOP solver (Bonnans et al. (2014)). We show that the optimal control structure depends on the difficulty of the separation task and on the column configuration. In certain cases the optimal control is almost of bang-bang type, which is very close to the cyclic operation mode reported by other authors. We test one of these optimal scenarios in dynamical simulation using ProSim software (ProSim, 2000), and found a significant amelioration with respect to the conventional optimal batch operation.

2. OPTIMAL CONTROL OF BATCH DISTILLATION

2.1 Dynamical model of batch distillation

The simple batch mode configuration considered in this paper is shown in Fig.1. It consist of a *reboiler*, a number of intermediate *plates* (or *trays*), used to bring the vapour and liquid phases into contact to enhance the mass transfer, a *condenser*, and an *accumulator tank* containing the distillate. For a given mixture, in order to assure the feasibility of the production of the distillate of a specified quality, the number of plates (including the reboiler and the condenser) must be greater than N_{min}. This number can be computed in relation to the initial composition and the desired purity of final product via the standard McCabe – Thiele method (Doherty, Malone (2001)). The greater is the difference in relative volatility of the components of the mixture, the easier is the *separation task*.

To describe the thermodynamic equilibrium between the vapour and the liquid phases we use the generalized constant relative volatility model due to Gmehling (Doherty, Malone (2001)):

$$y(x) = \frac{ax}{1 + (a-1)x} + bx(1-b). \tag{1}$$

We consider a batch distillation column under assumption of equimolar overflow and total condensation. The vapour hold-up as well as the pressure drop along the column are neglected, the liquid and the vapour phases are supposed to be perfectly mixed on the plates. These assumptions imply that the values U_1° and U_i°, $i = 2, \ldots, N-1$, representing the molar hold-ups for condenser and internal plates are constant, so the complete state space of the N-plates column can be defined as

$$X_0 = \{q = (x_0, x_1, \ldots, x_N, U_0, U_N) : x_i \in [0,1] \, \forall i\}.$$

Here $x_0, x_1, \ldots, x_N$ are molar concentrations of the light component in the liquid phase in accumulator, condenser, intermediate plates and the reboiler correspondingly, while U_0 and U_N are the liquid hold-up in the accumulator tank and in the reboiler. Below we mark with the superscript "°" the initial values of these variables. The mass balances over the column yield the system of differential equations (Mujtaba (2004)):

$$\frac{dx_0}{dt} = \frac{u}{U_0}(x_1 - x_0), \qquad \frac{dx_1}{dt} = \frac{V}{U_1^\circ}(y_2 - x_1),$$

$$\frac{dx_i}{dt} = \frac{1}{U_i^\circ}(L(x_{i-1} - x_i) + V(y_{i+1} - y_i)), \; i = 2, ..., N-1$$

$$\frac{dx_N}{dt} = \frac{1}{U_N}(L(x_{N-1} - x_N) - V(y_N - x_N)),$$

$$\frac{dU_0}{dt} = u, \qquad \frac{dU_N}{dt} = -u. \tag{2}$$

The value $V = const$ is the constant vapour rate between the plates of the column, L and u are variable liquid and distillate rates, and $u(t) + L(t) = V$ for all t. These values define the *reflux ratio* $R = L/u$. The total mass and the mass of the light component are preserved by (2). Note that these equations blow up if either the reboiler or the accumulation tank is empty. To avoid this difficulty we assume that at the beginning of the process the accumulator contains a small amount of distillate of
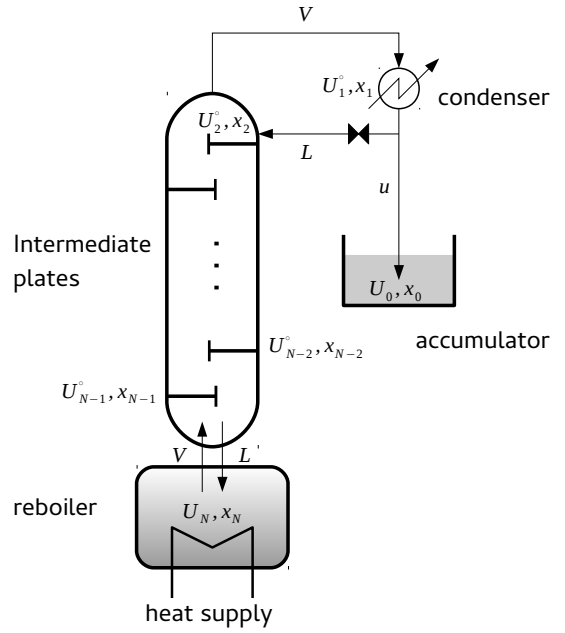

Fig. 1. A simple batch distillation column

the same concentrations as in the condenser (the *first drop* assumption).

2.2 Maximum distillate problem

Equations (2) define a control system where $u \in [0, V]$ plays the role of the control parameter. In this paper we consider the problem of maximization of the distillate production of desired purity y_* over in fixed interval of time t_f. The standard form of the purity constraint is the following (Converse, Gross (1963), Kim et al. (2001), Upreti (2011)) :

$$y_* \int_0^{t_f} u(t) dt = \int_0^{t_f} u(t) x_1(t) dt. \tag{3}$$

Note that y_* describe the purity of the liquid accumulated during the distillation process. The true concentration of the distillate depends on the concentration x_0° and the amount U_0° of the *first drop* of the distillate:

$$x_D = x_0^f = y_* + \frac{(x_0^\circ - y_*)U_0^\circ}{U_0(t_f)}.$$

In general, $x_D > y_*$ provided y_* is smaller than x_0°, which is always the case for the infinite reflux steady-state initial conditions considered below.

By introducing a new auxillary state variable z (the *purity deviation*) verifying

$$\frac{dz}{dt} = u(t)(x_1(t) - y_*), \qquad z(0) = 0 \tag{4}$$

the purity constraint (3) reduces to a terminal time condition $z(t_f) = 0$.

Assuming that at $t = 0$ the column was at steady state under infinite reflux ($u = 0$ for $t < 0$), the maximum distillate problem formulates as

$$\max_{u(\cdot) \in [0,V]} U_0(t_f), \qquad (5)$$

where the $N + 4$ state variables $U_0, U_N, x_0, \ldots x_N$ and z verify differential equations (2),(4) and the $(N + 4)$ steady state initial conditions at $t = 0$, as well as the final condition $z(t_f) = 0$. The infinite reflux steady state conditions can be easily computed via recurrence formulae : $x_i^\circ = y_{i+1}(x_{i+1}^\circ)$, $i = N - 1, \ldots, 1$, assuming that the initial concentration in reboiler x_N° is known.

Due to the mass conservation property of equations (2), the dimension of the state space can be reduced by two constants $m^\circ = U_N^\circ + U_0^\circ$, $m_x^\circ = \sum_{i=0}^{N} x_i^\circ U_i^\circ$, we restrict the original problem (5) to the level sets $U_N(t) + U_0(t) = m^\circ$, $m_x(t) = m_x^\circ$, obtaining the reduced state space

$$X_1 = \{q = (x_1, \ldots, x_N, U_N, z) : x_i \in [0,1] \, \forall i\}.$$

After reduction, the problem can be formulated as the following optimal control problem:

$$\min_{u(\cdot) \in [0,V]} q_{N+1}(t_f)$$

$$\frac{dq}{dt} = f_0(q) + u f_1(q), \quad q \in X_1, \qquad (6)$$

$$q(0) = q^{ss}, \qquad q_{N+2}(t_f) = 0$$

where t_f is fixed, and

$$f_0 = V \begin{bmatrix} \dfrac{y_2 - x_1}{U_1^\circ} \\ \vdots \\ \dfrac{x_{i-1} - x_i + y_{i+1} - y_i}{U_i^\circ} \\ \vdots \\ \dfrac{x_{N-1} - y_N}{U_N} \\ 0 \\ 0 \end{bmatrix}, \quad f_1 = \begin{bmatrix} 0 \\ \vdots \\ \dfrac{x_i - x_{i-1}}{U_i^\circ} \\ \vdots \\ \dfrac{x_N - x_{N-1}}{U_N} \\ -1 \\ x_1 - y_* \end{bmatrix}$$

2.3 Structure of optimal control by PMP

A preliminary idea about the structure of possible optimal controls of problem (6) can be obtained from Pontryagin's Maximum Principe (PMP). Problem (6) is a standard optimal control problem with space and state constraints, and affine (with respect to control u) dynamics. To simplify the mathematical analysis, the state constraints can be modified as $x_i \in]0,1[$, since the limit values are physically unrealizable. The Hamiltonian associated to (6) has the form $H(p, q, u) = H_0(q) + u H_1(q)$, where $H_i(p, q) = (p, f_i(q))$, $i = 0, 1$, and $p \in \mathbb{R}^{N+2}$ denotes the adjoint vector. According to PMP (Bonnard, Chyba (2003)), the optimal control can be found from the maximization condition for the Hamiltonian, which is necessarily valid along any optimal trajectory $\hat{q}(t)$ of (6):

$$H(p(t), \hat{q}(t), u_{opt}(t)) = \max_{v \in [0,V]} H(p(t), \hat{q}(t), v), \quad t \in [0, t_f].$$

The function $H_1(p, q)$ plays the the role of the switching function of the problem. If $H_1 \neq 0$ the optimal control is of *bang* type : $u = 0$ if $H_1 > 0$ and $u = V$ if $H_1 < 0$. If along some piece of the optimal trajectory $H_1 \equiv 0$, the corresponding optimal control is of *singular* type, i.e.

it verifies $\frac{\partial H}{\partial u} = 0$. Generically, in the affine case the singular control can be computed by the standard formula $u_s = -\frac{\{H_0, \{H_0, H_1\}\}}{\{H_1, \{H_0, H_1\}\}}$, where $\{.,.\}$ denotes the Poisson brackets.

The maximum distillate problem in the non reduced formulation has the same switching function and singular control as the reduced problem (6). Indeed, replacing the maximization condition for U_0 by the equivalent minimization condition for U_N, the hamiltonian of problem (5) takes the form

$$H^{full} = H_0 + u \left(\frac{p_{x_0}(x_1 - x_0)}{U_0} + p_{U_0} + H_1 \right).$$

where p_{x_0} and p_{U_0} are adjoint states associated to x_0 and U_0. According to PMP, they must verify the following differential equations:

$$\frac{dp_{x_0}}{dt} = \frac{u p_{x_0}}{U_0}, \qquad \frac{dp_{U_0}}{dt} = \frac{u p_{x_0}(x_1 - x_0)}{U_0^2}.$$

$U_0(t)$ and u are positive bounded values, so if $p_{x_0}(0) \neq 0$, then $p_{x_0}(t)$ is a monotone increasing (resp. decreasing) function if $p_{x_0}(0) > 0$ (resp. $p_{x_0}(0) < 0$). On the other hand, since the final values $x_0(t_f)$ and $U_0(t_f)$ are non prescribed, the transversality condition of PMP implies $p_{x_0}(t_f) = 0$, $p_{U_0}(t_f) = 0$. Therefore necessarily $p_{x_0}(t) \equiv 0$ and hence $p_{U_0} \equiv 0$. So, for the switching function we have $H_1^{full} = H_1$. Moreover, it is easy to verify that $\{H_0, H_1^{full}\} = \{H_0, H_1\}$, so the singular control is of the same form as in the reduced case.

It is worth to remark that several attempts were made in the past (Converse, Gross, 1963), (Diwekar, 1992) to apply the PMP to solve the maximum distillate problem for binary mixtures taking the *reflux ratio* $R = \frac{V}{u} - 1$, $R \in [0, +\infty)$, as the control parameter. The quasi steady-state approximation was used to describe the dynamics in the inner plates. Since $u = \frac{V}{R+1}$, the case $R = 0$ corresponds to $u = V$ and $R = +\infty$ (infinite reflux) to $u = 0$. In contrast with the distillate rate u, which can be easily implemented in practice, and which leads to a standard optimal control formulation, the use of R has serious disadvantages: the control system (2) is non-linear with respect to R, and R is unbounded from above though in reality the value $u = 0$ (infinite reflux) is attained. Indeed, the cited above authors found only the optimal control of singular type.

3. NUMERICAL RESULTS

Due to the dimension and high non-linearity of the problem, we first solve it numerically. To this end, we consider three homogeneous non-azeotropic binary mixtures, whose thermodynamic properties are well in accordance with the thermodynamic model (1).

3.1 The cases studied

Table 1.

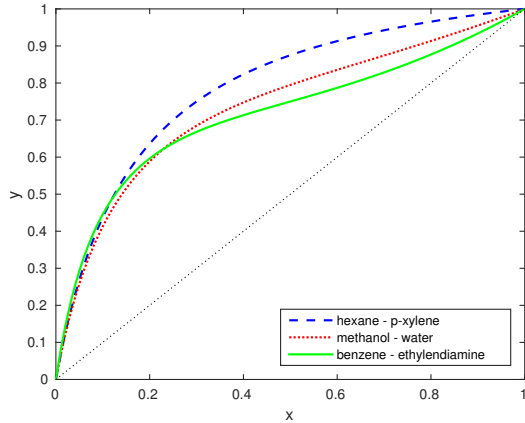| light component	heavy component	a	b	N_{min}
hexane	p-xylen	7	0	3
methanol	water	7.15	-0.33	4
benzene	ethylenediamine	9	-0.6	6

Fig. 2. Liquid-Vapour equilibrium curves for hexane - p-xylene (dashed), methanol - water (dotted), benzene - ethylendiamine (full)

Initially the reboiler is charged with $U_N^\circ = 10\,(mol)$ of liquid, whether each plate (including the condenser and the accumulator tank) contains $U_i^\circ = 0.1\,(mol)$, $i = 0, \ldots, N - 1$. For the vapour rate, the value $V = 11\,(mol/h)$ was taken. The final time $t_f = 0.8\,(h)$ assures that the reboiler is never empty: indeed, it corresponds to 88% the time of the complete emptying of the reboiler with maximal distillate rate $u = V$. In addition, we took $x_N^\circ = 0.1$ and $y_* = 0.95$. In order to obtain the distillate of desired purity, $N \geq N_{min}$ plates are needed. As it be seen from Tab.1 and Fig.2, the minimal number of plates depends on the location of the graph of the function $y(x)$ with respect to the diagonal, the separation is more difficult if this graph contains inflection points, as in the case of benzene - ethylendiamine.

3.2 Numerical solution by BOCOP solver

Table 2.

mixture	N	$U_a(t_f)$	recovery rate	discretization scheme
hexane -	5	1.39534	93.35%	Lobatto, 1600 points
p-xylen	9	1.88885	98.58%	Gauss, 1800 points
	12	2.20997	99.02%	Gauss, 1800 points
	14	2.42212	99.21%	Gauss, 1800 points
methanol -	6	1.3557	86.21%	Lobatto, 1600 points
water	9	1.8424	97.64%	Gauss, 1800 points
	12	2.1734	98.67%	Gauss, 1800 points
benzene -	9	1.7188	92.24%	Gauss, 1800 points
ethylediamine	12	2.1279	97.79%	Gauss, 1800 points

Numerical results discussed below were obtained with the BOCOP optimal control solver (Bonnans et al. (2014)). BOCOP is and open-source toolbox for solving optimal control problems by direct method (Trelat (2005)). The optimal control problem is approximated by a finite dimensional optimization problem (NLP) resulting from the time discretization of the dynamics of the system by an appropriate choice of the discretization scheme. In the current version different options are available starting from the 1-st order explicit Euler scheme until 6-th order implicit Lobatto algorithm. The NLP problem is then solved by Ipopt algorithm.
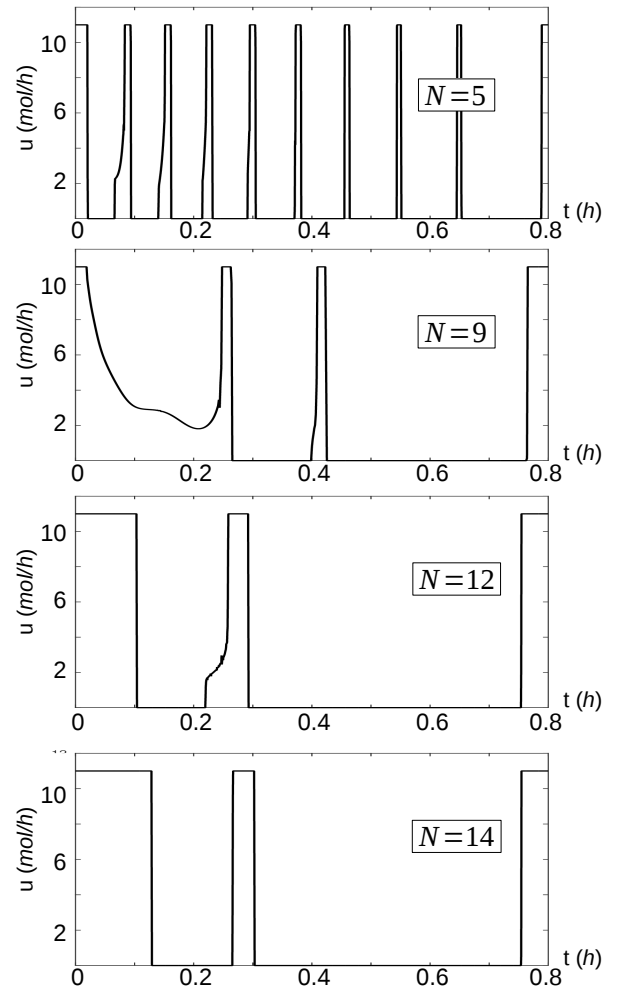

Fig. 3. Case hexane-p-xylene : the optimal distillate rate $u(t)$ for the distillation columns with 5, 9, 12 and 14 plates.

For the numerical resolution the full formulation (5) of the problem was used. Tab.2 displays the optimal costs and the recovery rates of the light component corresponding all examples considered below. In general, the convergence of the algorithm is very sensitive to the chosen discretisation scheme, and it is particularly difficult when the number of plates is close to the minimal one. The most results presented below were obtained with the 4-th order implicit Gauss method with 1800 discretisation points.

Case 1: hexane -P -hylene The optimal control policies of the maximum distillate problem for this mixture are shown in Fig. 3 for different number of plates. The first column ($N = 5$) has just enough plates to obtain the distillate of desired quality. We observe that in this case the optimal control is almost of *bang - bang* type with very short singular arcs, there are 21 commutations between different types of controls. The first and the last control arcs correspond to the maximal distillate rate $u = V$. As we will see below, this is always the case in all studied examples. In total there are 10 maximal distillate rate intervals alternating with zero distillation rate intervals. Very short singular control arcs precede the maximal
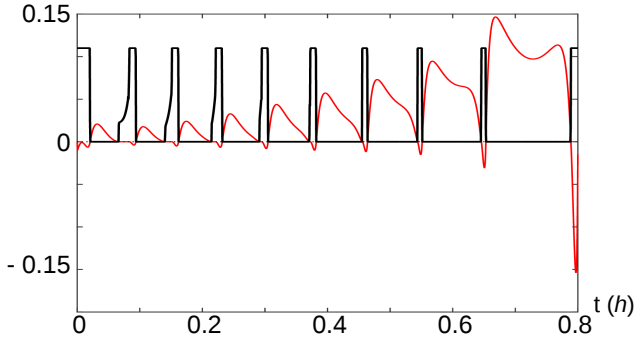
Fig. 4. Case hexane-p-xylene, $N = 5$: comparison of the switching function H_1 (thin red curve) and the rescaled optimal control $u/100$ (thick black curve)

distillate rate arcs 2, 3 and 4. Such a control policy is very similar to the so-called cyclic operating policy described in Sorensen (1999).

BOCOP allows to extract from its output data the values of the adjoint vector p along the optimal solution. This makes possible to perform the *a-posteriori* computation of the switching function $H_1(p, q)$. In Fig. 4 we compare the behaviour of this function with the optimal control $u(t)$ (taken with the rescaling factor 0.01). As expected, the sign-changes of H_1 are perfectly correlated with the switches between bang and singular arcs of the control function. So, the optimal control $u(t)$ computed numerically by direct method via complete discretization verifies the necessary optimality conditions of PMP.

With the second column (N=9) the separation is much easier. The corresponding optimal control has 4 bang arcs of maximal distillate rate, 2 arcs of zero control, and two singular arcs: the first one lasts about 25% of the total operation time t_f, and there are 8 switching points between different control types. In the case $N = 12$ the number of switchings drops to 5. The first bang arc (of maximal distillate rate) became longer, and there is only one short singular arc preceding the maximum distillate rate arc in the middle. Finally, with $N = 14$ the optimal control is of bang-bang type with one maximal distillate rate arc in the middle, which separates two long zero control arcs.

Case 2: methanol - water   The optimal distillate rate policy of this mixture is shown in Fig. 5. For the first configuration, $N = N_{min} + 2 = 6$ we found a bang-bang type control policy. The control switches 16 times between 9 maximal distillate rate arcs and 8 zero control arcs. Again, at the beginning and at the end the maximal control policy is used. The increment of the number of plates makes easier the separation. The optimal control policy becomes more tricky, but the number of switchings drops up to 9. Three singular arcs appear in the case $N = 9$. The first short singular arc connects two maximal rate arcs, the second connects the first zero control arc with the third maximal rate arc, and the third arc, of very duration, precedes the terminal bang arc. The duration of the second singular arc is about 43% of t_f. With the further increment of N ($N = 12$) the first and the third singular arcs becomes longer, while the second shortens.
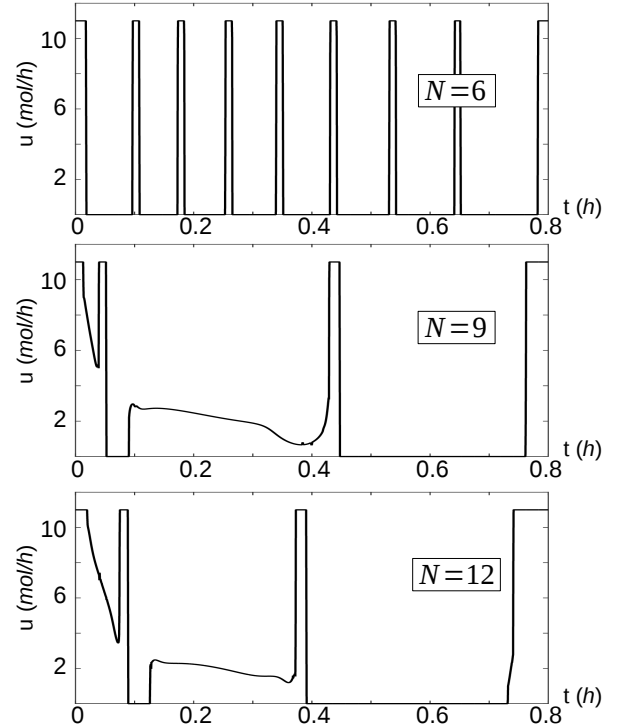

Fig. 5. Case methanol-water : the optimal distillate rate $u(t)$ for the distillation columns with 6, 9, 12 and 14 plates.
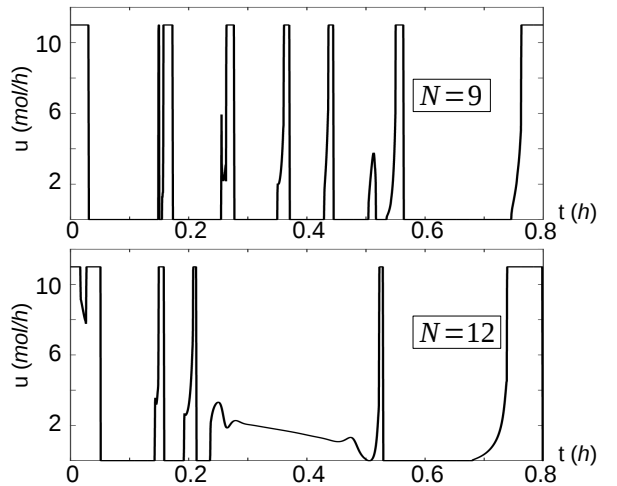

Fig. 6. Case benzene - ethylendiamine : the optimal distillate rate $u(t)$ for the distillation columns with 9, 12 and 14 plates.

Globally, we observe the same tendency as in the previous example, which make us think that with the bigger number of plates the optimal control policy would be of bang-bang type with very few switching points.

Case 3: benzene - ethylendiamine   Fig. 6 gives the idea of the optimal distillate rate policy for benzene - ethylendiamine mixture. For $N = 9$ plates we observe the picture similar to the previous two examples, though quality of the numerical solution is much worth compared to the

previous ones. Again, we observe a quasi bang-bang structure, which disappears with the increment of the number of plates. In the last picture ($N = 12$) the appearing singular arcs alternates with the maximal and zero distillate rate arcs. The series starts and terminates with two bang arcs $u = V$.

The above examples put in evidence the dependence of the optimal solution of the maximum distillate problem with the separation task difficulty and the column configuration. If the number of plates is close to the minimal one, the optimal control is almost of the bang-bang type with a number of short maximum rate arcs. With bigger number of plates the same quality of the final product can be achieved with smaller number of commutations by combining bang and singular type controls. If the number of plates in the columns is big enough, the operation can be done by a simple series of bang controls. In any case, the amount of the final product increases with the number of plates, as well as the recovery are of the light component. Such a mechanism cannot be (and was not) identified if the dynamics of the internal plates of the column is considered in a steady-state approximation, as was done in the literature so far.

3.3 Validation in dynamical simulation

The maximum distillate problem formulated in Section 2 is based on a series of quite strong assumption on the real dynamics of the column: equimolar overflow, total condensation, independence of the thermodynamic prosperities of the mixture on temperature. In order to test if the optimal solution obtained for the simplified problem can bring a real improvement to the conventional operation techniques, a more realistic simulation was done using the ProSim software (ProSim (2000)). We considered a 5 plates column charged with hexane and p-xylene, modeled by a UNIFAC equations. A bang-bang scenario approximating the optimal solution presented on the top of Fig. 3 was proposed. The column was driven to a steady-state and then the bang-bang scenario was realized. In Tab.3 the resuts of this simulation are compared with the solution obtained by BOCOP and with the solution obtained by ProSim with conventional operation policy. With almost the same control policy, the recovery rate in the "real" column is smaller than the one obtained by solving the simplified problem. In the same time, this strategy allowed to improve the recovery rate by 10% with respect to the conventional control used in the industrial context.

Table 3.

	BOCOP	ProSim (bang-bang)	Prosim (conventional control)
x_d	95.33%	96%	97%
recovery rate	93.35%	86%	76%

4. CONCLUSION

This paper focuses on the batch distillation of homogeneous binary mixtures. We discuss the correct optimal control formulation of the maximum distillate problem using the distillate rate instead of reflux as the control parameter. By introducing a new state variable, the purity deviation z, we reduce the distillate purity constrain to a simple terminal condition for z. According to PMP, the optimal controls can be of bang or singular types, which correspond to three types of reflux policy: infinite reflux, zero reflux and singular reflux that can be computed from PMP. We presented the results of the numerical resolution of the problem by direct method using the BOCOP optimal control solver. Our results put in evidence the strong correlation between the structure of the optimal distillate policy and the number of the plates in the column, in relation to the thermodynamics of the binary mixture to be separated. We show that a cyclic-like policy reported by other authors in a different context is very close the optimal solution of the problem if the number of plates is just sufficient or is big enough to assure the desired purity of the distillate. If the number of plates is relatively small, but far from N_{min}, the optimal way to obtain the product of the desired quality consist in the alternation of the singular controls with the periods of zero or maximal distillate rate policies.

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