

# Time (energy) requirements in closed batch distillation arrangements

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## Abstract

Batch time (energy) requirements are provided for the separation of a zeotropic mixture in three batch column configurations. The separation tasks were performed in two different multivessel column arrangements (with and without vapor bypass) and a rectifier column. All columns are operated as closed systems. The elimination of the vapor bypass in the multivessel column improves the composition dynamics in the middle vessel significantly. The modified multivessel column (without the vapor bypass) requires 30% less time than the conventional one (with the vapor bypass). The effect of the feed composition and product specification on the time requirements is also studied. The multivessel arrangements perform always better than the rectifier column, which requires from 35 to 100% more time to perform a given separation. All results are based on dynamic simulations of the processes.

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## 1. Introduction

Batch distillation is known to be less energy efficient than its continuous counterpart. However, it has received renewed interests last years due to the flexibility it offers. In a batch column, multicomponent mixtures can be separated in one single column and variations in the feed, the separation difficulty and the product specifications can be handled efficiently. This makes batch distillation especially suitable for pharmaceutical, fine and specialty chemicals industry where the demand and lifetime of the products can vary significantly with time and can also be uncertain.

Following these trends, new batch column configurations, like the multivessel column, and non-conventional operation modes, like closed operations, has received lately strong attention both in the industry and the academia. In this work, two different multivessel column configurations are compared to a rectifier batch column in terms of batch time (energy) requirements. The results are based on dynamic simulations for the separation of a zeotropic system. To the best of our knowledge, this is the first study where the dynamics in the middle vessel of multivessel configurations are discussed in details, and two different multivessel configurations are compared to each other and to a rectifier column.

The multivessel batch column can be viewed as a generalization of a batch rectifier and a batch stripper. The column has both a rectifying and a stripping section and therefore it is possible to obtain a light and a heavy fraction simultaneously from the top and the bottom of the column, while an intermediate fraction is also recovered in the middle vessel. Two modifications of the multivessel are studied here. The first one is the vapor bypass modification in which the vapor from the stripping section bypasses the middle vessel and enters the rectifying section, as shown in Fig. 1a. We refer to this configuration as conventional multivessel, since it is the one mostly studied in the literature. The second multivessel configuration is the one where both the liquid and the vapor streams enter the middle vessel. This configuration is illustrated in Fig. 1b and we refer to this one as modified multivessel. The third one is a rectifier column, shown in Fig. 1c, and hereafter called cyclic two-vessel column.

All columns are operated as closed systems, which simply means that there is no distillate or bottom stream out from the columns. The final products are accumulated in the vessels and discharged when the specifications are satisfied. In the multivessel column a ternary mixture can be separated simultaneously in one such close operation. No product change-overs are required and all products are accumulated in the three vessels at the end of the process. In the cyclic two-vessel column the separation is sequential, since there exist only two vessels. The products are

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**Nomenclature**

$a$	relative volatility vector
$F$	feed
$L_{\text{bot}}$	liquid flows in the stripping section
$L_{\text{top}}$	liquid flows in the rectifying section
$n_C$	number of components
$n_N$	number of total stages
$n_V$	number of vessels
$N_r$	number of stages in the rectifying section
$N_s$	number of stages in the stripping section
(s)	saddle point
(sn)	stable node
TC	temperature controller
(un)	unstable node
$V$	vapor flows
$x_B$	bottom vessel composition
$x_F$	feed composition
$x_{F2}$	composition at the beginning of cycle 2
$x_{i,n}$	composition in each stage
$x_M$	middle vessel composition
$x_{\text{spec},1}$	specification set 1
$x_{\text{spec},2}$	specification set 2
$x_{\text{spec},3}$	specification set 3
$x_T$	top vessel composition

separated one at each time and for a ternary mixture a sequence of two such closed operations is needed. The sequence chosen here resembles to the direct split in continuous columns. From the practical point of view, closed

operation modes are preferable over traditional open operation modes, like constant reflux, constant distillate or optimal reflux ratio policies. The closed operation mode requires minimum operator intervention and monitoring. There is a definite distinction between the product change-overs and it is easier to assure the product qualities.

In addition, closed operation modes can exhibit advantages in terms of separation time or energy requirements. Sorensen and Skogestad (1994) studied the performance of the rectifier column when it was operated under the cyclic policy. The proposed cyclic operation was characterized by repeating the following three periods: “filling up” of the condenser drum, “total reflux” operation of the column and “dumping” of the condenser drum product. Each sequence of these three periods was called a cycle and the number of the cycles could be predetermined or it could be optimized. The cyclic policy was shown to be superior to conventional open operation policies in some cases, like for example, difficult separations or when a small amount of light product is to be recovered. In some cases the reduction in the operating time was more than 30%, which simply indicates the potential energy savings by changing the operating policy. Sorensen and Prenzler (1997) applied this cyclic policy in an experimental batch column and they highlighted its much simpler operation and control. Hasebe et al. (1999) and Noda et al. (1999) provided comparisons between the closed (total reflux) operation of the rectifier, which is called cyclic two-vessel column in this study (Fig. 1c), and the conventional open operation of the rectifier or the stripper. They used the term “total reflux column” and they showed that it

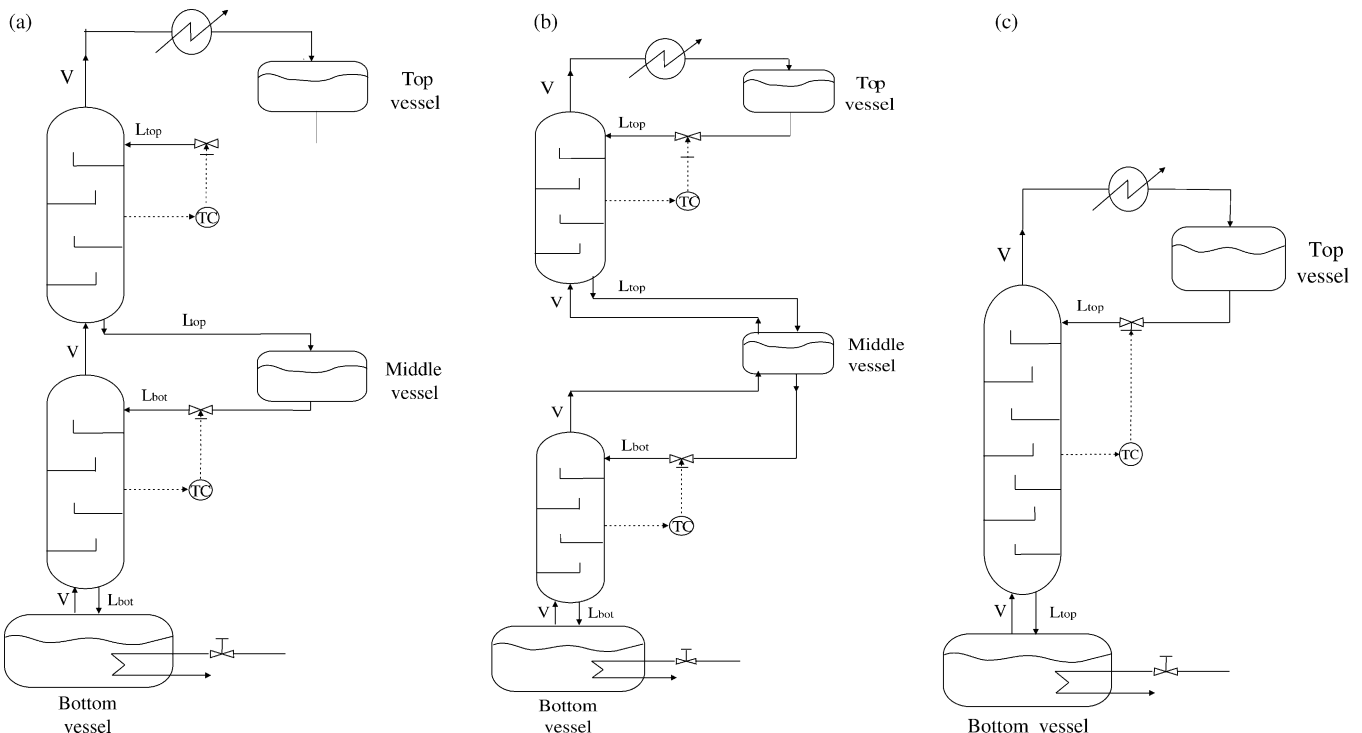


Fig. 1. (a) Conventional multivessel column with vapor bypass; (b) modified multivessel column without vapor bypass; (c) cyclic two-vessel column.

performs equal or better than the traditional columns when the operation was optimized in all columns.

Comparisons between the multivessel batch column and traditional batch columns, like rectifiers or strippers, have also been reported in the literature. In a series of papers, Hasebe et al. (1995), Hasebe et al. (1997), and Hasebe et al. (1999) investigated optimal operating modes for the multivessel column, called multi-effect batch distillation system (MEBAD) and they compared the novel column with both batch rectifiers and continuous columns. The energy consumption of the multivessel was almost half of that of a rectifier. Wittgens and Skogestad (1998) have reached the same conclusion that “a reduction of energy consumption of approximately 50% was found when using a multivessel column instead of a conventional batch distillation column”. The superiority of the multivessel column over the batch rectifier was further justified by the work of Furlonge et al. (1999). The rectifier required twice as much mean rate energy consumption as the multivessel for the separation of an equimolar quaternary ideal mixture. Finally, Hilmen (2000) provided a detailed comparison between the multivessel and the cyclic two-vessel column. The indirect split case was studied in their work and the multivessel column required less operating time than the cyclic two-vessel column. The time savings were more prominent for difficult separations, reaching a total of 50%.

The rest of this work is structured as follows. First, the model used in our simulation will be briefly presented along with the necessary information about the simulations. Then the paper is divided into two parts. In the first one, the base case of equimolar feed is studied. The separation procedure in the different column configurations is explained and the batch time comparisons are given. The effect of the elimination of the vapor bypass in the composition dynamics of the middle vessel is exhibited. In the second part, the effect of feed composition and product specification is investigated. Feeds rich in light, intermediate and heavy component are studied and the effect in the time requirements is presented. The paper ends with some concluding remarks.

## 2. Simulations

### 2.1. The model

The model used in our simulations consists of: overall and component material balances, vapor–liquid equilibrium modeled by NRTL activity coefficient model with binary parameters taken from the DECHEMA series, temperature estimations in each stage by a bubble point calculation under constant atmospheric pressure. The following assumptions has been made: staged column sections, constant vapor flows, constant liquid holdup on all stages, negligible vapor holdup, perfect mixing and equilibrium in all stages and ideal vapor phase.

The resulting mathematical model takes the form of a set of differential and algebraic equations (DAE system) with a total of  $(n_C + 1) \times (n_N + n_V)$  state variables, where  $n_C$  is the number of components,  $n_N$  is the total number of stages in the column sections and  $n_V$  is the number of vessels (two for the cyclic two-vessel column and three for the multivessel configurations). The resulting DAE system is solved in Matlab with the DAE solver ODE15s.

### 2.2. Simulation aspects

Batch time (energy) comparisons are provided for the separation of a ternary zeotropic system. The system under consideration is the mixture methanol/ethanol/1-propanol. A quick rough estimation of the relative volatilities of the system  $\alpha = [4.4, 2.3, 1]$  indicates a medium difficulty separation. However, the assumption of constant relative volatilities is not used in our model and this is the reason we avoid the term “ideal” mixture and we prefer the term “zeotropic” mixture.

We consider batch time, as a direct indication of energy consumption since the heat input (boilup) in the reboiler is constant. In order to minimize batch time, all columns are operated at maximum boilup (reboiler capacity). Thus, the vapor flow is constant and equal in all three columns. The ratio of the boilup flow over the initial feed (V/F) is a measure of how many times the feed is boiled every hour. This is chosen to be close to unity (once per hour) and is the same for both the multivessel and the cyclic column.

Theoretically, the minimum batch time is achieved for infinite number of stages. In practice, in our simulations, each column section has sufficient number of trays for the given separation and therefore the time calculations do not depend on the number of stages. Same number of stages was used in both the conventional multivessel and the cyclic two-vessel column. Thus, the number of stages in the cyclic two-vessel column is the sum of the stages in the two sections of the multivessel. The modified multivessel has one stage less than the conventional one since the middle vessel is an additional equilibrium stage in this case. Data for all three columns are given in Appendix.

The effect of the column liquid holdup is not included in this study. All columns have very small liquid holdup negligible compared to the initial feed (2% of the charge). In practice, this means that almost all the initial charge is recovered in the vessels at the end of the process. It also means that the dynamics inside the column sections are neglected and a change in the holdup in one of the vessel is almost instantaneously anticipated by a change in the holdup of another vessel.

The initial distribution of the feed in the vessels affects the separation time and no try has been done to optimize the initial feed distribution in the vessels. However, our simulation experience indicates that it either optimal or close to optimal, in terms of batch time, to charge most of the feed in the reboiler. In the multivessel column, 94% of the

total charge is fed in the reboiler, 5% in the middle vessel and only 1% in the top vessel. In the cyclic two-vessel column, 99% if the charge is fed in the reboiler and 1% in the top vessel. Furlonge et al. (1999) and Hasebe et al. (1995, 1999) provided optimization studies on this issue. In many cases the simple “feed in the reboiler” policy was proved to be either optimal or close to optimal for the closed multivessel. In an experimental work in the multivessel column, Wittgens (1999) found that it is easier to establish a good initial composition profile in the column by charging the feed in the reboiler. This feed policy also resembles to the one used in the cyclic two-vessel column with the feed charged in the reboiler. Based again on our simulation experience, the worst is to charge the feed in the middle vessel, while an equal distribution of the feed in the vessels is close to the “feed in the reboiler” policy. The results from Hilmen (2000) also support this simple feed policy. Of course in case of thermal decomposition or thermal sensitivity of the products it is wise to avoid the “feed in the reboiler” policy and implement other feed policies.

Four different feed compositions are considered. The base case presented first is for an equimolar feed. After the base case, feeds rich in light, intermediate and heavy component will be subsequently discussed. In the simulations initial compositions in all stages are equal to that of the feed mixture  $x_{i,n} = x_F$  and initial temperature estimations are that of the feed in boiling conditions. The solver ODE15s in Matlab was proved to be very robust in initializing the simulations in all cases and no problems were experienced, on this issue.

An indirect level control based on temperature feedback control is implemented in the vessels, as proposed by Skogestad et al. (1997). The feasibility of this control strategy was verified both by simulations and experiments in the multivessel column by Wittgens et al. (1996) and Wittgens and Skogestad (2000). The same simple control approach can be implemented for the cyclic two-vessel column, as proposed by Wittgens and Skogestad (2000) and it is shown in Fig. 1c. The main advantage of this feedback control strategy is the robustness in facing uncertainties in the feed composition. Furlonge et al. (1999) agreed on this issue but mentioned that this method is not always the best in terms of batch time (energy) consumption. The temperature measurements for the  $T$ -controllers are situated in the center of the column section for both the multivessel and the cyclic two-vessel column, as shown in Fig. 1. The temperature setpoints are set to the average of the boiling point of the two components separated in this column section.

The criterion for terminating the simulations was the fulfillment of the product specifications in the vessels. The product recoveries are also an important factor. In order to obtain comparable results, we tried to take care of the recoveries in the vessels but this was not always possible because of the different way the separation is performed in the columns. The separation is simultaneous in the multivessel and sequential in the cyclic two-vessel column, which

implies that it is much easier to handle the recoveries of the products, independently, in the latter case.

The batch time calculations do not include charging of the columns, preheating, product discharging and shutdown. These are considered to be the same for both the multivessel and the cyclic two-vessel column. The only exception is the product discharging period, which is higher for the cyclic two-vessel column because of the time required to discharge the top vessel holdup between the two cycles. This is an additional advantage for the multivessel column.

### 3. Results

#### 3.1. Base case—equimolar feed

The system methanol/ethanol/1-propanol is studied. An equimolar feed  $x_F = [1/3, 1/3, 1/3]$ , mainly placed in the reboiler, is considered and will be used as a base case in our work. Since this system exhibits no azeotropic behavior, the separation is proceeding according to the boiling temperatures of the components.

In the conventional multivessel column (Fig. 1a) the mixture is separated simultaneously in one closed operation. The three components are accumulated in the vessels at the end of the process. Methanol is the light component and is recovered in the top vessel, while ethanol and 1-propanol are the intermediate and heavy components recovered in the middle and bottom vessel, respectively. Fig. 2 shows how the separation is evolving with time. The top vessel is steadily enriched in methanol, the middle vessel in ethanol and the propanol is staying in the bottom vessel. The final column liquid profile is also shown with open circles. The rectifying (top) section of the column is performing the binary separation between methanol and ethanol. The top section liquid profile is therefore in the binary edge between

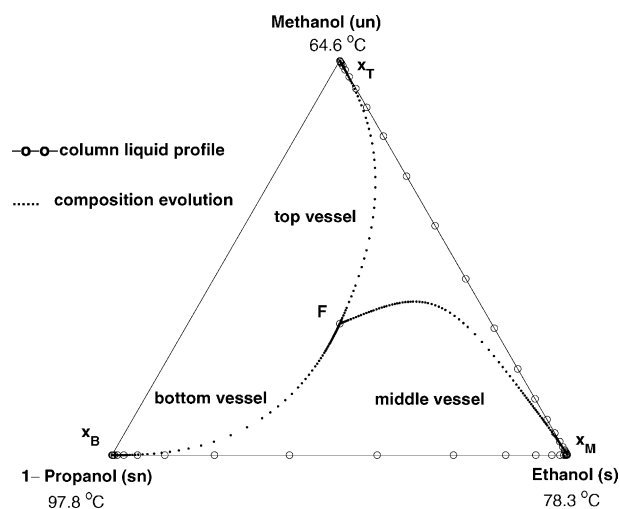


Fig. 2. Simultaneous separation of a zeotropic mixture in the multivessel column.

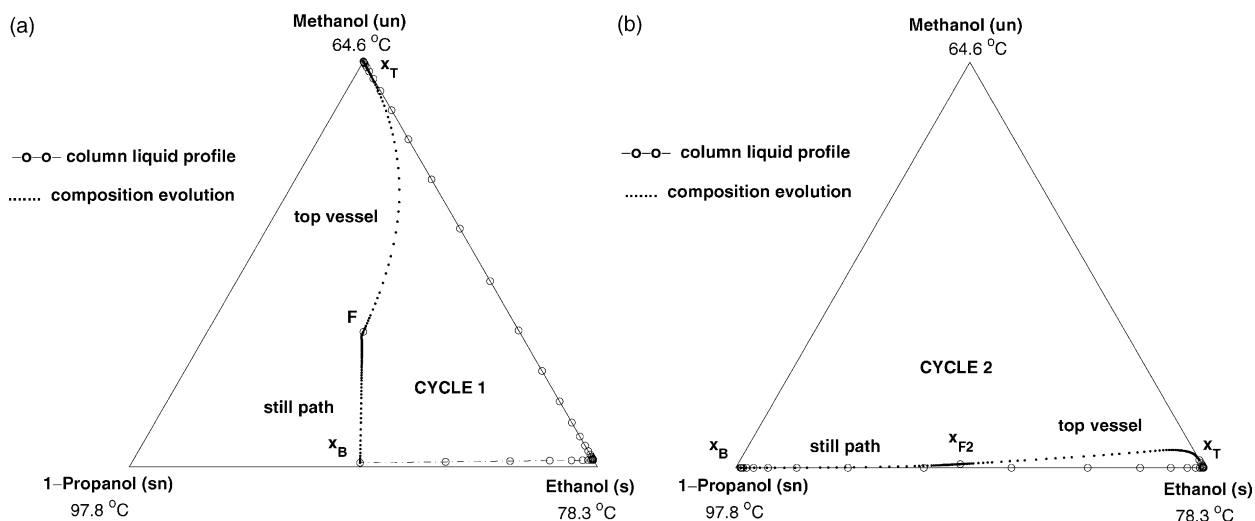


Fig. 3. (a) Separation of a zeotropic mixture in the cyclic column (cycle 1); (b) separation of a zeotropic mixture in the cyclic column (cycle 2).

methanol and ethanol. The stripping (bottom) section of the column is performing the binary separation between ethanol and 1-propanol and the bottom liquid profile is mainly in the binary edge between these two components.

The separation is performed exactly in the same way in the modified multivessel column (Fig. 1b). One closed operation is needed and the final products are simultaneously accumulated in the vessels. The reason for investigating this new multivessel configuration will become evident later after the analysis of the dynamics in the vessels.

In the cyclic two-vessel column (Fig. 1c) the separation is proceeding differently. The column has two vessels, which means that it is not possible to separate all three components simultaneously. Two closed operations, which will be called cycles hereafter, are needed and the separation resembles to the direct split in continuous columns.

During cycle 1 the light component (methanol) is accumulated in the top vessel, as shown in Fig. 3a. The still (bottom vessel) is following a linear path away from the component (methanol) accumulated in the top vessel. Cycle 1 is terminated when the specification for methanol is fulfilled. Then the vessel is emptied and the accumulated methanol is discharged in the product tank, instantaneously. A small amount of methanol still remains in the column and can contaminate the future products. Thus, a small off-cut fraction has to be removed. A closed operation is run for very short time with the same indirect level control and control parameters as for the second cycle. The off-cut fraction removed is equal to the total column holdup (0.1 kmol).

Cycle 2 is, then, an almost binary separation of the two components (ethanol and 1-propanol) left in the still after the off-cut fraction ( $x_{F2}$  in Fig. 3a). The intermediate component (ethanol) is accumulated in the top vessel, while the heaviest one (1-propanol) remains in the still, as shown in Fig. 3b.

The simulations were terminated when the composition specifications for all the products in the vessels were fulfilled. Results are provided for three specification sets.

$x_{\text{spec},1} = [0.99, 0.97, 0.99]$ ,  $x_{\text{spec},2} = [0.99, 0.99, 0.99]$  and  $x_{\text{spec},3} = [0.995, 0.995, 0.995]$ . In the second set, the specification in the middle vessel is stricter (0.97–0.99). In the third set the specifications are tighter in all vessels (0.99–0.995). The results are presented in Table 1.

### 3.1.1. Conventional multivessel versus cyclic two-vessel column

The batch time comparisons in Table 1 show that the conventional multivessel performs always better than the cyclic two-vessel column for equimolar feeds. The cyclic two-vessel column requires from 16 to 32% more time than the multivessel in order to perform the same separation. The most important difference is that the separation is performed simultaneously in the multivessel, in contrast to the cyclic two-vessel column, where two closed operations are required.

The time advantages of the multivessel are becoming smaller, as the specification in the middle vessel becomes stricter. For example, when the specification in the middle vessel increases from 0.97 to 0.99, the time advantages of the multivessel decrease from 32 to 16%. However, when the specification becomes strict in all vessels (third specification set) no more time gains can be expected for the two-vessel column. This happens because the increase in the separation time for the multivessel column, from 4.9 to 5.8 h, is outweighed by a proportionally equal increase mainly in cycle 1 of the two-vessel column. Cycle 1 has to be run for longer time in order to achieve the strict specification of 0.995 for the methanol in the top vessel.

### 3.1.2. Conventional multivessel versus modified multivessel

Table 1 shows clearly that the elimination of the vapor bypass in the multivessel column has a great effect in the batch time (energy) requirements. The modified multivessel is always faster than the conventional multivessel for equimolar feeds. The time savings vary from 26 to 33% depending on the specification, which indicate a weak dependence on the

Table 1  
Batch time calculations and time savings (basis: conventional multivessel)

Specification	Conventional multivessel (vapor bypass) (h)	Modified multivessel (no vapor bypass) (%)	Cyclic two-vessel (%)
Base case—equimolar $x_F = [1/3, 1/3, 1/3]$			
[0.99, 0.97, 0.99]	3.8	−26	+32
[0.99, 0.99, 0.99]	4.9	−31	+16
[0.995, 0.995, 0.995]	5.8	−33	+16
Rich in light $x_F = [0.7, 0.15, 0.15]$			
[0.99, 0.97, 0.99]	3.6	−19	+8
[0.99, 0.99, 0.99]	4.1	−22	+2
[0.995, 0.995, 0.995]	4.5	−22	+2
Rich in intermediate $x_F = [0.15, 0.7, 0.15]$			
[0.99, 0.97, 0.99]	4.0	−33	+28
[0.99, 0.99, 0.99]	6.6	−36	−2
[0.995, 0.995, 0.995]	7.9	−34	−8
Rich in heavy $x_F = [0.15, 0.15, 0.7]$			
[0.99, 0.97, 0.99]	2.4	0	+71
[0.99, 0.99, 0.99]	2.4	0	+104
[0.995, 0.995, 0.995]	2.8	0	+104

specifications. In average, the modified multivessel exhibits time advantages of around 30% over the conventional multivessel.

This is a rather surprising result since someone would expect minor differences, mainly attributed to the one additional equilibrium stage of the modified multivessel compared to the conventional. However, the situation is a bit more complicated. The middle vessel should not be considered simply as an additional equilibrium stage. It has a larger holdup compared to the stages inside the column and the dynamics in the vessels are playing a decisive role in the separation time requirements.

Fig. 4 illustrates what is happening in the vessels of the multivessel during the process. Fig. 4a shows the composition dynamics of the main component in each vessel for the conventional multivessel. The case of the first specification test [0.99, 0.97, 0.99] is considered. The methanol in the top vessel reaches the specification very fast, after 0.5 h, mainly because of the very small amount of initial holdup in the vessel. The bottom vessel is responding slowly the first 1 h. This is because it carries 94% of the total feed. However, the evolution is almost exponential after the first hour and finally, the propanol reaches its specification after 2.5 h. The middle vessel, which has the lowest specification, exhibits the slowest dynamics. It takes actually 3.8 h for ethanol to reach its low specification (0.97). At this time, all specifications are met and the separation task is ended. It is clear that the dynamics of the middle vessel are decisive for the whole process, since the middle vessel is the last one to reach the specification.

Fig. 4b provides even more insight into the process. The composition dynamics of the light component (methanol) in all vessels are shown. We see that the light methanol starts accumulating very fast in the top vessel and it is depleted rather fast (practically after 1.5 h) from the bottom vessel. However, the methanol insists on appearing in the middle vessel, which indicates an inherent inability, of the

middle vessel, to ‘boil-off’ the light component. The light component is in a sense trapped in the middle vessel and the process is significantly delayed. Why this is happening is becoming clear by looking in the design characteristics of the conventional multivessel in Fig. 1a. The vapor bypass from the stripping section to the rectifying section of the column is responsible for this. The light component in the middle vessel is depleted in a slow rate because there is no vapor phase coming in contact with the liquid holdup in it. This disadvantage of the conventional multivessel is removed in the so-called modified multivessel, where the vapor stream from the stripping section is entering the middle vessel.

The effect of the elimination of the vapor bypass is obvious in Fig. 4c, where the evolution of the compositions in the middle vessel is shown for both the conventional and the modified multivessel. The methanol is boiled-off faster when there is no vapor bypass and the main component (ethanol for the middle vessel) is reaching its specification faster. Almost 30% less time is required for performing this separation in the modified multivessel compared to the conventional one.

### 3.1.3. Discussion

In Fig. 4c it is obvious that the elimination of the vapor bypass has negligible effect in the composition dynamics of the heavy component (1-propanol). This is reasonable since the liquid flow out from the middle vessel remains almost unchanged in the two multivessel configurations. The problem however can be important in liquid bypass configurations. In this case, we would observe the reverse situation. The heavy component, entering the middle vessel, will be ‘trapped’ there and will be stripped down in the column in a slow rate.

The results presented in Hasebe et al. (1995) and Skogestad et al. (1997) indicate the slow dynamics in the middle vessel. In Fig. 7 of Hasebe et al. (1995) the middle

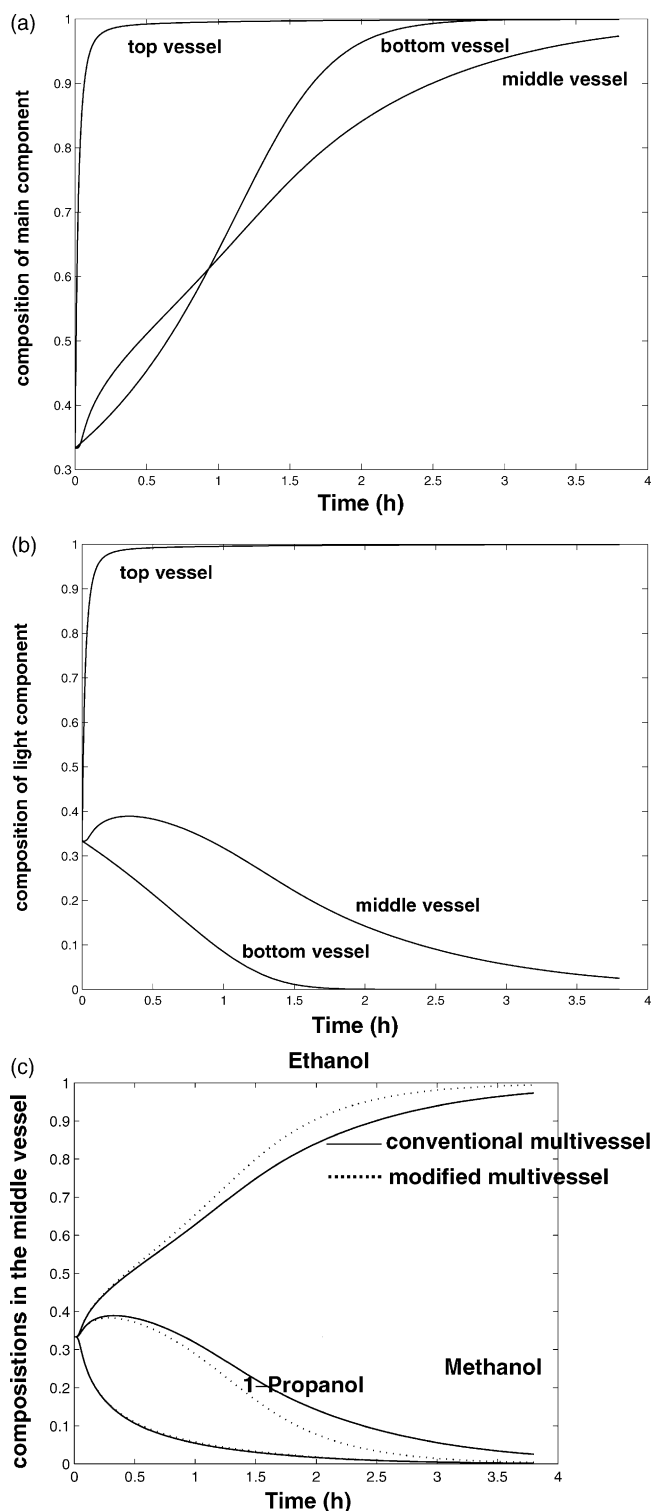


Fig. 4. (a) Composition of the main component in each vessel in the conventional multivessel; (b) composition of the light component (methanol) in each vessel in the conventional multivessel; (c) composition of all components in the middle vessel for the conventional and modified multivessel.

vessel product satisfies its specification last. Skogestad et al. (1997) provided simulated results for a quaternary mixture in a multivessel column with four vessels. In Figs. 3b and 4b of this work the light impurities in the two middle vessels persist for long time, thus delaying the process. The elimination of the vapor bypass enhances the composition dynamics in the middle vessel, thus making the process faster.

In the work of Hasebe, a set of existing batch rectifiers connected sequentially is proposed for a practical realization of the multivessel column. In this case it would be impractical to eliminate the vapor bypass since this will require significant changes in the existing rectifier columns. Nevertheless, in the case of a new multivessel column, built from scratch, the no vapor bypass configuration would be the best choice.

The knowledge of the slow dynamics in the middle vessel can serve as a very simple guideline for the initial feed distribution in the vessels. If the objective is to minimize batch time (energy demand) the advice we give is not to place the feed in the middle vessel. In the opposite case, e.g. placing the feed in the middle vessel, the dynamic response of the vessel is even slower, because of the large holdup to be accomplished and the large amount of light component that has to be boiled off in a slow rate. Hilmen (2000) supports this simple intuitive guideline. “For medium difficulty separations . . . charging the feed to the intermediate vessel was worst in all cases of feed composition” and “. . . for easy separations we found large time savings for feeds charged to the reboiler instead of the middle vessel”. In contrast, for difficult separations the initial distribution of the feed is not very important anymore. This happens because the actual batch time is determined by the difficulty of the separation task itself and not by the dynamics of the vessels.

In conclusion, the results for the base case indicate that the modified multivessel is the best alternative. The modified column requires 30% less time than the conventional one. By comparing now the cyclic two-vessel column with the modified multivessel, we see that the former requires around 70% more time than the latter for the same separation.

### 3.2. Effect of the feed composition

#### 3.2.1. Feed rich in light component

For a feed rich in light component the results in Table 1 reveal that the elimination of the vapor bypass is advantageous also in this case. The modified multivessel requires around 20% less time than the conventional multivessel and the time savings are independent of the specification.

The striking result for such feeds is the minor advantages of the multivessel column compared to the cyclic two-vessel column, which is marginally slower (8–2%) than the multivessel. This result is in agreement with the results presented by Hilmen (2000) that mentions “for medium difficult separations, the benefits of the multivessel column are low for feeds rich in light components and feed low in heavy component”. The comparison in Hilmen (2000) refers to

the indirect split in the cyclic column but our results show that it holds also for the direct split.

However, we see that there is a potential to save separation time (energy) by using multivessel configurations as long as the modified multivessel is employed, instead of the conventional one. Then, the potential savings in the modified multivessel compared to the cyclic two-vessel are around 25%.

### 3.2.2. Feed rich in intermediate component

Intuitively, the results for this case should reflect the fact that the middle vessel has slow dynamics and delays the separation task in multivessel configurations. The results in Table 1 support our intuition. The first thing to observe is the increase in the separation time values for the conventional multivessel. Notice, for example, the time requirements for the second specification set for different feeds  $x_F$ . The actual separation times increase from 4.1 to 4.9 h and finally to 6.6 h, as the intermediate component increase in the feed. This happens because of the large amount of intermediate component accumulated in the middle vessel during the process. The middle vessel has anyway very slow dynamics and the large holdup to be processed in the vessel is making the situation even worse.

As expected, the elimination of the vapor bypass is very effective in this case. The modified multivessel requires around 35% less time than the conventional one and this is the maximum time savings observed with the modified configuration, in this study. Consequently, the modified multivessel is strongly recommended for such feeds.

The comparison between the conventional multivessel and the cyclic two-vessel column is in favor of the former only for the first specification set. When the specifications become stricter in the middle vessel (from 0.97 to 0.99) or to all the vessels (from 0.99 to 0.995) then the two-vessel column requires slightly less time than the conventional multivessel.

The same was mentioned by Meski et al. (1998), who compared the multivessel column with the direct and indirect split (or combinations) in regular batch columns. They found that regular columns (e.g. the two-vessel column) were best for feeds rich in the intermediate component. In contrast, Hasebe et al. (1992) claimed that the multivessel column is more effective in removing light and heavy impurities from a feed than ordinary distillation. Our results, certainly do not support such a statement, neither do the results from Hilmen (2000) and Meski et al. (1998).

By comparing now the modified multivessel with the two-vessel column we see that even in this case, which is the worst one for the conventional multivessel, the modified multivessel is superior and requires around 35% less time than the two-vessel column.

### 3.2.3. Feed rich in heavy component

The results for this last case studied here are very interesting. This case represents the “ultimate” situation for multivessel configurations. The last rows in Table 1 show that the separation task is accomplished very fast in the conventional

multivessel. It takes only 2.4 h (for the second specification set) for the separation to be finished, compared to 6.6 or 4.9 or 4.1 h in previous cases.

The secret behind these numbers is that the separation is governed by the dynamics of the bottom vessel. The middle vessel is not anymore the slowest vessel and therefore the inherent disadvantage of the middle vessel is vanishing. The fact that the middle vessel dynamics are playing no role in this case is illustrated clearly by the zero time savings of the modified multivessel. The elimination of the vapor bypass has no effect in the separation task, which is determined by the bottom vessel dynamics.

The comparison between the conventional multivessel and the cyclic two-vessel column is strongly in favor of the former. The cyclic column requires from 70 to 104% more time depending on the specification. In addition, as the specification becomes stricter the results are even worse for the two-vessel column, a trend that is opposite of what we have seen until now. This case verifies the 50% time savings by using the multivessel column instead of a rectifier (e.g. two-vessel) reported before in the literature.

Hilmen (2000) also mentioned that large time savings are expected for the multivessel column compared to the cyclic for medium difficult and feeds rich in heavy components. Sorensen and Prenzler (1997) mention that this is a common separation task in the pharmaceutical and fine chemical industries where light solvents should be recovered from waste water streams and the specifications are very tight (few ppm for the solvent). The common practice is to perform these separations sequentially in conventional batch rectifiers. However, as the results indicate here, a multivessel configuration of serially connected existing rectifiers is actually a much better alternative. The potential time (energy) savings of such a rearrangement of existing rectifiers in the plant are remarkable (around 50%) and indicate that valuable process time can be saved without too much effort.

## 4. Conclusions

Batch time (energy) requirements, based on simulations, were provided for the separation of a zeotropic mixture in closed batch distillation arrangements. The batch arrangements studied were a conventional multivessel column with a vapor bypass, a modified multivessel without a vapor bypass and a cyclic two-vessel column. The task was to separate a mixture of methanol/ethanol/1-propanol.

The base case of equimolar feed was used for illustrating an important disadvantage of conventional multivessel configurations, namely, the slow dynamics in the middle vessel. Elimination of the vapor bypass in the middle was proposed and the results for the so-called modified multivessel supported this intuitive design modification. Multivessel configurations, either conventional or modified, perform always better than traditional configurations like the cyclic two-vessel column.



In the comparison between the modified multivessel (without vapor bypass) and the conventional multivessel (with vapor bypass), the former is superior to the latter, with the exception of feeds rich in heavy components where no differences are noticed in the time requirements.

The conventional multivessel performs worst for feeds rich in the intermediate component. This is the only case where the cyclic two-vessel column is faster and reflects the

slow dynamic response of the middle vessel. However, even in this case, the modified multivessel is the best alternative.

Feeds rich in the heavy component are the ultimate case for multivessel configurations. The potential time savings of processing such feeds in multivessel configurations instead of in regular batch columns are around 50%. This result should be of great practical importance in the pharmaceutical, fine and specialty chemicals industries.

## Appendix

Table A.1 Column data

	Conventional multivessel	Modified multivessel	Cyclic two-vessel column
Stages per section	$N_r = 25, N_s = 25$	$N_r = 25, N_s = 24$	$N_{\text{total}} = 50$
Initial feed	$M_F = 5.385 \text{ kmol}$	$M_F = 5.385 \text{ kmol}$	$M_F = 5.385 \text{ kmol}$
Initial condenser holdup	$M_{T0} = 0.035 \text{ kmol}$	$M_{T0} = 0.035 \text{ kmol}$	$M_{T0} = 0.035 \text{ kmol}$
Initial middle vessel holdup	$M_{F0} = 0.250 \text{ kmol}$	$M_{F0} = 0.250 \text{ kmol}$	No middle vessel
Initial reboiler holdup	$M_{B0} = 5.000 \text{ kmol}$	$M_{B0} = 5.000 \text{ kmol}$	$M_{B0} = 5.250 \text{ kmol}$
Trays holdup	$M_i = 1/500 \text{ kmol}$	$M_i = 1/500 \text{ kmol}$	$M_i = 1/500 \text{ kmol}$
Vapor flow	$V = 5 \text{ kmol h}^{-1}$	$V = 5 \text{ kmol h}^{-1}$	$V = 5 \text{ kmol h}^{-1}$

Table A.2 Temperature controller data

Controller gain for the rectifying section or cycle 1	$K_c = 0.365 \text{ kmol (h } ^\circ\text{C)}^{-1}$
Controller gain for the stripping section or cycle 2	$K_c = 0.256 \text{ kmol (h } ^\circ\text{C)}^{-1}$
Temperature setpoint for the rectifying section or cycle 1	$T_{\text{sp}} = 71.45 \text{ } ^\circ\text{C}$
Temperature setpoint for the stripping section or cycle 2	$T_{\text{sp}} = 88.05 \text{ } ^\circ\text{C}$

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